MOLYCORP INC - QUESTA NM
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MOLYCORP INC QUESTA
QUESTA NM

Chevron Mining

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3-4-13-1 FTW-01-01-032-2-014-01-004

Transfer#: PT-412-2010-1848 **Box:3 CC:R6**

ARR1-626901954 **Asset#:** AAC1-304444812 **Whole Container: N**

C/F: NM0022306 VOL 3 CHEVRON MINING $00087547 **Created:** 3/1/2013

THOUSE General Reference Temporary Loan of Records

Standard Other (contact FRC to arrange) BILL OUR UPS ACCOUNT

To: PATRICIA VINCENT
EPA / DALLAS , 1445 ROSS AVE., SUITE 1200
DALLAS, TX, 75202
P : (214) 665-6532 F :

P 662 037 136

RECEIPT FOR CERTIFIED MAIL

NO INSURANCE COVERAGE PROVIDED
NOT FOR INTERNATIONAL MAIL

(See Reverse)

Sent to	
Street and No.	
P.O., State and ZIP Code	
Postage	$
Certified Fee	
Special Delivery Fee	
Restricted Delivery Fee	
Return Receipt Showing to whom and Date Delivered	
Return receipt showing to whom, Date, and Address of Delivery	
TOTAL Postage and Fees	$
Postmark or Date	

PS Form 3800, Feb. 1982 ★ U.S.G.P.O. 1983-403-517

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

MAR 0 8 1985

CERTIFIED MAIL: RETURN RECEIPT REQUESTED (P 662 037 136)

Mr. C. R. Sacrison
General Manager
Molycorp, Inc.
P.O. Box 419
Questa, New Mexico 87556

Re: Application to Discharge to Waters of the United States
Permit No. NM0022306

Dear Mr. Sacrison:

Enclosed is the public notice, fact sheet, and a copy of the permit which this Agency has drafted under the authority of the National Pollutant Discharge Elimination System. A copy of the final permit will be mailed to you when the Agency has made a final permit decision.

Should you have any questions concerning any part of the permit, please feel free to contact the Permits Branch at the above address or telephone (214) 767-4375.

Sincerely,

/s/Myron O. Knudson

Myron O. Knudson, P.E.
Director, Water Management Division (6W)

Enclosures

cc w/permit copy:
New Mexico Environmental Inprovement Division

8/13/84

8/11/84:HUMKE:kw:202:NM22306-1/-15

CONCURRENCES

SYMBOL	6W-PI	6W-P		6W-E				
SURNAME	Becker	Hannesschlager						
DATE	12/3/84	12/4		2/7	2/21			

EPA Form 1320-1 (12-70)

OFFICIAL FILE COPY

GPO : 1983 O - 403-201

Advertising Order Number 5T-3216-NNLX
U.S. Environmental Protection Agency
Public Notice of Draft NPDES Permit(s)

MARCH 9, 1985

This is to give notice that the U.S. Environmental Protection Agency, Region 6, has formulated a Draft Permit for the following facility (facilities) under the National Pollutant Discharge Elimination System. Development of the draft permit(s) was based on a preliminary staff review by EPA, Region 6, and consultation with the State of NEW MEXICO. The State of NEW MEXICO is currently reviewing the draft permit(s) for the purpose of certifying or denying certification of the permit(s). The permit(s) will become effective within 30 days after the close of the comment period unless:

a. The State of NEW MEXICO denies certification, or requests an extension for certification prior to that date.

b. Comments received prior to APRIL 9, 1985 warrant a public notice of EPA's final permit decision.

c. A public hearing is held requiring delay of the effective date.

EPA's contact person for submitting written comments, requesting information regarding the draft permit, and/or obtaining copies of the permit and the Statement of Basis or Fact Sheet is:

Ms. Ellen Caldwell
Permits Branch (6W-PS)
U.S. Environmental Protection Agency
Interfirst Two Building
1201 Elm Street
Dallas, Texas 75270
(214) 767-2765

EPA's comments and public hearing procedures may be found at 40 CFR 124.10 and 124.12 (48 Federal Register 14264, April 1, 1983, as amended at 49 Federal Register 38051, September 26, 1984). The comment period during which written comments on the draft permit may be submitted extends for 30 days from the date of this Notice. During the comment period, any interested person may request a Public Hearing by filing a written request which must state the issues to be raised. A public hearing will be held when EPA finds a significant degree of public interest.

EPA will notify the applicant and each person who has submitted written comments or requested notice of the final permit decision. A final permit decision means a final decision to issue, deny, modify, revoke or reissue, or terminate a permit. Any person may request an Evidentiary Hearing on the agency's final permit decision. However, the request must be submitted within 30 days of the date of the final permit decision and be in accordance with the requirements of 40 CFR 124.74. Any condition(s) contested in a request for an evidentiary hearing on an existing Source may be stayed if the request for a hearing is granted. If any condition(s) contested in a request for an evidentiary hearing are granted on a New Source, New Discharger, or Recommencing Discharger the applicant shall be without a permit.

Further information including the administrative record may be viewed at the above address between 8 a.m. and 4:30 p.m., Monday through Friday.

NPDES authorization to discharge to waters of the United States, permit No. NM0022306 .

The applicant's mailing address is: Molycorp, Inc.
P.O. Box 419
Questa, New Mexico 87556

The discharge from this molybdenum milling facility is made into the Red River, a water of the United States classified for coldwater fishing; fish culture; livestock and wildlife watering; and secondary contact recreation. The discharge is located on that water approximately one mile above the bridge at the Red River Fish Hatchery. A fact sheet is available. Under the standard industrial classification (SIC) code 1061, the applicant's activities are mining and milling operations which produce molybdenum disulfide concentration.

The changes from the previously issued permit are:

1. Concentration limits for molybdenum and manganese are suspended during periods of zero flow from outfall 001.

2. The definition of the term "composite sample" is revised to "a minimum of two grab samples of effluents collected not less than four hours apart over a normal eight hour operating day-----."

3. pH limitations are changed to within the range of 6.0 to 9.0 S.U.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION VI
INTERFIRST TWO BUILDING, 1201 ELM STREET
DALLAS, TEXAS 75270

FACT SHEET

For proposed National Pollutant Discharge Elimination System (NPDES) Permit No. NM0022306 to discharge to waters of the United States.

Issuing office: U. S. Environmental Protection Agency
Region VI
InterFirst Two Building
1201 Elm Street
Dallas, Texas 75270

Applicant: Molycorp, Inc.
P.O. Box 469
Questa, New Mexico 87556

1. The applicant currently operates a molybdenum mining and milling operation.

2. As described in the application, the plant site is located in Taos County, New Mexico. Discharge is to the Red River of the Rio Grande Basin.

3. The known uses of the receiving waters are:

Coldwater Fishery.
Fish Culture.
Livestock and wildlife watering.
Secondary contact recreation.

4. Stream standards are:

The general and specific stream standards are provided in "New Mexico Water Quality Standards," New Mexico Water Quality Control Commission, August 1973.

5. The following is a quantitative description of the discharge described in the application:

a. Outfall	Flow Frequency	Avg/Daily (MGD)	Max/(MGD)	Min/(MGD)
001	Intermittent	3.31	4.32	N/A
002	Intermittent	N/A	0.61	N/A

b. Outfall	Temp. °F Avg/Summer	Temp. °F Avg/Winter	Temp. °F Max	Temp. °F Min
001	N/A	N/A	N/A	N/A
002	N/A	N/A	N/A	N/A

c.

Outfall	Parameter	Effluent Characteristics Daily Avg (mg/l)	Daily Max (mg/l)
001	Chemical Oxygen Demand	30	38
001	Total Suspended Solids	11	29
001	Total Arsenic	N/A	0.56
001	Total Cadmium	<0.01	<0.01
001	Total Copper	N/A	0.04
001	Total Cyanide	0.046	0.046
001	Flouride	2.90	2.90
001	Total Iron	0.56	0.80
001	Total Lead	N/A	0.18
001	Total Manganese	1.20	1.39
001	Total Mercury	<0.06	<0.06
001	Total Molybdenum	4.13	4.36
001	Total Zinc	0.08	0.14
	pH within range of 6.6 to 8.3 S.U.		
002	Chemical Oxygen Demand	8	15
002	Total Suspended Solids	7	20
002	Total Arsenic	<0.04	N.D.
002	Total Cadmium	<0.01	<0.01
002	Total Copper	<0.03	N.D.
002	Total Cyanide	0.004	0.005
002	Flouride	2.20	2.30
002	Total Iron	0.36	0.54
002	Total Lead	N/A	0.21
002	Total Manganese	1.37	1.42
002	Total Mercury	<0.01	N.D.
002	Total Molybdenum	2.71	2.84
002	Total Zinc	0.0	0.09
	pH within range of 7.1 to 7.8 S.U.		

6. On the basis of preliminary staff review, the Environmental Protection Agency, after consultation with the State of New Mexico, has made a tentative determination to issue a permit for the discharge described in the application.

7. The proposed effluent limitations for those pollutants proposed to be limited are as follows:

See attached draft permit.

8. A brief explanation follows of the express statutory or regulatory provision on which permit requirements are based, including appropriate supporting references to the Administrative Record required by 40 CFR §124.9:

a. NPDES permit No. NM0022306 issued to Molycorp on July 29, 1983.
b. Consolidated NPDES application No. NM0022306 received from Molycorp on January 19, 1982.
c. Ore Mining & Dressing Point Source Category Guidelines, 40 CFR Part 440, dated December 3, 1982.

d. Request for permit modification from Molycorp, dated June 29, 1984, and received on July 5, 1984.

9. The following is an explanation of calculations or other necessary explanation of the derivation of specific effluent limitations and conditions, including a citation to the applicable effluent limitation guideline or performance standard provisions as required under 40 CFR §122.44 and §122.45 and reasons why these are applicable:

1. In accordance with the modification request, concentration limits for molybdenum and manganese are suspended during periods of zero flow from Outfall 001. When only Outfall 002 is being discharged, the levels of these small flow seepage sources may exceed the concentration limits for the normal composite of Outfalls 001 and 002 without any significant effect on the receiving stream. The economics of re-routing and treatment are not feasible or warranted. Therefore, based on best professional judgment (BPJ), concentration limits only are suspended during periods of zero flow from Outfall 001.

2. In accordance with the modification request, based on BPJ the definition of "composite sample" is revised to "a sample consisting of a minimum of two grab samples of effluents collected not less than four hours apart over a normal operating day-----." This modification is made to allow the permittee to utilize personnel normally available at the monitoring site since the two-part composite sampling has provided representative, consistent samples in the past.

3. In accordance with the modification request and the concurrence of the NMEID, pH monitoring limits are revised to within the range of 6.0 to 9.0 S.U.

10. The requested variance(s) N/A appear justified for the following reason(s):

N/A.

11. The permit is in the process of certification by the State agency. A draft permit and draft public notice will be sent to the District Engineer, Corps of Engineers, and to the Regional Director of the U. S. Fish and Wildlife Service, and the National Marine Fisheries Service, prior to the publication of that notice.

12. The public notice describes the procedures for the formulation of final determinations.

PROPOSED PERMIT

Permit No. NM0022306
Application No. NM0022306

AUTHORIZATION TO DISCHARGE UNDER THE
NATIONAL POLLUTANT DISCHARGE ELIMINATION SYSTEM

In compliance with the provisions of the Federal Water Pollution Control Act, as amended, (33 U.S.C. 1251 et. seq; the "Act"),

Molycorp, Inc.
P.O. Box 469
Questa, New Mexico 87556

is authorized to discharge from a facility located at Questa, Taos County, New Mexico

to receiving waters named Red River

in accordance with effluent limitations, monitoring requirements and other conditions set forth in Parts I, II, and III hereof.

This permit shall become effective on

This permit and the authorization to discharge shall expire at midnight, August 30, 1988

Signed this day of

Myron O. Knudson, P.E.
Director, Water Management Division (6W)

PART I
REQUIREMENTS FOR NPDES PERMITS

SECTION A. EFFLUENT LIMITATIONS AND MONITORING REQUIREMENTS - Outfall 001 & 002

During the period beginning the effective date and lasting through ***** the permittee is authorized to discharge from Outfall(s) serial number(s) 001, process water from milling operation and tailings disposal; 002 - seepage from tailing impoundment.

Such discharges shall be limited and monitored by the permittee as specified below:

Effluent Characteristic	Discharge Limitations			
	kg/day(lbs/day)		Other Units (Specify)	
	Daily Avg	Daily Max	Daily Avg	Daily Max
Flow-m^3/Day(MGD)	N/A	N/A	*	*
Temperature	N/A	N/A	* °C (*°F)	* °C(* °F)
Chemical Oxygen Demand	N/A	N/A	60 mg/l	90 mg/l
Total Suspended Solids	N/A	N/A	20 mg/l	30 mg/l
Total Arsenic	N/A	N/A	0.5 mg/l	1.0 mg/l
Total Cadmium	N/A	N/A	N/A	.05 mg/l
Total Copper	N/A	N/A	0.15 mg/l	0.30 mg/l
Total Cyanide	N/A	N/A	0.025 mg/l	0.05 mg/l
Fluoride	N/A	N/A	N/A	3.0 mg/l
Total Iron	N/A	N/A	N/A	0.6 mg/l
Total Lead	N/A	N/A	0.3 mg/l	0.6 mg/l
Total Manganese	N/A	N/A	1.0 mg/l	1.5 mg/l
Total Mercury	N/A	N/A	0.001 mg/l	0.002 mg/l
Total Molybdenum	35(78)****	71(156)***	* mg/l	* mg/l
Total Zinc	N/A	N/A	N/A	0.2 mg/l
Biomonitoring	N/A	N/A	N/A	N/A

Effluent Characteristic	Monitoring Requirements	
	Measurement Frequency	Sample Type
Flow-m^3/Day(MGD)	(1)	Record
Temperature	1/week	In Situ
Chemical Oxygen Demand	1/week	Composite**
Total Suspended Solids	1/week	Composite**
Total Arsenic	1/week	Composite**
Total Cadmium	1/week	Composite**
Total Copper	1/week	Composite**

Continued from page 2

Total Cyanide	(2)	Composite**
Fluoride	1/week	Composite**
Total Iron	1/week	Composite**
Total Lead	1/week	Composite**
Total Manganese	1/week	Composite**
Total Mercury	1/week	Composite**
Total Molybdenum	1/week	Composite**
Total Zinc	1/week	Composite**
Biomonitoring	(3)	(4)

* Report
** See Part III, Paragraph B
*** Sum total for Outfalls 001 & 002
**** Sum total average for Outfalls 001 & 002 for six months preceeding reporting period end date; also report daily average mass for each month.
(1) Continuous and totalized monitoring for Outfall 001; daily estimate for Outfall 002
(2) 2/week for Outfall 001; 1/week for Outfall 002.
(3) See Part III, Paragraph D.
(4) See Part III, Paragraph E.
***** One year from date of mill start-up.

The pH shall not be less than 6.0 standard units nor greater than 9.0 standard units and shall be monitored 1/week by grab sample.

There shall be no discharge of floating solids or visible foam in other than trace amounts.

Samples taken in compliance with the monitoring requirements specified above shall be taken at the following location(s):

<u>Outfall 001</u> Latitude 36° 40' 61" and Longitude 105° 38' 30" which is the discharge spillway from Pope Lake.

<u>Outfall 002</u> Latitude 36° 40' 61" and Longitude 105° 37' 30" which is the collected and combined seepage from the tailings impoundment.

PART I
REQUIREMENTS FOR NPDES PERMITS

SECTION A. EFFLUENT LIMITATIONS AND MONITORING REQUIREMENTS - Outfall 001 & 002

During the period beginning ***** and lasting through the expiration date, the permittee is authorized to discharge from Outfall(s) serial number(s) 001, process water from milling operation and tailings disposal; 002, seepage from tailing impoundment.

Such discharges shall be limited and monitored by the permittee as specified below:

Effluent Characteristic	Discharge Limitations			
	kg/day(lbs/day)		Other Units (Specify)	
	Daily Avg	Daily Max	Daily Avg	Daily Max
Flow-m^3/Day(MGD)	N/A	N/A	*	*
Temperature	N/A	N/A	* °C (*°F)	* °C (*°F)
Chemical Oxygen Demand	N/A	N/A	60 mg/l	90 mg/l
Total Suspended Solids	N/A	N/A	20 mg/l	30 mg/l
Total Arsenic	N/A	N/A	0.5 mg/l	1.0 mg/l
Total Cadmium	N/A	N/A	N/A	.05 mg/l
Total Copper	N/A	N/A	0.15 mg/l	0.30 mg/l
Total Cyanide	N/A	N/A	0.025 mg/l	0.05 mg/l
Fluoride	N/A	N/A	N/A	3.0 mg/l
Total Iron	N/A	N/A	N/A	0.6 mg/l
Total Lead	N/A	N/A	0.3 mg/l	0.6 mg/l
Total Manganese	N/A	N/A	1.0 mg/l	1.5 mg/l
Total Mercury	N/A	N/A	0.001 mg/l	0.002 mg/l
Total Molybdenum	11(25)****	23(50)***	1.0 mg/l	2.0 mg/l
Total Zinc	N/A	N/A	N/A	0.2 mg/l
Biomonitoring	N/A	N/A	N/A	N/A

Effluent Characteristic	Monitoring Requirements	
	Measurement Frequency	Sample Type
Flow-m^3/Day(MGD)	(1)	Record
Temperature	1/week	In-Situ
Chemical Oxygen Demand	1/week	Composite**
Total Suspended Solids	1/week	Composite**
Total Arsenic	1/week	Composite**
Total Cadmium	1/week	Composite**
Total Copper	1/week	Composite**
Total Cyanide	(2)	Composite**

Continued from page 4

Fluoride	1/week	Composite**
Total Iron	1/week	Composite**
Total Lead	1/week	Composite**
Total Manganese	1/week	Composite**
Total Mercury	1/week	Composite**
Total Molybdenum	1/week	Composite**
Total Zinc	1/week	Composite**
Biomonitoring	(3)	(4)

* Report
** See Part III, paragraph B.
*** Sum total for Outfalls 001 & 002.
**** Sum total average for Outfalls 001 & 002 for six months preceeding reporting period end date; also report daily average mass for each month.
(1) Continuous and totalized monitoring for Outfall 001; daily estimate or Outfall 002.
(2) 2/week for Outfall 001; 1/week for Outfall 002.
(3) See Part III, Paragraph D.
(4) See Part III, Paragraph E.
***** One year from date of mill start-up.

The pH shall not be less than 6.0 standard units nor greater than 9.0 standard units and shall be monitored 1/week by grab sample.

There shall be no discharge of floating solids or visible foam in other than trace amounts.

Samples taken in compliance with the monitoring requirements specified above shall be taken at the following location(s):

<u>Outfall 001</u> Latitude 36° 40' 61" and Longitude 105° 38' 30" which is the discharge spillway from Pope Lake.

<u>Outfall 002</u> Latitude 36° 40' 61" and Longitude 105° 37' 30" which is the collected and combined seepage from the tailings impoundment.

SECTION B. SCHEDULE OF COMPLIANCE

The permittee shall achieve compliance with the effluent limitations specified for discharges in accordance with the following schedule:

Report mill start-up

Progress Report	3 mos. after mill start-up
Progress Report	6 mos. after mill start-up
Progress Report	9 mos. after mill start-up
Achieve Compliance	12 mos. after mill start-up

PART II
STANDARD CONDITIONS FOR NPDES PERMITS

SECTION A. GENERAL CONDITIONS

1. Duty to Comply

The permittee must comply with all conditions of this permit. Any permit noncompliance constitutes a violation of the Clean Water Act and is grounds for enforcement action; for permit termination, revocation and reissuance, or modification; or for denial of a permit renewal application.

2. Penalties for Violations of Permit Conditions

The Clean Water Act provides that any person who violates a permit condition implementing Sections 301, 302, 306, 307, 308, 318, or 405 of the Clean Water Act is subject to a civil penalty not to exceed $10,000 per day of such violation. Any person who willfully or negligently violates permit conditions inplementing Sections 301, 302, 306, 307, or 308 of the Clean Water Act is subject to a fine of not less than $2,500 nor more than $25,000 per day of violation, or by imprisonment for not more than 1 year, or both.

3. Permit Actions

This permit may be modified, revoked and reissued, or terminated for cause including, but not limited to, the following:

a. Violation of any terms or conditions of this permit;

b. Obtaining this permit by misrepresentation or failure to disclose fully all relevant facts; or

c. A change in any condition that requires either a temporary or a permanent reduction or elimination of the authorized discharge; or

d. A determination that the permitted activity endangers human health or the environment and can only be regulated to acceptable levels by permit modification or termination.

The filing of a request by the permittee for a permit modification, revocation and reissuance, or termination, or a notification of planned changes or anticipated noncompliance, does not stay any permit condition.

9. Severability

The provisions of this permit are severable, and if any provision of this permit or the application of any provision of this permit to any circumstance is held invalid, the application of such provision to other circumstances, and the remainder of this permit, shall not be affected thereby.

10. Definitions

The following definitions shall apply unless otherwise specified in this permit:

a. "Daily Discharge" means the discharge of a pollutant measured during a calendar day or any 24-hour period that reasonably represents the calendar day for purposes of sampling. For pollutants with limitations expressed in terms of mass, the "daily discharge" is calculated as the total mass of the pollutant discharged over the sampling day. For pollutants with limitations expressed in other units of measurement, the "daily discharge" is calculated as the average measurement of the pollutant over the sampling day. "Daily discharge" determination of concentration made using a composite sample shall be the concentration of the composite sample. When grab samples are used, the "daily discharge" determination of concentration shall be the arithmetic average (weighted by flow value) of all samples collected during that sampling day.

b. "Daily Average" discharge limitation means the highest allowable average of "daily discharges" over a calendar month, calculated as the sum of all "daily discharges" measured during a calendar month divided by the number of "daily discharges" measured during that month.

c. "Daily Maximum" discharge limitation means the highest allowable "daily discharge" during the calendar month.

c. Notice

(1) Anticipated bypass. If the permittee knows in advance of the need for a bypass, it shall submit prior notice, if possible at least ten days before the date of the bypass.

(2) Unanticipated bypass. The permittee shall submit notice of an unanticipated bypass as required in Section D, paragraph 6 (24-hour notice).

d. Prohibition of bypass.

(1) Bypass is prohibited, and the Director may take enforcement action against a permittee for bypass, unless:

(a) Bypass was unavoidable to prevent loss of life, personal injury, or severe property damage;

(b) There were no feasible alternatives to the bypass, such as the use of auxiliary treatment facilities, retention of untreated wastes, or maintenance during normal periods of equipment downtime. This condition is not satisfied if adequate back-up equipment should have been installed in the exercise of reasonable engineering judgment to prevent a bypass which occured during normal periods of equipment downtime or preventive maintenance; and

(c) The permittee submitted notices as required under Section B, paragraph 4.c.

(2) The Director may approve an anticipated bypass, after considering its adverse effects, if the Director determines that it will meet the three conditions listed above in Section B, paragraph 4.d.(1).

5. Upset Conditions

a. Definition. "Upset" means an exceptional incident in which there is unintentional and temporary noncompliance with technology-based permit effluent limitations because of factors beyond the reasonable control of the permittee. An upset does not include noncompliance to the extent caused by operational error, improperly designed treatment facilities, inadequate treatment facilities, lack of preventive maintenance, or careless or improper operation.

SECTION C. MONITORING AND RECORDS

1. Representative Sampling

Samples and measurements taken as required herein shall be representative of the volume and nature of the monitored dishcarge. All samples shall be taken at the monitoring points specified in this permit and, unless otherwise specified, before the effluent joins or is diluted by any other wastestream, body of water, or substance. Monitoring points shall not be changed without notification to and the approval of the Director.

2. Flow Measurements

Appropriate flow measurement devices and methods consistent with accepted scientific practices shall be selected and used to ensure the accuracy and reliability of measurements of the volume of monitored discharges. The devices shall be installed, calibrated, and maintained to insure that the accuracy of the measurements are consistent with the accepted capability of that type of device. Devices selected shall be capable of measuring flows with a maximum deviation of less than ± 10% from true discharge rates throughout the range of expected discharge volumes. Guidance in selection, installation, calibration, and operation of acceptable flow measurement devices can be obtained from the following references:

a. "A Guide to Methods and Standards for the Measurement of Water Flow", U.S. Department of Commerce, National Bureau of Standards, NBS Special Publication 421, May 1975, 97 pp. (Available from the U.S. Government Printing Office, Washington, D.C. 20402. Order by SD catalog No. C13.10:421).

b. "Water Measurement Manual", U.S. Department of Interior, Bureau of Reclamation, Second Edition, Revised Reprint, 1974, 327 pp. (Available from the U.S. Government Printing Office, Washington, D.C. 20402. Order by Catalog No. I27.19/2:W29/2, Stock No. S/N 24003-0027).

c. "Flow Measurement in Open Channels and Closed Conduits", U.S. Department of Commerce, National Bureau of Standards, NBS Special Publication 484, October 1977, 982 pp. (Available in paper copy or microfiche from National Technical Information Service (NTIS), Springfield, VA 22151. Order by NTIS No. PB-273 535/5ST).

d. "NPDES Compliance Sampling Manual", U.S. Environmental Protection Agency, Office of Water Enforcement, Publication

7. Averaging of Measurements

Calculations for all limitations which require averaging of measurements shall utilize an arithmetic mean unless otherwise specified by the Director in the permit.

8. Retention of Records

The permittee shall retain records of all monitoring information, including all calibration and maintenance records and all original strip chart recordings for continuous monitoring instrumentation, copies of all reports required by this permit, and records of all data used to complete the application for this permit, for a period of at least 3 years from the date of the sample, measurement, report, or application. This period may be extended by request of the Director at any time.

9. Record Contents

Records of monitoring information shall include:

a. The date, exact place, and time of sampling or measurements;

b. The individual(s) who performed the sampling or measurements;

c. The date(s) analyses were performed;

d. The individual(s) who performed the analyses;

e. The analytical techniques or methods used; and

f. The results of such analyses.

10. Inspection and Entry

The permittee shall allow the Director, or an authorized representative, upon the presentation of credentials and other documents as may be required by law, to:

a. Enter upon the permittee's premises where a regulated facility or activity is located or conducted, or where records must be kept under the conditions of this permit;

b. Have access to and copy, at reasonable times, any records that must be kept under the conditions of this permit;

c. Inspect at reasonable times any facilities, equipment (including monitoring and control equipment), practices, or operations regulated or required under this permit; and

d. Sample or monitor at reasonable times, for the purposes of assuring permit compliance or as otherwise authorized by the Clean Water Act, any substances or parameters at any location.

6. Twenty Four Hour Reporting

The permittee shall report any noncompliance which may endanger health or the environment. Any information shall be provided orally within 24 hours from the time the permittee becomes aware of the circumstances. A written submission shall also be provided within 5 days of the time the permittee becomes aware of the circumstances. The written submission shall contain a description of the noncompliance and its cause; the period of noncompliance, including exact dates and times, and if the noncompliance has not been corrected, the anticipated time it is expected to continue; and steps taken or planned to reduce, eliminate, and prevent reoccurrence of the noncompliance. The Director may waive the written report on a case-by-case basis if the oral report has been received within 24 hours.

The following shall be included as information which must be reported within 24 hours:

a. Any unanticipated bypass which exceeds any effluent limitation in the permit.

b. Any upset which exceeds any effluent limitation in the permit.

c. Violation of a maximum daily discharge limitation for any of the pollutants listed by the Director in Part III of the permit to be reported within 24 hours.

7. Other Noncompliance

The permittee shall report all instances of noncompliance not reported under Section D, paragraphs 4, 5, and 6, at the time monitoring reports are submitted. The reports shall contain the information listed in Section D, paragraph 6.

8. Changes in Discharges of Toxic Substances

The permittee shall notify the Director as soon as it knows or has reason to believe:

a. That any activity has occured or will occur which would result in the discharge, in a routine or frequent basis, of any toxic pollutant which is not limited in the permit, if that discharge will exceed the highest of the "notification levels" described in 40 CFR Part 122.42(a)(1) [48 FR 14153, April 1, 1983, as amended at 49 FR 38046, September 26, 1984].

b. That any activity has occured or will occur which would result in any discharge, on a non-routine or infrequent basis, of a toxic pollutant which is not limited in the permit, if

(3) For a municipality, State, Federal, or other public agency: by either a principal executive officer or ranking elected official. For purposes of this section, a principal executive officer of a Federal agency includes:

(i) The chief executive officer of the agency, or

(ii) a senior executive officer having responsibility for the overall operations of a principal geographic unit of the agency.

b. All reports required by the permit and other information requested by the Director shall be signed by a person described above or by a duly authorized representative of that person. A person is a duly authorized representative only if:

(1) The authorization is made in writing by a person described above.

(2) The authorization specifies either an individual or a position having responsibility for the overall operation of the regulated facility or activity, such as the position of plant manager, operator of a well or a well field, superintendent, or position of equivalent responsibility, or an individual or position having overall responsibility for environmental matters for the company. A duly authorized representative may thus be either a named individual or any individual occupying a named position; and

(3) The written authorization is submitted to the Director.

c. Certification. Any person signing a document under this section shall make the following certification:

"I certify under penalty of law that this document and all attachments were prepared under the direction or supervision in accordance with a system designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who manage the system, or those persons directly responsible for gathering the information, the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations."

PART III
OTHER CONDITIONS

A. The "daily average" concentration means the arithmetic average (weighted by flow value) of all the daily determinations of concentration made during a calendar month. Daily determinations of concentration made using a composite sample shall be the concentration of the composite sample. When grab samples are used, the daily determination of concentration shall be the arithmetic average (weighted by flow value) of all the samples collected during that calendar day.

The "daily maximum" concentration means the daily determination of concentration for any calendar day.

B. The term " composite sample" means a sample consisting of a minimum of two grab samples of effluent collected not less than four hours apart over a normal eight hour operating day and combined proportional to flow or a sample continously collected proportional to flow over a normal eight hour operating day. All such samples shall be typical and representative of effluent generated during the period since the last sample was collected.

C. Analysis and reporting for Outfalls 001 and 002 shall be accomplished separately for each outfall. In addition, the permittee shall report the flow weighted average results for Outfalls 001 plus 002. Effluent limitations on weight of total molybdenum shall be the sum total for Outfalls 001 and 002. For determination of compliance with other effluent limitations the following shall apply:

1. Discharge from Outfall 001 shall be in compliance.

2. A composite sample of discharges from Outfalls 001 and 002 monitoring samples combined in flow weighted proportion shall be in compliance, except that concentration limits for molybdenum and manganese are suspended during periods of zero flow from Outfall 001.

   During periods when no flow occurs from Outfall 001, monthly average reporting of concentration for molybdenum and manganese shall be based on the average of all composite samples obtained in the month for Outfalls 001 and 002; and monthly maximum reporting of concentration for molybdenum and manganese shall be based on the maximum composite sample obtained in that month for Outfalls 001 and 002. In conjuction with the DMR for each month, the permittee shall report periods, if any, when no flow exists at Outfall 001.

D. The permittee shall determine if eighty (80) percent or greater of the culture of test organisms will survive by use of the "Range-Finding Screening Test," set out in "Methods for Measuring the Acute Toxicity of Effluents to

Aquatic Organisms, "EPA-600/4-78-012 (Rev. July, 1978). Organisms for this test shall be Daphnia sp. if the effluent is less than five (5) parts per thousand salinity or Mysidopsis sp. if the effluent is equal to or greater than five (5) parts per thousand salinity. This screening test will be conducted within sixty (60) days of effectiveness of the biomonitoring requirements. Tests will be conducted once each quarter for a duration of two years utilizing a static method for 24 hours and following this dilution scheme only:

Effluent sample* - 100 percent by volume
Dilution water - 0 percent by volume

*24-hour composite; refrigerated after collection

If at any time during the two year testing period a test shows a survival of eighty (80) percent or less of the test organisms, the permittee shall within twenty-four (24) hours conduct a replacement static 48-hour median lethal concentration (LC50) test on the orginially collected sample. Replacement of effluent samples shall be once per 24 hours. Organisms for this test shall be Daphnia sp. if the effluent is less than five (5) parts per thousand salinity and reconstituted fresh water (EPA-600/4-78-012 Section 4) shall be used for dilution. If the effluent is equal to or greater than five (5) parts per thousands salinity, Mysidopsis sp. shall be used as the test organism, and reconstituted seawater will be used as dilution water (EPA-600/4-78-012 Section 4). The remaining LC50 methodology is available in EPA-600/4-78-012.

All screening and LC50 test results shall be reported with the Discharge Monitoring Reports. The test results should include the chemical and physical data as specified in Section 7 of EPA-600/4-78-012.

E. Sample taken in compliance with this section shall consist of 24-hour composite samples taken at the discharge spillway from Pope Lake.

F. The Molycorp thiocyanate colorimetric method is approved for the analysis of molybdenum unless subsequently determined to be inappropriate by the NMEID or EPA.

G. As soon as practicable after the arrival of Molycorp's environmental staff at the site of a tailings spill that reaches the Red River, but no later than two (2) hours after arrival at the site, water quality sampling shall commence. Samples shall be taken at three sites:

(1) Approximately 100 feet above the point where tailings enter the river;
(2) Approximately 100 feet below the point where tailings enter the river; and
(3) Approximately one-half mile below the point where tailings enter the river.

All samples shall be properly preserved and analyzed for:

Chemical Oxygen Demand (COD)
Total Suspended Solids (TSS)
Cyanide (CN)
Total Iron (Fe)
Manganese, Dissolved (Mn)
Total Molybdenum (Mo)
pH
Temperature
Conductivity
Total Zinc (Zn)

The results of the analyses shall be submitted to the United States Environmental Protection Agency and the New Mexico Environmental Improvement Division within 30 days following a tailings spill.

Consistent with the procedures described in the Preventative Maintenance and Surveillance Plan and Contingency Action and Reporting Plans (June 1975), a written report containing the following information will be sent to the US EPA and NMEID within ten (10) days following any spill:

(1) Date of spill.

(2) Time when the spill was observed and time when tailings flow into the river was stopped.

(3) Location (pipe or coupling number).

(4) Estimated amount of tailings that entered the river.

(5) Sketch and dimension of size of hole or failure that cuased the spill.

(6) Position of failure in the pipe or coupling.

(7) Copy of the latest computer printout covering the pipe or coupling which failed.

(8) Comments, if required for clarification.

Molycorp, Inc.

Questa Division
P.O. Box 469
Questa, New Mexico 87556
Telephone: (505) 586-0212

exp. 8.30.88

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MOLYCORP

CERTIFIED MAIL --
RETURN RECEIPT REQUESTED

September 28, 1987

Mr. Robert Layton Jr.
Regional Administrator
U. S. EPA Region VI
1445 Ross Avenue
Dallas, Texas 75202

Mr. Michael Burkhart, Director
New Mexico EID
P.O. Box 968
Santa Fe, New Mexico 87504

Gentlemen:

Enclosed please find a permit application for renewal of Molycorp's Questa Division NPDES permit NM0022306.

Regulations under 40 CFR Part 122.53 require submission of this permit application "180 days before the expiration date of the existing permit". NPDES permit NM0022306 will expire August 15, 1988.

The application includes EPA application forms 1 and 2C; however, please be advised that Molycorp's mining and milling operations have ceased as of February, 1986, and there has been no discharge from Outfall 001, and no milling operation impact on Outfall 002, which is currently discharging. Molycorp at this time seeks a deferral on the analysis requirements under Parts A, B, C, and GC/MS fraction analysis on form 2C for Outfall 001 and Outfall 002. Molycorp, upon resuming normal milling operations and normal continuous flow from Outfall 001, will submit samples from both Outfalls to comply with required analysis for permit renewal. Molycorp, however, is submitting Biological Toxicity testing data for Outfall 002, the Red River, the receiving waters for both Outfalls, and the West Diversion Channel; a final outlet that feeds decant mill process water at our Ion Exchange Plant. Due to the phase down of milling operations and ultimate final mill shutdown on February, 1986, no Biological Toxicity data is available for Outfall 001 from August 1985, when U.S. EPA Region 6 NPDES requirements went into effect.

Please note that we have included a copy of a letter from Mr. R. G.

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OCT 9 1987
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Molycorp, Inc.

Questa Division
P.O. Box 469
Questa, New Mexico 87556
Telephone: (505) 586-0212

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CONTENTS OF CONSOLIDATED PERMIT APPLICATION

FOR

MOLYCORP, INC. NMD 002699094

SECTION I EPA STANDARD FORM 1 - GENERAL INFORMATION

SECTION II EPA STANDARD FORM 2c - WASTEWATER DISCHARGE INFORMATION

SECTION III DESCRIPTION OF TAILINGS DISPOSAL AND ION EXCHANGE PLANT OPERATIONS AND FLOW DIAGRAM OF OPERATION

SECTION IV LETTER OF AUTHORIZATION FOR MINE MANAGER AS SIGNATOR

SECTION V REQUEST FOR CONTINUING ALTERNATIVE MOLYBDENUM TEST PROCEDURE

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SECTION VII RESULTS OF BIOLOGICAL TOXICITY TESTING OF OUTFALL 002, THE RED RIVER AND WEST DIVERSION CHANNEL

SECTION I

EPA STANDARD FORM 1

GENERAL INFORMATION

NM22306

Please print or type in the unshaded areas only (fill-in areas are spaced for elite type, i.e., 12 characters/inch).

Form Approved OMB No. 158-R0175

FORM 1 GENERAL

EPA

U.S. ENVIRONMENTAL PROTECTION AGENCY

GENERAL INFORMATION

Consolidated Permits Program

(Read the "General Instructions" before starting.)

I. EPA I.D. NUMBER F NMD002699094 D

LABEL ITEMS

I. EPA I.D. NUMBER

III. FACILITY NAME

V. FACILITY MAILING ADDRESS

VI. FACILITY LOCATION

NMD 002699094

MOLYCORP, INC.

P. O. BOX 469

QUESTA, NEW MEXICO 87556

GENERAL INSTRUCTIONS

If a preprinted label has been provided, affix it in the designated space. Review the information carefully; if any of it is incorrect, cross through it and enter the correct data in the appropriate fill-in area below. Also, if any of the preprinted data is absent *(the area to the left of the label space lists the information that should appear)*, please provide it in the proper fill-in area(s) below. If the label is complete and correct, you need not complete Items I, III, V, and VI *(except VI-B which must be completed regardless)*. Complete all items if no label has been provided. Refer to the instructions for detailed item descriptions and for the legal authorizations under which this data is collected.

II. POLLUTANT CHARACTERISTICS

INSTRUCTIONS: Complete A through J to determine whether you need to submit any permit application forms to the EPA. If you answer "yes" to any questions, you must submit this form and the supplemental form listed in the parenthesis following the question. Mark "X" in the box in the third column if the supplemental form is attached. If you answer "no" to each question, you need not submit any of these forms. You may answer "no" if your activity is excluded from permit requirements; see Section C of the instructions. See also, Section D of the instructions for definitions of bold-faced terms.

SPECIFIC QUESTIONS	MARK 'X' YES	NO	FORM ATTACHED
A. Is this facility a **publicly owned treatment works** which results in a **discharge** to **waters of the U.S.**? (FORM 2A)		X	
B. Does or will this facility *(either existing or proposed)* include a **concentrated animal feeding operation** or **aquatic animal production facility** which results in a **discharge** to **waters of the U.S.**? (FORM 2B)		X	
C. Is this a facility which currently results in **discharges** to **waters of the U.S.** other than those described in A or B above? (FORM 2C)	X		
D. Is this a proposed facility *(other than those described in A or B above)* which will result in a **discharge** to **waters of the U.S.**? (FORM 2D)		X	
E. Does or will this facility treat, store, or dispose of **hazardous wastes**? (FORM 3)		X	
F. Do you or will you inject at this facility industrial or municipal effluent below the lowermost stratum containing, within one quarter mile of the well bore, underground sources of drinking water? (FORM 4)		X	
G. Do you or will you inject at this facility any produced water or other fluids which are brought to the surface in connection with conventional oil or natural gas production, inject fluids used for enhanced recovery of oil or natural gas, or inject fluids for storage of liquid hydrocarbons? (FORM 4)		X	
H. Do you or will you inject at this facility fluids for special processes such as mining of sulfur by the Frasch process, solution mining of minerals, in situ combustion of fossil fuel, or recovery of geothermal energy? (FORM 4)		X	
I. Is this facility a proposed **stationary source** which is one of the 28 industrial categories listed in the instructions and which will potentially emit 100 tons per year of any air pollutant regulated under the Clean Air Act and may affect or be located in an attainment area? (FORM 5)		X	
J. Is this facility a proposed stationary source which is NOT one of the 28 industrial categories listed in the instructions and which will potentially emit 250 tons per year of any air pollutant regulated under the Clean Air Act and may affect or be located in an attainment area? (FORM 5)		X	

III. NAME OF FACILITY

C 1 SKIP MOLYCORP INC

IV. FACILITY CONTACT

A. NAME & TITLE *(last, first, & title)*: C 2 DAVID R SHOEMAKER MINE MANAGER

B. PHONE *(area code & no.)*: 505 586 0212

V. FACILITY MAILING ADDRESS

A. STREET OR P.O. BOX: C 3 PO BOX 469

B. CITY OR TOWN: C 4 QUESTA

C. STATE: NM

D. ZIP CODE: 87556

VI. FACILITY LOCATION

A. STREET, ROUTE NO. OR OTHER SPECIFIC IDENTIFIER: C 5 EAST OF QUESTA ON HIGHWAY 38

B. COUNTY NAME: TAOS

C. CITY OR TOWN: C 6 QUESTA

D. STATE: NM

E. ZIP CODE: 87556

F. COUNTY CODE *(if known)*:

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VII. SIC CODES *(4-digit, in order of priority)*

A. FIRST	B. SECOND
7 1061 (specify) METAL MINING - FERRO ALLOY ORES	7 (specify)
C. THIRD	**D. FOURTH**
7 (specify)	7 (specify)

VIII. OPERATOR INFORMATION

A. NAME: 8 MOLYCORP INC

B. Is the name listed in Item VIII-A also the owner? [X] YES [ ] NO

C. STATUS OF OPERATOR *(Enter the appropriate letter into the answer box; if "Other", specify.)*
F = FEDERAL
S = STATE
P = PRIVATE
M = PUBLIC *(other than federal or state)*
O = OTHER *(specify)*

P (specify)

D. PHONE *(area code & no.)*: A 505 586 0212

E. STREET OR P.O. BOX: PO BOX 469

F. CITY OR TOWN: B QUESTA

G. STATE: NM

H. ZIP CODE: 87556

IX. INDIAN LAND

Is the facility located on Indian lands? [ ] YES [X] NO

X. EXISTING ENVIRONMENTAL PERMITS

A. NPDES *(Discharges to Surface Water)*: 9 N NM0022306	D. PSD *(Air Emissions from Proposed Sources)*: 9 P
B. UIC *(Underground Injection of Fluids)*: 9 U	E. OTHER *(specify)*: 9 (specify)
C. RCRA *(Hazardous Wastes)*: 9 R	E. OTHER *(specify)*: 9 (specify)

XI. MAP

Attach to this application a topographic map of the area extending to at least one mile beyond property boundaries. The map must show the outline of the facility, the location of each of its existing and proposed intake and discharge structures, each of its hazardous waste treatment, storage, or disposal facilities, and each well where it injects fluids underground. Include all springs, rivers and other surface water bodies in the map area. See instructions for precise requirements.

XII. NATURE OF BUSINESS *(provide a brief description)*

MINING AND MILLING OPERATIONS PRODUCING MOLYBDENUM DISULFIDE CONCENTRATION.

XIII. CERTIFICATION *(see instructions)*

I certify under penalty of law that I have personally examined and am familiar with the information submitted in this application and all attachments and that, based on my inquiry of those persons immediately responsible for obtaining the information contained in the application, I believe that the information is true, accurate and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment.

A. NAME & OFFICIAL TITLE *(type or print)*	B. SIGNATURE	C. DATE SIGNED
DAVID R. SHOEMAKER, MINE MANAGER	David R Shoemaker	9/22/87

COMMENTS FOR OFFICIAL USE ONLY

C

EPA Form 3510-1 (6-80) REVERSE

Additional certification: R. G. Dewey, President, Mining, Molycorp, Inc.

SECTION II

EPA STANDARD FORM 2C

WASTEWATER DISCHARGE INFORMATION

EPA I.D. NUMBER *(copy from Item 1 of Form 1)*

Please print or type in the unshaded areas only.

Form Approved.
OMB No. 2000-0059
Approval expires 12-31-85

FORM 2C NPDES

EPA

U.S. ENVIRONMENTAL PROTECTION AGENCY
APPLICATION FOR PERMIT TO DISCHARGE WASTEWATER
EXISTING MANUFACTURING, COMMERCIAL, MINING AND SILVICULTURAL OPERATIONS
Consolidated Permits Program

I. OUTFALL LOCATION

For each outfall, list the latitude and longitude of its location to the nearest 15 seconds and the name of the receiving water.

A. OUTFALL NUMBER *(list)*	B. LATITUDE			C. LONGITUDE			D. RECEIVING WATER *(name)*
	1. DEG.	2. MIN.	3. SEC.	1. DEG.	2. MIN.	3. SEC.	
001	36	40	61	105	38	30	RED RIVER
002	36	40	61	105	37	30	RED RIVER

II. FLOWS, SOURCES OF POLLUTION, AND TREATMENT TECHNOLOGIES

A. Attach a line drawing showing the water flow through the facility. Indicate sources of intake water, operations contributing wastewater to the effluent, and treatment units labeled to correspond to the more detailed descriptions in Item B. Construct a water balance on the line drawing by showing average flows between intakes, operations, treatment units, and outfalls. If a water balance cannot be determined *(e.g., for certain mining activities)*, provide a pictorial description of the nature and amount of any sources of water and any collection or treatment measures.

B. For each outfall, provide a description of: (1) All operations contributing wastewater to the effluent, including process wastewater, sanitary wastewater, cooling water, and storm water runoff; (2) The average flow contributed by each operation; and (3) The treatment received by the wastewater. Continue on additional sheets if necessary.

1. OUTFALL NO. *(list)*	2. OPERATION(S) CONTRIBUTING FLOW		3. TREATMENT		
	a. OPERATION *(list)*	b. AVERAGE FLOW *(include units)*	a. DESCRIPTION	b. LIST CODES FROM TABLE 2C-1	
001	PROCESS WATER FROM MILLING	2000 GPM	SETTLING AND TAILINGS		I-U
	OPERATIONS. TAILINGS DIS-		IMPOUNDMENT		
	POSAL				
	SEE DISCUSSION SECTION III		ION EXCHANGE		2-J
002	SEEPAGE FROM TAILINGS	350 GPM	NONE REQUIRED		
	IMPOUNDMENT				

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OFFICIAL USE ONLY *(effluent guidelines sub-categories)*

CONTINUED FROM THE FRONT

C. Except for storm runoff, leaks, or spills, are any of the discharges described in Items II-A or B intermittent or seasonal?

☐ YES *(complete the following table)* ☐ NO *(go to Section III)*

1. OUTFALL NUMBER *(list)*	2. OPERATION*(s)* CONTRIBUTING FLOW *(list)*	3. FREQUENCY a. DAYS PER WEEK *(specify average)*	3. FREQUENCY b. MONTHS PER YEAR *(specify average)*	4. FLOW a. FLOW RATE *(in mgd)* 1. LONG TERM AVERAGE	4. FLOW a. FLOW RATE *(in mgd)* 2. MAXIMUM DAILY	4. FLOW b. TOTAL VOLUME *(specify with units)* 1. LONG TERM AVERAGE	4. FLOW b. TOTAL VOLUME *(specify with units)* 2. MAXIMUM DAILY	4. FLOW c. DURATION *(in days)*
001	SEE DISCUSSION IN SECTION III							

III. PRODUCTION

A. Does an effluent guideline limitation promulgated by EPA under Section 304 of the Clean Water Act apply to your facility?

☒ YES *(complete Item III-B)* ☐ NO *(to to Section IV)*

B. Are the limitations in the applicable effluent guideline expressed in terms of production *(or other measure of operation)*?

☐ YES *(complete Item III-C)* ☒ NO *(go to Section IV)*

C. If you answered "yes" to Item III-B, list the quantity which represents an actual measurement of your level of production, expressed in the terms and units used in the applicable effluent guideline, and indicate the affected outfalls.

1. AVERAGE DAILY PRODUCTION a. QUANTITY PER DAY	b. UNITS OF MEASURE	c. OPERATION, PRODUCT, MATERIAL, ETC. *(specify)*	2. AFFECTED OUTFALLS *(list outfall numbers)*

IV. IMPROVEMENTS

A. Are you now required by any Federal, State or local authority to meet any implementation schedule for the construction, upgrading or operation of wastewater treatment equipment or practices or any other environmental programs which may affect the discharges described in this application? This includes, but is not limited to, permit conditions, administrative or enforcement orders, enforcement compliance schedule letters, stipulations, court orders, and grant or loan conditions.

☐ YES *(complete the following table)* ☒ NO *(go to Item IV-B)*

1. IDENTIFICATION OF CONDITION, AGREEMENT, ETC.	2. AFFECTED OUTFALLS a. NO.	2. AFFECTED OUTFALLS b. SOURCE OF DISCHARGE	3. BRIEF DESCRIPTION OF PROJECT	4. FINAL COMPLIANCE DATE a. REQUIRED	4. FINAL COMPLIANCE DATE b. PROJECTED

B. OPTIONAL: You may attach additional sheets describing any additional water pollution control programs *(or other environmental projects which may affect your discharges)* you now have underway or which you plan. Indicate whether each program is now underway or planned, and indicate your actual or planned schedules for construction. ☐ MARK "X" IF DESCRIPTION OF ADDITIONAL CONTROL PROGRAMS IS ATTACHED

 CONTINUE ON PAGE 3

CONTINUED FROM PAGE 2

V. INTAKE AND EFFLUENT CHARACTERISTICS

A, B, & C: See instructions before proceeding — Complete one set of tables for each outfall — Annotate the outfall number in the space provided.
NOTE: Tables V-A, V-B, and V-C are included on separate sheets numbered V-1 through V-9.

D. Use the space below to list any of the pollutants listed in Table 2c-3 of the instructions, which you know or have reason to believe is discharged or may be discharged from any outfall. For every pollutant you list, briefly describe the reasons you believe it to be present and report any analytical data in your possession.

1. POLLUTANT	2. SOURCE	1. POLLUTANT	2. SOURCE
NONE			

VI. POTENTIAL DISCHARGES NOT COVERED BY ANALYSIS

Is any pollutant listed in Item V-C a substance or a component of a substance which you currently use or manufacture as an intermediate or final product or byproduct?

☐ YES *(list all such pollutants below)* ☒ NO *(go to Item VI-B)*

CONTINUED FROM THE FRONT

VII. BIOLOGICAL TOXICITY TESTING DATA

Do you have any knowledge or reason to believe that any biological test for acute or chronic toxicity has been made on any of your discharges or on a receiving water in relation to your discharge within the last 3 years?

[X] YES *(identify the test(s) and describe their purposes below)* [ ] NO *(go to Section VIII)*

ASSESSMENT OF THE ACUTE TOXICITY OF OUTFALL 002 TO DAPHNIA MAGNA, AS REQUIRED UNDER OUR PRESENT PERMIT, PART III, PAGE 22 OF 23, PERMIT NO. NM 0022306, RESULTS OF TEST ARE ENCLOSED. ALSO A SAMPLE FROM OUR RECEIVING WATERS THE RED RIVER, WAS ANALYZED FOR ACUTE TOXICITY TO DAPHNIA MAGNA ON JANUARY 1987, TEST REPORT IS ENCLOSED.

VIII. CONTRACT ANALYSIS INFORMATION

Were any of the analyses reported in Item V performed by a contract laboratory or consulting firm?

[ ] YES *(list the name, address, and telephone number of, and pollutants analyzed by, each such laboratory or firm below)* [X] NO *(go to Section IX)*

A. NAME	B. ADDRESS	C. TELEPHONE *(area code & no.)*	D. POLLUTANTS ANALYZED *(list)*

IX. CERTIFICATION

I certify under penalty of law that this document and all attachments were prepared under my direction or supervision in accordance with a system designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who manage the system or those persons directly responsible for gathering the information, the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations.

A. NAME & OFFICIAL TITLE *(type or print)*	B. PHONE NO. *(area code & no.)*
David R. Shoemaker, Mine Manager Molycorp, Inc.	(505) 586-0212
C. SIGNATURE	**D. DATE SIGNED**
David R. Shoemaker	9/22/87

SECTION III

DESCRIPTION OF TAILINGS DISPOSAL AND ION EXCHANGE PLANT OPERATIONS AND FLOW DIAGRAM OF OPERATION

DESCRIPTION OF TAILINGS DISPOSAL

~~More than 90% of the water taken into the mill from the four wells, spring and river water and mine drainage is used, or will be used, for extracting and processing ore.~~ Relatively small quantities of water are used for domestic purposes; wastewater from these processes is treated in either a septic tank or a wastewater treament plant permitted by the State of New Mexico under Ground Water Discharge Plan (DP-132).

All water discharged from Outfall 001 and 002 is process water from the mill. Tailings, which is a slurry of approximately 60% water and 40% crushed waste rock, are transported to the tailings disposal area. The tailings are deposited in an impoundment where the solids are allowed to settle and the water is decanted off. The water then flows to a second settling pond; is transported to a new Ion Exhange Plant (see attached Appendix I), and finally to Pope Lake. Pope Lake will be stocked with rainbow trout as a final water quality check before discharge into the Red River.

~~Cyanide is not presently being used~~ in the milling operations. ~~If use is resumed,~~ inadequate photodecomposition of residual cyanide may be experienced in cold months. In order to meet effluent limitations for cyanide in winter months, water treatment may be required. Hydrogen Peroxide will be added to the influent of Pope Lake to oxidize the free cyanide. The Pope Lake influent will be carefully monitored during the winter months to control the peroxide addition.

As is generally experienced with any impoundment, seepage within the impoundment occurs. One of the major design considerations for dam construction is collection of seepage to control flow through the dam. In order to insure dam stability, a chimney drain is built into the dam, channeling seepage into collection drains at the toe of the dam. This water is collected and discharges through Outfall 002. Water from Outfall 002 is untreated and has consistently stayed within all permit limitations of the existing permit.

Management of the tailings impoundment requires careful attention to many factors. The primary purpose of the process is disposal of the crushed waste rock from which the molybdenum is extracted. While water treatment, recovery, and discharge are important considerations in tailings management, of ultimate concern is maintaining the stability of the dam structure.

In the Questa operations, tailings discharge locations are alternately selected ~~to insure that water decanted from the tailings flows away from the dam and collects in a settling pond.~~ By varying the tailings discharge point, even distribution of solids is achieved throughout the pond while still maintaining a settling pond near the water decant ditch. Retention time of the ponded water can also be controlled by varying the discharge points. Adequate settling time is required for maintaining water quality.

Often, settled tailings will dry to a depth of several feet between discharge times. A dust control system has been implemented at the impoundment to stabilize dry tailings. New discharge on a dry section of tailings will cause retention of some water in the clay and slime portions of the

settled tailings, thus reducing the amount of water which is decanted and discharged. Depending upon the distance between the tailings discharge point and the water decant ditch, varying flows may be experienced through the settling ponds. Since the mill has been inoperative since February 1986, the settled tailings in the impoundment have dried significantly. During the normal mill operations, flow through Pope Lake and Outfall 001, is continuous. Approximately 300 acres of dry tailings have been capped with varying thicknesses of topsoil or other borrow material such as alluvial gravels for dust control. About half of the covered areas have been seeded in different grass and clover mixtures. The remaining uncapped areas are either kept wet or are being kept controlled through the use of a chemical stabilizer. These capped and seeded areas will be used for future tailings storage upon resumption of mining and milling operations.

Dewey, President of Molycorp, Inc. authorizing Mr. David R. Shoemaker, Mine Manager, Questa Operations, to sign all correspondence regarding the NPDES permit, and a copy of a Power of Attorney authorizing Mr. Shoemaker to act as "the true, sufficient and lawful agent and attorney for the Company". Also included is a request for continued approval of the alternative molybdenum test procedure which was approved by EPA in 1977 for the existing permit.

Please call me at (505) 586-0212 if you have any questions regarding this application or if you need additional information or clarification.

Respectfully submitted,

David R. Shoemaker

David R. Shoemaker,
Mine Manager

Encl:

cc: John Ackerman
LeRoy E. Apodaca

files

MOLYCORP ION EXCHANGE PLANT

1.0 INTRODUCTION

Molybdenum in the form of the solid mineral molybdenite (MoS_2) is recovered by a conventional crushing, grinding and flotation process in the main Molycorp plant. During this process, however, a small proportion of the molybdenum dissolves in the water used for the grinding and flotation process and results in water contaminated by molybdate ions ($MoO_4^=$). This water, together with suspended barren tailings particles, then flows to the tailings impoundment area where the slurry is collected in tailings ponds. The solids are allowed to settle in these ponds with the now clear but contaminated water overflowing the tailings ponds for discharge to the Red River after being decontaminated by the ion exchange plant. Hence, the purpose of the ion exchange plant is to remove the environmentally unacceptable molybdate ions ($MoO_4^=$) from the water before discharge to the Red River.

2.0 THE ION EXCHANGE PROCESS

The molybdate ($MoO_4^=$) ions are removed from the water by passing it through beds of ion exchange resin. The resin can be described as billions of tiny spherical "sponge-like" beads which are saturated with environmentally acceptable bisulphate ions (HSO_4^-). When the molybdate ions come in contact with the resin, they preferentially attach to the resin and in so doing displace harmless bisulphate ions into the water. Thus, as the name ion exchange resin implies, the process of decontamination is achieved by exchanging harmful ions in solution for harmless bisulphate ions.

This exchange process is most efficient under slightly acid conditions and hence very small quantities of sulphuric acid (H_2SO_4) are added to the water prior to contact with the resin and, after the ion exchange is complete, this acid is neutralized by lime ($Ca(OH)_2$) addition prior to discharge to Red River.

3.0 REGENERATING THE RESIN

When one of the four beds of resin has become saturated by molybdate ions, it is known as loaded resin and is transferred to the elution vessel where sodium hydroxide (NaOH) solution is passed through the resin. The sodium hydroxide solution contains hydroxyl ions (OH-) which displace the molybdate (MoO_4=) ions from the resin in a very concentrated form. This solution is referred to as eluant and is stored in one of the three eluant tanks for further processing. Having removed the molybdate ions from the resin and replaced them with hydroxyl ions, the resin is now in the "free-base" form and hence must be converted back to the bisulphate form before it is ready to resume its duty of removing molybdate ions from the contaminated water. This is achieved by passing dilute sulphuric acid through the resin while it is still in the elution vessel thus once again saturating it with bisulphate (HSO_3-) ions and converting it to the form which is active for the removal of molybdate ions. The eluted resin is then transferred back to the loading column to resume its normal duty.

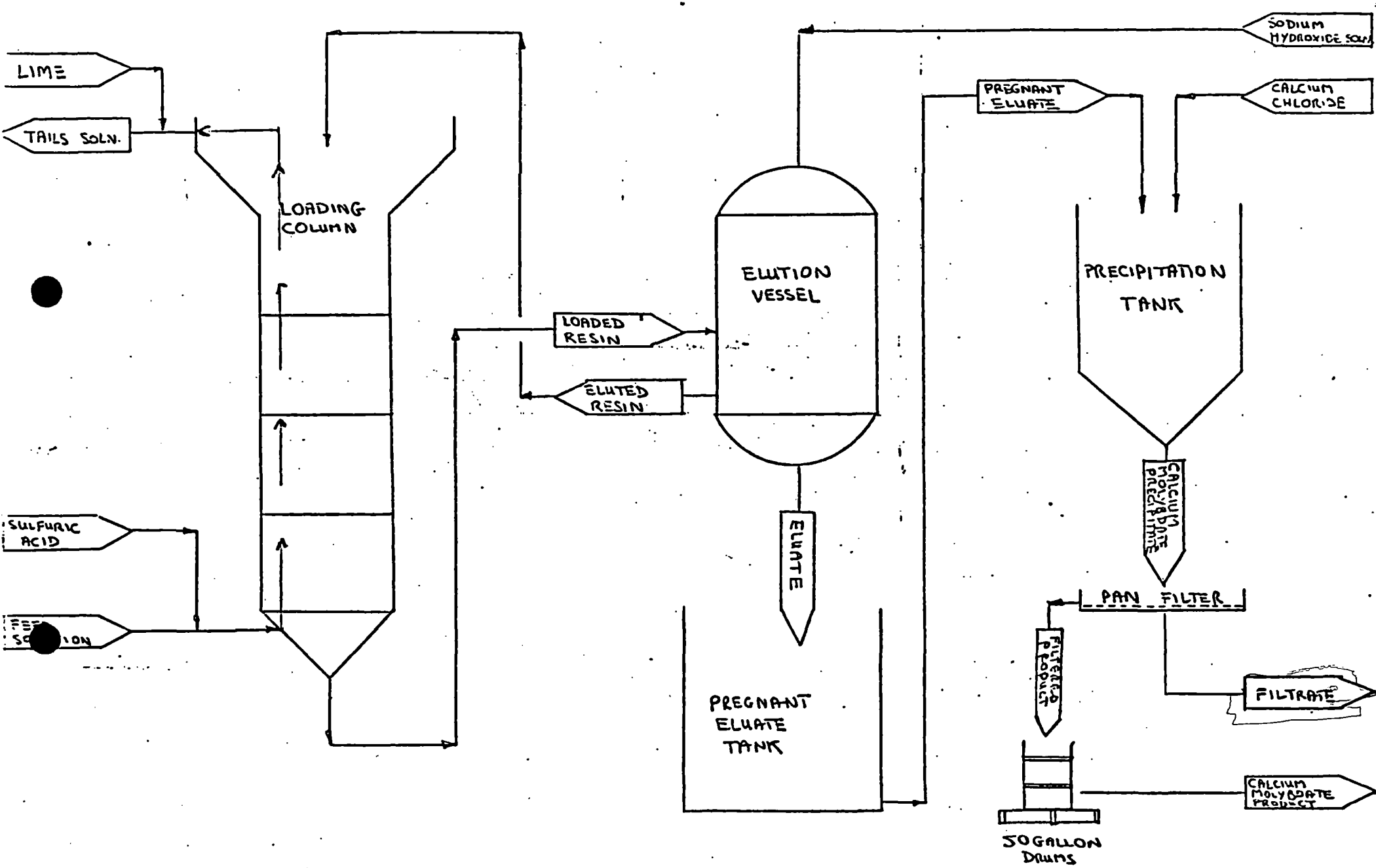
4.0 PRECIPITATING THE MOLYBDENUM

The final step in the process is removing the molybdate ions from the concentrated pregnant eluate and converting them to the solid form for disposal or sale.

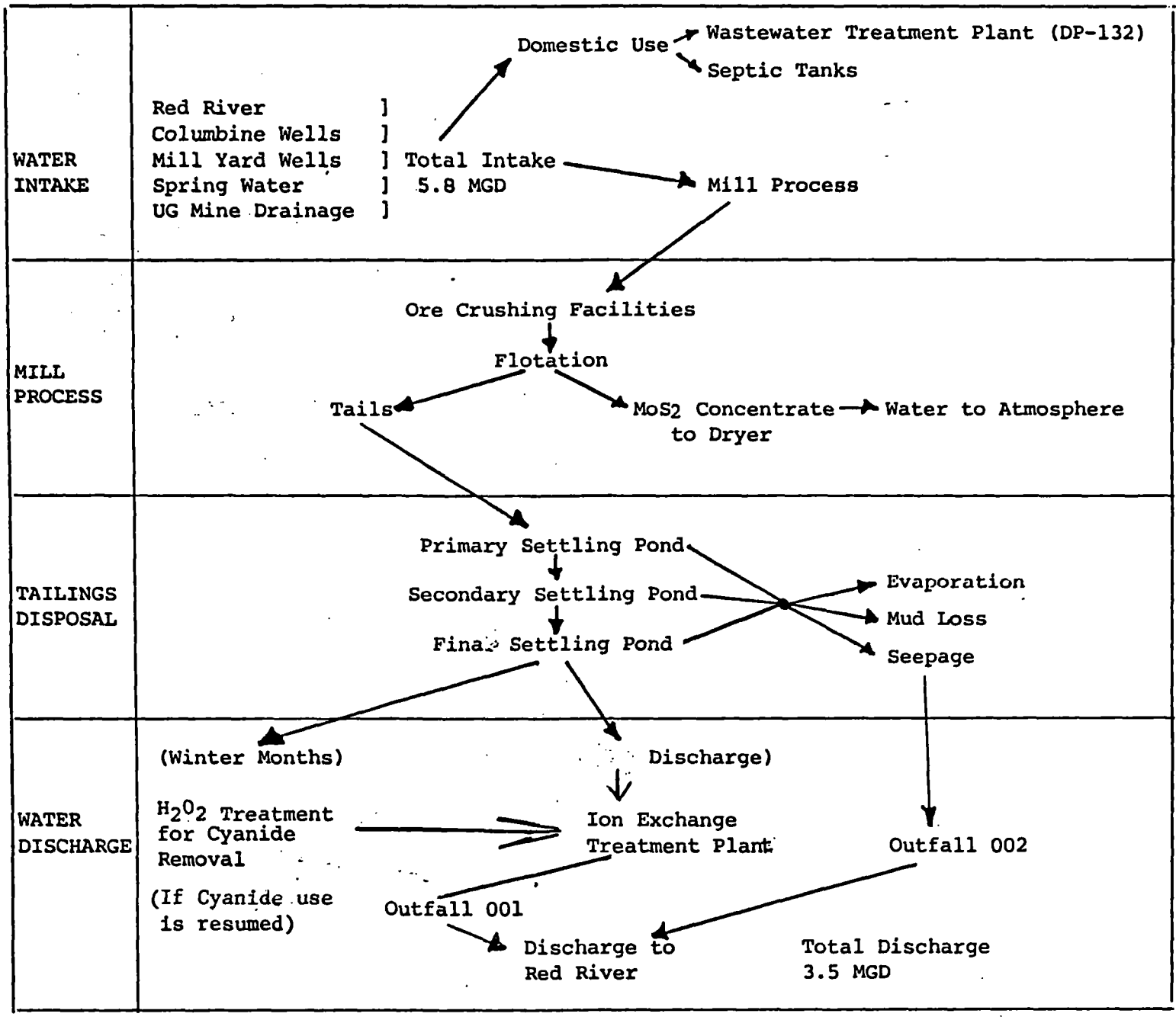
The pregnant eluate is pumped from the pregnant eluate tank to the precipitation tank where solid calcium chloride ($CaCl_2$) is added by conveyor. The calcium chloride dissolves in the pregnant eluate to form calcium ions (Ca++) and chloride ions (Cl-). The calcium ions then combine with the molybdate ions to form calcium molybdate ($CaMoO_4$) which is insoluble and precipitates as a pink/white solid in the precipitation tank. The calcium molybdate is then allowed to settle and is drained from the tank onto pan filters where excess moisture is drawn from the solid calcium molybdate prior to discharge into drums for transport away from the plant.

MOLYCORP ION EXCHANGE PLANT.

SIMPLIFIED FLOW DIAGRAM



FLOW DIAGRAM OF MOLYCORP, INC. QUESTA OPERATION



SECTION V

LETTER OF AUTHORIZATION FOR MINE MANAGER AS SIGNATOR

P/A

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

DATE: June 12, 1998
NPDES NUMBER: NM0022306 - Molycorp
SUBJECT: EXPIRING PERMIT
EXPIRATION DATE: 101498
DATE APP. REC'D: 052198
FROM: JACKIE GREENSAGE (6WQ-CA)
TO: Jane Watson
RETURN TO JACKIE GREENSAGE (6WQ-CA)

The attached application is for the expiring NPDES permit reference above.

In accordance with the new consolidated regulations, a completeness review is required and a response must be given to the applicant within 60 days from the receipt of this application.

	COMPLETED DATE	INITIALS
6W-PI Completeness Review Completed	6-17-98	JGW
6W-PS Mailing of Completeness/ Deficiency Determination		

PLEASE CIRCLE:

Is this application COMPLETE or INCOMPLETE? JGW

If incomplete, please provide explanation:

Should this application be sent for New Source Review? YES or NO

JGW

PLEASE CIRCLE:

At last review was determination MAJOR or MINOR? SCORE _____

Has this Major/Minor Determination changed? YES or NO

Indicate new determination. MAJOR or MINOR SCORE _____

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 6
1445 ROSS AVENUE, SUITE 1200
DALLAS, TX 75202-2733

SEP 22 1992

NM0022306
MOLYCORP INC. QUESTA DIV.
DAVID R SHOEMAKER, GEN. SUPT.
P. O. BOX 469
QUESTA NM 87556

Dear Permittee:

A review of our records indicates that your National Pollutant Discharge Elimination System (NPDES) permit will soon expire. If you wish to continue to discharge subsequent to the expiration date of your permit, you must re-apply for your permit not less than **180** days prior to the permit expiration date.

This will be the only notice you will receive from the Agency advising you of the need for re-applying for your permit. If you anticipate your facility will be discharging after your permit expires, you should submit the enclosed application forms to the office not less than **180** days prior to the expiration date on your current permit. If however, you determine a permit will no longer be required for your facility, you should notify the Agency of this fact. It is considered unlawful to continue discharging from your facility without timely reapplication for your permit. Applications not received in a timely manner will be referred to our Enforcement Branch for appropriate action.

Once again, this is the **only** notice the Agency will issue advising you of the expiration of your permit. Subsequent correspondence will be from our Enforcement Branch.

Your cooperation in this matter will be appreciated. If you have any questions, please contact me at the above address or telephone (214) 655-7190.

Sincerely,

Stephanie Abbott

Jenaie Franke
Environmental Protection Specialist
Permits Issuance Section (6W-PS)

Enclosure

Printed on Recycled Paper

MAR 6 1985

CERTIFIED MAIL - RETURN RECEIPT REQUESTED (P 423 868 806)

Mr. C. R. Sacrison
General Manager
Molycorp, Inc.
P.O. Box 469
Questa, New Mexico 87556

Re: NPDES Permit Application (NM-0022306)
New Source Determination (NM9-29)

Dear Mr. Sacrison:

We have reviewed the information dated November 26, 1984, for the proposed addition of a facility to further reduce the lead content from molybdenum concentrate produced at the Questa facility. The proposed action is an existing source and not a new source as defined at Section 306 of the Clean Water Act and 40 CFR Part 440, Subpart J. This is a preliminary determination which may be challenged by a request for an evidentiary hearing in accordance with 40 CFR 122.21(k)(4) (45 Federal Register 33444, May 19, 1980) within thirty (30) days of the issuance of the public notice.

If you need assistance in this matter, please contact Hector Pena at (214) 767-6661.

Sincerely yours,

Clinton B. Spotts
Chief, Federal Activities Branch (6ES-FA)

bcc: Becker (6W-PI)
Jack Ferguson

RECEIVED
MAR 11 1985
6W-PS

6ES-FA:Pena:X6661:ba:X2716:3/1/85:FA-118

6ES-FA

MINTZ

HEALTH & ENVIRONMENT department

STATE OF NEW MEXICO

ENVIRONMENTAL IMPROVEMENT DIVISION
P.O. Box 968, Santa Fe, New Mexico 87504-0968
(505) 984-0020

TONEY ANAYA
GOVERNOR

DENISE D. FORT
DIRECTOR

April 8, 1985

Mr. Myron Knudson
U.S. Environmental Protection Agency
Interfirst Two Building
1201 Elm Street
Dallas, Texas 75270

Re: State Certification

Dear Mr. Knudson,

Enclosed please find state certification for the following industrial permits

Molycorp	NM0022306
City of Farmington/Animas Steam Plant	NM0000043
Kaiser Steel Corp.	NM0000205

Other minor changes are enclosed on separate sheets. Also enclosed are comments submitted by our state Energy and Minerals Department (EMD) suggesting additional effluent limitations for Kaiser Steel.

We request that the Energy and Minerals Department recommendations be given careful consideration.

Sincerely,

Charles Nylander

Charles Nylander
Chief
Surface Water Quality Bureau

cc: EID District Managers, I & II
Farmington EID Field Office
Taos EID Field Office
Raton EID Field Office

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APR 11 1985
6W-PS

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION VI
ALLIED BANK TOWER AT FOUNTAIN PLACE
1445 ROSS AVENUE
DALLAS, TEXAS 75202

August 4, 1987

FACT SHEET

for proposed National Pollutant Discharge Elimination System (NPDES) Permit No. NM0022306 to discharge to waters of the United States.

Issuing office: U. S. Environmental Protection Agency
Region VI
Allied Bank Tower
1445 Ross Avenue
Dallas, Texas 75202-2733

Applicant: Molycorp, Inc.
P.O. Box 469
Questa, New Mexico 87556

1. The applicant currently operates a molybdenum mining and milling operation.

2. As described in the application, the plant site is located in Taos County, New Mexico. Discharge is to the Red River in Segment No. 2-120 of the Rio Grande Basin.

3. The known uses of the receiving waters are: coldwater fishing, fish culture, livestock and wildlife watering, and secondary contact recreation.

4. Stream standards are: the general and specific stream standards are provided in "Water Quality Standards for Interstate and Intrastate Streams in New Mexico", New Mexico Water Quality Control Commission, February 15, 1985.

5. The following is a quantitative description of the discharge described in the application:

a.

Outfall	Flow Frequency	Avg/Daily (MGD)	Max/(MGD)	Min/(MGD)
001	Intermittent	3.31	4.32	N/A
002	Intermittent	N/A	0.61	N/A

b.

Outfall	Temp. °F Avg/Summer	Temp. °F Avg/Winter	Temp. °F Max	Temp. °F Min
001	N/A	N/A	N/A	N/A
002	N/A	N/A	N/A	N/A

PN 3-9-85 Hunke 4-11-85

Richard Whittington, Regional Administrator
Environmental Protection Agency
1201 Elm Street
Dallas, TX 75270

April 4, 1985

STATE CERTIFICATION

Re:
Molycorp, Inc.
P.O. Box 469
NM0022306 March 8, 1985

Dear Mr. Whittington:

The New Mexico Environmental Improvement Division has examined the application for and the proposed NPDES permit NM0022306 above. The following conditions are necessary to assure compliance with the applicable provisions of the Clean Water Act Sections 208(e), 301, 302, 303, 306, and 307 and with appropriate requirements of State law. Compliance with the terms and conditions of the permit and this certification, will provide reasonable assurance that the permitted activities, will be conducted in a manner which will not violate applicable water quality standards.

The State of New Mexico

( ) includes the following more stringent conditions and citation to the State or Federal requirements upon which those conditions are based (see attachments).

(X) certifies that the discharge will comply with the applicable provisions of Sections 208(e), 301, 302, 303, 306 and 307 of the Clean Water Act and with apropriate requirements of State law.

( ) waives its right to certify

( ) denies certification for the reasons stated in the attachment

In order to meet the requirements of State law, including water quality standards and appropriate basin plan, each of the conditions cited in the draft permit and the State certification, shall not be made less stringent.

Please contact John Gould if you have any questions concerning this certification. Please refer to the enclosed sheet for typographical and minor changes which are to be made before the final permit is issued.

Sincerely,

Charles Nylander

Charles Nylander, Chief
Surface Water Quality Bureau

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Molycorp Permit # NM0022306

1. Page 1 To "receiving water named Red River" add " . . . Segment 2-119 of Upper Rio Grande Basin

2. Page 1 Fill in effective date and expiration date of permit

3. Page 3 3 month and 6 month Progress Report due dates will have passed by effective date of new permit.

4. Page 14 Molycorp is a major industrial discharger. Please include appropriate day of the month DMR is due and date of first DMR.

5. Page 14 Fill in state reporting address:

   Program Manager
   Surface Water Section
   Surface Water Quality Bureau
   N.M. Environmental Improvement Division
   P.O. Box 968
   Santa Fe, New Mexico 87504-0968

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APR 11 1985
6W-PS

~~I~~ ~~6 mo AVE.~~

II For Mo & Mn — See Part III.

① When 001 does not Q (flow) for a mo. then the [ ] limits are suspended. (This is clear).

② But what if 001 does not Q for only 1 day. Are [ ] limits suspended for the DA for that mo or just DM, or what?

Further, No further reporting reqmt's. in Part III to require Co. to tell us when a no Q exists at 001. So how can our Enf. people know how to Enforce?

To typing
1/17/85

~~Roger~~ 6W-E

Jack - See MOK's note inside -

Should I concur?

What is relationship to C.D.??

Roy

12/14

ROUTING AND TRANSMITTAL SLIP

Date 1/3/85

TO: (Name, office symbol, room number, building, Agency/Post)	Initials	Date
1. Shirley		
2.		
3.		
4.		
5.		

	Action		File		Note and Return
	Approval		For Clearance		Per Conversation
	As Requested		For Correction		Prepare Reply
	Circulate		For Your Information		See Me
	Comment		Investigate		Signature
	Coordination		Justify		

REMARKS

Permit requirements will requirement some extra work to load into PCS but should not present any problems with data entry or DMR preprinting.

DO NOT use this form as a RECORD of approvals, concurrences, disposals, clearances, and similar actions

FROM: (Name, org. symbol, Agency/Post)	Room No.—Bldg.
Tom L	Phone No.

5041-102

OPTIONAL FORM 41 (Rev. 7-76)
Prescribed by GSA
FPMR (41 CFR) 101-11.206

☆ U.S.G.P.O.: 1984-421-529/412

Jack - You had 3 concerns

* 1. Molybdenum limits are not tech. based but are W.Q. based, via previous Evi. Hearing settlement with State. So the 6 mo. ave. limit is W.Q. based. They do have to meet a D.M. limit, however.

2. Asterisks added for clarification per your request.

3. Re: 002 suspension of [ ] limits for Molybdenum — they are still subject to mass limits so they are not without regulation at this outfall during "0" flow at 001.

* As you know, we have very few permits that deviate from normal DA / DM. This is one of those exceptions.

Jim

Molycorp, Inc.

Questa Division
P.O. Box 469
Questa, New Mexico 87556
Telephone: (505) 586-0212

union 76
MOLYCORP

March 13, 1985

Permit File
NM0022306

Mr. Dick Whittington
Regional Administrator
U. S. Environmental Protection Agency
Region VI
1201 Elm Street
Dallas, TX 75270

Dear Mr. Whittington:

Enclosed is the Discharge Monitoring Report for the month of February, 1985, NPDES Permit #NM 0022306.

For outfall 002, the following excursions occurred during the month of February.

1. Total manganese daily average of 1.0 mg/L, reported at 1.2 mg/L.
2. Total molybdenum daily average of 1.0 mg/L, reported at 2.3 mg/L.
3. Total molybdenum daily maximum of 2.0 mg/L, reported at 2.6 mg/L.

These parameters are directly addressed in my June 29, 1984 letter to your Agency requesting permit modifications. As I indicated to you in the January report, Molycorp is anxious to resolve this issue and requests that either the manganese and molybdenum limits for 002 be voided during periods when outfall 001 is not discharging or that the daily average and daily maximum limitations be increased on a permanent basis.

If you have any questions, please feel free to call me or Lee Lockie.

Sincerely,

C. R. Sacrison
General Manager

CRS:pcr

cc: Myron Knudson, Water Management Division
Charles Nylander, Environmental Improvement Division
Fred Humke, Water Management Division

Molycorp, Inc.
Questa Division
P. O. Box 469
Questa, New Mexico 87556
Telephone: (505) 586-0212

Permit File
NM00 22306

UNION 76
MOLYCORP

February 12, 1985

Mr. Dick Whittington
Regional Administrator
U. S. Environmental Protection Agency
Region VI
1201 Elm Street
Dallas, TX 75270

Dear Mr. Whittington:

Enclosed is the Discharge Monitoring Report for the month of January, 1985, NPDES Permit #NM 0022306.

For outfall 002, the following excursions occurred during the month of January.

1. Total manganese daily average of 1.0 mg/L, reported at 1.4 mg/L.
2. Total molybdenum daily average of 1.0 mg/L, reported at 2.6 mg/L.
3. Total molybdenum daily maximum of 2.0 mg/L, reported at 2.7 mg/L.

These parameters are directly addressed in my June 29, 1984 letter to your Agency requesting permit modifications. As I indicated to you in the December report, Molycorp is anxious to resolve this issue and requests that either the manganese and molybdenum limits for 002 be voided during periods when outfall 001 is not discharging or that the daily average and daily maximum limitations be increased on a permanent basis.

If you have questions, please feel free to call me or Lee Lockie.

Sincerely,

C. R. Sacrison
General Manager

CRS:pcr

cc: Myron Knudson, Water Management Division
Charles Nylander, Environmental Improvement Division

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FEB 19 1985
6W-PS

Molycorp, Inc.
Questa Division
Post Office Box 469
Questa, New Mexico 87556

QUESTA
FEB 15 '85
U.S. POSTAGE
.37

union 76
MOLYCORP

Molycorp, Inc.
Questa Division
P.O. Box 469
Questa, New Mexico 87556

union
MOLYCORP

Mr. Fred Humke
Water Management Division
U. S. Environmental Prot. Agency
Region VI
1201 Elm Street
Dallas, TX 75270

J.P.

Molycorp, Inc.

Questa Division
P. O. Box 469
Questa, New Mexico 87556
Telephone: (505) 586-0212

Permit File
NM0022306

union 76 MOLYCORP

February 4, 1985

Mr. Fred Humke
Environmental Engineer
U. S. Environmental Protection Agency
1201 Elm Street
Dallas, TX 75270

Dear Mr. Humke:

Enclosed please find a copy of the letter we discussed in our conversation of last month.

Please call me if you have questions.

Lee Lockie

Lee Lockie
Environmental Coordinator

LL:pcr
Enclosure

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FEB 7 1985
6W-PS

Molycorp, Inc.

Questa Division
P. O. Box 469
Questa, New Mexico 87556
Telephone: (505) 586-0212

UNION 76
MOLYCORP

November 26, 1984

Mr. Dick Whittington
Regional Administrator
U. S. Environmental Protection Agency
1201 Elm Street
Dallas, TX 75270
Attn: Myron O. Knudson, Director
Water Management Division

RE: NPDES Permit No. NM 022306

Dear Mr. Whittington:

As required under Part II, Section D of the company's NPDES Permit No. NM 0022306, Molycorp, Inc. hereby notifies the Environmental Protection Agency of a planned physical alteration to its Questa concentrate processing facility. For your information, I am enclosing a detailed description of this process change and an analysis of its impact on water quality.

In short, the steel industry has established specifications for molybdenum oxide (MoO_3) usage in alloying which Molycorp is unable to meet without further treatment of the mill concentrate. In order to provide a competitive and saleable product, it has become necessary to construct a facility to reduce lead content to specification levels. Construction of the facility has begun and it should be in operation by July of 1985.

Since this lead removal plant is so substantially integrated into the existing concentrate processing facility, Molycorp believes that this process change does not meet the necessary criteria for designation as a "new source" under 40 CFR 122.66(b).

Please contact me or Lee Lockie if you have questions concerning this change.

Very truly yours,

C. R. Sacrison
General Manager

CRS:pcr

cc: Charles Nylander, EID
Enclosures

RECEIVED
FEB 7 1985
6W-PS

DESCRIPTION OF PROCESS CHANGE

I. BACKGROUND

Molycorp, Inc. operates an underground mining and milling operation near Questa, New Mexico. The product from the milling operation, molybdenum disulfide (MoS_2) concentrate is shipped by rail to a Molycorp roasting facility in Washington, Pennsylvania. At the roasting facility, the concentrate is converted to MoO_3 for use as an alloying agent in the steel industry. The steel industry has established specifications for MoO_3 usage in alloying which Molycorp is unable to meet without further treatment of the mill concentrate. Alloying specifications call for a MoO_3 product which contains less than 0.045% lead (Pb). Molycorp's concentrate (and roasted product) contains $\pm$ 0.10% Pb. In order to provide a competitive and saleable product, it has become necessary to construct a facility to reduce lead content to specification levels. For several years Molycorp has performed laboratory and pilot plant test work to establish the most feasible method for attaining desirable lead levels in Questa concentrate. In mid-1984 Molycorp contracted with Mountain States Engineering of Tucson, Arizona, to review all previous low lead test work, to perform additional testing deemed necessary, and design a lead removal facility for the Questa operations. Construction of the facility has begun and it should be in operation by July of 1985.

II. DESCRIPTION

The lead removal plant will utilize a 1.2N hydrochloric acid leach solution to treat the concentrate for approximately one hour at a tempurature of 80°C. The plant would be located at the Questa operations site and immediately adjacent to the existing concentrate drying facility. A flow sheet for the proposed leaching plant is attached. As indicated by the flow sheet, the plant's operations will be entirely integrated with those of the existing concentrate processing facilities.

Present plans are to subject 70% of the concentrates to low-lead leaching. However, for purposes of the discussion below, leaching of all concentrates is considered. The hydrochloric acid leach system would require a bleed system to control the spent acid, dissolved constituents from the acid leach, and filtrate wash water. A material balance for the leach system shows a total bleed stream entering the mill tailings of 15.6 gpm. The nominal liquid fraction of the mill tailings if 4,352 gpm. Thus, the dilution ratio of the bleed stream is approximately 280:1.

III. ANALYSIS

A series of laboratory leaching tests was performed on molybdenum concentrate to confirm calculated stoichiometric balances. A molybdenum concentrate containing 52% Mo, 0.31% Pb, 0.29% cu, and 1.51% Fe was subjected to locked cycle leach testing. The "bleed" effluent from this series of tests had the following analysis in Mg/ml (ppm): Mo = 193, Pb = 85, Cu = 44, Fe = 485. A simulated mill tailings sample was used for dilution to determine the effects of varying ratios of dilution. The following table summarizes the results:

MIXING EFFLUENT WITH MILL TAILINGS

Effluent Analysis Mg/Ml (ppm)(2) Mo: 1983 Pb: 85 Cu: 44 Fe: 485

		pH		Final Analysis g/ml (ppm)(3)				
Test	Ratio(1)	Initial	Final	Mo	Pb	Cu	Fe	Cl^-
161-86-1	Effluent(4)	8.7	8.7	1.1	0.02	0.04	0.01	7
161-86-2	40:1	8.5	7.5	4.1	0.05	0.04	0.01	245
161-86-3	80:1	8.5	7.7	2.6	0.02	0.04	0.01	131
161-87-1	100:1	8.7	7.7	2.3	0.02	0.04	0.01	102

(1) Mill tailings solution to effluent.

(2) Analysis by atomic absorption.

(3) Analysis by DC plasma except Cl^- determined by Cl^- electrode.

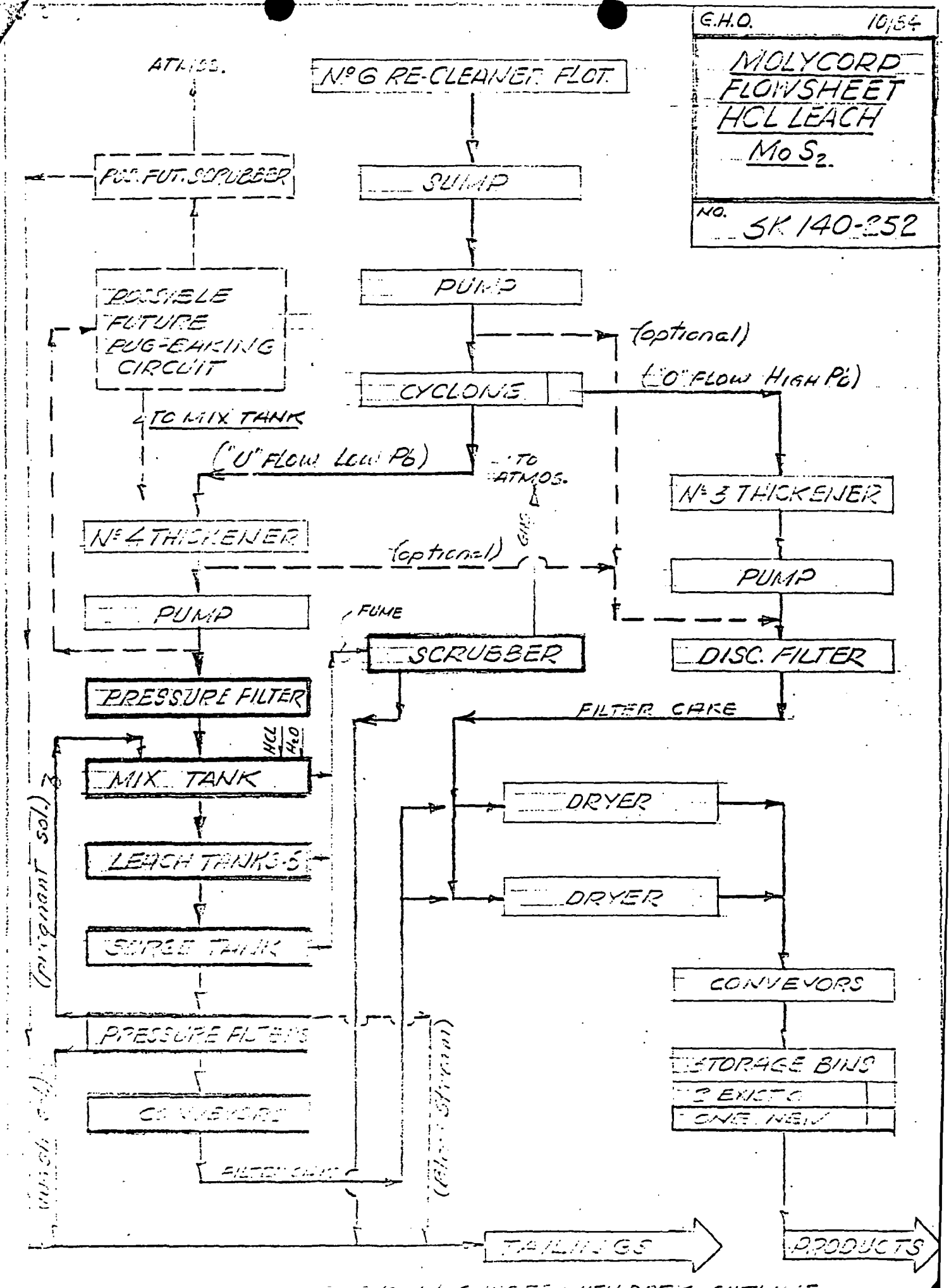
(4) Simulated mill tailings effluent.

Projecting this table to a 1:280 ratio as would be experienced in the leach plant, the calculated final analysis would be as follows:

	pH		Final Analysis ppm				
Ratio	Initial	Final	Mo	Pb	Cu	Fe	Cl
280:1	8.6	8.0	1.54	0.02	0.04	] 0.01	40

The laboratory tests demonstrate that the heavy metal content (Pb, Cu, Fe) of the effluent will not be affected by the addition of the leach plant bleed stream to the mill tailings slurry. The heavy metals will precipitate as hydroxides and settle with tailings solids. The pH of the effluent will remain mid-range at about 8.0. Only a portion of the Mo will precipitate and overall Mo levels in the effluent would increase between 0.4 and 0.5 ppm. However, treatment of the effluent in the ion exchange plant prior to discharge will lower the Mo concentrations in the discharge (outfall 001) to current discharge levels (1.82 ppm, 1984 average concentration). In fact, the only constituent in the effluent discharge that will be increased from present discharge levels is chloride. Chloride levels will be increased from 30 to 35 ppm. It should be noted, however, that Molycorp's NPDES Permit does not contain an effluent limit for chloride and the 35 ppm discharge will be well below the 250 ppm secondary maximum contaminant level for chloride in drinking water specified in the Federal standards (40 C.F.R. § 143.3).

In the future, if a baking step is added prior to leaching, only one of the parameters making up the leach plant bleed stream would change. The concentration of chlorides would increase somewhat but still remain well below the Federal standards.


G.H.O. 10/54
MOLYCORP FLOWSHEET HCL LEACH MoS2
NO. SK 140-252
ATMOS.
Nº 6 RE-CLEANER FLOT.
POS. FUT. SCRUBBER
SUMP
PUMP
POSSIBLE FUTURE PUG-BAKING CIRCUIT
(optional)
CYCLONE
("O" FLOW HIGH Pb)
TO MIX TANK
("U" FLOW LOW Pb)
TO ATMOS.
Nº 3 THICKENER
Nº 4 THICKENER
(optional)
PUMP
PUMP
FUME
SCRUBBER
DISC. FILTER
PRESSURE FILTER
FILTER CAKE
HCL
H2O
MIX TANK
DRYER
LEACH TANKS 5
DRYER
SURGE TANK
(pregnant sol.)
CONVEYORS
PRESSURE FILTERS
STORAGE BINS
2 EXIST.
ONE NEW
CONVEYORS
FILTER CAKE
TAILINGS
PRODUCTS

Molycorp, Inc.

Questa Division
P. O. Box 469
Questa, New Mexico 87556
Telephone: (505) 586-0212

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OCT 16 1984
6W-EA

union 76
MOLYCORP

October 10, 1984

Mr. Dick Whittington
Regional Administrator
U.S. Environmental Protection Agency
Region VI
1201 Elm Street
Dallas, TX 75270

Dear Mr. Whittington:

Enclosed is the Discharge Monitoring Report for the month of September, 1984, NPDES Permit #NM0022306.

Please be advised that outfall 001 did not discharge during the entire month of September. Also, Molycorp is aware that the manganese daily average of 1.0 mg/L was exceeded in September (outfall 002 daily average for manganese in September is reported at 1.3 mg/L). This issue is addressed in Molycorp's June 29, 1984 letter requesting permit modifications. Molycorp is anxious to resolve this issue and requests that either the manganese limits be voided during periods when outfall 001 is not discharging or that the daily average be brought up at least to the daily maximum limits of 1.5 mg/L.

If you have any questions, please feel free to call me.

Sincerely,

C. R. Sacrison
General Manager

cc: Environmental Improvement Division
P. O. Box 968
Santa Fe, NM 87504

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correspondence 15

Molycorp, Inc.

Questa Division
P. O. Box 469
Questa, New Mexico 87556
Telephone: (505) 586-0212

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CERTIFIED MAIL
RETURN RECEIPT REQUESTED

union 76
MOLYCORP

May 15, 1984

Mr. Dick Whittington
Regional Administrator
U.S. EPA
Region VI
1201 Elm Street
Dallas, TX 75270

RE: NPDES Permit - NM 0022306

Dear Mr. Whittington:

Enclosed is the Discharge Monitoring Report for the month of April, 1984.

Conditions of the new permit which have been included in Molycorp's Request For Evidentiary Hearing, submitted to your agency on August 26, 1983, have not been addressed in this report. Those permit conditions include:

1. Molybdenum concentration limitations
2. Total arsenic
3. Definition of composite
4. Biomonitoring
5. Temperature

Under Part II D(8)b of our NPDES Permit, Molycorp is required to notify the Director of changes in discharges which were not reported in the permit application. (Note: pages 15 and 16 of the final permit are misnumbered). Molycorp is currently testing a mill reagent with the trade name Orfom D8 manufactured by Phillips Chemical Company. The product has performed satisfactorily in preliminary mill tests and Molycorp is considering using the product full time. A Material Safety Data Sheet about the product is enclosed for your information. The EPA will be notified if a decision is made to use this reagent full time.

If you have any questions about this information, please contact me.

Sincerely,

C. R. Sacrison
General Manager

CRS/lm

xc: EID-Steve Asher - Santa Fe

DATE June , 1982

MATERIAL SAFETY DATA SHEET

("ESSENTIALLY SIMILAR" TO FORM OSHA-20)

S (1)

NM-22306

WHERE APPLICABLE, THIS PRODUCT HAS BEEN REPORTED FOR THE EPA'S CHEMICAL SUBSTANCE INVENTORY.

SECTION I - IDENTIFICATION OF PRODUCT

MANUFACTURERS NAME: PHILLIPS CHEMICAL COMPANY	EMERGENCY TELEPHONE NUMBERS: DURING BUSINESS HOURS (918) 661-3865; OUTSIDE BUSINESS HOURS (918) 661-8118
ADDRESS (NUMBER, STREET, CITY, STATE, & ZIP CODE): BARTLESVILLE, OK 74004	PRODUCT NO.
TRADE NAME: Orfom® D8	CHEMICAL NAME AND SYNONYMS: Mixture
CHEMICAL FAMILY: Petrosulfur compound	CHEMICAL FORMULA
DOT SHIPPING NAME: Combustible Liquid, n.o.s.	HAZARD CLASS: Combustible Liquid; ID NUMBER: NA 1993

SECTION II - HAZARDOUS COMPONENTS OF MIXTURES

INGREDIENTS	CAS NUMBER	% BY WT.	THRESHOLD LIMIT VALUE (UNITS)
Sodium Hydroxide	1310-73-2	5	

SECTION III - TYPICAL PHYSICAL DATA

BOILING POINT (°F): >212°F	APPEARANCE AND ODOR: Red liquid
VAPOR PRESSURE	SPECIFIC GRAVITY (H_2O = 1): 60/60 F 1.3106
VAPOR DENSITY (AIR = 1): >1	PERCENT VOLATILE BY VOLUME (%): 60
SOLUBILITY IN WATER: Appreciable	EVAPORATION RATE (ethyl ether = 1): <1

SECTION IV - FIRE AND EXPLOSION - HAZARD DATA

FLASH POINT (METHOD): 110°F (TCC, ASTM D56)	FLAMMABLE LIMITS (% BY VOLUME)	Lel	Uel

FIRE EXTINGUISHING MEDIA: Dry chemical, CO_2, foam.

SPECIAL FIRE FIGHTING PROCEDURES: Use water fog or spray to cool exposed equipment and containers.

UNUSUAL FIRE AND EXPLOSION HAZARDS: SO_x released as a combustible product. Use appropriate respiratory protection when fighting a fire.

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NO GUARANTY IS MADE AS TO THE ACCURACY OF ANY DATA OR STATEMENT CONTAINED HEREIN. WHILE THIS MATERIAL IS FURNISHED IN GOOD FAITH, NO WARRANTY EXPRESS OR IMPLIED, OF MERCHANTABILITY, FITNESS OR OTHERWISE IS MADE. THIS MATERIAL IS OFFERED ONLY FOR YOUR CONSIDERATION, INVESTIGATION AND VERIFICATION AND PHILLIPS, INCLUDING ITS DIVISIONS, AFFILIATES AND SUBSIDIARIES, SHALL NOT IN ANY EVENT BE LIABLE FOR SPECIAL, INCIDENTAL OR CONSEQUENTIAL DAMAGES IN CONNETION WITH ITS PUBLICATION. LIKEWISE, NO SATEMENT MADE HEREIN SHALL BE CONSTRUED AS A PERMISSION OR RECOMMENDATION FOR THE

SECTION V - HEALTH HAZARD DATA

THRESHOLD LIMIT VALUE: None established

EFFECTS OF OVEREXPOSURE: Exposure may cause chemical burns.

EMERGENCY AND FIRST AID PROCEDURES:

Eyes: Gently flush with water for at least 15 min. Seek immediate medical assistance.

Skin: Rinse affected area with clean running water. Remove all contaminated clothing and shoes.

Ingestion: Do not induce vomiting. Encourage the victim to drink several glasses of water. Seek immediate medical assistance.

Inhalation: Monitor breathing and initiate artificial respiration if necessary. Transport to a physician as quickly as possible.

SECTION VI - REACTIVITY DATA

STABILITY			CONDITIONS TO AVOID:
	UNSTABLE		
	STABLE	X	pH ≤ 7

INCOMPATIBILITY (MATEPIALS TO AVOID FOR PURPOSES OF TRANSPORT, HANDLING AND STORAGE ONLY): Strong acids or oxidants

HAZARDOUS DECOMPOSITION PRODUCTS: Sulfur oxides, carbon disulfide

HAZARDOUS POLYMERIZATION			CONDITIONS TO AVOID:
	MAY OCCUR		
	WILL NOT OCCUR	X	

SECTION VII - SPILL OR LEAK PROCEDURES

STEPS TO BE TAKEN IN CASE MATERIAL IS RELEASED OR SPILLED:

Contain spill. Protect from ignition. Keep out of water sources and sewers.

WASTE DISPOSAL (INSURE CONFORMITY WITH ALL APPLICABLE DISPOSAL REGULATIONS):

Burn under controlled conditions.

SECTION VIII - PERSONAL PROTECTION INFORMATION

RESPIRATORY PROTECTION:

VENTILATION		
	LOCAL EXHAUST	Recommended
	MECHANICAL (GENERAL)	
	SPECIAL OR OTHER	

PROTECTIVE GLOVES: Neoprene or rubber

EYE PROTECTION: Goggles

OTHER PROTECTIVE EQUIPMENT:

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SECTION IX - HANDLING AND STORAGE PRECAUTIONS

PRECAUTIONS TO BE TAKEN IN HANDLING AND STORING:

Protect from sources of ignition. Provide means of controlling leaks and spills. Store in cool, well-ventilated area. Bond and ground during liquid transfer.

OTHER PRECAUTIONS:

~~(1) Peter~~
~~(2) Hum & FYI~~
JUN 26 1984 (3) Permit File

Assistance for Arranging Acute and Chronic Bioassays

Robert B. Elliott
Chief, Water Quality Management Branch, 6W-Q

James E. Stiebing
Chief, Surveillance Branch, 6ES-S

We request your assistance in arranging for two acute bioassays using Daphnia and two chronic bioassays using Ceriodaphnia to be performed at the Houston Laboratory and ERL-Duluth, respectively. These bioassays, which were requested by the State of New Mexico, Environmental Improvement Division, will serve to test the toxic nature of the Molycorp Mill tailings pond effluents which are discharged into the Red River, near Questa, New Mexico. The State would prefer to collect the samples in about two to three weeks if this time frame is workable with the two labs. For both acute and chronic tests, the State will collect effluent samples and samples for use as dilution water.

Further assistance will be needed at a later date, some time between August and October. In this case, approximately six additional chronic boassays are requested. Precise number of bioassays to be performed would depend on EMR Duluth's capabilities at the time. These bioassays would function to better evaluate the potential instream toxicity due to the high metals concentrations which are characteristic of the Red River.

We would appreciate a response to this request as soon as possible so that the State may plan their sampling trip.

Thank you for your assistance. Please contact Philip Crocker at X7-9916 if there are any questions.

bcc: David Neleigh, 6W-QT
Robert Hannesschlager, 6W-P
Mac Holman, 6ES-SA
Roger Hartung, 6W-E

6W-QT:Crocker:X9916:at:6/26/84 PC-801

6W-QT
Neleigh

5-2-84 Blanke

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION VI
INTERFIRST TWO BUILDING, 1201 ELM STREET
DALLAS, TEXAS 75270

July 24, 1984

Mr. C. R. Sacrison, General Manager
Union Molycorp
Questa Division
P.O. Box 469
Questa, New Mexico 87556

Re: NPDES Permit No. NM0022306

Dear Mr. Sacrison:

This is to acknowledge receipt of your permit modification request dated June 29, 1984, for Union Molycorp.

It is possible that processing of your request may require time as modification requests are reviewed in accordance with Federal and State priorities for water pollution abatement. Also, please be advised that a request for permit modification does not relieve your obligation meet current permit requirements. Therefore, you must attempt to meet all permit requirements to the best of your ability.

Should additional information be required to complete processing of your request, you will be contacted.

Sincerely,

Jayne Watson

Jayne Watson, Chief
Permits Issuance Section (6W-PS)

(1) Slaven (2) Hamle (3) Permit File

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

DATE: 7-11-84

SUBJECT: MODIFICATION REQUEST FOR AN ISSUED PERMIT

FROM: JENAIE SLAVEN, 6W-PS.

TO: Sam Becker, 6W-PS

The referenced application and/or letter (see attached copy) has been submitted to this office requesting a modification to their existing NPDES permit, NM0022306.

Please return this form to 6W-PS initialing your desired course of action.

Name of Facility: Union Molycorp

_____ From the information submitted, it would appear that a new pwemit should be written and issued independently from the previously issued permit. A new NPDES number should be assigned to this request.

_____ The information received requesting a modification to the permit does not include enough information to make a determination. Please request additional information from permittee in the form of a Short Form A, B, C, or D, or Standard Form A or C. (Please circle appropriate form.)

✓ JK EPA staff will handle the request and modify the permit if necessary. Please refer to Hamke. (Optional)

_____ EPA is in agreement with changing the permit. Send a copy of the request to the appropriate state agency for drafting of a new permit.

_____ Send a copy of the request to the appropriate state agency to draft a new permit if the state agency determines that a change is warranted.

_____ The request does not warrant a change to the permit. Please place in permit file for future reference.

Attachment(s)

Molycorp, Inc.

Questa Division
P. O. Box 469
Questa, New Mexico 87556
Telephone: (505) 586-0212

MR 2

union 76
MOLYCORP

June 29, 1984

Mr. Myron Knudson
Director, Water Management Div. (6W)
U.S. Environmental Protection Agency
Region VI
1201 Elm Street
Dallas, Texas 75270

Re: Molycorp/Questa
NPDES Permit NM0022306
Request for Modification

Dear Mr. Knudson:

The purpose of this letter is to request certain minor modifications to Molycorp's NPDES Permit for the Questa mine and milling facility. This request is being filed pursuant to 40 C.F.R. 122.63 and 40 C.F.R. 124.5. However, Region VI determines that this request must be considered under 40 C.F.R. 122.62, Molycorp consents to the consideration of this request on that basis.

The current NPDES Permit became effective on August 31, 1983. Though the permit was acceptable to Molycorp in almost all respects, there were a few points of disagreement. Consequently, Molycorp filed an Evidentiary Hearing Request on August 26, 1983. Molycorp received notice on June 11, 1984 that the Regional Administrator had denied the request, on the basis that the request did not set forth material issues of fact. Though Molycorp does not agree with the reasons for that decision, we are anxious to resolve the few remaining minor issues at the regional level without further delay. Accordingly, Molycorp is filing this request in the hope that it can be considered prior to the July 8, 1984 deadline to seek review of the Regional Administrator's June 8, 1984 denial. However, if that tight schedule cannot be met, Molycorp will make a protective filing with the Administrator.

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JUL 5 1984
6W-PS

The portions of the permit for which Molycorp requests modification, and the reasons therefore, are set out below.

1. Suspension of the molybdenum and manganese concentration Limitations for Outfall 002 When Outfall 001 is not Discharging

Molybdenum limitations set forth in the development program of the existing permit were based on molybdenum loading to the Red River, calculated by dilution factors and irrigation use downstream. Compliance with molybdenum loading and concentration limitations is determined by a calculation based on the combined effects of Outfalls 001 and 002. This reasoning is retained in the new permit (Part 111.C.2).

Molycorp has recently completed a new ion exchange water treatment plant to remove molybdenum from water discharged through Outfall 001. The plant represents state of the art technology and is designed to remove sufficient quantities of molybdenum to comply with loading requirements and concentration limitations when calculated as stated in Part 111.C.2., when both Outfalls are discharging.

However, during normal operation, there will be times when there will be no discharge from Outfall 001. Under those circumstances, the flow weighted average reported for molybdenum would equal the total discharge from Outfall 002. This discharge will easily meet the loading limitations (25 pounds per day average), but not the limitations for concentrations (1.0 mg/l average). This situation commonly occurs during periods of construction on the tailings dams, and the result will be a situation similar to the recent mill shutdown. During such times, the seepage collected and discharged from Outfall 002 will continue, discharging less than ten pounds per day of molybdenum.

The molybdenum concentration limitations, as shown in the Stipulated Agreement on NPDES Permit NM0022306, June 16, 1977, Section IV.(2), were "based on Best Available Technology Economically Achievable as presently defined." BATEA is based on economics, and the concentration limitations are indeed economically achievable as calculated for combined Outfalls 001 and 002.

Outfall 002, when it represents the sole discharge from the operation, cannot economically meet the molybdenum concentration requirements for two reasons. First, the capital investment and operating expenses necessary to comply are not reasonable costs, and will have significant non-water quality impacts, particularly energy requirements. A description of the engineering aspects, costs and energy requirements is attached hereto as Exhibit 1.

Second, in order to treat the relatively low flows from Outfall 002 in the ion exchange treatment plant, Molycorp would be required to impound the water until sufficient quantity was collected to operate the treatment plant. Pumping costs to impound this water are included in Exhibit 1. At design flow, the treatment plant would operate approximately one hour per day to treat Outfall 002.

The treatment plant is designed for continuous flow and could practicably not be operated only one hour per day. The treatment plant operates most efficiently with continuous flow throughout a complete operating cycle before shutdown. An operating cycle is estimated to be four days at 4,000 gallons per minute, or larger periods of lesser flows down to a minimum of ±1,500 gallons per minute. Operating the plant on a short cycle could damage the ion exchange resin through osmotic shock and/or algae growth brought about by non-continuous operation. Severe osmotic shock or algae growth could destroy most of the resin, and thus render the plant inoperable to treat the significant flows from Outfall 001. Total replacement of resin would result in costs in excess of $0.5 million. The ion exchange resin is not a stock item with the manufacturer, but is made to order, requiring three to four months lead time. The end result could be a discharge of inadequately treated water if resin is destroyed more rapidly than anticipated.

In view of the environmental, engineering and economic risks associated with intermittent use of the ion exchange plant, Molycorp requests that the concentration limitations be suspended during periods of zero flow from Outfall 001. No impact on the receiving water will occur as the result of this change. The discharge from 002 does not change when 001 is not discharging. Total loadings to the Red River are much <u>lower</u> during such periods.

Manganese limits at 1.0 mg/l liter daily average a 1.5 mg/l liter daily maximum present no particular problem when 001 and 002 discharges are flow weighted. However, when only 002 is discharging, the daily average and maximum limits can be exceeded. Outfall 001 during 1984 (Outfall commenced in February) through May has averaged 0.42 mg/l liter manganese. Outfall 002 obviously picks up some manganese from the substrata since it was approximately 1.3 mg/l liter during the same period. It is requested that either the manganese limits be voided during periods when 001 is not discharging or that the daily average be brought up at least to the daily maximum limits of 1.5 mg/l liter.

2. <u>Molycorp Requests Suspension of the Arsenic Limitations Contained in Part I.A of the Permit for Two Years From the Date the Mill was Restarted (October 1985).</u>

EPA has included a discharge limitation of 0.5 mg/l daily average and 1.0 mg/l daily maximum in the permit, stating that "arsenic was present in significant levels in Outfall 001" in the analysis

submitted in the NPDES application. (See Response to Comments Issue No. 3) Molycorp notes that the analytical data submitted for arsenic was a single composite sample, as required by 40 C.F.R. 122.21 (g). Arsenic was not included as an effluent limitation in the existing permit, and Molycorp was given no indication that it would be added to the new permit. Consequently, very limited previous test work regarding arsenic has been conducted.

Since arsenic limitations were proposed in the draft permit, Molycorp began testing of the effect of the ion exchange pilot plant on arsenic solutions. There are no conclusive results from this limited test work to date. Because the mill was not operating for some time, it was impossible until recently to test the arsenic removal effect on the actual effluent. The results of monitoring performed since mill startup in October 1983 indicate arsenic levels in the effluent are 0.01 or less in both 001 and 002 Outfalls. These levels are at least an order of magnitude below the standards in the New Mexico Water Quality Control regulations for Human Health Standards (Section 3-103).

Sampling by EPA consultants conducted at two separate times, and by Molycorp consultants in 1970-72, indicates arsenic levels significantly below the levels detected in the sampling for the NPDES Permit application submitted to EPA in January 1982. On April 19, 1977, Calspan, an EPA consultant, collected monitoring samples pursuant to the Toxic Substances Control Act. Tailings pond effluent was analyzed for many constituents, including arsenic concentration, which was determined to be 0.005 mg/l. A copy of the results is attached as Exhibit 2.

In June 1970, Molycorp retained Thorne Ecological Institute to perform an environmental assessment of the Questa operation. The assessment included an extensive sampling program to define water quality aspects. Arsenic was included in the sampling program. Samples collected, which were analyzed for arsenic, showed concentrations below the detection limit of 0.02 parts per million. The arsenic concentrations were so consistently below detection limits that arsenic monitoring was dropped from the program. Excerpts from the final Thorne Report are attached as Exhibit 3.

Limited test work has been performed to date to determine the effect of the ion exchange treatment plant on arsenic. Molycorp needs additional time to define what arsenic limitations can be achieved by the plant. It is, however, reasonable to assume that the ion exchange plant will have some effect on arsenic levels which will then be below the "significant levels." As Molycorp noted in its comments on the draft permit, EPA expects arsenic contol to occur. (See 47 Fed. Reg. 54605.) Molycorp urges suspension of these limitations for two years to permit arsenic levels in the actual effluent to be adequately defined.

3. Molycorp Requests Part 111.B of the Permit be Amended to Read as follows:

The term "composite sample" means a sample consisting of a minimum of two grab samples of effluents collected not less than four hours apart over a normal eight hour operating day and combined proportional to flow or a sample continuously collected proportional to flow over a normal eight hour operating day. All such samples shall be typical and representative of effluent generated during the period since the last sample was collected.

Sampling of Molycorp's effluent over the past six years pursuant to the previous permit was collected by obtaining a sample consisting of a minimum of two grab samples collected, not less than four hours apart over a normal eight hour operating day. Effluent from Outfall 001 is, in effect, a composite, since the wastewater is retained in a 50 acre-foot holding pond called Pope Lake. Retention time in the combined ponds is estimated to be at least ten days or more, sufficient time for mixing and stabilization of the water before discharge.

The definition of composite sampling (Part 11.B of the previous Permit) was carefully developed during discussions with EPA and EID regarding the conditions of that permit. It was determined that a two-part composite was an appropriate, representative sample of the effluent for two reasons. First, Outfalls 001 and 002 discharge water from impoundments with holding times of greater than 24 hours. Second, Molycorp environmental personnel work eight hours per day, five days per week and are best qualified to collect samples for compliance with the permit. Continuous samplers operate poorly in the harsh weather conditions experienced at the discharge locations, and samples must be manually composited by Environmental Department staff.

The four-part composite sample required in the new permit over a 24 hour operating day means that the sampling would be performed by the 24 hour tailings patrol. The position of tailings patrol is a union (hourly) position subject to shift rotation, bidding procedures and seniority. The weekly samples would be collected by a different group of samplers every week, depending upon the individuals on each shift. Variable sample quality could be experienced as a result. The samples, if collected by the tailings patrols, could be less representative under a four-part compositing scheme than under the previous sampling program under direct control of Molycorp environmental staff. The time of tailings patrol is best spent on surveillance of the tailings lines.

Because both Outfalls 001 and 002 discharge water from retention ponds with a retention period greater than 24 hours, and since EPA proposed that "a minimum of one grab sample will generally be sufficient to ensure a representative sample" from such impoundments

for permit application (See 47 FR 52076, November 18, 1982, EPA Proposal to Revise Consolidated Permit Regulations in Accordance with Settlement, Suspended NPDES Application Rules), the two-part composite sampling under the previous permit provides the most representative, consistent samples and should be the sampling required in the existing permit.

4. Molycorp Requests that Region VI Consider an Alternative Method for Biomonitoring, as Described Below

Section 308 of the Clean Water Act gives EPA the authority to require monitoring, including biomonitoring, as necessary, to carry out the objectives of the Clean Water Act. The objective of the Act is "to restore and maintain the chemical, physical and biological integrity of the Nation's water." (Section 101) In proposing the alternate method, Molycorp carefully considered that objective and believes that the alternate method provides a more valuable scientific tool with which to measure attainment of the goals of the Act.

In the comments on the new permit, Molycorp included a summary of biomonitoring results of a long-term comprehensive study of the Red River done by Dr. R. W. Pennak. Molycorp proposes the continuation of Dr. Pennak's research as an alternate method. Molycorp has taken the initiative, beginning in 1971, to determine the impact of its effluent on the receiving water. The study was conducted over 12 years and has included periods of inactivity of the mine and mill operations. Through Dr. Pennak's work, environmental scientists can compare the biological quality of the Red River during periods of operation and inactivity, and can compare the Red River with other mountain streams and determine not only toxicity of the effluent but its overall impact on the Red River and aquatic life.

While Molycorp understands EPA's need to develop a data base for toxic materials in effluent, we believe that the standard method proposed by EPA is a less effective means of developing a useful data base to determine the effects on the Red River of Molycorp's discharge. EPA has recognized the complexity of effluents and the synergistic effects between toxic substances in such effluents. Molycorp believes that using a simplistic biomonitoring method based on a non-indigenous organism does not (1) yield information which would be of value to EPA to determine synergistic effects of constituents, (2) apply to other operations or (3) provide insight into the toxicity of Molycorp's effluent in a reasonable context.

The proposed alternate method brings forth 12 years of collected data as a base, information regarding biological, physical and chemical data directly applicable to the quality of aquatic organisms found in the Red River. Molycorp's proposed alternate method reveals information of far more scientific value than Region VI's standardized method, which is not based on a specific receiving water. The

proposed alternate method is at least as expensive as EPA's standardized method and provides a more valuable measurement of the effect of any effluent.

5. Molycorp Requests that the pH limitations be Revised from 6.6 - 8.6 to 6.0 - 9.0 S.U.

Molycorp has requested that the State of New Mexico waive these requirements, and the State has agreed. The waiver documentation was provided to Region VI by our letter of October 31, 1983.

Thank you for your consideration of these requested modifications. We look forward to discussing them with you and your staff in the near future. Please call if you have any questions.

Sincerely,

C. R. Sacrison
General Manager

CRS/lm

EXHIBIT I

UNION MOLYCORP
P.O. BOX 469
QUESTA, NEW MEXICO 87556

INTEROFFICE CORRESPONDENCE

FROM: G. F. Eisebraun

TO: C. R. Sacrison

DATE: August 22, 1983

SUBJECT: RECYCLE PUMP SYSTEM FOR 002/003 OUTFALL — COST ESTIMATE

The following pump system provides for recycle of the ±300 GPM flow from the pipe junction of the 003/002 seeps below the Number 1 Dam to the Number 4 tailings pond area.

General:

1. Pipe route is 2000 feet with the intake at the 7280-foot elevation and the discharge at the 7530-foot elevation.

2. Pipe is six inch diameter schedule 40, of mild steel in 21-foot lengths, fitted with victaulic couplings.

3. Concrete works consists of a buried 3000-gallon collection tank, and pipe line foundations on 20-foot centers along the route.

4. Pumping operations would be controlled by automation at the pump site with all required monitoring data transmitted to the Molycorp mill control room.

5. Pump data follows:

a.	Total hydraulic head	268'
b.	PSIG at pump	116
c.	Brake horsepower required	25.5
d.	Pump horsepower at 1750 RPM	30
e.	Pump Stages	8
f.	Power to site (voltage)	230/460

g. Proposed pumps are equipped with 9LA bowls, WT1 weather enclosures, basked screens, 6-inch casing with one-inch drive line, 6 x 6 x 12 dish head, and six-inch discharge flanges rated at 125 PSI.

Project Cost Estimate:

Pumps (2 each w/one on standby)	$ 12,200
Pipe w/victaulic grooves @ $190/length	18,100
Couplings (Model 77 @ $16 each)	1,600
Earthwork (route preparation, tank excavating, etc.)	30,000
Concrete works (reinforced)	
Tank - 70 C.Y. Conc. @ $300/C.Y. installed	21,000
Pipe foundations - 100 each @ $200/each installed	20,000
230/460 V power - powerline (transformers, switch gear) 1200' @ $10/ft. installed	12,000
Instrumentation - site control/valving installed	5,000
- mill read out system (FMX-Mission)	8,000
	$127,900
Labor - pipeline and pump installation (5 man crew, 6 days @ $120/man day) (Rate 9 mechanic plus 20% fringe)	3,600
	$131,500
Design and field engineering @ 10% of Cost	13,200
	$144,700
Contingency @ 10%	14,500
ESTIMATED PROJECT COST	$159,200

Operating Cost:

Power - Electrical	$ 10,000
Maintenance, etc.	$ 10,000
ANNUAL COST	$ 20,000

xc: T. R. Thomas - Legal Dept.

EXHIBIT II

Calspan

17 October 1977

Ms. Obbey Davidson
Environmental Engineer
Molycorp
Questa, NM 87556

Dear Ms. Davidson:

Results of chemical analyses of water samples that we collected last spring on behalf of EPA at your facilities have recently become available. In accordance with previous agreements, EPA has authorized us to transmit the analytical results to you, with the proviso that they are to be treated as preliminary data. This is particularly important in those cases where some analyses (e.g., for asbestos) are to be repeated.

The enclosed copies of the analytical results are accompanied by one table showing the laboratory that performed the analysis and by another that lists the lowest detectable quantity of those constituents for which this number can be stated. When a target constituent is listed as "not detectable" in the record for your facility, its concentration was found to be less than the detection limit.

Thank you again for your cooperation and hospitality during this survey program. If you should have questions regarding this material, please contact me.

Very truly yours,

D.H. Bock

D.H. Bock

DHB:eb-32
Enclosure

SUMMARY DATA SHEET

Operation: Molycorp of America, Questa, New Mexico Date Visited: April 18-19, 1977

Sample-Site Description: Influent to tails pond

Calspan Sample No. W58-X1-FA-01A MC EPA Sample No. T002039 (17380)

Remarks: 24-hour composite

SUBSTANCE	CONCENTRATION	SUBSTANCE	CONCENTRATION
pH	7.40	1,3-dichlorobenzene	
total suspended solids	349,000	1,4-dichlorobenzene	
volatile suspended solids	4300	3,3'-dichlorobenzidine	
COD	1180	1,1-dichloroethylene	
TOC	19	1,2-trans-dichloroethylene	
radium 226 (total)	------	2,4-dichlorophenol	
radium 226 (dissolved)	------	1,2-dichloropropane	
antimony (total)	<100 µg/L	1,3-dichloropropylene (1,3-dichloropropene)	
arsenic (total)	10 µg/L		
beryllium (total)	130 µg/L	2,4-dimethylphenol	
cadmium (total)	13 µg/L	2,4-dinitrotoluene	
chromium (total)	8300 µg/L	2,6-dinitrotoluene	
copper (total)	10000 µg/L	1,2-diphenylhydrazine	
lead (total)	11000 µg/L	ethylbenzene	
mercury (total)	<0.5 µg/L	fluroanthene	
nickel (total)	3500 µg/L	4-chlorophenyl phenyl ether	
selenium (total)	40 µg/L	4-bromophenyl phenyl ether	
silver (total)	<250 µg/L	bis (2-chloroisopropyl) ether	
thallium (total)	<100 µg/L	bis (2-chloroethoxy) methane	
zinc (total)	13000 µg/L	methylene chloride (dichloromethane)	
asbestos (fibrous)		methyl chloride (chloromethane)	
cyanide (total)	<0.02	methyl bromide (bromomethane)	
phenol (total)	0.02	bromoform (tribromomethane)	
aldrin	NOT DETECTED	dichlorobromomethane	
dieldrin		trichlorofluoromethane	
chlordane (technical mixture and metabolites)		dichlorodifluororomethane	
		chlorodibromomethane	
4,4'-DDT		hexachlorobutadiene	
4,4'-DDE (p,p'-DDX)		hexachlorocyclopentadiene	
4,4'-DDD (p,p'-TDE)		isophorone	
α-endosulfan		naphthalene	
β-endosulfan		nitrobenzene	
endosulfan sulfate		2-nitrophenol	
endrin		4-nitrophenol	

FA - 01A MC

[illegible]	NOT DETECTED		hexachlorobutadiene		
4,4'-DDT			hexachlorocyclopentadiene		
4,4'-DDE (p,p'-DDX)			isophorone		
4,4'-DDD (p,p'-TDE)			napthalene		
α-endosulfan			nitrobenzene		
β-endosulfan			2-nitrophenol		
endosulfan sulfate			4-nitrophenol		
endrin			2,4-dinitrophenol		
endrin aldehyde			4,6-dinitro-o-cresol		
heptachlor			N-nitrosodimethylamine		
heptachlor epoxide			N-nitrosodiphenylamine		
α-BHC			N-nitrosodi-n-propylamine		
β-BHC			pentachlorophenol		
γ-BHC (lindane)			phenol		
δ-BHC			bis (2-ethylhexyl) phthalate	100 µg/L	
PCB-1242 (Arochlor 1242)			butyl benzyl phthalate	not detected	believed [illegible]
PCB-1254 (Arochlor 1254)			di-n-butyl phthalate	15 µg/L	sampling [illegible]
acenaphthene	not detected		diethyl phthalate	not detected	contamin[illegible]
acrolein			dimenthyl phthalate		
acrylonitrile			1,2-benzanthracene		
benzene			benzo (a) pyrene (3,4-benzopyrene)		
benzidine			3,4-benzofluoranthene		
carbon tetrachloride (tetrachloromethane)			11,12-benzofluoranthene		
chlorobenzene			chrysene		
1,2,4-trichlorobenzene			acenaphthylene		
hexachlorobenzene			anthracene		
1,2-dichloroethane			1,12-benzoperylene		
1,1,1-trichloroethane			fluorene		
hexachloroethane			phenanthrene		
1,1-dichloroethane			1,2:5,6-dibenzanthracene		
1,1,2-trichloroethane			indeno (1,2,3-C,D) pyrene		
1,1,2,2-tetrachloroethane			pyrene		
chloroethane			2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD)		
bis (chloromethyl) ether			tetrachloroethylene		
bis (chloroethyl) ether			toluene		
2-chloroethyl vinyl ether (mixed)			trichloroethylene		
2-chloronaphthalene			vinyl chloride		
2,4,6-trichlorophenol			toxaphene		
parachlorometa cresol					
chloroform (trichloromethane)					
[illegible]					

SUMMARY DATA SHEET

Operation: Molycorp of America, Questa, New Mexico Date Visited: April 18-19, 1977

Sample-Site Description: Effluent from tails pond

Calspan Sample No. W58 - X1 - FA - 028 MC EPA Sample No. T002031 (17381)

Remarks: 24 - hour composite

SUBSTANCE	CONCENTRATION	SUBSTANCE	CONCENTRATION
pH	6.95	1,3-dichlorobenzene	not detected
total suspended solids	68	1,4-dichlorobenzene	
volatile suspended solids	4	3,3'-dichlorobenzidine	
COD	20	1,1-dichloroethylene	
TOC	7	1,2-trans-dichloroethylene	
radium 226 (total)	—	2,4-dichlorophenol	
radium 226 (dissolved)	—	1,2-dichloropropane	
antimony (total)	70 µg/L	1,3-dichloropropylene (1,3-dichloropropene)	
arsenic (total)	5 µg/L	2,4-dimethylphenol	
beryllium (total)	<20 µg/L	2,4-dinitrotoluene	
cadmium (total)	<5 µg/L	2,6-dinitrotoluene	
chromium (total)	20 µg/L	1,2-diphenylhydrazine	
copper (total)	270 µg/L	ethylbenzene	
lead (total)	<20 µg/L	fluroanthene	
mercury (total)	<0.5 µg/L	4-chlorophenyl phenyl ether	
nickel (total)	1400 µg/L	4-bromophenyl phenyl ether	
selenium (total)	<5 µg/L	bis (2-chloroisopropyl) ether	
silver (total)	<250 µg/L	bis (2-chloroethoxy) methane	
thallium (total)	<100 µg/L	methylene chloride (dichloromethane)	
zinc (total)	5900 µg/L	methyl chloride (chloromethane)	
asbestos (fibrous) total/chrysotile	$3.3 \cdot 10^{10}$/$2.0 \cdot 10^{9}$ fibers/liter	methyl bromide (bromomethane)	
cyanide (total)	<0.02	bromoform (tribromomethane)	
phenol (total)	0.01	dichlorobromomethane	
aldrin	NOT DETECTED	trichlorofluoromethane	
dieldrin		dichlorodifluoromethane	
chlordane (technical mixture and metabolites)		chlorodibromomethane	
4,4'-DDT		hexachlorobutadiene	
4,4'-DDE (p,p'-DDX)		hexachlorocyclopentadiene	
4,4'-DDD (p,p'-TDE)		isophorone	
α-endosulfan		napthalene	
β-endosulfan		nitrobenzene	
endosulfan sulfate		2-nitrophenol	
endrin			

FA-02B MC

(technical mixture and metabolites)	not detected				
4,4'-DDT			hexachlorobutadiene		
4,4'-DDE (p,p'-DDX)			hexachlorocyclopentadiene		
4,4'-DDD (p,p'-TDE)			isophorone		
α-endosulfan			napthalene		
β-endosulfan			nitrobenzene		
endosulfan sulfate			2-nitrophenol		
endrin			4-nitrophenol		
endrin aldehyde			2,4-dinitrophenol		
heptachlor			4,6-dinitro-o-cresol		
heptachlor epoxide			N-nitrosodimethylamine		
α-BHC			N-nitrosodiphenylamine		
β-BHC			N-nitrosodi-n-propylamine		
γ-BHC (lindane)			pentachlorophenol		
δ-BHC			phenol		
PCB-1242 (Arochlor 1242)			bis (2-ethylhexyl) phthalate		
PCB-1254 (Arochlor 1254)			butyl benzyl phthalate		
acenaphthene	not detected		di-n-butyl phthalate	15 μg/l - from sampler	
acrolein			diethyl phthalate	not detected	
acrylonitrile			dimenthyl phthalate		
benzene			1,2-benzanthracene		
benzidine			benzo (a) pyrene (3,4-benzopyrene)		
carbon tetrachloride (tetrachloromethane)			3,4-benzofluorathene		
			11,12-benzofluoranthene		
chlorobenzene			chrysene		
1,2,4-trichlorobenzene			acenaphthylene		
hexachlorobenzene			anthracene		
1,2-dichloroethane			1,12-benzoperylene		
1,1,1-trichloroethane			fluorene		
hexachloroethane			phenanthrene		
1,1-dichloroethane			1,2:5,6-dibenzanthracene		
1,1,2-trichloroethane			indeno (1,2,3-C,D) pyrene		
1,1,2,2-tetrachloroethane			pyrene		
chloroethane			2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD)		
bis (chloromethyl) ether					
bis (chloroethyl) ether			tetrachloroethylene		
2-chloroethyl vinyl ether (mixed)			toluene		
2-chloronaphthalene			trichloroethylene		
2,4,6-trichlorophenol			vinyl chloride		
parachlorometa cresol			toxaphene		
chloroform (trichloromethane)					

LOE Task XL

SUMMARY DATA SHEET

Operation: _____ Date Visited: _____

Sample-Site Description: Chemical Laboratory Listing for Analytical Work Performed

Calspan Sample No. _____ EPA Sample No. _____

Remarks: _____

SUBSTANCE	CONCENTRATION	SUBSTANCE	CONCENTRATION
pH	Calspan	1,3-dichlorobenzene	Gulf South Research Instit.
total suspended solids		1,4-dichlorobenzene	
volatile suspended solids		3,3'-dichlorobenzidine	
COD		1,1-dichloroethylene	
TOC		1,2-trans-dichloroethylene	
radium 226 (total)	Teledyne Isotopes	2,4-dichlorophenol	
radium 226 (dissolved)		1,2-dichloropropane	
antimony (total)	Calspan	1,3-dichloropropyiene (1,3-dichloropropene)	
arsenic (total)			
beryllium (total)	EPA	2,4-dimethylphenol	
cadmium (total)	Calspan	2,4-dinitrotoluene	
chromium (total)		2,6-dinitrotoluene	
copper (total)		1,2-diphenylhydrazine	
lead (total)		ethylbenzene	
mercury (total)		fluroanthene	
nickel (total)		4-chlorophenyl phenyl ether	
selenium (total)		4-bromophenyl phenyl ether	
silver (total)		bis (2-chloroisopropyl) ether	
thallium (total)		bis (2-chloroethoxy) methane	
zinc (total)		methylene chloride (dichloromethane)	
asbestos (fibrous)		methyl chloride (chloromethane)	
cyanide (total)		methyl bromide (bromomethane)	
phenol (total)		bromoform (tribromomethane)	
aldrin		dichlorobromomethane	
dieldrin		trichlorofluoromethane	
chlordane (technical mixtures and metabolites)		dichlorodifluororomethane	
		chlorodibromomethane	
4,4'-DDT		hexachlorobutadiene	
4,4'-DDE (p,p'-DDX)		hexachlorocyclopentadiene	
4,4'-DDD (p,p'-TDE)		isophorone	
α-endosulfan		napthalene	
β-endosulfan		nitrobenzene	
endosulfan sulfate		2-nitrophenol	
		4-nitrophenol	

4,4'-DDT			hexachlorobutadiene		
4,4'-DDE (p,p'-DDX)			hexachlorocyclopentadiene		
4,4'-DDD (p,p'-TDE)			isophorone		
α-endosulfan			napthalene		
β-endosulfan			nitrobenzene		
endosulfan sulfate			2-nitrophenol		
endrin			4-nitrophenol		
endrin aldehyde			2,4-dinitrophenol		
heptachlor			4,6-dinitro-o-cresol		
heptachlor epoxide			N-nitrosodimethylamine		
α-BHC			N-nitrosodiphenylamine		
β-BHC			N-nitrosodi-n-propylamine		
γ-BHC (lindane)			pentachlorophenol		
δ-BHC			phenol		
PCB-1242 (Arochlor 1242)			bis (2-ethylhexyl) phthalate		
PCB-1254 (Arochlor 1254)			butyl benzyl phthalate		
acenaphthene	Gulf South Research Inst.		di-n-butyl phthalate		
acrolein			diethyl phthalate		
acrylonitrile			dimenthyl phthalate		
benzene			1,2-benzanthracene		
benzidine			benzo (a) pyrene (3,4-benzopyrene)		
carbon tetrachloride (tetrachloromethane)			3,4- benzofluorathene		
			11,12-benzofluoranthene		
chlorobenzene			chrysene		
1,2,4-trichlorobenzene			acenaphthylene		
hexachlorobenzene			anthracene		
1,2-dichloroethane			1,12-benzoperylene		
1,1,1-trichloroethane			fluorene		
hexachloroethane			phenanthrene		
1,1-dichloroethane			1,2:5,6-dibenzanthracene		
1,1,2-trichloroethane			indeno (1,2,3-C,D) pyrene		
1,1,2,2-tetrachloroethane			pyrene		
chloroethane			2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD)		
bis (chloromethyl) ether					
bis (chloroethyl) ether			tetrachloroethylene		
2-chloroethyl vinyl ether (mixed)			toluene		
2-chloronaphthalene			trichloroethylene		
2,4,6-trichlorophenol			vinyl chloride		
parachlorometa cresol			toxaphene		
chloroform (trichloromethane)					
2-chlorophenol					

LOE Task XI

SUMMARY DATA SHEET

Operation: _____

Sample-Site Description: _____

Available Limits of Detection

Calspan Sample No. _____ EPA Sample No. _____

Remarks: _____

SUBSTANCE	CONCENTRATION	SUBSTANCE	CONCENTRATION
pH		1,3-dichlorobenzene	
total suspended solids	1 mg/L.	1,4-dichlorobenzene	
volatile suspended solids	10 mg/L	3,3'-dichlorobenzidine	
COD	2 mg/L	1,1-dichloroethylene	0.50
TOC	1 mg/L	1,2-trans-dichloroethylene	0.35
radium 226 (total)	2 piC/L	2,4-dichlorophenol	
radium 226 (dissolved)	2 piC/L	1,2-dichloropropane	0.02
antimony (total)		1,3-dichloropropylene (1,3-dichloropropene)	0.35
arsenic (total)		2,4-dimethylphenol	0.40
beryllium (total)		2,4-dinitrotoluene	0.20
cadmium (total)		2,6-dinitrotoluene	0.75
chromium (total)		1,2-diphenylhydrazine	0.20
copper (total)		ethylbenzene	0.10
lead (total)		fluroanthene	0.20
mercury (total)		4-chlorophenyl phenyl ether	0.06
nickel (total)		4-bromophenyl phenyl ether	
selenium (total)		bis (2-chloroisopropyl) ether	
silver (total)		bis (2-chloroethoxy) methane	
thallium (total)		methylene chloride (dichloromethane)	0.08
zinc (total)		methyl chloride (chloromethane)	0.35
asbestos (fibrous)		methyl bromide (bromomethane)	0.08
cyanide (total)	0.02 mg/L	bromoform (tribromomethane)	0.40
phenol (total)	0.002 mg/L	dichlorobromomethane	0.05
aldrin	0.1 ug/L	trichlorofluoromethane	
dieldrin	0.5 ug/L	dichlorodifluororomethane	
chlordane (technical mixture and metabolites)	1 ug/L	chlorodibromomethane	
4,4'-DDT	1 ug/L	hexachlorobutadiene	0.08
4,4'-DDE (p,p'-DDX)	1 ug/L	hexachlorocyclopentadiene	
4,4'-DDD (p,p'-TDE)	1 ug/L	isophorone	
α-endosulfan	1 ug/L	napthalene	0.15
β-endosulfan	1 ug/L	nitrobenzene	0.85
endosulfan sulfate	1 ug/L	2-nitrophenol	
		4-nitrophenol	

4,4'-DDT	1 ug/L	hexachlorobutadiene	0.08
4,4'-DDE (p,p'-DDX)	1 ug/L	hexachlorocyclopentadiene	
4,4'-DDD (p,p'-TDE)	1 ug/L	isophorone	
α-endosulfan	1 ug/L	napthalene	0.15
β-endosulfan	1 ug/L	nitrobenzene	0.85
endosulfan sulfate	1 ug/L	2-nitrophenol	
endrin	0.5 ug/L	4-nitrophenol	
endrin aldehyde	0.5 ug/L	2,4-dinitrophenol	
heptachlor	0.1 ug/L	4,6-dinitro-o-cresol	
heptachlor epoxide	0.1 ug/L	N-nitrosodimethylamine	
α-BHC	0.1 ug/L	N-nitrosodiphenylamine	
β-BHC	0.1 ug/L	N-nitrosodi-n-propylamine	
γ-BHC (lindane)	0.1 ug/L	pentachlorophenol	ug/L
δ-BHC	0.1 ug/L	phenol	
PCB-1242 (Arochlor 1242)	1 ug/L	bis (2-ethylhexyl) phthalate	0.20
PCB-1254 (Arochlor 1254)	1 ug/L	butyl benzyl phthalate	0.25
acenaphthene	0.03	di-n-butyl phthalate	
acrolein		diethyl phthalate	0.20
acrylonitrile		dimenthyl phthalate	0.35
benzene	0.04	1,2-benzanthracene	0.05
benzidine		benzo (a) pyrene (3,4-benzopyrene)	
carbon tetrachloride (tetrachloromethane)	0.35	3,4-benzofluorathene	
chlorobenzene	0.15	11,12-benzofluoranthene	
1,2,4-trichlorobenzene		chrysene	0.20
hexachlorobenzene	ug/L	acenaphthylene	
1,2-dichloroethane	0.08	anthracene	0.05
1,1,1-trichloroethane	0.15	1,12-benzoperylene	
hexachloroethane		fluorene	0.10
1,1-dichloroethane	0.15	phenanthrene	
1,1,2-trichloroethane	0.10	1,2:5,6-dibenzanthracene	
1,1,2,2-tetrachloroethane	0.90	indeno (1,2,3-C,D) pyrene	
chloroethane		pyrene	0.40
bis (chloromethyl) ether		2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD)	
bis (chloroethyl) ether		tetrachloroethylene	1.10
2-chloroethyl vinyl ether (mixed)		toluene	0.35
2-chloronaphthalene		trichloroethylene	0.35
2,4,6-trichlorophenol		vinyl chloride	
parachlorometa cresol		toxaphene	
chloroform (trichloromethane)	0.05		
2-chlorophenol	1.00		

OLOGICAL
STITUTE

EXHIBIT III

140[illegible] Broadway

7325

[illegible] BOULDER, COLORADO 80302 (303) 44[illegible]

FINAL REPORT ON ECOLOGICAL RESEARCH AND REHABILITATION

DONE FOR

THE MOLYBDENUM CORPORATION OF AMERICA

1970 - 1972

October 1972

USTEES
[illegible]
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I. INTRODUCTION

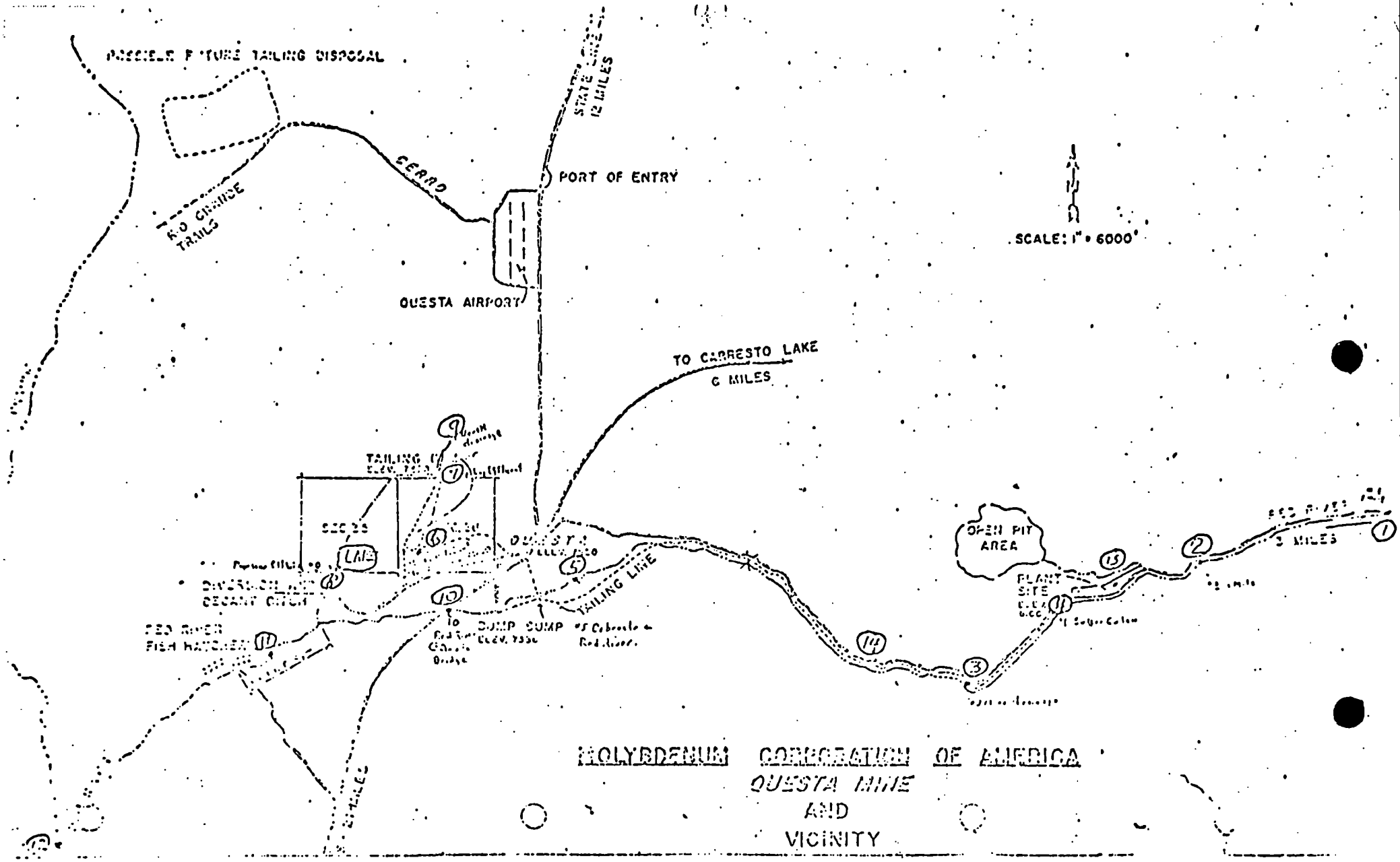
A. Background: In June of 1970, the Thorne Ecological Institute (TEI) was invited to send a professional ecologist to the Questa mine of the Molybdenum Corporation of America to make an assessment of the kinds of environmental problems that the mine was occasioning. In July Dr. Beatrice E. Willard, staff ecologist, visited the site and examined it thoroughly in the company of Mr. Colin Campbell, General Manager, and several of his associates. At that time, she was authorized to draw up a study plan for the company. August through October, three investigators were authorized to make reconnaissance of the area and draw up study proposals submitted to TEI. These were Dr. Wallace H. Johnson, plant ecologist of Ft. Collins, Dr. Robert W. Pannak, fresh water biologist of the University of Colorado, Boulder, and Dr. Cooper H. Wayman, water quality expert of G. W. Squared, Inc. in Golden. These men completed their work by November.

Dr. Willard took their plans and amalgamated them into a composite TEI proposal which she submitted to the company in early December, 1970. This proposal for ecological research and rehabilitation was reviewed by the company and approved in substance in April, 1971, when Thorne Ecological Institute was authorized to implement the proposed program. Implementation was done in two parts. The first part started in May, 1971 and went through October of 1971, after which an interim report would be submitted to the Company for its review and decision about how much of the second part was to be done. This interim report was submitted in late fall of 1971, and recommended some changes in the second phase. It was carried on during the spring and summer of 1972. The present report is a review of all data gathered during this 14-month period of research and rehabilitation activity at the Questa mine.

B. Approach: It is the policy of the Thorne Ecological Institute to make an initial reconnaissance of any environmental situation, then to select the very best possible investigators with special expertise in solving the types of problems found. This was done at the Questa site by selecting the above three-named men who have special expertise in the particular problems that were presented by the Questa mine. Secondly, TEI seeks to discover and use the ecological concepts and practices necessary for maintaining or recovering a high quality of environmental health in whatever situation it is requested to take part. Under no condition does the Thorne Ecological Institute solicit research. Its Guidelines and Criteria for accepting Applied Ecology projects are appended to this report.

II. METHODS

The selection of specific methods was left entirely to the investigators retained. In the case of the vegetation, Dr. Willard as a plant ecologist, had considerable input with Dr. Johnson on the methods and materials used in revegetating certain sites. The specific methods are described individually by the investigators in their particular sections of the report. It is important to recognize that standard scientific methods have been used in all cases. A characteristic of this method is to gather data, to analyze them, and to interpret them in an objective, scientific manner. In this way, it is hoped that environmental problems can be solved by use of ecological principles substantiated by facts gathered in the field, thus eliminating the emotional component so frequently connected to environmental problems today.



MOLYBDENUM CORPORATION OF AMERICA
QUESTA MINE
AND
VICINITY

Spring 1972: All of the areas seeded to grass in August, 1971 were examined for general appearance in the spring. The seeding on the flood control dam looked very good at this time. The plants were robust and vigorous and approximately 4 inches tall. The stand was still spotty as was observed last fall. With the extreme droughty situation that existed, it may not fill in. The plants present now should be able to stand considerable drought, but moisture is needed for full establishment and development.

The seeding on the Sugar Shack berm is very good at the present time. The plants were robust, vigorous and about 3 inches tall. The stand is spotty as observed last fall. The seeding establishment was so good in places on the berm that it actually shows up green. It should be noted that quite a bit of grazing by deer has already taken place on the plants on the berm. Again, moisture is needed badly.

The seeding on the old mine dump is a failure as was anticipated. Only a very few weak, spindly seedlings were found and these mostly in areas where large quantities of mulch had accumulated. The disturbance of digging the tree holes and refilling them destroyed some of the seedlings that were present. However, there should be enough seed of grasses and other plants in soil hauled in that spots of quite luxuriant vegetation will develop. This may be all that is needed.

B. Water Quality: Within the time span of May, 1971 through July, 1972, the G. W. Squared Corporation of Golden, Colorado was retained as a water quality investigator by Thorne Ecological Institute of Boulder, Colorado, to assess water quality in the Red River and Rio Grande River near Questa, New Mexico.

The study consisted of a 17 mile stretch of the Red River approximately 3 miles east of the Moly Corp plant site to a point of confluence between the Red River and Rio Grande River. Sampling was also performed on the tailing pond and adjacent points of the Moly Corp settling pond area.

Sampling points were selected on a basis to represent possible background concentration best above the plant site (run-off before the mill site) and to represent points representative of run-off at sources contiguous to the mine property.

The attached map represents and identifies sampling points utilized in the report. Two ground water well samples (13, 14) were periodically taken to supply information with respect to effects of possible leaching of upper formations into ground water. The sample points on the map are identified as follows:

#1 Three miles above plant site

#2 One mile above plant site

#3 Mine drainage

#4 Sulphur Gulch

#5 Confluence of Cabresto Creek and Red River

#6 Influent to tailing pond

#7 Effluent from tailing pond

#8 Pope Lake (represents the Moly Corp discharge to Red River)

#9 North drainage

#10 Red River at Questa Bridge

#11 Fish Hatchery on Red River

#12 Confluence of Red River and Rio Grande River

#13 Moly Corp plant site well

#14 Columbine well near Cottonwood

1. Background

To the time of investigation only meager data are available on water quality aspects of the Red River. The Federal Water Pollution Control Administration (now EPA) carried out chemical, bacteriological and biological studies in November, 1965 (FWPCA, 1966). This study was continued in terms of additional biological studies on the Rio Grande and Red Rivers in October, 1966 in that portion of the river immediately above and below the mouth of the Red River (FWPCA, 1966). The most detailed report completed by a government agency consists of a water quality study during November, 1970 at seven stations along the Red River and at selected points to assess metals content in mine tailings (EPA, 1971).

The most detailed study with respect to water quality on the Red River is this report. This report describes a number of stations both above and below the plant site and reflects a reasonable approach to evaluate water quality on the Red River over nearly a continuous one-year span through sampling on a monthly basis. It is significant to point out that the data obtained in EPA, 1971 comprises a variety of sampling schemes, e.g. sampling at 2-hour intervals, and 8-hour and 24-hour sample composites. It is significant to note the similarity in results between the EPA investigation data obtained by grab samples * and data in this report which are based upon a systematic sampling program. The point of identification signifies that grab samples, if properly obtained, are equivalent to longer term composite samples. The latter are not always practical or economical on detailed water quality studies.

2. Characteristics of drainage area

An excellent and concise description of the drainage system of the Red River is found in the EPA Report. Because this description is apropos of this report, it is satisfactory to utilize it as a direct quote as follows (EPA 1971, p.5-6):

*grab sample: a totally haphazard sampling method with no schedule for sampling station defined.

"The drainage area of the Red River is mountainous and ranges in elevation from 13,151 feet at Wheeler Peak to 6,500 at the confluence with the Rio Grande. Most of the area is forested except for that part above timberline and the area on both sides of State Highway 3 north of the river. Part of the area on each side of Highway 3 is irrigated for crops and pasture. The forested (sic) and rocky nature of most of the drainage basin produces fine material which contributes to the silt load of the stream. Landslides or construction activities may, however, contribute large quantities of silt from small areas.

The runoff from rainfall and snowmelt flows into the Red River via several small creeks with steep gradients. The distribution of summer rainfall and the slow melting of winter snows tend to prevent extremes of high or low flows in the river. The numerous springs and seeps tend to support a stable base flow throughout the year.

Total drainage area for the Red River is 190 square miles. Cabresto Creek, a major tributary to the Red River, has a drainage area of 35.3 square miles. The area is subject to a severe winter climate. Average mean rainfall is 21.22 inches (town of Red River). The average mean temperatures for the area is 39. 6° F (4° C). Elevation of the river bed varies from 8,600 feet (above town of Red River) to 7,100 feet near the Fish Hatchery."

3. Methods of chemical analysis

Either method 1 or method 2 (see Appendix 1) was employed for elemental analysis. A brief description of each method is given in the Appendix. The Appendix also states whether or not a different method from that of methods 1 or 2 was employed. Because the cyanide method employed may be deemed significant to future analysis, it is also given in Appendix 1.

A total of 80 samples were taken to a residue and studied by semi-quantitative analysis using the technique of emission spectrometry.

Temperature and dissolved oxygen (D.O.) were measured with the aid of a Yellow Springs Instrument D.O. meter. The in situ pH was determined with an Orion type specific ion electrode meter using a combination Ag-Ag Cl electrode system. Flow discharge measurements were made with a U.S.G.S. type flow-meter employing gear-agitations and amplified sound phones.

When the study was commenced in June, 1971, the following determinations were made on each sample:

suspended solids	chloride
hardness	cyanide
sulphate	chromium
nitrate	copper
phosphate	iron
barium	mercury
silver	maganese
arsenic	molybdenum
boron	nickel
coliform bacteria	lead
gross beta activity	cadmium
emission spec on residues	selenium
pH, D.O., temperature, flow discharge	zinc

However, after January, 1972 analyses were completed, it was concluded that a predictable trend was attained for certain determinations. Commencing with the February, 1972 sampling no analyses were made on barium, silver, arsenic, boron, cadmium, mercury, nickel, lead, chromium, gross beta activity or emission spectrographic analyses.

4. Water quality standards

With respect to effluent quality, New Mexico Regulation 4A (March 4, 1968) specifies that for a single sample the following limitations are imposed:

(1) Settleable Solids -- less than 1.0 ml/l
(2) Fecal Coliforms -- less than 500 per 100 ml
(3) pH -- between 6.6 and 8.6

In this study suspended solids and non-settleable solids were measured. The distinction should be made that settleable solids are measured on a volume/volume basis and suspended solids are based upon a mass/volume basis. Though the parameters are similar, they are not identical as a result of density differences, but corrections can be made when one knows the density of solids.

The other and most important regulation is New Mexico Number 6 (August, 1971), which states that a single sample must conform to the following:

As	0.05 mg/l or less
Ba	1.0 mg/l or less
B	0.75 mg/l or less
Cd	0.01 mg/l or less
Cr (total)	0.01 mg/l or less
Cu	0.05 mg/l or less
Pb	0.05 mg/l or less
Mn	0.1 mg/l or less
Hg	0.001 mg/l or less
Mo	0.01 mg/l or less
Ni	0.1 mg/l or less
Se	0.01 mg/l or less
Ag	0.05 mg/l or less
Zn	0.1 mg/l or less

Wayman would like to make one comment on these regulations. If the intent is to protect water quality and abate pollution, then why should community sewerage systems be given a license to exceed the copper concentration and zinc concentration by factors of 2 and 5, respectively, to wit:

> "provided that effluents from a community sewerage system may contain 0.1 mg/l copper and 0.5 mg/l zinc."

A very inte...ive and extensive report was prepared by Wayman in May, 1971 to evaluate criteria significant to New Mexico's Regulation No. 6. This report highlighted some of the inconsistencies and shortcomings in the type of criteria employed to establish standards in Regulation 6 in addition to providing background material on why a "single sample for regulatory purposes" should be criticized (Wayman, 1971).

Stream standards to be distinguished from effluent standards require that dissolved oxygen levels not be less than 6 mg/l for trout waters nor less than 5 mg/l for warm water fisheries. If the Red River constitutes a cold water fishery, then the stream standard would be 70° F. (21° C).

5. Results and discussion

Twelve sample points were selected to provide data on background (concentrations of elements) in the Red River above the Moly Corp plant and mine, to provide data on how the mine operation may exceed background concentrations, to provide information on concentrations of elements at various locations in the settling pond area, and to determine if the element concentration in the Rio Grande is substantially different from those in the Red River.

The sample points will be discussed in pairs or in combinations because of similar predictable behavior of these points or because of the manner in which the study was designed.

a) Sample Points 1,2

These two samples were selected to provide background information on the Red River above the Moly Corp plant and mine. The samples were approximately one and three miles above the plant site and should reflect natural concentrations of elements in the Red River without influence from a mechanical or man-made influences upon the river. In essence, these samples should indicate what happens as a result of the combination of stream run-off, baseflow (seepage on ground water), and from upstream leaching as distinguished from concentrations reaching the river from either mining and/or other activities prior to entrance onto the Moly Corp property and the Rio Grande.

Certain parameters analyzed were not plotted because these remained essentially constant throughout the sampling period and also because these elements were always below the heavy metal standards set by New Mexico Regulation Number 6. The actual concentrations of the various metals measured for these two points and all others were less than: (see page 17 for comparison with New Mexico standard)

Element	Concentration (ppm)
Ba	1.0
Ag	0.005
As	0.02
B	0.5
Cd	0.002
Hg	0.0001
Ni	0.005
Pb	0.005
Cr	0.01

The temperature range over the one year period ranged from 0° C in November, 1971 to 16° C in July, 1971. (see pertinent figures 1, 2). The EPA figure (large triangle) is about 2° C which is in reasonably good agreement with our data. These temperature data show about what the temperature range for a mountain stream is like over a period of a year. The data indicate further that the Red River above the Moly Corp plant is below stream standards for cold water fisheries.

The pH ranged from about 7.5 to a high of 8.4. It is significant to point out that this stream is somewhat on the basic side which is not uncommon for streams in this area. The high figure (8.4), also indicates that background nearly exceeds the standard imposed for effluent quality. The value of EPA of 7.3 for November, 1970 is substantially below our figures.. Our study employed very elegant instrumentation to eliminate streaming potentials and measurements were done in such a manner to reflect equilibrium readings. If the EPA measurements were not done in a similar manner, the values in their report may not represent equilibrium, which might explain the differences.

The D.O. measurements ranged from a low of 6 ppm to 11 ppm. The EPA value for November, 1970 was 12.1 which is in good agreement with our November, 1971 value of 10.8 ppm. The value of 6 ppm in April, 1972 is just about equivalent to the allowable stream standard for trout.

Coliform bacteria ranged from zero per 100 ml to a high of about 300 per 100 ml in October, 1971. The EPA value of 50 per 100 ml (November, 1970) is well in agreement with our values. Red River is currently below the value of 500 per 100 ml for effluent standards, but at least one measurement reflects a value that exceeds the stream standard of 200 per 100 ml for water used for body contact sports.

Suspended solids ranged from 0 to about 10 ppm. The data shown for these sample points indicates the value of 1 ml/l of settleable solids for effluent standards was exceeded for a significant portion of the sampling period.

One important thing should be indicated at this point. Samples were taken during high storm run-off above the plant on Red River and at the Ranger Station on Red River on September 30, 1971. The total solids contents of these samples were 6400 ppm and 7500 ppm, respectively. This indicates that uncontrollable natural conditions can temporarily cause problems on a stream which must be taken into consideration by regulatory agencies. A very significant and concommitant effect occurs during high run-off. Certain heavy metals concentrations also tend to increase. In this particular instance concentrations for cadmium, copper, magnanese, molybdenum (plant sample) and zinc would all have exceeded the heavy metal effluent standards indicating how background can easily fluctuate on a stream, due to natural conditions alone, and may cause serious and misleading consequences if a single sample were taken for regulatory purposes (see data Appendix 2).

The data for sulphate, phosphate, and nitrate are about what would be predictable as background for a mountain stream. The value for cyanide is also below the drinking water standard of 0.02 ppm. New Mexico has no stream or effluent standard for cyanide at the present time. The EPA data for suspended solids, sulphate, and nitrate was very similar to the data reported herein.

With respect to copper, the data indicate that the background value for copper does not presently exceed the effluent standard except during period of high run-off.

Iron was originally proposed at 0.3 ppm for Regulation 6, but never became part of that regulation. The background iron for this stream exceeds a value of 0.1 ppm for substantial amounts of time. The one reported EPA value for November, 1970 was greater than 0.1 ppm.

The regulation sets manganese at a value of 0.1 ppm. It is important to indicate that 7 of our samples were about equivalent or exceeded this standard. In addition, the value was significantly exceeded during high run-off.

The value for zinc ranged from about 0.02 to 0.1 ppm (one sample). Most of the time the stream was below the effluent standard set for the element.

The detailed results on sample 1 for Mo are as follows:

June 1971	.005 ppm
July "	.002 "
August "	.024 "
September "	.010 "
October "	.003 "
November "	.002 "
January 1972	.002 "
February "	.003 "
March "	.004 "
April "	.005 "

The effluent standard for Mo for this river has been set at 0.01 ppm. The above data indicate that there are times when the background concentration on the river can exceed the limit. Let us admit for purposes of a calculation that one background value for this stream could be as much as 0.024 ppm. This value is without significance unless the flow X concentration can be related because its the amount that is discharged and not just the concentration which alludes to a serious limitation

of concentration only as employed in effluent standards. The flow rate measured for the month of August, 1971 for the related Mo value was 12.5 CFS. The amount of Mo passing this point in one second is as follows:

$$12.5\ ft.^3 \times \frac{1728\ in.^3}{ft.^3} \times \frac{16.61\ cm^3}{in^3} \times \frac{1\ l}{1000\ cm^3} = 358.8\ l/sec.$$

$$358.8\ l/sec. \times 0.024\ mg/l = 8.61\ mg/sec.$$

$$8.61\ mg/sec. = 0.00861\ g/sec. \times 1\ \#/454\ oz.$$

$$= 1.95 \times 10^{-5}\ \#/sec.$$

Therefore, the background concentration may attain at least a value of 1.9×10^{-5} lb./sec. The regulation should allow a value of 7.9×10^{-6} lb./sec. for the flow rate, but does not consider flow x concentration. The lowest value observed for Mo during the sampling program was 0.002 ppm. If the same flow rate were applied to this value 1.6×10^{-6} lb./sec. of Mo would flow at this point. However, the actual flow rate measured was 7.8 CFS which give 1.0×10^{-6} lb/sec. Thus, the calculation shows that a setting of effluent standards by concentration alone is without merit. In reality, the numbers indicate that if the number of pounds is what we are concerned with, then if the flow rate decreases a higher concentration should be permitted in the effluent and vice-versa. This type of logic has been epitomized in a recent paper by Vogeli and King (1969), whereas the authors state on pages N3 and N5:

> "If natural conditions, including the rate of stream discharge, under which molybdenum arrives at the sampling point were to remain constant it would be possible to calculate an accurate total weight of molybdenum carried past the sampling point per minute, hour, day, and year. However, the natural conditions vary greatly, and the quantity of molybdenum in water changes. Only through constant monitoring of the molybdenum content in water and of stream discharge can accurate weight totals of molybdenum for unit time be determined."

b) Sample points 3,4

These two points were selected to contrast the Red River above the plant (background), a point below the plant (Sulphur Gulch), and leaching of country rock with flow from an underground stream (point 3). Most of the data indicate that the temperature, pH, D.O., flow rate, coliform bacteria, suspended solids, sulphate, phosphate, cyanide, copper and iron contents between sample points 1, 2 and 4 are similar. These data indicate that mining and milling activities of the plant do not change the stream concentration, in its natural state. It is also significant to indicate the similarity in our results in contrast to the EPA data of November, 1970.

The curves do indicate a striking difference between natural background of the Red River and the leaching from underground streams which might reach the river and impair water quality. During the colder months of the year, the temperature of the underground water is markedly higher than temperatures of the Red River. Underground leaching in this case produces anomalous values (in contrast to background) for manganese (4 to 50 ppm), molybdenum (4 to 10 ppm), zinc (1 to 6 ppm), nitrate (1 to 20 ppm), and sulphate (1300 to 1600 ppm). These observations are important. It indicates that leakage from underground streams at even as low flow rates at these high concentrations, could significantly change the concentrations of contaminants in a surface stream due to natural causes. The reasons for these high values in sample point number 3 most likely are leaching of the ore and/or country rock. The high sulphate value probably results from dissolution of gypsum or anydrite.

c) Sample points 5,10

These two points were selected distant from the mining and milling operations along Red River to determine if a combination of mining activities and local man-made effects (essentially agricultural) would materially change the water quality from background.

The field measurements (Temperature, D.O., pH, and flow rate) are similar to those at sample points 1, 2, and 4. The exception is that the flow rate at point 10 is somewhat higher as a result of additional tributaries, e.g. Cabresto Creek. Coliform bacteria were extremely high for sample point 5 in September, 1971; this high value probably was the result of activity from cattle grazing nearby. These data are similar

to th[illegible] of EPA with the exception of [illegible]l. Possible reasons for this discrepancy have been explained above.

Sample points 5 and 10 have nitrate values generally higher than background. Possibly the enhanced values may be the result of extensive use of fertilizers and cattle grazing in this area.

Sample point 10 contained a copper concentration of nearly 0.100 ppm in October, 1971 which would exceed the standard set forth in Regulation 6. Sample point number 5 contained excessive concentrations of manganese and zinc in November, 1971. These data indicate that at times the Red River may temporarily fluctuate in concentrations in heavy metals, even at points distantly removed from the effects of mining operations. These observations again taint with uncertainty the hazard of a single sample for regulatory purposes. The two sample identities of the EPA are similar in concentration to our data.

d) Sample points 13, 14

These two samples were taken to supply information with respect to possible leaching of upper formations by underground waters. Sampling of the wells was abandoned after October, 1971 because of higher priority attached to sample points 5 and 10 and because no significant change in trend with three sampling times.

The data indicate values predictable for ground-water in this type locale, with the exception of manganese and zinc in sample point 13. This is probably the result of underground leaching of the ore. Dissolved oxygen measurements were not made because samples were obtained by mechanical pumping which functioned as a sparger or aerator.

e) Sample points 6,7,8,9

These sample points represent what happens to tailing discharged effluent (sample point 8 for all practical purposes). A definitive analysis of these series of sample points indicates the potential for clarification by natural processes (flocculation - sedimentation) prior to discharge from Pope Lake into Red River.

The important sample point to consider is 8 because this is effluent quality. Temperature for the sample ranged from 0° C to 21° C, which does not exceed the temperature of 70° F (21° C) for the stream standard, but is just on the border line. The D.O. concentration never fell below the stream standard of 6.0 ppm for trout waters with the exception of one sample for June, 1971. Coliform bacteria for the sample was only slightly above or less than 100 per 100 ml which indicates that neither the effluent standard (500 per 100 ml) nor the stream standard of 200 per 100 ml was ever exceeded.

It is important to contrast sample points 6 and 8 for suspended solids. The suspended solids content of the influent ranged from 12 grams per liter to 680 grams per liter. In the early portion of the sampling program, sample 8 contained 4 to 5 mg/l at the point of discharge. These values for the effluent would exceed the effluent discharge standard for settleable solids of 1.0 ml/l. However, sampling from January, 1972 through April, 1972 indicated that the standard was just about being met at 1.0 ml/l. The suspended solids content should be carefully considered in the future so that the standard is not exceeded.

Nitrate and phosphate are two compounds believed to induce eutrophication (accelerated aging) of water. Though no stream or effluent standards are set for these compounds, values of 0.1 ppm and 1.0 ppm for phosphate and nitrate, respectively, should be considered. The level for phosphate is exceeded at times in North Drainage probably as a result of its content of domestic sewage. Sample point 8 also has phosphate concentrations of concern that should be carefully monitored in the future. The lowest concentration of nitrate in sample 8 during the study was 2.0 ppm. The observed concentrations are considered to be high and should be carefully checked in the future.

Cyanide does not seem to present any serious problem. There are no effluent standards or stream standards for this compound at the present time. However, the U. S. Public Health Drinking Water Standard is 0.02 ppm. The influent concentration contained as much as 1.0 ppm (June, 1971), but sample 8 was less than 0.02 ppm in all instances with the exception of August, 1971 (0.1 ppm) and October, 1971 (0.05 ppm). Cyanide probably finds its source from the flotation circuit where it is employed as a depressant. Low concentrations of cyanide are known to be toxic to fish (Nelson and Lysyj, 1971). This is particularly important at pH values less than 9.0 wherein HCN is formed. The EPA value for November, 1970 is in very good

agreement with our value measured in November, 1971.

Most heavy metals occur in the discharged effluent in insignificant amounts (same conclusion by EPA, 1971, p. 15) with the exception of Mo. Therefore, unless explained below, heavy metals contents in sample point 8 will presently meet the standards of Regulation 6.

The copper concentration in the influent since October, 1971 has been about 0.1 ppm. This value would exceed the 0.05 ppm for Regulation 6. However, the clarification process in the settling area reduces the influent concentration to values less than 0.05 ppm prior to discharge into the Red River. The EPA value in the tailing is nearly identical to our observations.

The iron concentration in sample point 8 for most of the sampling times was about 0.05 ppm, a very good value. As has been indicated before, no heavy metal standard was set for iron.

Regulation 6 sets manganese at 0.1 ppm or less. Concentrations of manganese in the influent for the most part were equal to or less than the standard. However, the manganese concentrations in the effluent (sample point 8) indicated that a substantial amount of time this standard is exceeded. The disparity between influent and effluent concentrations can most likely be attributed to the North Drainage leakage (see sample point 9). Apparently, this natural drainage may produce high concentrations. The one sample value by EPA is much lower than those found in the study though for the months of July, 1971 and September, 1971 our effluent values were less than those of the EPA study.

The data for sample 8 indicate that most values for zinc ranged from about 0.02 to 0.04 ppm which is consistent with the EPA value reported for November, 1970. These data place zinc below the standard in New Mexico Regulation 6.

The most serious problem with the discharged effluent to Red River is with Mo. New Mexico Regulation 6 sets an effluent standard of 0.1 ppm or less. Sample point 8 discharged a concentration of Mo ranging from about 1 to 2 ppm during the course of this study. Becuase of low discharge flows, only limited flows were measureable. An average value of about 2.0 CFS can be employed for purposes of this discussion. It is of interest to calculate the amount of Mo discharged to the Red River in contrast to the amount flowing past sample

points 1, 2 considered background. The calculation is made on the basis of at upper limit of 2.0 ppm Mo at a discharge rate of 2.0 CFS:

2.0 CFS = 57.4 l/sec.

57.4 l/sec. X 0.002 g/l = 0.1148 g/sec.

$$\frac{0.115 \text{ g/sec.}}{454 \text{ g/\#}} = 2.7 \times 10^{-4} \text{ \#/sec.}$$

or

$$2.7 \times 10^{-4} \text{ \#/sec. X } 86{,}400 \text{ sec./day} = 23.32 \text{ \# Mo/day}$$

At this flow rate, Regulation 6 would permit 1.2×10^{-6} #/Sec. or 0.103 #/day. Thus, the amount being discharged exceeds the regulation by about two orders of magnitude.

It was previously shown that the background concentration of Mo at sample points 1, 2 may attain a value of 1.9×10^{-5} #/sec. or 1.64 # Mo/day. <u>It is difficult to see how this standard could ever be achieved when the background concentration in Red River itself may, at times, exceed the standard by more than one order of magnitude.</u>

An important point possibly to be pursued is <u>what amount in pounds per day can a body of water tolerate</u> in a manner similar to setting pounds per day of allowable B.O.D. If this were done, then one could treat to an acceptable concentration and adjust flow rate to meet effluent standards. This logic would be a sensible and reasonable means for setting and/or meeting standards.

f) <u>Sample points 11, 12</u>

Sample point 11 was taken at the Fish Hatchery on the Red River. This sample would reflect any deleterious consequences that the discharged effluent from sample point 8 might have on the hatchery. Sample point 12 was taken at the confluence of the Red River and the Rio Grande, from June, 1971 through August, 1971. After that date it was not possible to get up and down the river bottom. The Rio Grande was then sampled above a point at its confluence with the Rio Hondo Creek, which is below Red River; this point gave a reasonable representation of the composition at the Red River - Rio Grande confluence.

Temperature ranged from about 5° C to about 21° C indicating that the Red River itself and its flow does not impair the stream standard of 21° C (cold water fishery). The EPA measured value of November, 1970 is nearly identical to our value.

The pH range was 7.5 to 8.5 and did not exceed either the stream standard or effluent standard of 6.6 to 8.6. The EPA was 7.0 in contrast to our measurements.

The C.O. content for trout waters was never below the 6.0 ppm except the June, 1971 measurement. The EPA measurement was very similar to our data.

The flow rate at sample point 11 ranged from 16.5 CFS to 23.5 CFS. One measurement by the EPA indicates a value of 45.55 CFS. This value exceeds our highest value by a factor of two. The difference most likely indicates their measurement was made at a deep and wide portion of the Red River.

Coliform bacteria at the Fish Hatchery (sample point 11) never exceeded 200 per 100 ml. This indicates that neither the effluent standard for stream standard was impaired. The data given for the Rio Grande at Rio Hondo show values from 860 to 4950 per 100 ml. These values considerably exceed the stream standard of 500 per 100 ml and are explicable on the basis of intense grazing activity in this area.

The concentrations of suspended solids, sulphate, and phosphate do not differ appreciably from background data and are in good agreement with the EPA data.

The results for nitrate exceed 1.0 ppm for much of the time period sampled. These data are substantially different from the EPA data. The high nitrate content at the Rio Grande (sample point 12) results from a combination of the high grazing activity and as a result of contamination from the Fish Hatchery itself as indicated by the EPA (ref. 3, p.4):

> "The State Fish Hatchery, located on the Red River some three or four stream miles above its mouth, is contributing to degradation of that portion of the stream, and consequently, to the Rio Grande. This degradation is increased when fish ponds are drained and cleaned for restocking. This discharge contains organics and solids and also has a fertilizing effect by the addition of nutrients."

The concentration of cyanide was less than 0.02 ppm in all instances indicating no pollution problem from sample point 8 (Red River) to as far down the Rio Grande at Rio Hondo.

No problems exist with heavy metals at either of these sample points with the exception of Mo and Mn. At sample point 11 the manganese effluent standard is exceeded by 0.04 ppm -- based on 12 samples, range 0.01 ppm to 0.04 ppm, mean 0.02 ppm -- probably as a result of natural stream contributions and in part at times from discharged effluent at sample point 8. As a result of dilution from the Rio Grande, the Mn standard is not exceeded.

The concentration of Mo at the Fish Hatchery ranged from about 0.04 ppm to 0.15 ppm which exceeds the effluent standards of 0.01 ppm. These values may be explainable from the natural background itself exceeding the standard and in part from the effluent at sample point 8. The EPA data on heavy metals is in reasonably good agreement with our data.

g) Other Analyses

Other analyses were made on the residues, but will not be discussed because there is nothing to add to the previous discussion already made on similar or identical elements.

Gross beta activity was also determined and is included in the tabulations. None of the samples analyzed approaches the value of 1000 picocuries per liter normally set by health authorities. The values usually were of the order of magnitude of ten picocuries per liter.

It is important to indicate that the EPA has classified all of its stations as biotically clean. On page 15, reference 3, it states that all stations along the Red River are considered clean based upon a numerical biotic index of 10 or more which classifies a station as clean. This would apparently indicate that effluent from sample point 8 has no impairment on stream biota.

6. Summary

This report summarizes a one year study of the measurement of various field parameters, the determination of various chemical constituents and coliform bacteria along the Red River from above the Moly Corp plant to the confluence of the Red River - Rio Grande to a point on the Rio Grande at the confluence with the Rio Hondo. The sampling program also assessed effects of underground leaching of the ore body.

The natural background concentration of the Red River at times will exceed the current effluent standard (Regulation 6) with respect to molybdenum and at other times be below the standard, suspended solids (settleable solids), and manganese.

Underground waters have a tendency to leach substantial amounts of Mo, Mn, nitrate and Zn and may contribute to pollution of the Red River from base flow considerations. The most serious problems are with Mo and Mn which were observed to discharge at concentrations as much as 10 ppm and 50 ppm, respectively.

The field measurements, with the exception of pH, coliform counts, and chemical analyses of this study are in good agreement with those reported by the EPA in their November, 1970 study (EPA, 1971).

With the exception of Mn and Mo, the discharged effluent (Pope Lake) into the Red River meets all current effluent regulations and stream standards. The suspended solids content is just about on the borderline of meeting effluent standards of 1 ml/l. The Mo concentration presently exceeds the allowable effluent standard by two magnitudes amounting to 23.32 pounds per day using an effluent concentration of 2.0 ppm at a flow rate of 2.0 CFS. The EPA report also indicates that heavy metals content is insignificant with the exception of Mo.

The EPA report indicates that the discharged effluent from Pope Lake maintains a "clean" condition on the Red River by not being less than 10 in Biotic index though a slight fertilization (mild pollution) effect is noted.

7. References

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REPORT ON WATER ANALYSES
FOR SAMPLING OF JANUARY 15, 1972

Sample	Temp °C	DO	pH	CFS	ROE	SS	Hardness
1	1.6	8.6	8.1	7.6	117	1	135
2	2.2	8.8	8.3	8.8	107	1	160
3	11.5	7.4	7.4	-	3180	3	1700
4	1.1	7.9	7.8	10.7	181	1	120
6	-	-	-	-	440	680[a]	215
7	1.5	5.8	7.6	-	1110	2	775
8	1.9	6.9	7.5	-	1260	1	715
9	2.6	7.4	8.2	-	1080	3	860
10	4.8	8.1	8.0	12.6	126	2	118
11	5.4	7.4	7.9	16.5	190	3	340
12	6.9	7.7	8.1	-	165	3	135

SS = suspended solids
[a] = total solids

Sample	Nitrate	Phosphate	Sulfate	Ag	As	B	Ba
1	0.4	<0.02	45	<0.005	<0.02	<0.1	<1
2	0.5	<0.02	125	<0.005	<0.02	<0.1	<1
3	17.6	<0.02	1530	<0.005	<0.02	<0.1	<1
4	0.3	<0.02	130	<0.005	<0.02	<0.1	<1
6	0.4	<0.02	300	<0.005	<0.02	<0.1	<1
7	5.8	<0.02	665	<0.005	<0.02	<0.1	<1
8	4.6	0.21	790	<0.005	<0.02	<0.1	<1
9	0.9	<0.02	640	<0.005	<0.02	<0.1	<1
10	1.5	<0.02	147	<0.005	<0.02	<0.1	<1
11	1.8	<0.02	226	<0.005	<0.02	<0.1	<1
12	2.4	0.05	91	<0.005	<0.02	<0.1	<1

Sample	Cd	Cl	CN	Cr	Cu	Fe	Hg
1	<0.005	1	<.01	<.01	<.02	.08	<.0001
2	<0.005	1	<.01	<.01	<.02	<.04	<.0001
3	0.005	8	<.01	<.01	.03	.05	<.0001
4	<0.005	1	<.01	<.01	<.02	<.04	<.0001
6	<0.005	1	<.01	<.01	.14	<.04	<.0001
7	<0.005	1	<.01	<.01	.03	<.04	<.0001
8	<0.005	2	<.01	<.01	<.02	<.04	<.0001
9	<0.005	1	<.01	<.01	<.02	<.04	<.0001
10	<0.005	1	<.01	<.01	<.02	<.04	<.0001
11	<0.005	3	<.01	<.01	<.02	<.04	<.0001
12	<0.005	4	<.01	<.01	<.02	.06	<.0001

Sample	Mn	Mo	Ni	Pb	Se	Zn	Coliform per 100/ml
1	.09	<.002	.008	<.005	<.005	.02	155
2	.25	.004	.007	<.005	<.005	.08	72
3	19.0	7.6	.050	<.005	<.005	5.6	41
4	.14	.003	.004	<.005	<.005	.05	170
6	.15	3.4	.073	<.005	<.005	.03	1
7	.40	1.8	.038	<.005	<.005	.05	18
8	.42	2.1	.029	<.005	<.005	.05	115
9	.36	.38	.018	<.005	<.005	.09	142
10	.28	.006	.015	<.005	<.005	.07	108
11	.18	.18	.010	<.005	<.005	.03	65
12	.06	.004	.024	<.005	<.005	.02	1860

Sample	Gross Beta Activity pC/l
1	3.1
2	6.9
3	19.0
4	4.4
6	6.2
7	10.8
8	4.9
9	12.8
10	1.6
11	6.1
12	11.9

Data

SPECTROGRAPHIC WATER ANALYSES
(Values in ppm or % as indicated)

Element	1	2	3	4	6	8	11	12
Fe	0.1%	0.02%	0.02%	0.04%	0.8%	0.1%	0.07%	0.3%
Ca	18.0%	9.0%	17.0%	18.0%	12.0%	19.0%	18.0%	19.0%
Mg	3.0%	2.0%	1.5%	2.0%	3.0%	0.9%	2.4%	1.0%
Ag	<1	<1	<1	<1	<1	<1	<1	<1
As	<500	<500	<500	<500	<500	<500	<500	<500
B	15	<10	15	10	10	20	40	70
Ba	180	40	10	60	100	40	60	130
Be	<2	<2	20	<2	<2	<2	<2	<2
Bi	<10	<10	<10	<10	<10	<10	<10	<10
Cd	<50	<50	<50	<50	<50	<50	<50	<50
Co	<5	<5	20	<5	10	<5	<5	<5
Cr	<10	<10	50	<10	40	<10	<10	<10
Cu	60	40	60	10	140	10	10	20
Ga	<10	<10	<10	<10	<10	<10	<10	<10
Ge	<20	<20	<20	<20	<20	<20	<20	<20
La	20	40	20	30	20	20	20	20
Mn	900	1100	9500	600	800	900	300	200
Mo	<2	<2	850	6	1600	1100	800	600
Nb	<20	<20	<20	<20	<20	<20	<20	<20
Ni	<5	<5	<5	10	90	<5	10	30
Pb	<10	<10	60	<10	<10	<10	<10	<10

Peter R.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

DATE: June 27, 1984

SUBJECT: Effluent Toxicity Testing at Molycorp NM0022306

FROM: James E. Stiebing, P.E.
Chief, Surveillance Branch (6ES-S)

TO: Roger Hartung, 6W-E
Bob Hanneschlager, 6W-P

Attached is a request for effluent toxicity testing of the Molycorp Mill tailing ~~and~~ pond effluents. It's been 3 years since an EPA inspection was made of this facility and there is no indication that this activity has been coordinated with your branch even though you may be involved.

Since there is no request for EPA presence and all the work, save the bioassay, will be done by state personnel, I do not have the same level of concern here as was true for the Tyler Pipe permit. It does appear to me, however, that this could present potential enforcement and/or permitting problems if only biotoxicity determinations are made. I would encourage you to get involved in these kinds of discharge monitoring activities to reduce the potential for problems in your respective areas of responsibility.

ESD is prepared to provide technical assistance to you and the state in planning a more meaningful and useful sampling of the Molycorp effluent for enforcement and permitting purposes if you so desire. Please contact me at X7-9784 if you wish assistance.

cc: Bruce Elliott (6W-Q)

Attachment

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

DATE: June 26, 1984

SUBJECT: Assistance for Arranging Acute and Chronic Bioassays

Control to Holman

FROM: Robert B. Elliott RBE
Chief, Water Quality Management Branch, 6W-Q

TO: James E. Stiebing
Chief, Surveillance Branch, 6ES-S

We request your assistance in arranging for two acute bioassays using Daphnia and two chronic bioassays using Ceriodaphnia to be performed at the Houston Laboratory and ERL-Duluth, respectively. These bioassays, which were requested by the State of New Mexico, Environmental Improvement Division, will serve to test the toxic nature of the Molycorp Mill tailings pond effluents which are discharged into the Red River, near Questa, New Mexico. The State would prefer to collect the samples in about two to three weeks if this time frame is workable with the two labs. For both acute and chronic tests, the State will collect effluent samples and samples for use as dilution water.

Further assistance will be needed at a later date, some time between August and October. In this case, approximately six additional chronic bioassays are requested. Precise number of bioassays to be performed would depend on EMR Duluth's capabilities at the time. These bioassays would function to better evaluate the potential instream toxicity due to the high metals concentrations which are characteristic of the Red River.

We would appreciate a response to this request as soon as possible so that the State may plan their sampling trip.

Thank you for your assistance. Please contact Philip Crocker at X7-9916 if there are any questions.

Mac,
See me on this--ASAP.
Jim D.

RECEIVED
SURVEILLANCE BRANCH
'84 JUN 26 PM 3 53

EPA Form 1320-6 (Rev. 3-76)

ROUTING AND TRANSMITTAL SLIP

Date 5-3-85

TO: (Name, office symbol, room number, building, Agency/Post)	Initials	Date
1. ~~Ellen Caldwell~~		
2. ~~Sam Coker~~		
3. Fred Humke		
4.		

	Action		File		Note and Return
	Approval		For Clearance		Per Conversation
	As Requested		For Correction		Prepare Reply
	Circulate		For Your Information		See Me
	Comment		Investigate		Signature
	Coordination		Justify		

REMARKS

Attached is a copy of an Evidentiary Hearing Request received from:

Molycorp Inc.

Please have the assigned Engineer provide Pat Hudson (x9970) with a Technical Response to the Evidentiary Hearing Request within two weeks.

Thank you.

DO NOT use this form as a RECORD of approvals, concurrences, disposals, clearances, and similar actions

FROM: (Name, org. symbol, Agency/Post)	Room No.—Bldg.
	2735
Toni G. Gould, Paralegal Spec.	Phone No. x9024

5041-102

OPTIONAL FORM 41 (Rev. 7-76)
Prescribed by GSA
FPMR (41 CFR) 101-11.206

U.S. GOVERNMENT PRINTING OFFICE : 1984 O - 431-986 (346)

4/10 5-4-85 Mamke 4-16-85

CONCERNED CITIZENS OF QUESTA

MR. MANUEL R. ORTEGA - MR. GEORGE WEISS (REPRESENTATIVES)

U.S.E.P.A. - Region VI
MS. Ellen Caldwell
Inter First - two Bldg.
1201 Elm Street
Dallas, Texas 75270

April 8, 1985

RE: NPDES Permit No. 022306; Moly Corp. Inc.

Dear Ms. Caldwell,

In Reference to Public Notice of Draft NPDES Permit Advertised in Order No. 5T-8216-NNLX, we as concerned citizens of Questa would strongly urge U.S.E.P.A. to deny certification or in the alternative to modify Permit No. 022306 presently held by Moly Corp. Inc for the following reasons:

(1.) Permit does not allow for Reclaimation of Land(s) in and around the Village of Questa

(2) U.S.E.P.A. does not have Enforcement Powers pertaining to Reclaimation of Lands in the State of New Mexico for Mining Industry.

(3) Spill Prevention Plan, for mine "Tailings", filed by Moly Corp Inc. with the U.S.E.P.A. is inadequate, since it does not protect Questa's Irrigation System from Abuse and/or serious contamination. (Acequias)

(4.) This Permit, under U.S.E.P.A. guidelines, does not allow for the full application of the New Mexico (State) Water Quality Act or the N.M. State Air Quality Act or their Regulatory Provisions.

Appropriate Supporting References on the Serious Effects of Water, Air and Soil Pollution caused by Moly Corp in and around Questa from mining and milling activities, are enclosed.

To further substantiate our concerns through slides, photos and documented evidence, a Request for an Evidentiary Hearing is hereby made. If you have any questions please contact Messrs. Ortega or Weiss at 586-0699 or 586-1095, anytime.

Thank You,

George Weiss — Manuel R. Ortega

P. O. BOX 1025 ◦ QUESTA, NEW MEXICO ◦ 87556

Date 5/7/85

ROUTING AND TRANSMITTAL SLIP

TO: (Name, office symbol, room number, building, Agency/Post)	Initials	Date
1. Becker (GW-PI)		
2. Hudson/Gould (GORC)		
3.		
4.		
5.		

	Action		File		Note and Return
	Approval		For Clearance		Per Conversation
	As Requested		For Correction		Prepare Reply
	Circulate		For Your Information		See Me
	Comment		Investigate		Signature
	Coordination		Justify		

REMARKS Evid. Request - Molycorp, NM0022306
All issues raised are addressed in issues #6 thru #8 of the attached response to comments. This is merely a permit modification and all issues raised are irrelevant to the modification (see Part 124.5(c)) as well as to basic NPDES permitting. Furthermore, a request for an evidentiary hearing is not timely during the public notice comment period.

DO NOT use this form as a RECORD of approvals, concurrences, disposals, clearances, and similar actions

FROM: (Name, org. symbol, Agency/Post)	Room No.—Bldg.
Fred Hunke (GW-PI	Phone No.

5041-102

OPTIONAL FORM 41 (Rev. 7-76)
Prescribed by GSA
FPMR (41 CFR) 101-11.206

☆ U.S. GOVERNMENT PRINTING OFFICE : 1982 O - 361-529 (214)

CERTIFIED/RETURN RECEIPT REQUESTED

MAY 25 1985

Mssrs. Manuel R. Ortega and George Weiss
Concerned Citizens of Questa
P. O. Box 1026
Questa, New Mexico

Re: Molycorp, Inc.
NPDES Permit No. NM0022306

Dear Mssrs. Ortega and Weiss:

This will acknowledge receipt of your request for an evidentiary hearing dated April 8, 1985 on NPDES permit NM0022306 which was received and filed April 16, 1985.

Your request has been determined to be deficient in that it does not conform with the requirements of 40 CFR Part 124, Subpart E, a copy of which is enclosed. Specifically, your request fails to comply with §124.74(a) in that the Regional Administrator has neither issued the permit nor served notice of EPA's final permit decision. Your request has been made too early and is, thus, untimely. Therefore, your request for an evidentiary hearing is denied. If upon the issuance of EPA's final permit decision you are still not satisfied with the permit, at that time you may request an evidentiary hearing.

This denial may be appealed to the Administrator under 40 CFR 124.91 within thirty (30) days of service of this letter.

The above discussion refers only to the Evidentiary Hearing Request portion of your letter. The remainder of your letter is being considered as comments prior to the issuance of the final permit. Your comments are greatly appreciated. Thank you.

Sincerely yours,

/s/ Frances E. Phillips for

Dick Whittington, P.E.
Regional Administrator

Enclosure

cc: Regional Hearing Clerk

bcc: S.Becker (6W-PI)
✓J.Watson (6W-PS)
W.Poole (6A)

6ORC:GOULD:x9024:05/06/85:revised HUDSON:05/06/85:revised SEALS:05/20/85 disk # EH

~~Attorney~~
② File

Permit file
NM0022306

CERTIFIED/RETURN RECEIPT REQUESTED

MAY 25 1985

Mssrs. Manuel R. Ortega and George Weiss
Concerned Citizens of Questa
P. O. Box 1025
Questa, New Mexico

Re: Molycorp, Inc.
NPDES Permit No. NM0022306

Dear Mssrs. Ortega and Weiss:

This will acknowledge receipt of your request for an evidentiary hearing dated April 8, 1985 on NPDES permit NM0022306 which was received and filed April 16, 1985.

Your request has been determined to be deficient in that it does not conform with the requirements of 40 CFR Part 124, Subpart E, a copy of which is enclosed. Specifically, your request fails to comply with §124.74(a) in that the Regional Administrator has neither issued the permit nor served notice of EPA's final permit decision. Your request has been made too early and is, thus, untimely. Therefore, your request for an evidentiary hearing is denied. If upon the issuance of EPA's final permit decision you are still not satisfied with the permit, at that time you may request an evidentiary hearing.

This denial may be appealed to the Administrator under 40 CFR 124.91 within thirty (30) days of service of this letter.

The above discussion refers only to the Evidentiary Hearing Request portion of your letter. The remainder of your letter is being considered as comments prior to the issuance of the final permit. Your comments are greatly appreciated. Thank you.

Sincerely yours,

/s/ Frances E. Phillips for

Dick Whittington, P.E.
Regional Administrator

Enclosure

cc: Regional Hearing Clerk

bcc: ✓S.Becker (6W-PI)
J.Watson (6W-PS)
W.Poole (6A)

6ORC:GOULD:x9024:05/06/85:revised HUDSON:05/06/85:revised SEALS:05/20/85 disk # EH

P 175 453 664

RECEIPT FOR CERTIFIED MAIL

NO INSURANCE COVERAGE PROVIDED
NOT FOR INTERNATIONAL MAIL

(See Reverse)

Sent to	
Street and No.	
P.O., State and ZIP Code	
Postage	$
Certified Fee	
Special Delivery Fee	
Restricted Delivery Fee	
Return Receipt Showing to whom and Date Delivered	
Return receipt showing to whom, Date, and Address of Delivery	
TOTAL Postage and Fees	$
Postmark or Date	

PS Form 3800, Feb. 1982 ★ U.S.G.P.O. 1984-446-014

U ED STATES ENVIRONMENTAL PROTE ON AGENCY

JUL 0 5 1985

CERTIFIED MAIL: RETURN RECEIPT REQUESTED (P 176 453 664)

Mr. C. R. Sacrison
General Manager
Molycorp, Inc.
P.O. Box 469
Questa, New Mexico 87556

Re: Application to Discharge to Waters of the United States
Permit No. NM0022306

Dear Mr. Sacrison:

Enclosed is the public notice of the Agency's final permit decision and a copy of our response to comments and the final permit. This public notice describes any substantial changes from the draft permit.

If you intend to request an evidentiary hearing, please follow the requirements outlined in the public notice of the draft permit.

Should you have any questions please feel free to contact the Permits Branch at the above address or telephone (214) 767-4375.

Sincerely,

/s/Myron O. Knudson

Myron O. Knudson, P.E.
Director, Water Management Division (6W)

Enclosures

cc: w/permit copy
New Mexico Environmental Improvement Division

JGH 4/15/85

4/12/85:HUMKE:JR:J13#3

CONCURRENCES

SYMBOL	6W-PI	6W-P		6W-EC	6ORC		
SURNAME	Becker	Hannesschlager		RANESH	Corley		
DATE	5/20/85	5/24	6/7	6/13	6/17		

EPA Form 1320-1 (12-70) OFFICIAL FILE COPY

*U.S. GPO : 1984-436-836

Advertising Order Number 5T-3338-NNLX
U.S. Environmental Protection Agency - Region 6
Public Notice of Final Permit Decision

JULY 6, 1985

This is to give notice that the U.S. Environmental Protection Agency, Region 6, has made a final permit decision and will issue the following ONE (1) Proposed Permit(s) under the National Pollutant Discharge Elimination System. The permit(s) will become effective 30 days from the date of this Public Notice. Any substantial changes from the Draft Permit are cited.

This issuance is based on a final staff review of the administrative record and comments received. A Response to Comments is available by writing to:

Ms. Ellen Caldwell
Permits Branch (6W-PS)
U.S. Environmental Protection Agency - Region 6
Interfirst Two Building
1201 Elm Street
Dallas, Texas 75270
(214) 767-2765

Any person may request an Evidentiary Hearing on this final permit decision. However, the request must be submitted within 30 days from the date of this Notice. The request should be in accordance with the requirements of 40 CFR 124.74 (Federal Register Vol. 45, No. 98, Monday, May 19, 1980). The original public notice contains the stay provisions of a granted evidentiary hearing request.

Further information including the administrative record may be viewed at the above address between 8 a.m. and 4:30 p.m., Monday through Friday.

NPDES authorization to discharge to waters of the United States, permit No. NM0022306.

The applicant's mailing address is: Molycorp, Inc.
P.O. Box 469
Questa, New Mexico 87556

The discharge from this molybdenum milling facility is made into the Red River, a water of the United States classified for coldwater fishing; fish culture; livestock and wildlife watering; and secondary contact recreation. The discharge is located on that water approximately one mile above the bridge at the Red River Fish Hatchery. Under the standard industrial classification (SIC) code 1061, the applicant's activities are mining and milling operations which produce molybdenum disulfide concentration.

There are no substantial changes from the draft permit.

N/A

This is our response to the comments received on the subject draft NPDES permit in accordance with our regulations.

RESPONSE TO COMMENTS
DRAFT NPDES PERMIT

Permit No.:	NM0022306
Permittee:	Molycorp, Inc.
Facility Name/Location:	Questa, Taos County, New Mexico
Draft Permit Public Notice Date:	March 9, 1985
Prepared by:	Fred Humke

Issue #1

NMEID requested that on the cover page following "receiving waters named Red River" an addition, "... Segment 2-119 of the Upper Rio Grande Basin" be made.

Response #1

EPA concurs.

Issue #2

NMEID requests that the effective date and expiration date be filled in on the cover sheet.

Response #2

These dates are not assigned until the final permit decision.

Issue #3

NMEID notes that the 3 month and 6 month Progress Report due date will have passed by the effective date of the new permit.

Response #3

This is not a new permit but is merely a modified permit. The schedule dates are still incumbent upon the permitee.

Issue #4

NMEID states that Molycorp is a major discharger and requests that the appropriate day of the month DMR is due and date of the first DMR be included.

Response #4

These are not assigned until the final permit decision.

Issue #5

NMEID requests that the state reporting address be included on Page 14.

Response #5

EPA concurs.

Issue #6

Concerned Citizens of Questa requested that EPA not modify the permit because the permit does not allow for reclamation of lands in and around the village of Questa and EPA does not have enforcement power pertaining to reclamation of lands in the State of New Mexico for the mining industry.

Response #6

The NPDES permit is issued by EPA to regulate the point source discharges to surface waters, which in this case is the Red River. EPA does not have authority to regulate the restoration of land associated with molybdenum mining.

Issue #7

Concerned Citizens of Questa requested that EPA not modify the permit because the spill prevention plan filed with EPA is not adequate to protect the irrigation system.

Response #7

This permit modification does not involve the spill prevention plan: Spill prevention is dealt with as an enforcement matter and is presently being addressed through the enforcement functions.

Issue #8

Concerned Citizens of Questa requested that EPA does not modify this permit because the permit, under EPA guidelines, does not allow for the full application of the State of New Mexico Water Quality Act or the New Mexico State Air Quality Act.

Response #8

The NPDES permit addresses point source discharges which are requested in accordance with national technology and New Mexico Water Quality Standards. Other laws and acts are addressed by other media.

Permit No. NM0022306
Application No. NM0022306

AUTHORIZATION TO DISCHARGE UNDER THE
NATIONAL POLLUTANT DISCHARGE ELIMINATION SYSTEM

In compliance with the provisions of the Federal Water Pollution Control Act, as amended, (33 U.S.C. 1251 et. seq; the "Act"),

Molycorp, Inc.
P.O. Box 469
Questa, New Mexico 87556

is authorized to discharge from a facility located at Questa, Taos County, New Mexico

to receiving waters named Red River, Segment 2-119 of the Upper Rio Grande Basin

in accordance with effluent limitations, monitoring requirements and other conditions set forth in Parts I, II, and III hereof.

This permit shall become effective on August 6, 1985

This permit and the authorization to discharge shall expire at midnight, August 30, 1988

Signed this 5th day of July 1985

Myron O. Knudson, P.E.
Director, Water Management Division (6W)

PART I
REQUIREMENTS FOR NPDES PERMITS

SECTION A. EFFLUENT LIMITATIONS AND MONITORING REQUIREMENTS - Outfall 001 & 002

During the period beginning the effective date and lasting through *****
the permittee is authorized to discharge from Outfall(s) serial number(s) 001, process water from milling operation and tailings disposal; 002 - seepage from tailing impoundment.

Such discharges shall be limited and monitored by the permittee as specified below:

Effluent Characteristic	Discharge Limitations			
	kg/day(lbs/day)		Other Units (Specify)	
	Daily Avg	Daily Max	Daily Avg	Daily Max
Flow-m^3/Day(MGD)	N/A	N/A	*	*
Temperature	N/A	N/A	* °C (*°F)	* °C(* °F)
Chemical Oxygen Demand	N/A	N/A	60 mg/l	90 mg/l
Total Suspended Solids	N/A	N/A	20 mg/l	30 mg/l
Total Arsenic	N/A	N/A	0.5 mg/l	1.0 mg/l
Total Cadmium	N/A	N/A	N/A	.05 mg/l
Total Copper	N/A	N/A	0.15 mg/l	0.30 mg/l
Total Cyanide	N/A	N/A	0.025 mg/l	0.05 mg/l
Fluoride	N/A	N/A	N/A	3.0 mg/l
Total Iron	N/A	N/A	N/A	0.6 mg/l
Total Lead	N/A	N/A	0.3 mg/l	0.6 mg/l
Total Manganese	N/A	N/A	1.0 mg/l	1.5 mg/l
Total Mercury	N/A	N/A	0.001 mg/l	0.002 mg/l
Total Molybdenum	35(78)****	71(156)***	* mg/l	* mg/l
Total Zinc	N/A	N/A	N/A	0.2 mg/l
Biomonitoring	N/A	N/A	N/A	N/A

Effluent Characteristic	Monitoring Requirements	
	Measurement Frequency	Sample Type
Flow-m^3/Day(MGD)	(1)	Record
Temperature	1/week	In Situ
Chemical Oxygen Demand	1/week	Composite**
Total Suspended Solids	1/week	Composite**
Total Arsenic	1/week	Composite**
Total Cadmium	1/week	Composite**
Total Copper	1/week	Composite**

Continued from page 2

Total Cyanide	(2)	Composite**
Fluoride	1/week	Composite**
Total Iron	1/week	Composite**
Total Lead	1/week	Composite**
Total Manganese	1/week	Composite**
Total Mercury	1/week	Composite**
Total Molybdenum	1/week	Composite**
Total Zinc	1/week	Composite**
Biomonitoring	(3)	(4)

* Report
** See Part III, Paragraph B
*** Sum total for Outfalls 001 & 002
**** Sum total average for Outfalls 001 & 002 for six months preceeding reporting period end date; also report daily average mass for each month.
(1) Continuous and totalized monitoring for Outfall 001; daily estimate for Outfall 002
(2) 2/week for Outfall 001; 1/week for Outfall 002.
(3) See Part III, Paragraph D.
(4) See Part III, Paragraph E.
***** One year from date of mill start-up.

The pH shall not be less than 6.0 standard units nor greater than 9.0 standard units and shall be monitored 1/week by grab sample.

There shall be no discharge of floating solids or visible foam in other than trace amounts.

Samples taken in compliance with the monitoring requirements specified above shall be taken at the following location(s):

<u>Outfall 001</u> Latitude 36° 40' 61" and Longitude 105° 38' 30" which is the discharge spillway from Pope Lake.

<u>Outfall 002</u> Latitude 36° 40' 61" and Longitude 105° 37' 30" which is the collected and combined seepage from the tailings impoundment.

PART I
REQUIREMENTS FOR NPDES PERMITS

SECTION A. EFFLUENT LIMITATIONS AND MONITORING REQUIREMENTS - Outfall 001 & 002

During the period beginning ***** and lasting through the expiration date, the permittee is authorized to discharge from Outfall(s) serial number(s) 001, process water from milling operation and tailings disposal; 002, seepage from tailing impoundment.

Such discharges shall be limited and monitored by the permittee as specified below:

Effluent Characteristic	Discharge Limitations			
	kg/day(lbs/day)		Other Units (Specify)	
	Daily Avg	Daily Max	Daily Avg	Daily Max
Flow-m^3/Day(MGD)	N/A	N/A	*	*
Temperature	N/A	N/A	* °C (*°F)	* °C (*°F)
Chemical Oxygen Demand	N/A	N/A	60 mg/l	90 mg/l
Total Suspended Solids	N/A	N/A	20 mg/l	30 mg/l
Total Arsenic	N/A	N/A	0.5 mg/l	1.0 mg/l
Total Cadmium	N/A	N/A	N/A	.05 mg/l
Total Copper	N/A	N/A	0.15 mg/l	0.30 mg/l
Total Cyanide	N/A	N/A	0.025 mg/l	0.05 mg/l
Fluoride	N/A	N/A	N/A	3.0 mg/l
Total Iron	N/A	N/A	N/A	0.6 mg/l
Total Lead	N/A	N/A	0.3 mg/l	0.6 mg/l
Total Manganese	N/A	N/A	1.0 mg/l	1.5 mg/l
Total Mercury	N/A	N/A	0.001 mg/l	0.002 mg/l
Total Molybdenum	11(25)****	23(50)***	1.0 mg/l	2.0 mg/l
Total Zinc	N/A	N/A	N/A	0.2 mg/l
Biomonitoring	N/A	N/A	N/A	N/A

Effluent Characteristic	Monitoring Requirements	
	Measurement Frequency	Sample Type
Flow-m^3/Day(MGD)	(1)	Record
Temperature	1/week	In-Situ
Chemical Oxygen Demand	1/week	Composite**
Total Suspended Solids	1/week	Composite**
Total Arsenic	1/week	Composite**
Total Cadmium	1/week	Composite**
Total Copper	1/week	Composite**
Total Cyanide	(2)	Composite**

Continued from page 4

Fluoride	1/week	Composite**
Total Iron	1/week	Composite**
Total Lead	1/week	Composite**
Total Manganese	1/week	Composite**
Total Mercury	1/week	Composite**
Total Molybdenum	1/week	Composite**
Total Zinc	1/week	Composite**
Biomonitoring	(3)	(4)

* Report
** See Part III, paragraph B.
*** Sum total for Outfalls 001 & 002.
**** Sum total average for Outfalls 001 & 002 for six months preceeding reporting period end date; also report daily average mass for each month.
(1) Continuous and totalized monitoring for Outfall 001; daily estimate or Outfall 002.
(2) 2/week for Outfall 001; 1/week for Outfall 002.
(3) See Part III, Paragraph D.
(4) See Part III, Paragraph E.
***** One year from date of mill start-up.

The pH shall not be less than 6.0 standard units nor greater than 9.0 standard units and shall be monitored 1/week by grab sample.

There shall be no discharge of floating solids or visible foam in other than trace amounts.

Samples taken in compliance with the monitoring requirements specified above shall be taken at the following location(s):

<u>Outfall 001</u> Latitude 36° 40' 61" and Longitude 105° 38' 30" which is the discharge spillway from Pope Lake.

<u>Outfall 002</u> Latitude 36° 40' 61" and Longitude 105° 37' 30" which is the collected and combined seepage from the tailings impoundment.

SECTION B. SCHEDULE OF COMPLIANCE

The permittee shall achieve compliance with the effluent limitations specified for discharges in accordance with the following schedule:

Report mill start-up

Progress Report	3 mos. after mill start-up
Progress Report	6 mos. after mill start-up
Progress Report	9 mos. after mill start-up
Achieve Compliance	12 mos. after mill start-up

PART II
STANDARD CONDITIONS FOR NPDES PERMITS

SECTION A. GENERAL CONDITIONS

1. Duty to Comply

The permittee must comply with all conditions of this permit. Any permit noncompliance constitutes a violation of the Clean Water Act and is grounds for enforcement action; for permit termination, revocation and reissuance, or modification; or for denial of a permit renewal application.

2. Penalties for Violations of Permit Conditions

The Clean Water Act provides that any person who violates a permit condition implementing Sections 301, 302, 306, 307, 308, 318, or 405 of the Clean Water Act is subject to a civil penalty not to exceed $10,000 per day of such violation. Any person who willfully or negligently violates permit conditions inplementing Sections 301, 302, 306, 307, or 308 of the Clean Water Act is subject to a fine of not less than $2,500 nor more than $25,000 per day of violation, or by imprisonment for not more than 1 year, or both.

3. Permit Actions

This permit may be modified, revoked and reissued, or terminated for cause including, but not limited to, the following:

a. Violation of any terms or conditions of this permit;

b. Obtaining this permit by misrepresentation or failure to disclose fully all relevant facts; or

c. A change in any condition that requires either a temporary or a permanent reduction or elimination of the authorized discharge; or

d. A determination that the permitted activity endangers human health or the environment and can only be regulated to acceptable levels by permit modification or termination.

The filing of a request by the permittee for a permit modification, revocation and reissuance, or termination, or a notification of planned changes or anticipated noncompliance, does not stay any permit condition.

4. Toxic Pollutants

Notwithstanding Section A, paragraph 3 above, if any toxic effluent standard or prohibition (including any schedule of compliance specified in such effluent standard or prohibition) is promulgated under Section 307(a) of the Clean Water Act for a toxic pollutant which is present in the discharge and that standard or prohibition is more stringent than any limitation on the pollutant in this permit, this permit shall be modified or revoked and reissued to conform to the toxic effluent standard or prohibition and the permittee so notified.

The permittee shall comply with effluent standards or prohibitions established under Section 307(a) of the Clean Water Act for toxic pollutants within the time provided in the regulations that established those standards or prohibitions, even if the permit has not yet been modified to incorporate the requirement.

5. Civil and Criminal Liability

Except as provided in permit conditions on "Bypassing" Section B, paragraph 4.b. and "Upsets" Section B, paragraph 5.b., nothing in this permit shall be construed to relieve the permittee from civil or criminal penalties for noncompliance.

6. Oil and Hazardous Substance Liability

Nothing in this permit shall be construed to preclude the institution of any legal action or relieve the permittee from any responsibilities, liabilities, or penalties to which the permittee is or may be subject under Section 311 of the Clean Water Act.

7. State Laws

Nothing in this permit shall be construed to preclude the institution of any legal action or relieve the permittee from any responsibilities, liabilities, or penalties established pursuant to any applicable State law or regulation under authority preserved by Section 510 of the Clean Water Act.

8. Property Rights

The issuance of this permit does not convey any property rights of any sort, or any exclusive privileges, nor does it authorize any injury to private property or any invasion of personal rights, nor any infringement of Federal, State, or local laws or regulations.

9. Severability

The provisions of this permit are severable, and if any provision of this permit or the application of any provision of this permit to any circumstance is held invalid, the application of such provision to other circumstances, and the remainder of this permit, shall not be affected thereby.

10. Definitions

The following definitions shall apply unless otherwise specified in this permit:

a. "Daily Discharge" means the discharge of a pollutant measured during a calendar day or any 24-hour period that reasonably represents the calendar day for purposes of sampling. For pollutants with limitations expressed in terms of mass, the "daily discharge" is calculated as the total mass of the pollutant discharged over the sampling day. For pollutants with limitations expressed in other units of measurement, the "daily discharge" is calculated as the average measurement of the pollutant over the sampling day. "Daily discharge" determination of concentration made using a composite sample shall be the concentration of the composite sample. When grab samples are used, the "daily discharge" determination of concentration shall be the arithmetic average (weighted by flow value) of all samples collected during that sampling day.

b. "Daily Average" discharge limitation means the highest allowable average of "daily discharges" over a calendar month, calculated as the sum of all "daily discharges" measured during a calendar month divided by the number of "daily discharges" measured during that month.

c. "Daily Maximum" discharge limitation means the highest allowable "daily discharge" during the calendar month.

SECTION B. OPERATION AND MAINTENANCE OF POLLUTION CONTROLS

1. Proper Operation and Maintenance

The permittee shall at all times properly operate and maintain all facilities and systems of treatment and control (and related appurtenances) which are installed or used by the permittee to achieve compliance with the conditions of this permit. Proper operation and maintenance also includes adequate laboratory controls and appropriate quality assurance procedures. This provision requires the operation of backup or auxiliary facilities or similar systems which are installed by a permittee only when the operation is necessary to achieve compliance with the conditions of the permit.

2. Need to Halt or Reduce not a Defense

It shall not be a defense for a permittee in an enforcement action that it would have been necessary to halt or reduce the permitted activity in order to maintain compliance with the conditions of this permit.

3. Duty to Mitigate

The permittee shall take all reasonable steps to minimize or prevent any discharge in violation of this permit which has a reasonable likelihood of adversely affecting human health or the environment.

4. Bypass of Treatment Facilities

a. Definitions

(1) "Bypass" means the intentional diversion of waste streams from any portion of a treatment facility.

(2) "Severe property damage" means substantial physical damage to property, damage to the treatment facilities which causes them to become inoperable, or substantial and permanent loss of natural resources which can reasonably be expected to occur in the absence of a bypass. Severe property damage does not mean economic loss caused by delays in production.

b. Bypass not exceeding limitations. The permittee may allow any bypass to occur which does not cause effluent limitations to be exceeded, but only if it also is for essential maintenance to assure efficient operation. These bypasses are not subject to the provisions of Section B, paragraphs 4.c. and 4.d. of this section.

c. Notice

(1) Anticipated bypass. If the permittee knows in advance of the need for a bypass, it shall submit prior notice, if possible at least ten days before the date of the bypass.

(2) Unanticipated bypass. The permittee shall submit notice of an unanticipated bypass as required in Section D, paragraph 6 (24-hour notice).

d. Prohibition of bypass.

(1) Bypass is prohibited, and the Director may take enforcement action against a permittee for bypass, unless:

(a) Bypass was unavoidable to prevent loss of life, personal injury, or severe property damage;

(b) There were no feasible alternatives to the bypass, such as the use of auxiliary treatment facilities, retention of untreated wastes, or maintenance during normal periods of equipment downtime. This condition is not satisfied if adequate back-up equipment should have been installed in the exercise of reasonable engineering judgment to prevent a bypass which occured during normal periods of equipment downtime or preventive maintenance; and

(c) The permittee submitted notices as required under Section B, paragraph 4.c.

(2) The Director may approve an anticipated bypass, after considering its adverse effects, if the Director determines that it will meet the three conditions listed above in Section B, paragraph 4.d.(1).

5. Upset Conditions

a. Definition. "Upset" means an exceptional incident in which there is unintentional and temporary noncompliance with technology-based permit effluent limitations because of factors beyond the reasonable control of the permittee. An upset does not include noncompliance to the extent caused by operational error, improperly designed treatment facilities, inadequate treatment facilities, lack of preventive maintenance, or careless or improper operation.

b. Effect of an upset. An upset constitutes an affirmative defense to an action brought for noncompliance with such technology-based permit effluent limitations if the requirements of Section B, paragraph 5.c. are met. No determination made during administrative review of claims that noncompliance was caused by upset, and before an action for noncompliance, is final administrative action subject to judicial review.

c. Conditions necessary for a demonstration of upset. A permittee who wishes to establish the affirmative defense of upset shall demonstrate, through properly signed, contemporaneous operating logs, or other relevant evidence that:

(1) An upset occurred and that the permittee can identify the cause(s) of the upset;

(2) The permitted facility was at the time being properly operated; and

(3) The permittee submitted notice of the upset as required in Section D, paragraph 6.

(4) The permittee complied with any remedial measures required under Section B, paragraph 3.

d. Burden of proof. In any enforcement proceeding the permittee seeking to establish the occurrence of an upset has the burden of proof.

6. Removed Substances

Solids, sludges, filter backwash, or other pollutants removed in the course of treatment or control of wastewaters shall be disposed of in a manner such as to prevent any pollutant from such materials from entering navigable waters.

SECTION C. MONITORING AND RECORDS

1. Representative Sampling

Samples and measurements taken as required herein shall be representative of the volume and nature of the monitored dishcarge. All samples shall be taken at the monitoring points specified in this permit and, unless otherwise specified, before the effluent joins or is diluted by any other wastestream, body of water, or substance. Monitoring points shall not be changed without notification to and the approval of the Director.

2. Flow Measurements

Appropriate flow measurement devices and methods consistent with accepted scientific practices shall be selected and used to ensure the accuracy and reliability of measurements of the volume of monitored discharges. The devices shall be installed, calibrated, and maintained to insure that the accuracy of the measurements are consistent with the accepted capability of that type of device. Devices selected shall be capable of measuring flows with a maximum deviation of less than ± 10% from true discharge rates throughout the range of expected discharge volumes. Guidance in selection, installation, calibration, and operation of acceptable flow measurement devices can be obtained from the following references:

a. "A Guide to Methods and Standards for the Measurement of Water Flow", U.S. Department of Commerce, National Bureau of Standards, NBS Special Publication 421, May 1975, 97 pp. (Available from the U.S. Government Printing Office, Washington, D.C. 20402. Order by SD catalog No. C13.10:421).

b. "Water Measurement Manual", U.S. Department of Interior, Bureau of Reclamation, Second Edition, Revised Reprint, 1974, 327 pp. (Available from the U.S. Government Printing Office, Washington, D.C. 20402. Order by Catalog No. I27.19/2:W29/2, Stock No. S/N 24003-0027).

c. "Flow Measurement in Open Channels and Closed Conduits", U.S. Department of Commerce, National Bureau of Standards, NBS Special Publication 484, October 1977, 982 pp. (Available in paper copy or microfiche from National Technical Information Service (NTIS), Springfield, VA 22151. Order by NTIS No. PB-273 535/5ST).

d. "NPDES Compliance Sampling Manual", U.S. Environmental Protection Agency, Office of Water Enforcement, Publication

MCD-51, 1977, 140 pp. (Available from the General Services Administration [8FFS], Centralized Mailing Lists Services, Building 41, Denver Federal Center, Denver, CO 80225).

3. Monitoring Procedures

Monitoring must be conducted according to test procedures approved under 40 CFR Part 136, unless other test procedures have been specified in this permit.

4. Penalties for Tampering

The Clean Water Act provides that any person who falsifies, tampers with, or knowingly renders inaccurate, any monitoring device or method required to be maintained under this permit shall, upon conviction, be punished by a fine of not more than $10,000 per violation, or by imprisonment for not more than 6 months per violation, or by both.

5. Reporting of Monitoring Results

Monitoring results must be reported on a Discharge Monitoring Report (DMR) Form EPA No. 3320-1. Monitoring results obtained during the previous month shall be summarized and reported on a DMR form postmarked no later than the 15th day of the month following the completed reporting period. The first report is due September 15, 1985 Duplicate copies of DMR's signed and certified as required by Section D, paragraph 11, and all other reports required by Section D. Reporting Requirements, shall be submitted to the Director and to the State (if listed) at the following address(es):

Director
Water Management Division (6W)
U.S. Environmental Protection Agency
Region VI
InterFirst Two Building
1201 Elm Street
Dallas, Texas 75270

Ann Young, Acting Program Manager
Surface Water Section
Surface Water Quality Bureau
New Mexico Environmental
Improvement Division
P.O. Box 968
Santa Fe, New Mexico 87504-0968

6. Additional Monitoring by the Permittee

If the permittee monitors any pollutant more frequently than required by this permit, using test procedures approved under 40 CFR Part 136 or as specified in this permit, the results of this monitoring shall be included in the calculation and reporting of the data submitted in the DMR. Such increased monitoring frequency shall also be indicated on the DMR.

7. Averaging of Measurements

Calculations for all limitations which require averaging of measurements shall utilize an arithmetic mean unless otherwise specified by the Director in the permit.

8. Retention of Records

The permittee shall retain records of all monitoring information, including all calibration and maintenance records and all original strip chart recordings for continuous monitoring instrumentation, copies of all reports required by this permit, and records of all data used to complete the application for this permit, for a period of at least 3 years from the date of the sample, measurement, report, or application. This period may be extended by request of the Director at any time.

9. Record Contents

Records of monitoring information shall include:

a. The date, exact place, and time of sampling or measurements;

b. The individual(s) who performed the sampling or measurements;

c. The date(s) analyses were performed;

d. The individual(s) who performed the analyses;

e. The analytical techniques or methods used; and

f. The results of such analyses.

10. Inspection and Entry

The permittee shall allow the Director, or an authorized representative, upon the presentation of credentials and other documents as may be required by law, to:

a. Enter upon the permittee's premises where a regulated facility or activity is located or conducted, or where records must be kept under the conditions of this permit;

b. Have access to and copy, at reasonable times, any records that must be kept under the conditions of this permit;

c. Inspect at reasonable times any facilities, equipment (including monitoring and control equipment), practices, or operations regulated or required under this permit; and

d. Sample or monitor at reasonable times, for the purposes of assuring permit compliance or as otherwise authorized by the Clean Water Act, any substances or parameters at any location.

SECTION D. REPORTING REQUIREMENTS

1. Planned Changes

The permittee shall give notice to the Director as soon as possible of any planned physical alterations or additions to the permitted facility. Notice is required only when:

a. The alteration or addition to a permitted facility may meet one of the criteria for determining whether a facility is a new source in 40 CFR Part 122.29(b) [48 FR 14153, April 1, 1983, as amended at 49 FR 38046, September 26, 1984]; or

b. The alteration or addition could significantly change the nature or increase the quantity of pollutants discharged. This notification applies to pollutants which are subject neither to effluent limitations in the permit, nor to notification requirements under 40 CFR Part 122.42(a)(1) [48 FR 14153, April 1,1983, as amended at 49 FR 38046, September 26, 1984].

2. Anticipated Noncompliance

The permittee shall give advance notice to the Director of any planned changes in the permitted facility or activity which may result in noncompliance with permit requirements.

3. Transfers

This permit is not transferable to any person except after notice to the Director. The Director may require modification or revocation and reissuance of the permit to change the name of the permittee and incorporate such other requirements as may be necessary under the Clean Water Act.

4. Monitoring Reports

Monitoring results shall be reported at the intervals and in the form specified in Section C, paragraph 5 (Monitoring).

5. Compliance Schedules

Reports of compliance or noncompliance with, or any progress reports on, interim and final requirements contained in any compliance schedule of this permit shall be submitted no later than 14 days following each schedule date. Any reports of noncompliance shall include the cause of noncompliance, any remedial actions taken, and the probability of meeting the next scheduled requirement.

6. Twenty Four Hour Reporting

The permittee shall report any noncompliance which may endanger health or the environment. Any information shall be provided orally within 24 hours from the time the permittee becomes aware of the circumstances. A written submission shall also be provided within 5 days of the time the permittee becomes aware of the circumstances. The written submission shall contain a description of the noncompliance and its cause; the period of noncompliance, including exact dates and times, and if the noncompliance has not been corrected, the anticipated time it is expected to continue; and steps taken or planned to reduce, eliminate, and prevent reoccurrence of the noncompliance. The Director may waive the written report on a case-by-case basis if the oral report has been received within 24 hours.

The following shall be included as information which must be reported within 24 hours:

a. Any unanticipated bypass which exceeds any effluent limitation in the permit.

b. Any upset which exceeds any effluent limitation in the permit.

c. Violation of a maximum daily discharge limitation for any of the pollutants listed by the Director in Part III of the permit to be reported within 24 hours.

7. Other Noncompliance

The permittee shall report all instances of noncompliance not reported under Section D, paragraphs 4, 5, and 6, at the time monitoring reports are submitted. The reports shall contain the information listed in Section D, paragraph 6.

8. Changes in Discharges of Toxic Substances

The permittee shall notify the Director as soon as it knows or has reason to believe:

a. That any activity has occured or will occur which would result in the discharge, in a routine or frequent basis, of any toxic pollutant which is not limited in the permit, if that discharge will exceed the highest of the "notification levels" described in 40 CFR Part 122.42(a)(1) [48 FR 14153, April 1, 1983, as amended at 49 FR 38046, September 26, 1984].

b. That any activity has occured or will occur which would result in any discharge, on a non-routine or infrequent basis, of a toxic pollutant which is not limited in the permit, if

that discharge will exceed the highest of the "notification levels" described in 40 CFR Part 122.42(a)(2) [48 FR 14153, April 1,1983, as amended at 49 FR 38046, September 26, 1984).

9. Duty to Provide Information

The permittee shall furnish to the Director, within a reasonable time, any information which the Director may request to determine whether cause exists for modifying, revoking and reissuing, or terminating this permit, or to determine compliance with this permit. The permittee shall also furnish to the Director, upon request, copies of records required to be kept by this permit.

10. Duty to Reapply

If the permittee wishes to continue an activity regulated by this permit after the expiration date of this permit, the permittee must apply for and obtain a new permit. The application shall be submitted at least 180 days before the expiration date of this permit. The Director may grant permission to submit an application less than 180 days in advance but no later than the permit expiration date.

11. Signatory Requirements

All applications, reports, or information submitted to the Director shall be signed and certified.

a. All permit applications shall be signed as follows:

(1) For a corporation: by a responsible corporate officer. For the purpose of this section, a responsible corporate officer means:

(i) A president, secretary, treasurer, or vice-president of the corporation in charge of a principal business function, or any other person who performs similar policy or decision making functions for the corporation, or

(ii) the manager of one or more manufacturing, production, or operating facilities employing more than 250 persons or having gross annual sales or expenditures exceeding $25 million (in second-quarter 1980 dollars), if authority to sign documents has been assigned or delegated to the manager in accordance with corporate procedures.

(2) For a partnership or sole proprietorship: by a general partner or the proprietor, respectively.

(3) For a municipality, State, Federal, or other public agency: by either a principal executive officer or ranking elected official. For purposes of this section, a principal executive officer of a Federal agency includes:

(i) The chief executive officer of the agency, or

(ii) a senior executive officer having responsibility for the overall operations of a principal geographic unit of the agency.

b. All reports required by the permit and other information requested by the Director shall be signed by a person described above or by a duly authorized representative of that person. A person is a duly authorized representative only if:

(1) The authorization is made in writing by a person described above.

(2) The authorization specifies either an individual or a position having responsibility for the overall operation of the regulated facility or activity, such as the position of plant manager, operator of a well or a well field, superintendent, or position of equivalent responsibility, or an individual or position having overall responsibility for environmental matters for the company. A duly authorized representative may thus be either a named individual or any individual occupying a named position; and

(3) The written authorization is submitted to the Director.

c. Certification. Any person signing a document under this section shall make the following certification:

"I certify under penalty of law that this document and all attachments were prepared under the direction or supervision in accordance with a system designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who manage the system, or those persons directly responsible for gathering the information, the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations."

12. Availability of Reports

Except for data determined to be confidential under 40 CFR Part 2, all reports prepared in accordance with the terms of this permit shall be available for public inspection at the office of the Director. As required by the Clean Water Act, the name and address of any permit applicant or permittee, permit applications, permits, and effluent data shall not be considered confidential.

13. Penalties for Falsification of Reports

The Clean Water Act provides that any person who knowingly makes any false statement, representation, or certification in any record or other document submitted or required to be maintained under this permit, including monitoring reports or reports of compliance or noncompliance shall, upon conviction, be punished by a fine of not more than $10,000 per violation, or by imprisonment for not more than 6 months per violation, or by both.

PART III
OTHER CONDITIONS

A. The "daily average" concentration means the arithmetic average (weighted by flow value) of all the daily determinations of concentration made during a calendar month. Daily determinations of concentration made using a composite sample shall be the concentration of the composite sample. When grab samples are used, the daily determination of concentration shall be the arithmetic average (weighted by flow value) of all the samples collected during that calendar day.

The "daily maximum" concentration means the daily determination of concentration for any calendar day.

B. The term " composite sample" means a sample consisting of a minimum of two grab samples of effluent collected not less than four hours apart over a normal eight hour operating day and combined proportional to flow or a sample continously collected proportional to flow over a normal eight hour operating day. All such samples shall be typical and representative of effluent generated during the period since the last sample was collected.

C. Analysis and reporting for Outfalls 001 and 002 shall be accomplished separately for each outfall. In addition, the permittee shall report the flow weighted average results for Outfalls 001 plus 002. Effluent limitations on weight of total molybdenum shall be the sum total for Outfalls 001 and 002. For determination of compliance with other effluent limitations the following shall apply:

1. Discharge from Outfall 001 shall be in compliance.

2. A composite sample of discharges from Outfalls 001 and 002 monitoring samples combined in flow weighted proportion shall be in compliance, except that concentration limits for molybdenum and manganese are suspended during periods of zero flow from Outfall 001.

   During periods when no flow occurs from Outfall 001, monthly average reporting of concentration for molybdenum and manganese shall be based on the average of all composite samples obtained in the month for Outfalls 001 and 002; and monthly maximum reporting of concentration for molybdenum and manganese shall be based on the maximum composite sample obtained in that month for Outfalls 001 and 002. In conjuction with the DMR for each month, the permittee shall report periods, if any, when no flow exists at Outfall 001.

D. The permittee shall determine if eighty (80) percent or greater of the culture of test organisms will survive by use of the "Range-Finding Screening Test," set out in "Methods for Measuring the Acute Toxicity of Effluents to

Aquatic Organisms, "EPA-600/4-78-012 (Rev. July, 1978). Organisms for this test shall be Daphnia sp. if the effluent is less than five (5) parts per thousand salinity or Mysidopsis sp. if the effluent is equal to or greater than five (5) parts per thousand salinity. This screening test will be conducted within sixty (60) days of effectiveness of the biomonitoring requirements. Tests will be conducted once each quarter for a duration of two years utilizing a static method for 24 hours and following this dilution scheme only:

Effluent sample* - 100 percent by volume
Dilution water - 0 percent by volume

*24-hour composite; refrigerated after collection

If at any time during the two year testing period a test shows a survival of eighty (80) percent or less of the test organisms, the permittee shall within twenty-four (24) hours conduct a replacement static 48-hour median lethal concentration (LC50) test on the orginially collected sample. Replacement of effluent samples shall be once per 24 hours. Organisms for this test shall be Daphnia sp. if the effluent is less than five (5) parts per thousand salinity and reconstituted fresh water (EPA-600/4-78-012 Section 4) shall be used for dilution. If the effluent is equal to or greater than five (5) parts per thousands salinity, Mysidopsis sp. shall be used as the test organism, and reconstituted seawater will be used as dilution water (EPA-600/4-78-012 Section 4). The remaining LC50 methodology is available in EPA-600/4-78-012.

All screening and LC50 test results shall be reported with the Discharge Monitoring Reports. The test results should include the chemical and physical data as specified in Section 7 of EPA-600/4-78-012.

E. Sample taken in compliance with this section shall consist of 24-hour composite samples taken at the discharge spillway from Pope Lake.

F. The Molycorp thiocyanate colorimetric method is approved for the analysis of molybdenum unless subsequently determined to be inappropriate by the NMEID or EPA.

G. As soon as practicable after the arrival of Molycorp's environmental staff at the site of a tailings spill that reaches the Red River, but no later than two (2) hours after arrival at the site, water quality sampling shall commence. Samples shall be taken at three sites:

(1) Approximately 100 feet above the point where tailings enter the river;
(2) Approximately 100 feet below the point where tailings enter the river; and
(3) Approximately one-half mile below the point where tailings enter the river.

All samples shall be properly preserved and analyzed for:

Chemical Oxygen Demand (COD)
Total Suspended Solids (TSS)
Cyanide (CN)
Total Iron (Fe)
Manganese, Dissolved (Mn)
Total Molybdenum (Mo)
pH
Temperature
Conductivity
Total Zinc (Zn)

The results of the analyses shall be submitted to the United States Environmental Protection Agency and the New Mexico Environmental Improvement Division within 30 days following a tailings spill.

Consistent with the procedures described in the Preventative Maintenance and Surveillance Plan and Contingency Action and Reporting Plans (June 1975), a written report containing the following information will be sent to the US EPA and NMEID within ten (10) days following any spill:

(1) Date of spill.

(2) Time when the spill was observed and time when tailings flow into the river was stopped.

(3) Location (pipe or coupling number).

(4) Estimated amount of tailings that entered the river.

(5) Sketch and dimension of size of hole or failure that cuased the spill.

(6) Position of failure in the pipe or coupling.

(7) Copy of the latest computer printout covering the pipe or coupling which failed.

(8) Comments, if required for clarification.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

FEB 29 1984

Recommendations on evidentiary hearing request for Molycorp, NPDES No. NM0022306, Docket No. EH-6-83-005.

Fred Humke, 6W-P

Pat Hudson, 6ORC

1. Issue No. 1
Molybdenum concentration limitation for Outfall 002 (Part 1.A.) when Outfall 001 is not discharging, or is discharging intermittently.

Recommendtion: Deny this issue.

Rationale: This is a condition of the prior permit which was carried into the reissued perit. Under anti-backsliding provisions of Part 122.44 (1), Molycorp is required to continue meeting these same conditions. However, EPA will consider a subsequent permit modification request from Molycorp if proper and sufficient justification is provided in accordance with part 122.44(1).

2. Issue No. 2
Arsenic limitations for Outfalls 001 and 002.

Recommendation: Grant this issue for the limitations only.

Rationale: Arsenic is a toxic which was identified at significant levels by Molycorp in the NPDES application. Molycorp now feels that residual levels of arsenic will not be significant and will be far below the limited levels. The burden of monitoring is not justification for ignoring a toxic pollutant which has been identified in the application. EPA has authority to impose monitoring requirements under Section 308 of the CWA, independent of any challenge to the specific limitations. EPA grants this issue for Molycorp to challange the severity of specific limitations for total arsenic of 0.5 mg/l daily average and 1.0 mg/l daily maximum which EPA has applied under the provisions of Part 122.44(b) and (e). Therefore, arsenic limitations are stayed but daily average and daily maximum monitoring and reporting for arsenic concentration levels is retained.

3. Issue No. 3
Composite sampling definition, Part III.B.

Recommendation: Deny this issue.

Rationale: Molycorp objects to conducting composite sampling in accordance with current Regional policy because of the added burden of monitoring. EPA has the authority to impose appropriate monitoring requirements under Section 308 of the Clean Water Act. However, EPA will consider a subsequent permit modification request from Molycorp if proper and sufficient justification is provided.

02/29/84:HUMKEic:M11:060HUM01/02

CONCURRENCES

SYMBOL								
SURNAME								
DATE								

EPA Form 1320-1 (12-70)

OFFICIAL FILE COPY

GPO : 1983 O - 403-201

4. Issue No. 4
Biomonitoring - Parts I.A. and III.D., E. of this permit.

Recommendation: Deny this issue.

Rationale: EPA (Region VI) has established a specific biomonitoring program for BAT toxic effluents which has been applied uniformly across the Region. Molycorp requests an alternative program which Molycorp considers more appropriate to the receiving stream (Red River). General Counsel supports the authority of EPA to impose the biomonitoring program under the provision of Section 101 and 308 of the Clean Water Act.

5. Issue No. 5
Temperature monitoring - Part I.A.

Recommendation: Deny this issue.

Rationale: Temperature monitoring (without limitations) was applied in this permit as a condition of certification by the NMEID. Therefore, this issue must be resolved with the state and is not a suitable issue for an evidentiary hearing. In any event, under Section 308 of the Clean Water Act, EPA has the authority to impose all monitoring requirements which are deemed appropriate.

cc: Becker, 6W-PI
Hannesschlager

PROPOSED PERMIT

Permit No. NM0022306

AUTHORIZATION TO DISCHARGE UNDER THE
NATIONAL POLLUTANT DISCHARGE ELIMINATION SYSTEM

In compliance with the provisions of the Federal Water Pollution Control Act, as amended, (33 U.S.C... 1251 et. seq; the "Act"),

Molycorp, Inc.
P.O. Box 469
Questa, New Mexico 87556

is authorized to discharge from a facility located at Questa, Taos County, New Mexico

to receiving waters named Red River, Segment 2-119 of the Upper Rio Grande Basin

in accordance with effluent limitations, monitoring requirements and other conditions set forth in Parts I (6 pages), II (14 pages), and III (9 pages) hereof.

This permit shall become effective on

This permit and the authorization to discharge shall expire at midnight,

Signed this day of

Myron O. Knudson

Myron O. Knudson, P.E.
Director
Water Management Division (6W)

PART I
REQUIREMENTS FOR NPDES PERMITS

SECTION A. EFFLUENT LIMITATIONS AND MONITORING REQUIREMENTS

OUTFALL 002

During the period beginning the effective date and lasting until mill start-up, the permittee is authorized to discharge from Outfall 002 - seepage from tailing impoundment.

Such discharges shall be limited and monitored by the permittee as specified below:

Effluent Characteristic	Discharge Limitations			
	Mass(lbs/day)		Other Units (Specify)	
	Daily Avg	Daily Max	Daily Avg	Daily Max
Flow (MGD)	N/A	N/A	(*1)	(*1)
Chemical Oxygen Demand	N/A	N/A	60 mg/l	90 mg/l
Total Suspended Solids	N/A	N/A	20 mg/l	30 mg/l
Total Arsenic	N/A	N/A	0.5 mg/l	1.0 mg/l
Total Cadmium	N/A	N/A	N/A	0.05 mg/l
Total Copper	N/A	N/A	0.15 mg/l	0.30 mg/l
Total Cyanide	N/A	N/A	0.025 mg/l	0.05 mg/l
Fluoride	N/A	N/A	N/A	3.0 mg/l
Total Iron	N/A	N/A	N/A	0.6 mg/l
Total Lead	N/A	N/A	0.3 mg/l	0.6 mg/l
Total Manganese	N/A	N/A	1.0 mg/l	1.5 mg/l
Total Mercury	N/A	N/A	0.001 mg/l	0.002 mg/l
Total Molybdenum	N/A	N/A	1.0 mg/l	2.0 mg/l
Total Zinc	N/A	N/A	N/A	0.2 mg/l

Effluent Characteristic	Monitoring Requirements	
	Measurement Frequency	Sample Type
Flow (MGD)	(*2)	Record
Chemical Oxygen Demand	1/Month	Composite
Total Suspended Solids	1/Month	Composite
Total Arsenic	1/Month	Composite
Total Cadmium	1/Month	Composite
Total Copper	1/Month	Composite
Total Cyanide	1/Month	Composite
Fluoride	1/Month	Composite
Total Iron	1/Month	Composite
Total Lead	1/Month	Composite
Total Manganese	1/Month	Composite
Total Mercury	1/Month	Composite
Total Molybdenum	1/Month	Composite
Total Zinc	1/Month	Composite

OUTFALL 002

The pH shall not be less than 6.0 standard units nor greater than 9.0 standard units and shall be monitored 1/month by grab sample.

There shall be no discharge of floating solids or visible foam in other than trace amounts.

Samples taken in compliance with the monitoring requirements specified above shall be taken at the following location(s):

Outfall 002 Latitude 36° 40' 61" and Longitude 105° 37' 30" which is the collected and combined seepage from the tailings impoundment.

FOOTNOTES

(*1) Report.
(*2) daily estimate.

PART I
REQUIREMENTS FOR NPDES PERMITS

SECTION A. EFFLUENT LIMITATIONS AND MONITORING REQUIREMENTS

OUTFALLS 001 & 002

During the period beginning mill start-up and lasting through the expiration date, the permittee is authorized to discharge from Outfalls 001 - process water from milling operation and tailings disposal; 002 - seepage from tailing impoundment.

Such discharges shall be limited and monitored by the permittee as specified below:

Effluent Characteristic	Discharge Limitations			
	Mass(lbs/day)		Other Units (Specify)	
	Daily Avg	Daily Max	Daily Avg	Daily Max
Flow (MGD)	N/A	N/A	(*1)	(*1)
Chemical Oxygen Demand	N/A	N/A	60 mg/l	90 mg/l
Total Suspended Solids	N/A	N/A	20 mg/l	30 mg/l
Total Arsenic	N/A	N/A	0.5 mg/l	1.0 mg/l
Total Cadmium	N/A	N/A	N/A	0.05 mg/l
Total Copper	N/A	N/A	0.15 mg/l	0.30 mg/l
Total Cyanide	N/A	N/A	0.025 mg/l	0.05 mg/l
Fluoride	N/A	N/A	N/A	3.0 mg/l
Total Iron	N/A	N/A	N/A	0.6 mg/l
Total Lead	N/A	N/A	0.3 mg/l	0.6 mg/l
Total Manganese	N/A	N/A	1.0 mg/l	1.5 mg/l
Total Mercury	N/A	N/A	0.001 mg/l	0.002 mg/l
Total Molybdenum	25 (*4)	50 (*3)	1.0 mg/l	2.0 mg/l
Total Zinc	N/A	N/A	N/A	0.2 mg/l
Biomonitoring	N/A	N/A	N/A	N/A

Effluent Characteristic	Monitoring Requirements	
	Measurement Frequency	Sample Type
Flow (MGD)	(*5)	Record
Chemical Oxygen Demand	1/Week	Composite (*2)
Total Suspended Solids	1/Week	Composite (*2)
Total Arsenic	1/Week	Composite (*2)
Total Cadmium	1/Week	Composite (*2)
Total Copper	1/Week	Composite (*2)
Total Cyanide	(*6)	Composite (*2)
Fluoride	1/Week	Composite (*2)
Total Iron	1/Week	Composite (*2)
Total Lead	1/Week	Composite (*2)
Total Manganese	1/Week	Composite (*2)
Total Mercury	1/Week	Composite (*2)
Total Molybdenum	1/Week	Composite (*2)
Total Zinc	1/Week	Composite (*2)
Biomonitoring	1/Month	(*7)

OUTFALLS 001 & 002

The pH shall not be less than 6.0 standard units nor greater than 9.0 standard units and shall be monitored 1/week by grab sample.

There shall be no discharge of floating solids or visible foam in other than trace amounts.

Samples taken in compliance with the monitoring requirements specified above shall be taken at the following location(s):

Outfall 001 Latitude 36° 40' 61" and Longitude 105° 38' 30" which is the discharge spillway from Pope Lake.

Outfall 002 Latitude 36° 40' 61" and Longitude 105° 37' 30" which is the collected and combined seepage from the tailings impoundment.

FOOTNOTES

(*1) Report.
(*2) See Part III, Paragraph B.
(*3) Sum total for Outfalls 001 & 002.
(*4) Sum total average for Outfalls 001 & 002 for six months preceeding reporting period end date; also report daily average mass for each month.
(*5) Continuous and totalized monitoring for Outfall 001; daily estimate for Outfall 002.
(*6) 2/week for Outfall 001; 1/week for Outfall 002.
(*7) See Part III, Paragraph C.

SECTION B. SCHEDULE OF COMPLIANCE

The permittee shall achieve compliance with the effluent limitations specified for discharges in accordance with the following schedule:

Report mill start-up.

PART II
STANDARD CONDITIONS FOR NPDES PERMITS

SECTION A. GENERAL CONDITIONS

1. Duty to Comply

The permittee must comply with all conditions of this permit. Any permit noncompliance constitutes a violation of the Clean Water Act and is grounds for enforcement action; for permit termination, revocation and reissuance, or modification; or for denial of a permit renewal application.

2. Penalties for Violations of Permit Conditions

The Clean Water Act provides that any person who violates a permit condition implementing Sections 301, 302, 306, 307, 308, 318, or 405 of the Clean Water Act is subject to a civil penalty not to exceed $10,000 per day of such violation. Any person who willfully or negligently violates permit conditions implementing Sections 301, 302, 306, 307, or 308 of the Clean Water Act is subject to a fine of not less than $2,500 nor more than $25,000 per day of violation, or by imprisonment for not more than 1 year, or both.

3. Permit Actions

This permit may be modified, revoked and reissued, or terminated for cause including, but not limited to, the following:

a. Violation of any terms or conditions of this permit;

b. Obtaining this permit by misrepresentation or failure to disclose fully all relevant facts;

c. A change in any condition that requires either a temporary or a permanent reduction or elimination of the authorized discharge; or,

d. A determination that the permitted activity endangers human health or the environment and can only be regulated to acceptable levels by permit modification or termination.

The filing of a request by the permittee for a permit modification, revocation and reissuance, or termination, or a notification of planned changes or anticipated noncompliance, does not stay any permit condition.

4. Toxic Pollutants

Notwithstanding Part II.A.3, if any toxic effluent standard or prohibition (including any schedule of compliance specified in such effluent standard or prohibition) is promulgated under Section 307(a) of the Clean Water Act for a toxic pollutant which is present in the discharge and that standard or prohibition is more stringent than any limitation on the pollutant in this permit, this permit shall be modified or revoked and reissued to conform to the toxic effluent standard or prohibition and the permittee so notified.

The permittee shall comply with effluent standards or prohibitions established under Section 307(a) of the Clean Water Act for toxic pollutants within the time provided in the regulations that established those standards or prohibitions, even if the permit has not yet been modified to incorporate the requirement.

5. Civil and Criminal Liability

Except as provided in permit conditions on "Bypassing" (Part II.B.4.b) and "Upsets" (Part II.B.5.b), nothing in this permit shall be construed to relieve the permittee from civil or criminal penalties for noncompliance.

6. Oil and Hazardous Substance Liability

Nothing in this permit shall be construed to preclude the institution of any legal action or relieve the permittee from any responsibilities, liabilities, or penalties to which the permittee is or may be subject under Section 311 of the Clean Water Act.

7. State Laws

Nothing in this permit shall be construed to preclude the institution of any legal action or relieve the permittee from any responsibilities, liabilities, or penalties established pursuant to any applicable State law or regulation under authority preserved by Section 510 of the Clean Water Act.

8. Property Rights

The issuance of this permit does not convey any property rights of any sort, or any exclusive privileges, nor does it authorize any injury to private property or any invasion of personal rights, nor any infringement of Federal, State, or local laws or regulations.

9. Severability

The provisions of this permit are severable, and if any provision of this permit or the application of any provision of this permit to any circumstance is held invalid, the application of such provision to other circumstances, and the remainder of this permit, shall not be affected thereby.

10. Definitions

The following definitions shall apply unless otherwise specified in this permit:

a. "Daily Discharge" means the discharge of a pollutant measured during a calendar day or any 24-hour period that reasonably represents the calendar day for purposes of sampling. For pollutants with limitations expressed in terms of mass, the "daily discharge" is calculated as the total mass of the pollutant discharged over the sampling day. For pollutants with limitations expressed in other units of measurement, the "daily discharge" is calculated as the average measurement of the pollutant over the sampling day. "Daily discharge" determination of concentration made using a composite sample shall be the concentration of the composite sample. When grab samples are used, the "daily discharge" determination of concentration shall be the arithmetic average (weighted by flow value) of all samples collected during that sampling day.

b. "Daily Average" (also known as monthly average) discharge limitation means the highest allowable average of "daily discharges" over a calendar month, calculated as the sum of all "daily discharges" measured during a calendar month divided by the number of "daily discharges" measured during that month. When the permit establishes daily average concentration effluent limitations or conditions, the daily average concentration means the arithmetic average (weighted by flow) of all "daily discharges" of concentration determined during the calendar month.

c. "Daily Maximum" discharge limitation means the highest allowable "daily discharge" during the calendar month.

d. The term "MGD" shall mean million gallons per day.

e. The term "mg/l" shall mean milligrams per liter or parts per million (ppm).

f. The term "ug/l" shall mean micrograms per liter or parts per billion (ppb).

SECTION B. OPERATION AND MAINTENANCE OF POLLUTION CONTROLS

1. Proper Operation and Maintenance

The permittee shall at all times properly operate and maintain all facilities and systems of treatment and control (and related appurtenances) which are installed or used by the permittee to achieve compliance with the conditions of this permit. Proper operation and maintenance also includes adequate laboratory controls and appropriate quality assurance procedures. This provision requires the operation of backup or auxiliary facilities or similar systems which are installed by a permittee only when the operation is necessary to achieve compliance with the conditions of the permit.

2. Need to Halt or Reduce not a Defense

It shall not be a defense for a permittee in an enforcement action that it would have been necessary to halt or reduce the permitted activity in order to maintain compliance with the conditions of this permit.

3. Duty to Mitigate

The permittee shall take all reasonable steps to minimize or prevent any discharge in violation of this permit which has a reasonable likelihood of adversely affecting human health or the environment.

4. Bypass of Treatment Facilities

a. Definitions

(1) "Bypass" means the intentional diversion of waste streams from any portion of a treatment facility.

(2) "Severe property damage" means substantial physical damage to property, damage to the treatment facilities which causes them to become inoperable, or substantial and permanent loss of natural resources which can reasonably be expected to occur in the absence of a bypass. Severe property damage does not mean economic loss caused by delays in production.

b. Bypass not exceeding limitations. The permittee may allow any bypass to occur which does not cause effluent limitations to be exceeded, but only if it also is for essential maintenance to assure efficient operation. These bypasses are not subject to the provisions of Part II.B.4.c and 4.d.

c. Notice

(1) Anticipated bypass. If the permittee knows in advance of the need for a bypass, it shall submit prior notice, if possible at least ten days before the date of the bypass.

(2) Unanticipated bypass. The permittee shall submit notice of an unanticipated bypass as required in Part II.D.6 (24-hour notice).

d. Prohibition of bypass

(1) Bypass is prohibited, and the Director may take enforcement action against a permittee for bypass, unless:

(a) Bypass was unavoidable to prevent loss of life, personal injury, or severe property damage;

(b) There were no feasible alternatives to the bypass, such as the use of auxiliary treatment facilities, retention of untreated wastes, or maintenance during normal periods of equipment downtime. This condition is not satisfied if adequate back-up equipment should have been installed in the exercise of reasonable engineering judgment to prevent a bypass which occured during normal periods of equipment downtime or preventive maintenance; and,

(c) The permittee submitted notices as required by Part II.B.4.c.

(2) The Director may approve an anticipated bypass, after considering its adverse effects, if the Director determines that it will meet the three conditions listed at Part II.B.4.d.(1).

5. <u>Upset Conditions</u>

a. Definition. "Upset" means an exceptional incident in which there is unintentional and temporary noncompliance with technology-based permit effluent limitations because of factors beyond the reasonable control of the permittee. An upset does not include noncompliance to the extent caused by operational error, improperly designed treatment facilities, inadequate treatment facilities, lack of preventive maintenance, or careless or improper operation.

b. Effect of an upset. An upset constitutes an affirmative defense to an action brought for noncompliance with such technology-based permit effluent limitations if the requirements of Part II.B.5.c are met. No determination made during administrative review of claims that noncompliance was caused by upset, and before an action for noncompliance, is final administrative action subject to judicial review.

c. Conditions necessary for a demonstration of upset. A permittee who wishes to establish the affirmative defense of upset shall demonstrate, through properly signed, contemporaneous operating logs, or other relevant evidence that:

(1) An upset occurred and that the permittee can identify the cause(s) of the upset;

(2) The permitted facility was at the time being properly operated;

(3) The permittee submitted notice of the upset as required by Part II.D.6; and,

(4) The permittee complied with any remedial measures required by Part II.B.3.

d. Burden of proof. In any enforcement proceeding the permittee seeking to establish the occurrence of an upset has the burden of proof.

6. Removed Substances

Solids, sludges, filter backwash, or other pollutants removed in the course of treatment or control of wastewaters shall be disposed of in a manner such as to prevent any pollutant from such materials from entering navigable waters.

SECTION C. MONITORING AND RECORDS

1. Representative Sampling

Samples and measurements taken as required herein shall be representative of the volume and nature of the monitored discharge. All samples shall be taken at the monitoring points specified in this permit and, unless otherwise specified, before the effluent joins or is diluted by any other wastestream, body of water, or substance. Monitoring points shall not be changed without notification to and the approval of the Director.

2. Flow Measurements

Appropriate flow measurement devices and methods consistent with accepted scientific practices shall be selected and used to ensure the accuracy and reliability of measurements of the volume of monitored discharges. The devices shall be installed, calibrated, and maintained to insure that the accuracy of the measurements are consistent with the accepted capability of that type of device. Devices selected shall be capable of measuring flows with a maximum deviation of less than ± 10% from true discharge rates throughout the range of expected discharge volumes. Guidance in selection, installation, calibration, and operation of acceptable flow measurement devices can be obtained from the following references:

a. "A Guide to Methods and Standards for the Measurement of Water Flow", U.S. Department of Commerce, National Bureau of Standards, NBS Special Publication 421, May 1975, 97 pp. (Available from the U.S. Government Printing Office, Washington, D.C. 20402. Order by SD catalog No. C13.10:421).

b. "Water Measurement Manual", U.S. Department of Interior, Bureau of Reclamation, Second Edition, Revised Reprint, 1974, 327 pp. (Available from the U.S. Government Printing Office, Washington, D.C. 20402. Order by Catalog No. I27.19/2:W29/2, Stock No. S/N 24003-0027).

c. "Flow Measurement in Open Channels and Closed Conduits", U.S. Department of Commerce, National Bureau of Standards, NBS Special Publication 484, October 1977, 982 pp. (Available in paper copy or microfiche from National Technical Information Service (NTIS), Springfield, VA 22151. Order by NTIS No. PB-273 535/5ST).

d. "NPDES Compliance Sampling Manual", U.S. Environmental Protection Agency, Office of Water Enforcement, Publication MCD-51, 1977, 140 pp.

(Available from the General Services Administration [8FFS], Centralized Mailing Lists Services, Building 41, Denver Federal Center, Denver, CO 80225).

3. Monitoring Procedures

Monitoring must be conducted according to test procedures approved under 40 CFR Part 136, unless other test procedures have been specified in this permit.

4. Penalties for Tampering

The Clean Water Act provides that any person who falsifies, tampers with, or knowingly renders inaccurate, any monitoring device or method required to be maintained under this permit shall, upon conviction, be punished by a fine of not more than $10,000 per violation, or by imprisonment for not more than 6 months per violation, or by both.

5. Reporting of Monitoring Results

Monitoring results must be reported on a Discharge Monitoring Report (DMR) Form EPA No. 3320-1. Monitoring results obtained during the previous month shall be summarized and reported on a DMR form post-marked no later than the _____ day of the month following the completed reporting period. The first report is due on _____. Duplicate copies of DMR's signed and certified as required by Part II.D.11 and all other reports required by Part II.D (Reporting Requirements) shall be submitted to the Director and to the State (if listed) at the following address(es):

Director
Water Management Division (6W)
U.S. Environmental Protection Agency
Region VI
Allied Bank Tower
1445 Ross Avenue
Dallas, Texas 75202-2733

Program Manager
Surface Water Section
Surface Water Quality Bureau
New Mexico Environmental
Improvement Division
P.O. Box 968
Santa Fe, New Mexico 87504-0968

6. Additional Monitoring by the Permittee

If the permittee monitors any pollutant more frequently than required by this permit, using test procedures approved under 40 CFR Part 136 or as specified in this permit, the results of this monitoring shall be included in the calculation and reporting of the data submitted in the DMR. Such increased monitoring frequency shall also be indicated on the DMR.

7. Averaging of Measurements

Calculations for all limitations which require averaging of measurements shall utilize an arithmetic mean unless otherwise specified by the Director in the permit.

8. Retention of Records

The permittee shall retain records of all monitoring information, including all calibration and maintenance records and all original strip chart recordings for continuous monitoring instrumentation, copies of all reports required by this permit, and records of all data used to complete the application for this permit, for a period of at least 3 years from the date of the sample, measurement, report, or application. This period may be extended by request of the Director at any time.

9. Record Contents

Records of monitoring information shall include:

a. The date, exact place, and time of sampling or measurements;

b. The individual(s) who performed the sampling or measurements;

c. The date(s) analyses were performed;

d. The individual(s) who performed the analyses;

e. The analytical techniques or methods used; and,

f. The results of such analyses.

10. Inspection and Entry

The permittee shall allow the Director, or an authorized representative, upon the presentation of credentials and other documents as may be required by law, to:

a. Enter upon the permittee's premises where a regulated facility or activity is located or conducted, or where records must be kept under the conditions of this permit;

b. Have access to and copy, at reasonable times, any records that must be kept under the conditions of this permit;

c. Inspect at reasonable times any facilities, equipment (including monitoring and control equipment), practices, or operations regulated or required under this permit; and,

d. Sample or monitor at reasonable times, for the purposes of assuring permit compliance or as otherwise authorized by the Clean Water Act, any substances or parameters at any location.

SECTION D. REPORTING REQUIREMENTS

1. Planned Changes

The permittee shall give notice to the Director as soon as possible of any planned physical alterations or additions to the permitted facility. Notice is required only when:

a. The alteration or addition to a permitted facility may meet one of the criteria for determining whether a facility is a new source in 40 CFR Part 122.29(b) [48 FR 14153, April 1, 1983, as amended at 49 FR 38046, September 26, 1984]; or,

b. The alteration or addition could significantly change the nature or increase the quantity of pollutants discharged. This notification applies to pollutants which are subject neither to effluent limitations in the permit, nor to notification requirements under 40 CFR Part 122.42(a)(1) [48 FR 14153, April 1, 1983, as amended at 49 FR 38046, September 26, 1984].

2. Anticipated Noncompliance

The permittee shall give advance notice to the Director of any planned changes in the permitted facility or activity which may result in noncompliance with permit requirements.

3. Transfers

This permit is not transferable to any person except after notice to the Director. The Director may require modification or revocation and reissuance of the permit to change the name of the permittee and incorporate such other requirements as may be necessary under the Clean Water Act.

4. Monitoring Reports

Monitoring results shall be reported at the intervals and in the form specified at Part II.C.5 (Monitoring).

5. Compliance Schedules

Reports of compliance or noncompliance with, or any progress reports on, interim and final requirements contained in any compliance schedule of this permit shall be submitted no later than 14 days following each schedule date. Any reports of noncompliance shall include the cause of noncompliance, any remedial actions taken, and the probability of meeting the next scheduled requirement.

6. Twenty-Four Hour Reporting

The permittee shall report any noncompliance which may endanger health or the environment. Any information shall be provided orally within 24 hours from the time the permittee becomes aware of the circumstances. A written submission shall also be provided within 5 days of the time the permittee becomes aware of the circumstances. The written submission shall contain a description of the noncompliance and its cause; the period of noncompliance, including exact dates and times, and if the noncompliance has not been corrected, the anticipated time it is expected to continue; and steps taken or planned to reduce, eliminate, and prevent reoccurrence of the noncompliance. The Director may waive the written report on a case-by-case basis if the oral report has been received within 24 hours.

The following shall be included as information which must be reported within 24 hours:

a. Any unanticipated bypass which exceeds any effluent limitation in the permit;

b. Any upset which exceeds any effluent limitation in the permit; and,

c. Violation of a maximum daily discharge limitation for any of the pollutants listed by the Director in Part III of the permit to be reported within 24 hours.

7. Other Noncompliance

The permittee shall report all instances of noncompliance not reported under Part II.D.4, 5, and 6 at the time monitoring reports are submitted. The reports shall contain the information listed at Part II.D.6.

8. Changes in Discharges of Toxic Substances

The permittee shall notify the Director as soon as it knows or has reason to believe:

a. That any activity has occured or will occur which would result in the discharge, in a routine or frequent basis, of any toxic pollutant which is not limited in the permit, if that discharge will exceed the highest of the "notification levels" described in 40 CFR Part 122.42(a)(1) [48 FR 14153, April 1, 1983, as amended at 49 FR 38046, September 26, 1984].

b. That any activity has occured or will occur which would result in any discharge, on a non-routine or infrequent basis, of a toxic pollutant which is not limited in the permit, if that

discharge will exceed the highest of the "notification levels" described in 40 CFR Part 122.42(a)(2) [48 FR 14153, April 1, 1983, as amended at 49 FR 38046, September 26, 1984).

9. Duty to Provide Information

The permittee shall furnish to the Director, within a reasonable time, any information which the Director may request to determine whether cause exists for modifying, revoking and reissuing, or terminating this permit, or to determine compliance with this permit. The permittee shall also furnish to the Director, upon request, copies of records required to be kept by this permit.

10. Duty to Reapply

If the permittee wishes to continue an activity regulated by this permit after the expiration date of this permit, the permittee must apply for and obtain a new permit. The application shall be submitted at least 180 days before the expiration date of this permit. The Director may grant permission to submit an application less than 180 days in advance but no later than the permit expiration date. Continuation of expiring permits shall be governed by regulations promulgated at 40 CFR Part 122.6 [48 FR 14153, April 1, 1983] and any subsequent amendments.

11. Signatory Requirements

All applications, reports, or information submitted to the Director shall be signed and certified.

a. All permit applications shall be signed as follows:

(1) For a corporation - by a responsible corporate officer. For the purpose of this section, a responsible corporate officer means:

(a) A president, secretary, treasurer, or vice-president of the corporation in charge of a principal business function, or any other person who performs similar policy or decision making functions for the corporation; or,

(b) The manager of one or more manufacturing, production, or operating facilities employing more than 250 persons or having gross annual sales or expenditures exceeding $25 million (in second-quarter 1980 dollars), if authority to sign documents has been assigned or delegated to the manager in accordance with corporate procedures.

(2) For a partnership or sole proprietorship - by a general partner or the proprietor, respectively.

(3) For a municipality, State, Federal, or other public agency - by either a principal executive officer or ranking elected official. For purposes of this section, a principal executive officer of a Federal agency includes:

(a) The chief executive officer of the agency, or

(b) A senior executive officer having responsibility for the overall operations of a principal geographic unit of the agency.

b. All reports required by the permit and other information requested by the Director shall be signed by a person described above or by a duly authorized representative of that person. A person is a duly authorized representative only if:

(1) The authorization is made in writing by a person described above;

(2) The authorization specifies either an individual or a position having responsibility for the overall operation of the regulated facility or activity, such as the position of plant manager, operator of a well or a well field, superintendent, or position of equivalent responsibility, or an individual or position having overall responsibility for environmental matters for the company. A duly authorized representative may thus be either a named individual or any individual occupying a named position; and,

(3) The written authorization is submitted to the Director.

c. Certification. Any person signing a document under this section shall make the following certification:

"I certify under penalty of law that this document and all attachments were prepared under my direction or supervision in accordance with a system designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who manage the system, or those persons directly responsible for gathering the information, the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations."

12. Availability of Reports

Except for data determined to be confidential under 40 CFR Part 2, all reports prepared in accordance with the terms of this permit shall be available for public inspection at the office of the Director. As required by the Clean Water Act, the name and address of any permit applicant or permittee, permit applications, permits, and effluent data shall not be considered confidential.

13. Penalties for Falsification of Reports

The Clean Water Act provides that any person who knowingly makes any false statement, representation, or certification in any record or other document submitted or required to be maintained under this permit, including monitoring reports or reports of compliance or noncompliance shall, upon conviction, be punished by a fine of not more than $10,000 per violation, or by imprisonment for not more than 6 months per violation, or by both.

PART III
OTHER CONDITIONS

A. The term "composite sample" means a sample consisting of a minimum of two grab samples of effluent collected not less than four hours apart over a normal eight hour operating day and combined proportional to flow or a sample continuously collected proportional to flow over a normal eight hour operating day. All such samples shall be typical and representative of effluent generated during the period since the last sample was collected.

B. Analysis and reporting for Outfalls 001 and 002 shall be accomplished separately for each outfall. In addition, the permittee shall report the flow weighted average results for Outfalls 001 plus 002. Effluent limitations on weight of total molybdenum shall be the sum total for Outfalls 001 and 002. For determination of compliance with other effluent limitations the following shall apply:

1. Discharge from Outfall 001 shall be in compliance.

2. A composite sample of discharges from Outfalls 001 and 002 monitoring samples combined in flow weighted proportion shall be in compliance, except that concentration limits for molybdenum and manganese are suspended during periods of zero flow from Outfall 001.

   During periods when no flow occurs from Outfall 001, monthly average reporting of concentration for molybdenum and manganese shall be based on the average of all composite samples obtained in the month for Outfalls 001 and 002; and monthly maximum reporting of concentration for molybdenum and manganese shall be based on the maximum composite sample obtained in that month for Outfalls 001 and 002. In conjunction with the DMR for each month, the permittee shall report periods, if any, when no flow exists at Outfall 001.

C. <u>CHRONIC BIOMONITORING REQUIREMENTS</u>

a. The permittee shall test the effluent for toxicity in accordance with the provisions in this section. Such testing will determine if an appropriately dilute effluent sample affects the survival and reproduction or growth of the appropriate test organism. The permittee shall initiate the following series of tests within 60 days of the effective date of this

permit to evaluate wastewater toxicity. All test organisms, procedures, and water quality assurance criterion used shall be in accordance with the latest revision of "Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms", EPA/600/4-85/014. The following tests shall be used:

1) The permittee shall conduct a 7-day Ceriodaphnia dubia survival and reproduction test (Method 1002.0).

2) The permittee shall conduct a 7-day fathead minnow (Pimephales promelas) larval survival and growth test (Method 1000.0).

b. A minimum of 5 dilutions must be performed in addition to an appropriate control, using a minimum dilution factor of 0.3. Three dilutions consisting of 100%, 31%, and 18% of the final effluent must be contained in the test series.

c. The samples shall be collected at a point following the last treatment unit. Dilution water used in toxicity tests will be receiving stream water collected at a point upstream of the discharge. If receiving water is unsatisfactory as a result of pre-existing in-stream toxicity (greater than 20% mortality in the control), the permittee must substitute reconstituted dilution water, with hardness and alkalinity similar to that of the receiving stream water. The permittee shall also report to EPA the toxicity of the upstream receiving water.

d. Flow-weighted 24-hour composite samples representative of dry weather flows during normal operation will be collected from Outfalls 001 and 002. These composites shall be combined in proportion to the average flow from each outfall for the day the sample was collected. The toxicity tests shall be performed on the flow-weighted composite of outfall samples.

e. The toxicity tests specified in paragraphs (a) and (b) above shall be conducted once per month. The permittee shall prepare a full report of the results according to EPA/600/4-85/014, Section 10, Report Preparation. This full report need not be submitted unless requested and shall be retained following the provisions of Part II.C.8 of this permit.

f. The permittee shall submit the toxicity testing information contained in Table 1 of this permit to EPA along with the Discharge Monitoring Report (DMR) submitted for the end of the reporting period following the toxicity test.

g. Should no toxicity occur within the first year of toxicity testing, in accordance with paragraph (h) below, for both species tested at the effluent dilution equivalent to 1/2 of low flow (31%), the permittee shall certify this information in writing to EPA Region VI and these biomonitoring requirements shall expire.

h. For the purpose of this biomonitoring requirement, chronic toxicity is defined as a statistically significant difference at the 95% confidence level between the survival and growth or reproduction in the appropriate test organism exposed to the control and to an effluent dilution.

i. This permit shall be reopened to require further monitoring studies and/or effluent limits if biomonitoring data show actual or potential ambient toxicity to be the result of the permittee's discharge to the receiving stream. Modification or revocation of the permit is subject to the provisions of 40 CFR Part 122.62. Accelerated or intensified toxicity testing may be required in accordance with Section 308 of the Clean Water Act.

TABLE 1

BIOMONITORING REPORTING

CERIODAPHNIA DUBIA SURVIVAL AND REPRODUCTION TEST

Permittee: _____
NPDES No.: _____

Composite collected FROM: _____ am/pm _____ date
TO: _____ am/pm _____ date

Test initiated: _____ am/pm _____ date

Dilution water used: ☐ Receiving water ☐ Reconstituted water

NUMBER OF YOUNG PRODUCED PER FEMALE @ 7 DAYS

Percent effluent (%)

REP	0%	1%	3%	10%	30%	100%	% at low flow %	% at 1/2 low flow %
A								
B								
C								
D								
E								
F								
G								
H								
I								
J								

TABLE 1 (Continued)

BIOMONITORING REPORTING

CERIODAPHNIA DUBIA SURVIVAL AND REPRODUCTION TEST

Permittee: _____
NPDES No.: _____

PERCENT SURVIVAL

Percent effluent (%)

Time of Reading	0%	1%	3%	10%	30%	100%	% at low flow %	% at 1/2 low flow %
24h								
48h								
7-day								

1. Fisher's Exact Test:

   Is the mean survival at 7 days significantly different (p=0.05) than the control survival for the % effluent corresponding to:

   a. LOW FLOW: _____ YES _____ NO
   b. 1/2 LOW FLOW: _____ YES _____ NO

2. Dunnett's Procedure or Steel's Many-One Rank Test as appropriate:

   Is the mean number of young produced per female significantly different (p=0.05) than the control's number of young per female for the % effluent corresponding to:

   a. LOW FLOW: _____ YES _____ NO
   b. 1/2 LOW FLOW: _____ YES _____ NO

3. Enter percent effluent corresponding to each NOEL below and circle lowest number:

   a. NOEL survival = _____ % effluent
   b. NOEL reproduction = _____ % effluent

4. If you answered NO to 1.a. and 2.a., enter [N]; otherwise enter [Y]: ____

5. Enter response to item 4 on DMR Form, Parameter No. TCP3B.

6. If you answered NO to 1.b. and 2.b., enter [N]; otherwise enter [Y]: ____

7. Enter response to item 6 on DMR Form, Parameter No. TDP3B.

TABLE 1 (Continued)

BIOMONITORING REPORTING

FATHEAD MINNOW LARVAE GROWTH AND SURVIVAL TEST
(Pimephales promelas)

Permittee: _____
NPDES No.: _____

Composite collected FROM: _____ am/pm _____ date
TO: _____ am/pm _____ date

Test initiated: _____ am/pm _____ date

Dilution water used: ☐ Receiving water ☐ Reconstituted water

DATA TABLE FOR GROWTH OF FATHEAD MINNOWS

Effluent Conc. (%)	Average Dry Weight in milligrams in replicate chambers				MEAN DRY WEIGHT	
	A	B	C	D	mg	CV%*
0%						
1%						
3%						
10%						
30%						
100%						
Low Flow %						
1/2 Low Flow %						

* coefficient of variation = standard deviation x 100/mean

1. Dunnett's Procedure:

Is the mean dry weight (growth) at 7 days effluent significantly different (p=0.05) than the control's dry weight (growth) for the % effluent corresponding to:

a. LOW FLOW: _____ YES _____ NO
b. 1/2 LOW FLOW: _____ YES _____ NO

TABLE 1 (Continued)

BIOMONITORING REPORTING

FATHEAD MINNOW LARVAE GROWTH AND SURVIVAL TEST
(Pimephales promelas)

Permittee: _____
NPDES No.: _____

DATA TABLE FOR FATHEAD MINNOW SURVIVAL

Effluent Conc. (%)	Percent Survival in replicate chambers				MEAN PERCENT SURVIVAL			
	A	B	C	D	24h	48h	7-day	CV%*
0%								
1%								
3%								
10%								
30%								
100%								
Low Flow %								
1/2 Low Flow %								

* coefficient of variation = standard deviation x 100/mean

2. Dunnett's Procedure or Steel's Many-One Rank Test as appropriate:

   Is the mean survival at 7 days significantly different (p=0.05) than the control survival for the % effluent corresponding to:

   a. LOW FLOW: _____ YES _____ NO
   b. 1/2 LOW FLOW: _____ YES _____ NO

3. Enter percent effluent corresponding to each NOEL below and circle lowest number:

   a. NOEL survival = _____ % effluent
   b. NOEL growth = _____ % effluent

4. If you answered NO to 1.a. and 2.a., enter [N]; otherwise enter [Y]: ____

5. Enter response to item 4 on DMR Form, Parameter No. TCP6C.

6. If you answered NO to 1.b. and 2.b., enter [N]; otherwise enter [Y]: ____

7. Enter response to item 6 on DMR Form, Parameter No. TDP6C.

D. The Molycorp thiocyanate colorimetric method is approved for the analysis of molybdenum unless subsequently determined to be inappropriate by the NMEID or EPA.

E. As soon as practicable after the arrival of Molycorp's environmental staff at the site of a tailings spill that reaches the Red River, but no later than two (2) hours after arrival at the site, water quality sampling shall commence. Samples shall be taken at three sites:

(1) Approximately 100 feet above the point where tailings enter the river;

(2) Approximately 100 feet below the point where tailings enter the river; and

(3) Approximately one-half mile below the point where tailings enter the river.

All samples shall be properly preserved and analyzed for:

Chemical Oxygen Demand (COD)
Total Suspended Solids (TSS)
Cyanide (CN)
Total Iron (Fe)
Manganese, Dissolved (Mn)
Total Molybdenum (Mo)
pH
Temperature
Conductivity
Total Zinc (Zn)

The results of the analyses shall be submitted to the United States Environmental Protection Agency and the New Mexico Environmental Improvement Division within 30 days following a tailings spill.

Consistent with the procedures described in the Preventative Maintenance and Surveillance Plan and Contingency Action and Reporting Plans (June 1975), a written report containing the following information will be sent to the U.S. EPA and NMEID within ten (10) days following any spill:

(1) Date of spill.

(2) Time when the spill was observed and time when tailings flow into the river was stopped.

(3) Location (pipe or coupling number).

(4) Estimated amount of tailings that entered the river.

(5) Sketch and dimension of size of hole or failure that caused the spill.

(6) Position of failure in the pipe or coupling.

(7) Copy of the latest computer printout covering the pipe or coupling which failed.

(8) Comments, if required for clarification.

UNOCAL 76
MOLYCORP

(PART II) - SECTION VII

ASSESSMENT OF ACUTE TOXICITY OF 002, WEST DIVERSION CHANNEL AND RED RIVER

SECTION IV

RESULTS OF BIOLOGICAL TOXICITY TESTING OF OUTFALL 002, THE RED RIVER & WEST DIVERSION CHANNEL

Assessment of Acute Toxicity of 002
Daphnia Magna

September 1985

ANALYTICAL BIO-CHEMISTRY LABORATORIES, INC.
P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

Static Acute Toxicity Report
#33795

Assessment of the Acute Toxicity of 91185-002 to *Daphnia magna*

Submitted To:

Molycorp, Incorporated
Attn: Mr. John Stribling
3 miles E. of Questa, on State Road 38
P.O. Box 469
Questa, NM 87556

September 26, 1985

Submitted By: Analytical Bio-Chemistry Laboratories, Inc.
7200 East ABC Lane
P. O. Box 1097
Columbia, Missouri 65205
(314) 474-8579

Prepared By:

David Burgess 9/26/85
David Burgess Date
Biologist II

Alan Forbis (CT) 9-26-85
Alan D. Forbis Date
Aquatic Biologist III/Study Director

Approved By:

Carl M. Thompson 9-26-85
Carl M. Thompson Date
Supervisor, Aquatic Toxicology

Kathy Landholt 09/26/85
Kathy Landholt Date
Quality Assurance Officer

Michael Williams 9/26/85
Michael Williams, Ph.D. Date
Group Leader, Animal Toxicology

/lrm

SUMMARY

The acute toxicity of 91185-002 to Daphnia magna was assessed for U.S. EPA Region 6 NPDES permit requirements using the methods outlined by the Committee on Methods for Toxicity Tests with Aquatic Organisms. Water quality parameters of temperature, dissolved oxygen, conductivity, hardness and pH were measured on receipt of the sample and were within acceptable limits.

The results of the 24-hour static Daphnia magna toxicity screen indicated that the mortality was <20% for 91185-002. In fact, no mortality was observed after the 24-hour exposure period. Therefore, a definitive 48 hour static acute study was not required. All reported values were based upon nominal concentrations.

The no effect level observed for 91185-002 was 100% of effluent after 24 hours, which was based on the lack of mortality and abnormal effects.

INTRODUCTION

This static bioassay was performed by the Aquatic Toxicology Division of Analytical Bio-Chemistry Laboratories, Inc., Columbia, Missouri, for Molycorp, Inc. from September 12 to September 13, 1985. The purpose of this test was to assess toxicity of a 100% solution of 91185-002 to *Daphnia magna*. The assessment was designed to meet the NPDES permit process in U.S. EPA Region 6 (1) requiring biomonitoring of industrial discharges. The screen test was conducted following the procedures outlined in ABC protocol #8301, as approved by Mr. John Stribling, Molycorp, Inc.

METHODS AND MATERIALS

The procedures applied for the 24-hour toxicity screen were basically those described in *Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms* (2), *Methods for Acute Toxicity Tests with Fish, Macroinvertebrates, and Amphibians* (3), *Standard Practice for Conducting Acute Toxicity Tests with Fishes, Macroinvertebrates, and Amphibians* (4), *Proposed Standard Practice for Conducting Acute Toxicity Test on Aqueous Effluents with Fishes, Macroinvertebrates and Amphibians* (5), and *Standard Methods for the Examination of Water and Wastewater* (6). The test lot of *Daphnia magna* were obtained from an in-house culture at ABC. The taxonomic key presented by Pennak (7) was used to identify to species the test daphnids. All test daphnids were held in a controlled temperature area at 20 ±2°C. The lighting was 50-70 footcandles on a 16 hour daylight photoperiod. During the holding period, they received a suspension of algae (*Selenastrum capricornutum*) supplemented with a blended fish food/cerophyl suspension at least every 3 days. First-instar daphnids (<24-hours old) were selected for testing and were not fed during the test.

The static daphnid bioassay was conducted in 250 ml glass beakers containing 200 ml of sample and/or ABC well water. The ABC well water control contained water with chemical characteristics listed in Table 1. These vessels were kept at 20 (±2.0) °C in a temperature controlled area. The lighting was maintained at 50-70 footcandles on a 16 hour daylight photoperiod.

A 24-hour screen test, composed of duplicate 100% effluent was performed to estimate the toxicity of the effluent. Also included was a duplicate ABC well water control. A five-six concentration static acute toxicity study would be performed if ≧20% mortality was observed after 24 hours in the screen test.

The 91185-002 was received on September 12, 1985 as a clear liquid and was received at 5°C. The sample was allowed to warm to test temperature before addition of *Daphnia*.

RESULTS

After 24 hours, the results indicated that the mortality of *Daphnia magna* to 91185-002 was <20%. The no-effect concentration based on the lack of mortality and abnormal effects was 100%. No abnormal effects, such as mortality, surfacing, and daphnids lying on the bottom were observed after 24 hours. Since mortality was not ≧20% after 24 hours in the screen test, a definitive study was not required per biomonitoring guideslines established for U.S. EPA Region 6.

Table 2 presents the water quality parameters measured on sample receipt. Hardness, alkalinity (as $CaCO_3$) and conductivity for the 91185-002 sample were 946, 182 mg/l and 1400 µmhos, respectively. Dissolved oxygen and pH for this sample were 11.4 mg/l and 7.3, respectively, and were considered adequate for testing (3).

The study was conducted following the intent of the Good Laboratory Practice Regulations (8) and the final report was reviewed by Analytical Bio-Chemistry Laboratories' Quality Assurance Unit. All original raw data was provided to Molycorp, Inc. with a copy retained at Analytical Bio-Chemistry Laboratories.

TABLE 1: Chemical Characteristics of Well Water Used by ABC's Aquatic Toxicology Division.

Parameters	Concentration
Temperature	15-20°C[b]
Dissolved Oxygen[a]	9.2-10.1 ppm[b]
pH	7.8-8.3[b]
Hardness ($CaCO_3$)	225-275 ppm[b]
Alkalinity ($CaCO_3$)	325-375 ppm[b]
Conductivity	700 μmhos/cm
NO_3-N	0.74 ppm
NO_3-NO_2-N	0.74 ppm
PO_4-P	<0.10 ppm
Aluminum	<20 ppb
Arsenic	<0.2 ppb
Cadmium	<2 ppb
Chromium	<3 ppb
Cobalt	<4 ppb
Copper	<3 ppb
Iron	12 ppb
Lead	<5 ppb
Mercury	<0.5 ppb
Nickel	<15 ppb
Silver	<5 ppb
Zinc	11 ppb
Measured organophosphorus pesticides	c
Measured organochlorine pesticides plus PCB's	c

[a]After aeration.

[b]Represents seasonal variation, with the monthly range not exceeding 10%.

[c]Less than minimum detectable limits.

TABLE 2

Water Quality Measurements During the Acute Toxicity Screen Test of 91185-002 to *Daphnia magna*

	ABC Well Water		100% 91185-002		
	0 hr.	24 hr.	On Receipt	0 hr.	24 hr.
Temperature[a]	20	20	5	20	20
Dissolved Oxygen[b]	8.0	8.2	11.4	10.3	9.1
pH[c]	8.2	8.3	7.3	7.3	7.8
Conductivity[d]	750	750	1400	1500	1550
Hardness[e]	--	--	946	--	--
Alkalinity[e]	--	--	182	--	--

[a]Temperature °C - Monitored with a mercury thermometer.

[b]Dissolved oxygen concentrations (mg/l) - Dissolved Oxygen System (YSI Model 54). Saturation at 20°C is 9.2 mg/l.

[c]pH - pH Probe (Corning Model 476182) used with a Corning Model 125 pH and mV meter.

[d]Conductivity (μMHOS) - Conductivity Meter (YSI Model 33).

[e]Hardness and alkalinity (mg/l as $CaCO_3$) - HACH Model AL-DT and HAC-DT.

LITERATURE CITED

(1) U.S. Environmental Protection Agency. 1983. Effluent Toxicity Testing and Biomonitoring Workshop. Feb. 22-23, 1983, Dallas, Texas.

(2) Peltier, William. 1978. Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms. U.S. Environmental Protection Agency, EPA-660/4-78-012, revised July 1978. 52 p.

(3) Committee on Methods for Toxicity Tests with Aquatic Organisms. 1975. Methods for Acute Toxicity Tests with Fish, Macroinvertebrates and Amphibians. Environmental Protection Agency, Ecological Research Series EPA-660/3-75-009, April 1975. 61 p.

(4) American Society for Testing and Materials. 1980. Standard practice for conducting basic acute toxicity tests with fish, macroinvertebrates and amphibians. May 1980, ASTM Committee E-35.23. 25 p.

(5) American Society for Testing and Materials. 1983. Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates and Amphibians. November 1983, ASTM Committee E-47.01. 37 p.

(6) American Public Health Association. 1980. Standard Methods for the Examination of Water and Wastewater. 15th ed. Washington, DC. 1134 p.

(7) Pennak, R. W. 1978. Freshwater Invertebrates of the United States. John Wiley & Sons, New York, 2nd ed. 803 p.

(8) (a) U.S. Food and Drug Administration. 1978. Nonclinical Laboratory Studies, Good Laboratory Practice Regulations (21 CFR, Part 58). Federal Register, Vol. 43, No. 247:59986-60025.

(b) U.S. Environmental Protection Agency. 1983. Pesticide Programs; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 160). Federal Register, Vol. 48, No. 230:53946-53969.

(c) U.S. Environmental Protection Agency. 1983. Toxic Substances Control; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 792). Federal Register, Vol. 48; No. 230:53922-53944.

(d) Organization for Economic Cooperation and Development. 1981. OECD Guidelines for Testing of Chemicals, Principles of Good Laboratory Practice Annex 2, C(81) 30(Final):7-28.

QUALITY ASSURANCE STATEMENT

Quality Assurance Statement for final report #33795 entitled, "Acute Toxicity of 91185-002 to *Daphnia magna*," for John Stribling, Molycorp, Inc., Questa, New Mexico.

In accordance with ABC Laboratories intent that all studies conducted at our facilities are designed and function in conformance with Good Laboratory Practice regulations and the protocols for individual laboratory studies, an inspection of the final report for 91185-002 was conducted and found to be in acceptable form by a member of our Quality Assurance Unit. A final inspection of all data and records on September 25, 1985 indicates that the report submitted to you is an accurate reflection of the study as it was conducted by ABC Laboratories.

Should you have any questions relating to the information provided in this statement or the function of our Quality Assurance Unit, please contact the QA Unit at your convenience.

Kathy Landholt 09/26/85

Kathy Landholt Date
Quality Assurance Officer

STUDY COMPLIANCE STATEMENT

Study Compliance Statement for ABC report #33795 entitled, "Acute Toxicity of 91185-002 to Daphnia magna," for John Stribling, Molycorp Inc., Questa, New Mexico.

In accordance with ABC Laboratories' intent that all aquatic toxicity tests conducted by our facility follow good laboratory practices, ABC's study director for the above test herein confirms that the study was conducted in compliance with the U.S. E.P.A. Good Laboratory Practice Standard; Toxic Substances Control (40 CFR 792).

All original raw data were sent to Molycorp, Inc., with a copy retained at Analytical Bio-Chemistry Laboratories.

Alan D. Forbis (CT) 9-26-85

Alan D. Forbis Date
ABC Study Director

APPENDIX - RAW DATA

NOTE: Some of the records which appear in this raw data appendix have been provided as photocopies of original records on file at ABC. This has been done by necessity for certain data which are used commonly in several studies at ABC. Such records include reference toxicity test data, standard solution preparations, organism culture logs, and well water chemical screen results.

EFFLUENT TOXICITY TEST
MORTALITY AND BEHAVIORAL OBSERVATIONS

Effluent Designation: OUTFALL 91185-002

Industry: MOLYCORP

Study No.: 33795

ABC Study Director: A. FORBIS

Lab Form No.: 91

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P.O. Box 1097
Columbia, Missouri 65205

☐ Prelim. ☑ Definitive Test Species: D. magna (Lot #85-B-7)

Date Initiated: 9-12-85 Test Terminated: 9/13/85

Dilution Water: NONE No./Vessel: 10 Vessel Size: 250 ml

Effluent Conc. (%)	24 hours Dead	24 hours Obs.	48 hours Dead	48 hours Obs.	72 hours Dead	72 hours Obs.	96 hours Dead	96 hours Obs.
Control A	0	N						
B	0	N						
100 A	0	N						
B	0	N						
Observer	DB							
Date	9/13/85							

Remarks: N = Normal; LOE = Loss of Equilibrium; SUR = Surfacing

Prepared By: David Bunger Date: 9/12/85

Checked By: Alan D Fabis Date: 9/16/85

WATER QUALITY FOR EFFLUENT SAMPLES

Test Material: 91185-002

Study No.: 33795

Protocol No.: 8301

ABC Study Director: A. FORBIS

Lab Form No.: 28b

Analytical Bio-Chemistry Labs.
Aquatic Toxicology Division
7200 ABC Lane; P.O. Box 1097
Columbia, Missouri 65205

Concentration ~~mg/l~~ %	9/12/85 DAY 0 DB				9/13/85 DAY 1 DB				DAY				DAY			
	Temp °C	D.O.[a] mg/l	pH[b]	Cond. µMhos	Temp °C	D.O. mg/l	pH	Cond. µMhos	Temp °C	D.O. mg/l	pH	Cond. µMhos	Temp °C	D.O. mg/l	pH	
ABC CONTROL	20	8.0	8.2	750	20	8.2	8.3	750								
100	20	10.3	7.3	1500	20	9.1	7.8	1550								

[a]Dissolved oxygen concentrations - Dissolved Oxygen Probe (YSI Dissolved Oxygen System Model 54).

[b]pH-pH Probe (Corning Model 476182) used with Corning Model 125 pH and mV meter.

[c] 100% Effluent on Receipt	Temp °C	D.O. mg/l	pH	Cond. µMhos	Hard mg/l	Alk. mg/l	~~NH_3 mg/l~~			Date	Int.
	5	11.4	7.3	1400	146	182				9-12-85	DB

Prepared By: David Burger Date: 9/12/85

Checked By: A. Forbis Date: 9/16/85

Remarks:

STUDY# 033795

PREPARATION OF EFFLUENT CONCENTRATIONS (volume/volume)

Effluent Designation: OUTFALL 91155-002

Industry: MOLYCORP

Study No.: 33795

ABC Study Director: A. FORBIS

Lab Form No.: 94

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane; P.O. Box 1097
Columbia, MO 65205

☐ PRELIMINARY ☑ DEFINITIVE

Dilution Water Type:

☐ ABC Well Water ☐ ABC Recon. Water

☐ Effluent Receiving Water _____ ☑ Other NONE

Sample No.	Aliquot Volume (ml)	Dilution Volume (ml)	Final Conc. (%)
1	200	0	100
2			
3	0	200	ABC CONTROL
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			

Remarks: .

Prepared By: [illegible] Date: 9/12/85

Checked By: [illegible] Date: 9/16/85

Study Director: [illegible] Date: 9/16/85

INVERTEBRATE CULTURE RECORD

Species: Daphnia magna

Lot No.: 85-B-7

Date Initiated: 8/27/85

Lab Form No: 52

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P. O. Box 1097
Columbia, MO 65205

Date	No. Adult Alive	Instar Present	Fed & Changed	Study I.D.	Comments	Obs. By:
8/27	—	~30	✓		300 ml algae 2 ml yeast food	LG
8/29	—	25	✓		" "	LG
8-31	—	25	✓		" "	JHB
9-2	25	~20	✓		" "	JHB
9/4	25	200	✓		" "	DB
9/6	25	800	✓		" "	DB
9-8	21	~800	✓		" "	[illegible]
9/10	21	500	✓		" "	DB
9/11				33761-140		DB
9/11	21	1500	✓		" "	DB
9/12				33795-40		DB
9/12	21	200	✓		" "	DB
9/15	19	~600	✓		" "	PC
		"This is an exact copy of The original document"				
		By B. Blackstock date 9-16-85				

Prepared by: Jori Georgie Date: 8/27/85

Checked by: Darrel Burgess Date: 9/16/85

ANALYTICAL BIO-CHEMISTRY LABORATORIES, INC.
P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

Sample Date: April 2, 1985

ANALYSIS RESULTS FOR:

Aquatic Toxicology Division
Analytical Bio-Chemistry Laboratories, Inc.
7200 East ABC Lane
P. O. Box 1097
Columbia, Missouri 65205

Water Analysis Results

ABC Lab No.	33102
Sample I.D.	ABC Aquatic Lab Well Water
NO_3-N	0.74 mg/l
NO_3-NO_2-N	0.74 mg/l
PO_4-P	<0.10 mg/l
Al	<20 µg/l
Co	<4 µg/l
Fe	12 µg/l
As	<0.2 µg/l
Cd	<2 µg/l
Cr	<3 µg/l
Cu	<3 µg/l
Pb	<5 µg/l
Hg	<0.5 µg/l
Ni	<15 µg/l
Ag	<5 µg/l
Zn	11 µg/l

<Indicates less than, if present at all.

Del A. Koch
Chemist III

Please note that the original data for the analysis reported in this table are in ABC's archives.

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Sample Date: April 2, 1985

ANALYSIS RESULTS FOR:

Aquatic Toxicology Division
Analytical Bio-Chemistry Laboratories, Inc.
7200 East ABC Lane
P. O. Box 1097
Columbia, Missouri 65205

Analysis of Aquatic Lab Well Water for Organophosphates

ABC Lab No.	Customer I.D.	Compound	ppb
33103	Aquatic Lab Well Water	Diazinon	<0.10
		Methyl Parathion	<0.10
		Malathion	<0.10
		Ethyl Parathion	<0.10

Randy Ayling 6/25/85
Randy Ayling
Chemist I

Please note that the original data for the analysis reported in this table are in ABC's archives.

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P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

Sample Date: April 2, 1985

ANALYSIS RESULTS FOR:

Aquatic Toxicology Division
Analytical Bio-Chemistry Laboratories, Inc.
7200 East ABC Lane
P. O. Box 1097
Columbia, Missouri 65205

Analysis of Aquatic Lab Well Water for Nonionic
Chlorinated Hydrocarbon Residue

ABC Lab No.	33103
Customer I.D.	ABC Aquatic Lab Well Water
Compound	ppb
αBHC	<0.01
Lindane	<0.01
Heptachlor	<0.01
Heptachlor Epoxide	<0.02
γChlordane	<0.02
αChlordane	<0.02
Aldrin	<0.01
Dieldrin	<0.03
p,p'-DDE	<0.03
o,p'-DDT	<0.05
p,p'-DDD	<0.05
p,p'-DDT	<0.05
Endrin	<0.05
Methoxychlor	<0.20
Toxaphene	<0.50
Aroclor 1242	<0.40
Aroclor 1016	<0.40
Aroclor 1254	<0.40
Aroclor 1260	<0.40
HCB	<0.01
Mirex	<0.10

<indicates less than, if present at all.

Randy Ayling 6/25/85
Randy Ayling
Chemist I

Please note that the original data for the analysis reported in this table are in ABC's archives.

DEFINITIVE CONCENTRATION NOTIFICATION

Firm: MOLYCORP INC	Lab Form No.: 186
Address:	Analytical Bio-Chemistry Labs Aquatic Toxicology Division 7200 ABC Lane, P.O. Box 1097 Columbia, MO 65202
City/State/Zip: QUESTA NM	
Phone: ( )	

Sponsor's Study Director: MR. JOHN STRIBLING

Person notified: same(✓) Other:

Date of Notification: 9/16/85

Notification of preliminary testing results with the following definitive test levels agreed upon:

Test Material	ABC #	Test Species	Test Conc. (~~mg/l~~ %)
OUTFALL 91185-002	33795	D. magna	100 TOX SCREEN

NOTIFIED JOHN STRIBLING OF THE RESULTS OF THE 24 HR REGION 6 TOX SCREEN WITH OUTFALL 91185-002 TO Daphnia magna.

Notified By: David Burgess Date: 9/16/85

MOLYCORP, INC.
QUESTA DIVISION

This sheet represents a chain of custody for effluent samples collected at OUTFALL 002, located in Taos County, Questa, New Mexico on 9-11-85.

Sample bottles were prepared by Molycorp employees in accordance with guidance set forth by the U.S. Environmental Protection Agency for the existing program permit. Sampling procedures were also followed in accordance with permit requirements.

Sample(s) Identification: 91185-002

Sample(s) collected at: 1:15pm 1st portion

By: [signature]
Environmental Technician

Sample(s) handed over to carrier by Molycorp employee:

DATE: 9-11-85

TIME: 1:32 pm

CARRIER: David Stagy

EMPLOYER: AIR-DRAULICS Co.

MOLYCORP PERSONNEL: [illegible]

Sample(s) delivered to ABC Lab for analysis. Please sign and date this letter upon receipt of sample(s).

Sample received by: ABC Laboratories Inc.
7200 ABC Lane
P.O. Box 1097
Columbia, MO 65205
(314) 474-8579

for the following analyses: Toxicity testing

Received by: DAVID BURGESS
ABC Laboratories Inc.

Date: SEPT. 12, 1985 8:50 AM

Please return this letter with the analytical results, signed and dated below.

David Burgess
ABC Laboratories Inc.

9/16/85
Date

ANALYTICAL BIO-CHEMISTRY LABORATORIES, INC.
P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

ABC LABS
SEP 24 1985
RECEIVED

ABC PROTOCOL NO. 8301

(August 21, 1984)

Effluent Static Bioassay with *Daphnia magna* or Mysid Shrimp (*Mysidopsis bahia*) to Meet EPA Region 6 NPDES Biomonitoring Guidelines

ABC Study Number 33795

Test Material Outfall 91185-002

Test Species *Daphnia magna*

1.0 INTRODUCTION

The use of bioassays in the identification and control of industrial effluents is rapidly increasing. Indeed, aquatic toxicity tests are presently being incorporated into the NPDES guidelines of the Federal Clean Water Act (1). To assist industry in meeting requirements set forth by legislation and regulatory agencies, Analytical Bio-Chemistry Laboratories, Inc. offers a complete range of aquatic bioassay tests of industrial effluents.

As part of the NPDES permit process in U.S. EPA Region 6, a biomonitoring requirement has been imposed for certain permit holders in that area. Discharges subject to the new regulations in Region 6 (Texas, New Mexico, Oklahoma, Arkansas and Louisiana) will be required to submit aquatic toxicity data on their effluents to EPA on a periodic basis.

This ABC protocol discusses the biomonitoring guidelines in Region 6 and describes ABC's technical approach to assist industry in complying with these regulations.

2.0 OBJECTIVES

The primary objective of the toxicity test described herein is to evaluate the acute toxicity of an effluent to freshwater or marine invertebrates under static conditions. The species selected for this testing include *Daphnia magna* (freshwater effluent, <5 ppt salinity) and *Mysidopsis bahia* (saltwater effluent, ≥5 ppt salinity). The effluent will first be tested on a 100% effluent screen basis. If the screen results in ≥20% mortality of test organisms, a 48-hour, five concentration LC_{50} test will be conducted. An LC_{50} is the approximate concentration (percent) of test effluent that produces 50 percent mortality of test organisms after prescribed intervals (24 and 48 hours).

3.0 TESTING FACILITY

The study will be conducted by the Aquatic Toxicology Division of Analytical Bio-Chemistry Laboratories, Inc., 7200 East ABC Lane, P. O. Box 1097, Columbia, Missouri 65205 (314-474-8579).

4.0 REGION 6 BIOMONITORING REQUIREMENT

The following biomonitoring requirement is now being included in the NPDES permit process for many major industrial discharges in EPA Region 6. The permit language given here was that presented by the U.S. EPA at a regional workshop on the matter (2):

> The permittee shall determine if the effluent from this outfall is lethal to twenty (20) percent or greater of the culture of test organisms by use of the "Range-Finding Screening Test," set out in "Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms," EPA-600/4-78-012 (Rev. July, 1978) (3).

Organisms for this test shall be Daphnia sp. if the effluent is less than five (5) parts per thousand salinity or Mysid sp. if the effluent is greater than five (5) parts per thousand salinity. This screening test will be conducted within sixty (60) days of effectiveness of the biomonitoring requirements. Tests will be conducted once each quarter for a duration of two years utilizing a static method for 24 hours and following this dilution scheme only:

Effluent sample* - 100 percent by volume
Dilution water - 0 percent by volume

*24-hour composite; refrigerated after collection

If at any time during the two year testing period a test shows a lethality of twenty (20) percent or more of the test organisms, the permittee shall within twenty-four (24) hours conduct a replacement static 48-hour median lethal concentration (LC_{50}) test on the originally collected sample. Replacement of effluent samples shall be once per 24 hours. Organisms for this test shall be Daphnia sp. if the effluent is less than five (5) parts per thousand salinity and reconstituted fresh water (EPA-600/4-78-012 Section 4) shall be used for dilution. If the effluent contains greater than five (5) parts per thousands salinity, Mysid sp. shall be used as the test organism, and reconstituted seawater will be used as dilution water (EPA-600/4-78-012 Section 4). The remaining LC_{50} methodology is available in EPA-600/4-78-012.

All screening and LC_{50} test results shall be reported with the Discharge Monitoring Reports.

It should be noted by the permittee that slight changes from the above permit language (e.g., pH modification of effluent) may be incorporated into his specific permit. Attention should be given to the permit requirements for each effluent before testing is initiated. ABC requires that a copy of the permit's biomonitoring clause be forwarded to ABC prior to testing so that consideration can be given to pH modification, etc.

5.0 METHODS AND MATERIALS

5.1 Methodology. The methods applied for both the preliminary and definitive phases of this test are basically those described in Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms (3), Methods for Acute Toxicity Tests with Fish, Macroinvertebrates, and Amphibians (4), Standard Practice for Conducting Acute Toxicity Tests with Fishes, Macroinvertebrates, and Amphibians (5), Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates

and Amphibians (6), and Standard Methods for the Examination of Water and Wastewater (7). This protocol has been written to comply with the federal Good Laboratory Practice Regulations (8).

5.2 Test Organisms. The species recommended by the EPA for this testing are Daphnia sp. and Mysid sp. Daphnia sp. are to be used when the effluent salinity is <5 parts per thousand (ppt) and Mysid sp. are to be used when the effluent salinity is ≧5 ppt. The species maintained by ABC, and acceptable by EPA, for this testing are Daphnia magna and Mysidopsis bahia. The test lot of Daphnia magna will be obtained from an in-house culture at ABC. The taxonomic key presented by Pennak (9) will be used to identify to species the test daphnids. All test daphnids will be held in a controlled temperature area at 20 ±2°C. The lighting will be 50-70 footcandles on a 16 hour daylight photoperiod. During the holding period, they will receive a suspension of algae (Selenastrum capricornutum) supplemented with a blended fish food/cerophyl suspension at least every 3 days. First-instar daphnids (<24 hours old) will be selected for testing and will not be fed during the test.

Mysid shrimp (Mysidopsis bahia) will be obtained from a commercial supplier (e.g., Sea Plantations, Inc. or Gulf Breeze Specimens) and maintained in culture at ABC. Mysids will be cultured following the techniques described by the U.S. EPA (10). Test mysids will be selected from a sub-culture of immature individuals.

5.3 Effluent Sample. The sample submitted to ABC for testing must be a 24-hour composite sample, consisting of equal volumes taken every hour (2). The sample should be collected in a glass or polyethylene container, refrigerated after collection, and shipped to arrive at ABC within 24 hours after collection. A one-gallon sub-sample of the 24-hour composite will be satisfactory for the toxicity test(s) required. ABC can provide sample containers and mailing cartons and can assist plant personnel in shipping arrangements so that the sample arrives at ABC in a timely manner.

5.4 Dilution Water. It will not be necessary to collect actual receiving water for effluent dilutions for this test. The biomonitoring regulations recommend the use of reconstituted freshwater or saltwater listed in the EPA effluent methods manual (3). The freshwater dilution water (for Daphnia sp.) will be a hard reconstituted water of 160-180 mg/l hardness (as $CaCO_3$). The reconstituted saltwater (for Mysid sp.) will be prepared from a commercial salt mix similar in composition to that listed in the EPA effluent manual (3) and adjusted to a test salinity of 15-25 ppt.

5.5 Screen Test. The biomonitoring regulations require that a 24-hour 100% effluent screen be conducted before more definitive (LC_{50}) testing is considered. As stated in section 4.0, if ≧20% of the test organisms die in the 100% solution, the permittee shall then conduct a 48-hour static replacement LC_{50} test. This latter test is discussed in section 5.6.

The screen test will be conducted in 250 ml glass beakers containing 200 ml of solution. These test chambers will be held in a controlled temperature area at 20 ±2°C on a 16-hour daylight vs. 8-hour darkness photoperiod with 30-minute dawn/dusk transition periods. The screen test will include duplicate test chambers at 100% vs. duplicate control chambers containing dilution water only. Ten test organisms will be placed in each replicate; therefore, twenty total organisms will be exposed to the 100% effluent and control (dilution) water.

After a 24-hour exposure period, the test chambers will be observed for mortality and abnormal effects. If the combined replicate mortality is ≧20%, the 48-hour LC_{50} test will be initiated.

5.6 48-Hr LC_{50} Test. The definitive test will be conducted by exposing the test organisms to at least 5 effluent concentrations and a dilution water control. The test will be conducted using duplicate beakers per concentration containing 10 test organisms per beaker (=20 per concentration). The test concentrations used will follow one of the logarithmic series presented in Table 1. The most common concentration series used is 10-100% (10, 18, 32, 56 and 100%). Several effluent toxicity tests conducted by ABC have shown that most industrial effluents are toxic within this range.

The test organisms will be placed in the test chambers by stratified random assignment within 30 minutes after solution preparations. The test chambers will be observed for mortality and/or abnormal effects every 24 hours, with a record maintained of mortality and abnormal behavior for each concentration tested.

5.7 Test Procedure - Chemical and Physical. Upon receipt of the test effluent, the following parameters will be measured of the 100% effluent: temperature, dissolved oxygen, pH, conductivity and hardness. These same parameters, except hardness, will be measured at 0 and 48 hours of definitive testing in one replicate of the control, low concentration, middle concentration and high concentration solutions.

5.8 Analysis of Results. The results of the definitive study will be statistically analyzed for 24 and 48 hour LC_{50} values and their corresponding 95 percent confidence limits. These values will be determined by an LC_{50} computer program developed by Stephan et al. (11) or by manual methods described by Litchfield-Wilcoxon (12) and Stephan (13).

5.9 Report. One copy of the report will be submitted. The report will contain all original raw data. Additional copies will be provided, if requested by the sponsor, at a copy rate of $50 per copy. The report will include the following:

5.9.1 Study dates, name and address of test facility.

5.9.2 Objectives and test methods.

5.9.3 Reference to the statistical methods used for data analysis.

5.9.4 Description of test effluent (date of receipt, storage conditions, physical characteristics, and method of preparing test concentrations).

5.9.5 Description of test design.

5.9.6 Summary of the data analysis, mortality observations and test water quality.

5.9.7 Location of raw data.

5.9.8 List and signatures of study personnel.

5.9.9 GLP compliance statement by study director and a statement by ABC's Quality Assurance Unit.

5.9.10 The report appendix will contain the original raw data for mortality observations and water quality, letter of test authorization, letters of authorized protocol changes and a copy of the approved protocol.

5.10 Data Retention. All original raw data generated in the preliminary and definitive studies will be provided to the study sponsor in the appendix to the final report. A copy of the data will be retained in ABC's archives.

6.0 PROTOCOL CHANGES

In the event that modifications of this protocol are deemed necessary, a written statement of any changes and reason(s) proposed by the study sponsor or ABC will be submitted to the other party. All agreed changes will be expressed in writing, signed and dated by the sponsor's study director. The signed changes will be appended to the protocol and included with the final report. This protocol is subject to revision when felt necessary to comply with changes in federal regulations and state of the art methodology. The protocol for a specific study will not be changed while the study is in progress without discussion with the study sponsor.

7.0 SPONSOR AUTHORIZATIONS DURING THE STUDY

Should a problem develop while the study is in progress, ABC will notify the study director within 24 hours. The problem and suggested test modifications will be discussed by telephone. ABC will proceed with the changes felt necessary upon the verbal authorization of the study director. A letter for written authorization will then be submitted by ABC to the study director and handled in the same manner discussed in section 6.0.

8.0 TEST-SPECIFIC INFORMATION

8.1.1 General. The following items will be addressed for each static bioassay. This information is necessary to be in compliance with Good Laboratory Practice Regulations (8). Sections 8.2, 8.3, 8.7 and 8.9 are to be completed by the study sponsor. Sections 8.4, 8.5 and 8.6 will be completed by ABC.

8.1.2 GLP Compliance. To be in compliance with the Federal Good Laboratory Practice regulations, the report of the investigation conducted utilizing this protocol must contain a statement that the study was conducted in accordance with one of the following GLP's.

1. U.S. F.D.A. Good Laboratory Practice Regulations (21 CFR 58).

2. U.S. E.P.A. Good Laboratory Practice Standards; Toxic Substances Control (40 CFR 792).

3. U.S. E.P.A. Good Laboratory Practice Standards; Pesticide Programs (40 CFR 160).

4. OECD Principles of Good Laboratory Practice; Annex 2 C(81) 30(Final).

5. Any state or local GLP which may apply.

The specific GLP regulation, for which this study specific protocol and resultant study must be in compliance with, is to be designated by the study sponsor in section 8.8 of this protocol.

8.2 Study Sponsor:

8.2.1 Company Molycorp, Inc., Questa Division

8.2.2 Address P. O. Box 469
Questa, NM 87556

8.2.3 Sponsor's Study Director

John Stribling — Environmental Engineer
Name — Title

8.3 Effluent:

8.3.1 Sample Number Outfall 91185-002

8.3.2 NPDES Permit Number 002230 6

8.3.3 Physical Description clear liquid

8.3.4 Handling Precautions Normal precautions

8.4 Test Concentrations: To be determined after 24-hour screen of 100% effluent and consultation with study sponsor.

8.4.1 Sponsor's study director notified of screen results:

Date _____ ABC Biologist _____

100% effluent screen = _____ ≥20% mortality _____ <20% mortality

8.4.2 Definitive test concentrations agreed upon:

8.5 Test Organisms:

8.5.1 Species Daphnia magna

8.5.2 Supplier ABC in-house culture

8.6 Study Dates:

8.6.1 Proposed starting date of definitive study Sept., 1985

8.6.2 Proposed completion date of definitive study Oct., 1985

8.7 ABC Study Personnel:

8.7.1 Study Director

Alan D. Forbis — Aquatic Biologist III
Name — Title

8.7.2 Aquatic Supervisor

Carl M. Thompson — Supervisor, Aquatic Toxicology
Name — Title

8.8 GLP Compliance (check the most appropriate):

EPA-FIFRA____; EPA-TSCA____; FDA____; OECD____; Other UPES.

8.9 Protocol Approvals. The following is to be signed by the appropriate study personnel:

8.9.1 Sponsor's Study Director

[signature] — Environmental Eng. — 9-19-85
Name — Title — Date

8.9.2 ABC's Study Director

[signature] — Aquatic Biologist III — 9/16/85
Name — Title — Date

8.9.3 ABC's Group Leader

[signature] — Group Leader, Animal Toxicology — 9/16/85
Name — Title — Date

8.10 Authorization to return the test material and sample:

8.10.1 Sponsor's authorized agent to receive the test material and sample.

[signature] — Environmental Engineer
Name — Title

8.10.2 Shipping Address.

Molycorp Inc.
Company

3 miles East of Questa Hwy. 38
Street Address

Questa, N.M. 87556
City — State — Zip Code

505-586-0212
Phone Number

8.10.3 Hazardous shipment information.

Proper Shipping Name

Class of Restricted Article (e.g. flammable, poison, radioactive, etc.)

TABLE 1: Guide to selection of experimental concentrations based on progressive bisection of intervals on logarithmic scale.

Col. 1	Col. 2	Col. 3	Col. 4	Col. 5
10.0	10.0	10.0	10.0	10.0
				8.7
			7.5	7.5
				6.5
		5.6	5.6	5.6
				4.9
			4.2	4.2
				3.7
3.2	3.2	3.2	3.2	3.2
				2.8
			2.4	2.4
				2.1
		1.8	1.8	1.8
				1.55
			1.35	1.35
				1.15
1.0	1.0	1.0	1.0	1.0

TABLE 2: Chemical characteristics of well water used by ABC's Aquatic Toxicology Division.

Parameters	Concentration
Temperature	15-20°C[b]
Dissolved Oxygen[a]	9.2-10.1 ppm[b]
pH	7.8-8.3[b]
Hardness ($CaCO_3$)	225-275 ppm[b]
Alkalinity ($CaCO_3$)	325-375 ppm[b]
Conductivity	700 µmhos/cm
NH_3-N	0.60 ppm
NO_3-NO_2-N	0.20 ppm
Ortho-Phosphate	0.10 ppm
Aluminum	<0.010 ppm
Arsenic	<0.0005 ppm
Cadmium	<0.002 ppm
Chromium	<0.006 ppm
Cobalt	<0.003 ppm
Copper	<0.002 ppm
Iron	<0.004 ppm
Lead	0.017 ppm
Mercury	0.0008 ppm
Nickel	<0.013 ppm
Silver	<0.0004 ppm
Zinc	0.001 ppm
Measured organophosphorus pesticides	c
Measured organochlorine pesticides plus PCB's	c

[a]After aeration.

[b]Represents seasonal variation, with the monthly range not exceeding 10%.

[c]Below minimum detectable limits.

9.0 REFERENCES

(1) U.S. Congress. 1977. Clean Water Act. Public Law 95-217. Federal Register, Dec. 27, 1977, 91 STAT 1566-1611.

(2) U.S. Environmental Protection Agency. 1983. Effluent Toxicity Testing and Biomonitoring Workshop. Feb. 22-23, 1983, Dallas, Texas.

(3) Peltier, William. 1978. Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms. U.S. Environmental Protection Agency, EPA-660/4-78-012, revised July, 1978. 52 p.

(4) Committee on Methods for Toxicity Tests with Aquatic Organisms. 1975. Methods for Acute Toxicity Tests with Fish, Macroinvertebrates and Amphibians. Environmental Protection Agency, Ecological Research Series EPA-660/3-75-009, April, 1975. 61 p.

(5) American Society for Testing and Materials. 1980. Standard practice for conducting basic acute toxicity tests with fish, macroinvertebrates and amphibians. May, 1980, ASTM Committee E-35.23. 25 p.

(6) American Society for Testing and Materials. 1983. Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates and Amphibians. November, 1983, ASTM Committee E-47.01. 37 p.

(7) American Public Health Association. 1980. Standard Methods for the Examination of Water and Wastewater. 15th ed. Washington, D.C. 1134 p.

(8) (a) U.S. Food and Drug Administration. 1978. Nonclinical Laboratory Studies, Good Laboratory Practice Regulations (21 CFR, Part 58). Federal Register, Vol. 43, No. 247: 59986-60025.

(b) U.S. Environmental Protection Agency. 1983. Pesticide Programs; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 160). Federal Register, Vol 48, No. 230: 53946-53969.

(c) U.S. Environmental Protection Agency. 1983. Toxic Substances Control; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 792). Federal Register, Vol. 48; No. 230:53922-53944.

(d) Organization for Economic Cooperation and Development. 1981. OECD Guidelines for Testing of Chemicals, Principles of Good Laboratory Practice Annex 2, C(81) 30(Final):7-28.

(9) Pennak, R. W. 1978. Fresh-Water Invertebrates of the United States, 2nd Ed. 365 p.

(10) U.S. Environmental Protection Agency. 1979. Static culture methods for sheepshead minnows (Cyprinodon variegatus) and mysid shrimp (Mysidopsis bahia). Unpublished manuscript, U.S. EPA, Athens, GA.

(11) Stephan, C. E., K. A. Busch, R. Smith, J. Burke and R. W. Andrew. 1978. A computer program for calculating an LC_{50}. U. S. Environmental Protection Agency, Duluth, Minnesota, pre-publication manuscript, August, 1978.

(12) Litchfield, J. T., Jr. and F. Wilcoxon. 1949. A Simplified Method of Evaluating Dose-Effect Experiments. Jour. Pharm. Exp. Ther. 96:99-113.

(13) Stephan, C. 1977. Methods for calculating an LC_{50}, p. 65-84. In F. L. Mayer and J. L. Hamelick (eds.). Aquatic Toxicology and Hazard Evaluation. ASTM Special Technical Publication 634. ASTM. Philadelphia.

Assessment of Acute Toxicity of 002
Daphnia Magna

December 1985

ANALYTICAL BIO-CHEMISTRY LABORATORIES, INC.
P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

Static Acute Toxicity Report
#34190

Assessment of the Acute Toxicity of Outfall 002 to *Daphnia magna*

Submitted To:

Molycorp, Incorporated
Attn: Mr. John Stribling
3 miles East of Questa, on State Road 38
Questa, New Mexico 87556

December 26, 1985

Submitted By: Analytical Bio-Chemistry Laboratories, Inc.
7200 East ABC Lane
P. O. Box 1097
Columbia, Missouri 65205
(314) 474-8579

Prepared By:

Alan D. Forbis — 12/31/85
Alan D. Forbis — Date
Aquatic Biologist III/Study Director

David Burgess — 12/30/85
David Burgess — Date
Biologist II

Approved By:

Carl M. Thompson — 1-2-86
Carl M. Thompson — Date
Supervisor, Aquatic Toxicology

Kathy Landholt — 01/02/86
Kathy Landholt — Date
Quality Assurance Officer

Michael Williams — 1/2/86
Michael Williams, Ph.D. — Date
Group Leader, Animal Toxicology

/bsb

SUMMARY

The acute toxicity of Outfall 002 to Daphnia magna was assessed for U.S. E.P.A. Region 6 NPDES permit requirements using the methods outlined by the Committee on Methods for Toxicity Tests with Aquatic Organisms. Water quality parameters of temperature, dissolved oxygen, conductivity, hardness and pH were measured on receipt of the sample and were within acceptable limits.

The results of the 24-hour static Daphnia magna toxicity screen indicated that the mortality was <20% for Outfall 002. In fact, no mortality was observed after the 24-hour exposure period. Therefore, a definitive 48-hour static acute study was not required. All reported values were based upon nominal concentrations.

The no-effect level observed for Outfall 002 was 100% of effluent after 24-hours, which was based on the lack of mortality and abnormal effects.

INTRODUCTION

This static bioassay was performed by the Aquatic Toxicology Division of Analytical Bio-Chemistry Laboratories, Inc., Columbia, Missouri, for Molycorp, Inc. from December 11, 1985 to December 12, 1985. The purpose of this test was to assess toxicity of a 100% solution of Outfall 002 to Daphnia magna. The assessment was designed to meed the NPDES permit process in U.S. EPA Region 6 (1) requiring biomonitoring of industrial discharges. The screen test was conducted following the procedures outlined in ABC protocol #8301, as approved by Mr. John Stribling, Molycorp, Inc. on December 13, 1985.

METHODS AND MATERIALS

The procedures applied for the 24-hour toxicity screen were basically those described in Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms (2), Methods for Acute Toxicity Tests with Fish, Macroinvertebrates, and Amphibians (3), Standard Practice for Conducting Acute Toxicity Tests with Fishes, Macroinvertebrates, and Amphibians (4), Proposed Standard Practice for Conducting Acute Toxicity Test on Aqueous Effluents with Fishes, Macroinvertebrates, and Amphibians (5), and Standard Methods for the Examination of Water and Wastewater (6). The test lot of Daphnia magna were obtained from an in-house culture at ABC. The taxonomic key presented by Pennak (7) was used to identify to species the test daphnids. All test daphnids were held in a controlled temperature area at 20 ±2°C. The lighting was 50-70 footcandles on a 16-hour daylight photoperiod. During the holding period, they received a suspension of algae (Selenastrum capricornutum) supplemented with a blended fish food/cerophyl suspension at least every 3 days. First-instar daphnids (<24-hours old) were selected for testing and were not fed during the test.

The static daphnid bioassay was conducted in 250-ml glass beakers containing 200 ml of sample and/or ABC well water. The ABC well water control contained water with chemical characteristics listed in Table 1. These vessels were kept at 20 (±2.0) °C in a temperature controlled area. The lighting was maintained at 50-70 footcandles on a 16-hour daylight photoperiod.

A 24-hour screen test, composed of duplicate 100% effluent was performed to estimate the toxicity of the effluent. Also included was a duplicate ABC well water control. A five-six concentration static acute toxicity study would be performed if ≧20% mortality was observed after 24-hours in the screen test.

The Outfall 002 was received on December 11, 1985 as a clear liquid and was received at 3°C. The sample was allowed to warm to test temperature before addition of Daphnia.

RESULTS

After 24 hours, the results indicated that the mortality of *Daphnia magna* to Outfall 002 was <20%. The no-effect concentration based on the lack of mortality and abnormal effects was 100%. No abnormal effects, such as mortality, surfacing, and daphnids lying on the bottom were observed after 24-hours. Since mortality was not ≧20% after 24-hours in the screen test, a definitive study was not required per biomonitoring guidelines established for U.S. EPA Region 6.

Table 2 presents the water quality parameters measured on sample receipt. Hardness, alkalinity (as $CaCO_3$) and conductivity for the Outfall 002 sample were 508, 184 mg/l and 1300 μMHOS, respectively. Dissolved oxygen and pH for this sample were 10.0 mg/l and 7.2, respectively, and were considered adequate for testing (3).

The study was conducted following the intent of the Good Laboratory Practice Regulations (8) and the final report was reviewed by Analytical Bio-Chemistry Laboratories' Quality Assurance Unit. All original raw data was provided to Molycorp, Inc. with a copy retained at Analytical Bio-Chemistry Laboratories.

TABLE 1: Chemical Characteristics of Well Water Used by ABC's Aquatic Toxicology Division.

Parameters	Concentration
Temperature	15-20°C[b]
Dissolved Oxygen[a]	9.2-10.1 ppm[b]
pH	7.8-8.3[b]
Hardness ($CaCO_3$)	225-275 ppm[b]
Alkalinity ($CaCO_3$)	325-375 ppm[b]
Conductivity	700 μmhos/cm
NO_3-N	0.74 ppm
NO_3-NO_2-N	0.74 ppm
PO_4-P	<0.10 ppm
Aluminum	<20 ppb
Arsenic	<0.2 ppb
Cadmium	<2 ppb
Chromium	<3 ppb
Cobalt	<4 ppb
Copper	<3 ppb
Iron	12 ppb
Lead	<5 ppb
Mercury	<0.5 ppb
Nickel	<15 ppb
Silver	<5 ppb
Zinc	11 ppb
Measured organophosphorus pesticides	c
Measured organochlorine pesticides plus PCB's	c

[a]After aeration.

[b]Represents seasonal variation, with the monthly range not exceeding 10%.

[c]Less than minimum detectable limits.

TABLE 2

Water Quality Measurements During the Acute Toxicity Screen Test of Outfall 002 to *Daphnia magna*

	ABC Well Water		100% Outfall 002		
	0 hr.	24 hr.	On Receipt	0 hr.	24 hr
Temperature [a]	20	20	3	20	20
Dissolved Oxygen [b]	8.5	8.4	10.0	9.9	9.9
pH [c]	8.1	8.5	7.2	7.2	8.2
Conductivity [d]	350	400	1300	1300	1500
Hardness [e]	---	---	508	---	---
Alkalinity [e]	---	---	184	---	---

[a]Temperature °C - Monitored with a mercury thermometer.

[b]Dissolved oxygen concentrations (mg/l) - Dissolved Oxygen System (YSI Model 54). Saturation at 20°C is 9.2 mg/l.

[c]pH - pH Probe (Corning Model 476182) used with a Corning Model 125 pH and mV meter.

[d]Conductivity (μMHOS) - Conductivity Meter (YSI Model 33).

[e]Hardness and alkalinity (mg/l as $CaCO_3$) - HACH Model AL-DT and HAC-DT.

LITERATURE CITED

(1) U.S. Environmental Protection Agency. 1983. Effluent Toxicity Testing and Biomonitoring Workshop. Feb. 22-23, 1983, Dallas, Texas.

(2) Peltier, William. 1978. Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms. U.S. Environmental Protection Agency, EPA-660/4-78-012, revised July 1978. 52 p.

(3) Committee on Methods for Toxicity Tests with Aquatic Organisms. 1975. Methods for Acute Toxicity Tests with Fish, Macroinvertebrates and Amphibians. Environmental Protection Agency, Ecological Research Series EPA-660/3-75-009, April 1975. 61 p.

(4) American Society for Testing and Materials. 1980. Standard practice for conducting basic acute toxicity tests with fish, macroinvertebrates and amphibians. May 1980, ASTM Committee E-35.23. 25 p.

(5) American Society for Testing and Materials. 1983. Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates and Amphibians. November 1983, ASTM Committee E-47.01. 37 p.

(6) American Public Health Association. 1980. Standard Methods for the Examination of Water and Wastewater. 15th ed. Washington, DC. 1134 p.

(7) Pennak, R. W. 1978. Freshwater Invertebrates of the United States. John Wiley & Sons, New York, 2nd ed. 803 p.

(8) (a) U.S. Food and Drug Administration. 1978. Nonclinical Laboratory Studies, Good Laboratory Practice Regulations (21 CFR, Part 58). Federal Register, Vol. 48, No. 247:59986-60025.

(b) U.S. Environmental Protection Agency. 1983. Pesticide Programs; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 160). Federal Register, Vol. 48, No. 230:53946-53969.

(c) U.S. Environmental Protection Agency. 1983. Toxic Substances Control; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 792). Federal Register, Vol. 48; No. 230:53922-53944.

(d) Organization for Economic Cooperation and Development. 1981. OECD Guidelines for Testing of Chemicals, Principles of Good Laboratory Practice Annex 2, C(81) 30(Final):7-28.

QUALITY ASSURANCE STATEMENT

Quality Assurance Statement for final report #34190 entitled, "Acute Toxicity of Outfall 002 to *Daphnia magna*," for John Stribling; Molycorp, Inc., Questa, New Mexico.

In accordance with ABC Laboratories' intent that all studies conducted at our facilities are designed and function in conformance with good laboratory practice regulations and the protocols for individual laboratory studies, an inspection of the final report for Outfall 002 was conducted and found to be in acceptable form by a member of our Quality Assurance Unit. A final inspection of all data and records on December 20, 1985, indicates that the report submitted to you is an accurate reflection of the study as it was conducted by ABC Laboratories.

Should you have any questions relating to the information provided in this statement or the function of our Quality Assurance Unit, please contact the QA Unit at your convenience.

Kathy Landholt 01/02/86

Kathy Landholt Date
Quality Assurance Officer

STUDY COMPLIANCE STATEMENT

Study Compliance Statement for ABC report #34190 entitled, "Acute Toxicity of Outfall 002 to Daphnia magna," for John Stribling; Molycorp, Inc., Questa, New Mexico.

In accordance with ABC Laboratories' intent that all aquatic toxicity tests conducted by our facility follow good laboratory practices, ABC's study director for the above test herein confirms that the study was conducted in compliance with the U.S. E.P.A. Good Laboratory Practice Standard; Toxic Substances Control (40 CFR 792).

All original raw data was sent to Molycorp, Inc., with a copy retained at Analytical Bio-Chemistry Laboratories.

Alan D. Forbis 12/31/85

Alan D. Forbis Date
ABC Study Director

APPENDIX - RAW DATA

NOTE: Some of the records which appear in this raw data appendix have been provided as photocopies of original records on file at ABC. This has been done by necessity for certain data which are used commonly in several studies at ABC. Such records include reference toxicity test data, standard solution preparations, organism culture logs, and well water chemical screen results.

EFFLUENT TOXICITY TEST
MORTALITY AND BEHAVIORAL OBSERVATIONS

Effluent Designation: OUTFALL 002
Industry: MOLYCORP
Study No.: 34190
ABC Study Director: A. FORBIS

Lab Form No.: 91

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P.O. Box 1097
Columbia, Missouri 65205

☐ Prelim. ☑ Definitive Test Species: D. magna (Lot #85-C-9)
Date Initiated: 12-11-85 (4:00 pm) Test Terminated: 12-12-85
Dilution Water: NONE No./Vessel: 10 Vessel Size: 250 ml

Effluent Conc. (%)	24 hours Dead	24 hours Obs.	48 hours Dead	48 hours Obs.	72 hours Dead	72 hours Obs.	96 hours Dead	96 hours Obs.
Control A	0	N						
B	0	N						
100 A	0	N						
B	0	N						
Observer Date	DB 12/12/85 3:30pm							

Remarks: N = Normal; LOE = Loss of Equilibrium; SUR = Surfacing

Prepared By: Darrel Burgess Date: 12/11/85
Checked By: A. Forbis Date: 12/12/85

WATER QUALITY FOR EFFLUENT SAMPLES

Test Material: OUTFALL 002

Study No.: 34190

Protocol No.: 8301

ABC Study Director: A. FORBIS

Lab Form No.: 28b

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane; P.O. Box 1097
Columbia, Missouri 65205

Concentration ~~mg/l~~ c/o	12/11/85 DAY 0 DB				12/12/85 DAY 1 DB				DAY				DAY			
	Temp °C	D.O.[a] mg/l	pH[b]	Cond. µMhos	Temp °C	D.O. mg/l	pH	Cond. µMhos	Temp °C	D.O. mg/l	pH	Cond. µMhos	Temp °C	D.O. mg/l	pH	
CONTROL	20	8.5	8.1	350	20	8.4	8.5	400								
100 (A)	20	9.9	7.2	1500	20	9.9	8.2	1500								

[a]Dissolved oxygen concentrations - Dissolved Oxygen Probe (YSI Dissolved Oxygen System Model 54).

[b]pH-pH Probe (Corning Model 476182) used with Corning Model 125 pH and mV meter.

c 100% Effluent on Receipt	Temp °C	D.O. mg/l	pH	Cond. µMhos	Hard mg/l	Alk. mg/l	~~NH_3 mg/l~~			Date	Int.
	3	10.0	7.2	1300	508	184				12-11-85	DB

Prepared By: David Bunzen Date: 12/11/85

Checked By: A. Foley Date: 12/11/85

Remarks:

STUDY# 034190

PREPARATION OF EFFLUENT CONCENTRATIONS (volume/volume)

Effluent Designation: OUTFALL 002

Industry: MOLYCORP

Study No.: 34190

ABC Study Director: A. FORBIS

Lab Form No.: 94

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane; P.O. Box 1097
Columbia, MO 65205

☐ PRELIMINARY ☑ DEFINITIVE

Dilution Water Type:

☐ ABC Well Water ☐ ABC Recon. Water

☐ Effluent Receiving Water _____ ☑ Other NONE

Sample No.	Aliquot Volume (ml)	Dilution Volume (ml)	Final Conc. (%)
1	—	200	CONTROL
2	200	—	100
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			

Remarks:

Prepared By: David Bunzen Date: 12/11/85

Checked By: A. Forbis Date: 12/11/85

Study Director: A. Forbis Date: 12/11/85

INVERTEBRATE CULTURE RECORD

Species: Daphnia magna

Lot No.: 85-C-9

Date Initiated: 11-19-85

Lab Form No: 52

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P. O. Box 1097
Columbia, MO 65205

Date	No. Adult Alive	Instar Present	Fed & Changed	Study I.D.	Comments	Obs. By:
11/19	—	~25	✓		300 ml ALGAE 2ml MIDGE FOOD	DB
11/21	—	~25	✓		" "	SF
11/24	25	—	✓		" "	DB
11/26	23	—	✓		" "	SF
11/29	23	~150	✓		" "	SF
12/1	23	~500	✓		" "	[illegible]
12/3	23	~40	✓		" "	SF
12/5	23	900	✓		" "	DB
12-8	23	~600	✓		" "	[illegible]
12-9				34029 — 160		SF
12-10	22	~100	✓		" "	SF
12/11				33847 — 120 34190 — 70 31171 — 70		DB
12/11	22	700	✓		" "	DB

"This is an exact copy of The original document"

By C. Wilhelm date 12-12-85

Prepared by: Daniel Burgess Date: 11/19/85

Checked by: A. Fah Date: 12/12/85

ANALYTICAL BIO-CHEMISTRY LABORATORIES, INC.
P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

Sample Date: April 2, 1985

ANALYSIS RESULTS FOR:

Aquatic Toxicology Division
Analytical Bio-Chemistry Laboratories, Inc.
7200 East ABC Lane
P. O. Box 1097
Columbia, Missouri 65205

Water Analysis Results

ABC Lab No.	33102
Sample I.D.	ABC Aquatic Lab Well Water
NO_3-N	0.74 mg/l
NO_3-NO_2-N	0.74 mg/l
PO_4-P	<0.10 mg/l
Al	<20 µg/l
Co	<4 µg/l
Fe	12 µg/l
As	<0.2 µg/l
Cd	<2 µg/l
Cr	<3 µg/l
Cu	<3 µg/l
Pb	<5 µg/l
Hg	<0.5 µg/l
Ni	<15 µg/l
Ag	<5 µg/l
Zn	11 µg/l

<Indicates less than, if present at all.

Del A. Koch
Chemist III

Please note that the original data for the analysis reported in this table are in ABC's archives.

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Sample Date: April 2, 1985

ANALYSIS RESULTS FOR:

Aquatic Toxicology Division
Analytical Bio-Chemistry Laboratories, Inc.
7200 East ABC Lane
P. O. Box 1097
Columbia, Missouri 65205

Analysis of Aquatic Lab Well Water for Organophosphates

ABC Lab No.	Customer I.D.	Compound	ppb
33103	Aquatic Lab Well Water	Diazinon	<0.10
		Methyl Parathion	<0.10
		Malathion	<0.10
		Ethyl Parathion	<0.10

Randy Ayling 6/25/85
Randy Ayling
Chemist I

Please note that the original data for the analysis reported in this table are in ABC's archives.

ANALYTICAL BIO-CHEMISTRY LABORATORIES, INC.
P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

Sample Date: April 2, 1985

ANALYSIS RESULTS FOR:

Aquatic Toxicology Division
Analytical Bio-Chemistry Laboratories, Inc.
7200 East ABC Lane
P. O. Box 1097
Columbia, Missouri 65205

Analysis of Aquatic Lab Well Water for Nonionic
Chlorinated Hydrocarbon Residue

ABC Lab No.	33103
Customer I.D.	ABC Aquatic Lab Well Water
Compound	ppb
αBHC	<0.01
Lindane	<0.01
Heptachlor	<0.01
Heptachlor Epoxide	<0.02
γChlordane	<0.02
αChlordane	<0.02
Aldrin	<0.01
Dieldrin	<0.03
p,p'-DDE	<0.03
o,p'-DDT	<0.05
p,p'-DDD	<0.05
p,p'-DDT	<0.05
Endrin	<0.05
Methoxychlor	<0.20
Toxaphene	<0.50
Aroclor 1242	<0.40
Aroclor 1016	<0.40
Aroclor 1254	<0.40
Aroclor 1260	<0.40
HCB	<0.01
Mirex	<0.10

<indicates less than, if present at all.

Randy Ayling 6/25/85
Randy Ayling
Chemist I

Please note that the original data for the analysis reported in this table are in ABC's archives.

MOLYCORP, INC.
QUESTA DIVISION

This sheet represents a chain of custody for effluent samples collected at OUTFALL 002 and WEST DIVERSION CHANNEL located in Taos County, Questa, New Mexico on 12-10-85.

Sample bottles were prepared by Molycorp employees in accordance with guidance set forth by the U.S. Environmental Protection Agency for the existing program permit. Sampling procedures were also followed in accordance with permit requirements.

Sample(s) Identification: 1. 002-12-10-85
2. WEST DIVERSION CHANNEL 12-10-85

Sample(s) collected at: 1. 10:11 AM LAST PORTION
2. 10:13 AM " "

By: [signature]
Environmental ~~[illegible]~~ ENG.

Sample(s) handed over to carrier by Molycorp employee:

DATE: 12-10-85

TIME: 10:45 AM

CARRIER: Timothy J Sugg

EMPLOYER: GARRETT Ind.

MOLYCORP PERSONNEL: John [illegible]

Sample(s) delivered to ABC Lab for analysis. Please sign and date this letter upon receipt of sample(s).

Sample received by: ABC Laboratories Inc.
7200 ABC Lane
P.O. Box 1097
Columbia, MO 65205
(314) 474-8579

for the following analyses: Toxicity testing

Received by: David Burgess
ABC Laboratories Inc.

Date: 12-11-85 10:30 AM

Please return this letter with the analytical results, signed and dated below.

David Burgess
ABC Laboratories Inc.

12-16-85
Date

DEFINITIVE CONCENTRATION NOTIFICATION

Firm: MOLYCORP
Address: P.O Box 469
City/State/Zip: Questa, NM 87556
Phone: ( )

Lab Form No.: 186
Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P.O. Box 1097
Columbia, MO 65202

Sponsor's Study Director: JOHN STRIBLING
Person notified: same (✓) Other:

Date of Notification: 12/12/85

Notification of preliminary testing results with the following definitive test levels agreed upon:

Test Material	ABC #	Test Species	Test Conc. ~~(mg/l)~~ c/o
OUTFALL 002	34190	D. magna	100

NOTIFIED JOHN STRIBLING OF THE RESULTS OF THE 24 HR TOX SCREEN. MORTALITY WAS <20%, SO NO DEFINITIVE TEST WAS NEEDED.

Notified By: [illegible] Date: 12/12/85

ANALYTICAL BIO-CHEMISTRY LABORATORIES, INC.
P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 314 • Answer Back (ABCLAB UD)

ABC PROTOCOL NO. 8301

(August 21, 1984)

Effluent Static Bioassay with *Daphnia magna* or Mysid Shrimp (*Mysidopsis bahia*) to Meet EPA Region 6 NPDES Biomonitoring Guidelines

ABC Study Number 34190

Test Material Outfall 002

Test Species *Daphnia magna*

RECEIVED

1.0 INTRODUCTION

The use of bioassays in the identification and control of industrial effluents is rapidly increasing. Indeed, aquatic toxicity tests are presently being incorporated into the NPDES guidelines of the Federal Clean Water Act (1). To assist industry in meeting requirements set forth by legislation and regulatory agencies, Analytical Bio-Chemistry Laboratories, Inc. offers a complete range of aquatic bioassay tests of industrial effluents.

As part of the NPDES permit process in U.S. EPA Region 6, a biomonitoring requirement has been imposed for certain permit holders in that area. Discharges subject to the new regulations in Region 6 (Texas, New Mexico, Oklahoma, Arkansas and Louisiana) will be required to submit aquatic toxicity data on their effluents to EPA on a periodic basis.

This ABC protocol discusses the biomonitoring guidelines in Region 6 and describes ABC's technical approach to assist industry in complying with these regulations.

2.0 OBJECTIVES

The primary objective of the toxicity test described herein is to evaluate the acute toxicity of an effluent to freshwater or marine invertebrates under static conditions. The species selected for this testing include *Daphnia magna* (freshwater effluent, <5 ppt salinity) and *Mysidopsis bahia* (saltwater effluent, ≧5 ppt salinity). The effluent will first be tested on a 100% effluent screen basis. If the screen results in ≧20% mortality of test organisms, a 48-hour, five concentration LC_{50} test will be conducted. An LC_{50} is the approximate concentration (percent) of test effluent that produces 50 percent mortality of test organisms after prescribed intervals (24 and 48 hours).

3.0 TESTING FACILITY

The study will be conducted by the Aquatic Toxicology Division of Analytical Bio-Chemistry Laboratories, Inc., 7200 East ABC Lane, P. O. Box 1097, Columbia, Missouri 65205 (314-474-8579).

4.0 REGION 6 BIOMONITORING REQUIREMENT

The following biomonitoring requirement is now being included in the NPDES permit process for many major industrial discharges in EPA Region 6. The permit language given here was that presented by the U.S. EPA at a regional workshop on the matter (2):

> The permittee shall determine if the effluent from this outfall is lethal to twenty (20) percent or greater of the culture of test organisms by use of the "Range-Finding Screening Test," set out in "Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms," EPA-600/4-78-012 (Rev. July, 1978) (3).

Organisms for this test shall be Daphnia sp. if the effluent is less than five (5) parts per thousand salinity or Mysid sp. if the effluent is greater than five (5) parts per thousand salinity. This screening test will be conducted within sixty (60) days of effectiveness of the biomonitoring requirements. Tests will be conducted once each quarter for a duration of two years utilizing a static method for 24 hours and following this dilution scheme only:

Effluent sample* - 100 percent by volume
Dilution water - 0 percent by volume

*24-hour composite; refrigerated after collection

If at any time during the two year testing period a test shows a lethality of twenty (20) percent or more of the test organisms, the permittee shall within twenty-four (24) hours conduct a replacement static 48-hour median lethal concentration (LC_{50}) test on the originally collected sample. Replacement of effluent samples shall be once per 24 hours. Organisms for this test shall be Daphnia sp. if the effluent is less than five (5) parts per thousand salinity and reconstituted fresh water (EPA-600/4-78-012 Section 4) shall be used for dilution. If the effluent contains greater than five (5) parts per thousands salinity, Mysid sp. shall be used as the test organism, and reconstituted seawater will be used as dilution water (EPA-600/4-78-012 Section 4). The remaining LC_{50} methodology is available in EPA-600/4-78-012.

All screening and LC_{50} test results shall be reported with the Discharge Monitoring Reports.

It should be noted by the permittee that slight changes from the above permit language (e.g., pH modification of effluent) may be incorporated into his specific permit. Attention should be given to the permit requirements for each effluent before testing is initiated. ABC requires that a copy of the permit's biomonitoring clause be forwarded to ABC prior to testing so that consideration can be given to pH modification, etc.

5.0 METHODS AND MATERIALS

5.1 Methodology. The methods applied for both the preliminary and definitive phases of this test are basically those described in Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms (3), Methods for Acute Toxicity Tests with Fish, Macroinvertebrates, and Amphibians (4), Standard Practice for Conducting Acute Toxicity Tests with Fishes, Macroinvertebrates, and Amphibians (5), Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates

and Amphibians (6), and Standard Methods for the Examination of Water and Wastewater (7). This protocol has been written to comply with the federal Good Laboratory Practice Regulations (8).

5.2 Test Organisms. The species recommended by the EPA for this testing are Daphnia sp. and Mysid sp. Daphnia sp. are to be used when the effluent salinity is <5 parts per thousand (ppt) and Mysid sp. are to be used when the effluent salinity is ≧5 ppt. The species maintained by ABC, and acceptable by EPA, for this testing are Daphnia magna and Mysidopsis bahia. The test lot of Daphnia magna will be obtained from an in-house culture at ABC. The taxonomic key presented by Pennak (9) will be used to identify to species the test daphnids. All test daphnids will be held in a controlled temperature area at 20 ±2°C. The lighting will be 50-70 footcandles on a 16 hour daylight photoperiod. During the holding period, they will receive a suspension of algae (Selenastrum capricornutum) supplemented with a blended fish food/cerophyl suspension at least every 3 days. First-instar daphnids (<24 hours old) will be selected for testing and will not be fed during the test.

Mysid shrimp (Mysidopsis bahia) will be obtained from a commercial supplier (e.g., Sea Plantations, Inc. or Gulf Breeze Specimens) and maintained in culture at ABC. Mysids will be cultured following the techniques described by the U.S. EPA (10). Test mysids will be selected from a sub-culture of immature individuals.

5.3 Effluent Sample. The sample submitted to ABC for testing must be a 24-hour composite sample, consisting of equal volumes taken every hour (2). The sample should be collected in a glass or polyethylene container, refrigerated after collection, and shipped to arrive at ABC within 24 hours after collection. A one-gallon sub-sample of the 24-hour composite will be satisfactory for the toxicity test(s) required. ABC can provide sample containers and mailing cartons and can assist plant personnel in shipping arrangements so that the sample arrives at ABC in a timely manner.

5.4 Dilution Water. It will not be necessary to collect actual receiving water for effluent dilutions for this test. The biomonitoring regulations recommend the use of reconstituted freshwater or saltwater listed in the EPA effluent methods manual (3). The freshwater dilution water (for Daphnia sp.) will be a hard reconstituted water of 160-180 mg/l hardness (as $CaCO_3$). The reconstituted saltwater (for Mysid sp.) will be prepared from a commercial salt mix similar in composition to that listed in the EPA effluent manual (3) and adjusted to a test salinity of 15-25 ppt.

5.5 Screen Test. The biomonitoring regulations require that a 24-hour 100% effluent screen be conducted before more definitive (LC_{50}) testing is considered. As stated in section 4.0, if ≧20% of the test organisms die in the 100% solution, the permittee shall then conduct a 48-hour static replacement LC_{50} test. This latter test is discussed in section 5.6.

The screen test will be conducted in 250 ml glass beakers containing 200 ml of solution. These test chambers will be held in a controlled temperature area at 20 ±2°C on a 16-hour daylight vs. 8-hour darkness photoperiod with 30-minute dawn/dusk transition periods. The screen test will include duplicate test chambers at 100% vs. duplicate control chambers containing dilution water only. Ten test organisms will be placed in each replicate; therefore, twenty total organisms will be exposed to the 100% effluent and control (dilution) water.

After a 24-hour exposure period, the test chambers will be observed for mortality and abnormal effects. If the combined replicate mortality is ≧20%, the 48-hour LC_{50} test will be initiated.

5.6 48-Hr LC_{50} Test. The definitive test will be conducted by exposing the test organisms to at least 5 effluent concentrations and a dilution water control. The test will be conducted using duplicate beakers per concentration containing 10 test organisms per beaker (=20 per concentration). The test concentrations used will follow one of the logarithmic series presented in Table 1. The most common concentration series used is 10-100% (10, 18, 32, 56 and 100%). Several effluent toxicity tests conducted by ABC have shown that most industrial effluents are toxic within this range.

The test organisms will be placed in the test chambers by stratified random assignment within 30 minutes after solution preparations. The test chambers will be observed for mortality and/or abnormal effects every 24 hours, with a record maintained of mortality and abnormal behavior for each concentration tested.

5.7 Test Procedure - Chemical and Physical. Upon receipt of the test effluent, the following parameters will be measured of the 100% effluent: temperature, dissolved oxygen, pH, conductivity and hardness. These same parameters, except hardness, will be measured at 0 and 48 hours of definitive testing in one replicate of the control, low concentration, middle concentration and high concentration solutions.

5.8 Analysis of Results. The results of the definitive study will be statistically analyzed for 24 and 48 hour LC_{50} values and their corresponding 95 percent confidence limits. These values will be determined by an LC_{50} computer program developed by Stephan et al. (11) or by manual methods described by Litchfield-Wilcoxon (12) and Stephan (13).

5.9 Report. One copy of the report will be submitted. The report will contain all original raw data. Additional copies will be provided, if requested by the sponsor, at a copy rate of $50 per copy. The report will include the following:

5.9.1 Study dates, name and address of test facility.

5.9.2 Objectives and test methods.

5.9.3 Reference to the statistical methods used for data analysis.

5.9.4 Description of test effluent (date of receipt, storage conditions, physical characteristics, and method of preparing test concentrations).

5.9.5 Description of test design.

5.9.6 Summary of the data analysis, mortality observations and test water quality.

5.9.7 Location of raw data.

5.9.8 List and signatures of study personnel.

5.9.9 GLP compliance statement by study director and a statement by ABC's Quality Assurance Unit.

5.9.10 The report appendix will contain the original raw data for mortality observations and water quality, letter of test authorization, letters of authorized protocol changes and a copy of the approved protocol.

5.10 Data Retention. All original raw data generated in the preliminary and definitive studies will be provided to the study sponsor in the appendix to the final report. A copy of the data will be retained in ABC's archives.

6.0 PROTOCOL CHANGES

In the event that modifications of this protocol are deemed necessary, a written statement of any changes and reason(s) proposed by the study sponsor or ABC will be submitted to the other party. All agreed changes will be expressed in writing, signed and dated by the sponsor's study director. The signed changes will be appended to the protocol and included with the final report. This protocol is subject to revision when felt necessary to comply with changes in federal regulations and state of the art methodology. The protocol for a specific study will not be changed while the study is in progress without discussion with the study sponsor.

7.0 SPONSOR AUTHORIZATIONS DURING THE STUDY

Should a problem develop while the study is in progress, ABC will notify the study director within 24 hours. The problem and suggested test modifications will be discussed by telephone. ABC will proceed with the changes felt necessary upon the verbal authorization of the study director. A letter for written authorization will then be submitted by ABC to the study director and handled in the same manner discussed in section 6.0.

8.0 TEST-SPECIFIC INFORMATION

8.1.1 General. The following items will be addressed for each static bioassay. This information is necessary to be in compliance with Good Laboratory Practice Regulations (8). Sections 8.2, 8.3, 8.7 and 8.9 are to be completed by the study sponsor. Sections 8.4, 8.5 and 8.6 will be completed by ABC.

8.1.2 GLP Compliance. To be in compliance with the Federal Good Laboratory Practice regulations, the report of the investigation conducted utilizing this protocol must contain a statement that the study was conducted in accordance with one of the following GLP's.

1. U.S. F.D.A. Good Laboratory Practice Regulations (21 CFR 58).

2. U.S. E.P.A. Good Laboratory Practice Standards; Toxic Substances Control (40 CFR 792).

3. U.S. E.P.A. Good Laboratory Practice Standards; Pesticide Programs (40 CFR 160).

4. OECD Principles of Good Laboratory Practice; Annex 2 C(81) 30(Final).

5. Any state or local GLP which may apply.

The specific GLP regulation, for which this study specific protocol and resultant study must be in compliance with, is to be designated by the study sponsor in section 8.8 of this protocol.

8.2 Study Sponsor:

8.2.1 Company Molycorp, Inc.

8.2.2 Address 3 miles east of Questa on State Road 58
P.O. Box 469, Questa, New Mexico 87556

8.2.3 Sponsor's Study Director

Mr. John Stribling — Environmental Eng.
Name — Title

8.3 Effluent:

8.3.1 Sample Number Outfall 002

8.3.2 NPDES Permit Number NM-0022306

8.3.3 Physical Description Clear liquid

8.3.4 Handling Precautions Normal precautions

8.4 Test Concentrations: To be determined after 24-hour screen of 100% effluent and consultation with study sponsor.

8.4.1 Sponsor's study director notified of screen results:
Date 12-12-85 ABC Biologist [illegible]
100% effluent screen = ____ ≧20% mortality ✓ <20% mortality

8.4.2 Definitive test concentrations agreed upon:
NO DEFINITIVE TEST

8.5 Test Organisms:

8.5.1 Species Daphnia magna

8.5.2 Supplier ABC in-house culture

8.6 Study Dates:

8.6.1 Proposed starting date of definitive study 12-11-85

8.6.2 Proposed completion date of definitive study 12-31-85

8.7 ABC Study Personnel:

8.7.1 Study Director

Alan D. Forbis — Aquatic Biologist III
Name — Title

8.7.2 Aquatic Supervisor

Carl M. Thompson	Aquatic Toxicology Supervisor
Name	Title

8.8 GLP Compliance (check the most appropriate):

EPA-FIFRA____; EPA-TSCA____; FDA____; OECD____; Other NPDES.

8.9 Protocol Approvals. The following is to be signed by the appropriate study personnel:

8.9.1 Sponsor's Study Director

John Strickling	Environmental Eng.	12-13-85 ①
Name	Title	Date

8.9.2 ABC's Study Director

Alan D. Forbes	Aquatic Biologist III	12-10-85
Name	Title	Date

8.9.3 ABC's Group Leader

Michael Wilson	Group Leader, Animal Toxicology	12-10-85
Name	Title	Date

8.10 Authorization to return the test material and sample:

8.10.1 Sponsor's authorized agent to receive the test material and sample.

John Strickling	Environmental Eng.
Name	Title

8.10.2 Shipping Address.

Molycorp Inc
Company

3 miles East of Questa Hwy. 38
Street Address

Questa	N.M.	87556
City	State	Zip Code

505-586-0212
Phone Number

8.10.3 Hazardous shipment information.

Proper Shipping Name

Class of Restricted Article (e.g. flammable, poison, radioactive, etc.)

① Due to the time restrictions for effluent testing following receipt of sample (within 24 hours), this protocol could [illegible] to the test

STUDY# 034190 0028 of 0032

TABLE 1: Guide to selection of experimental concentrations based on progressive bisection of intervals on logarithmic scale.

Col. 1	Col. 2	Col. 3	Col. 4	Col. 5
10.0	10.0	10.0	10.0	10.0
				8.7
			7.5	7.5
				6.5
		5.6	5.6	5.6
				4.9
			4.2	4.2
				3.7
3.2	3.2	3.2	3.2	3.2
				2.8
			2.4	2.4
				2.1
		1.8	1.8	1.8
				1.55
			1.35	1.35
				1.15
1.0	1.0	1.0	1.0	1.0

TABLE 2: Chemical characteristics of well water used by ABC's Aquatic Toxicology Division.

Parameters	Concentration
Temperature	15-20°C[b]
Dissolved Oxygen[a]	9.2-10.1 ppm[b]
pH	7.8-8.3[b]
Hardness ($CaCO_3$)	225-275 ppm[b]
Alkalinity ($CaCO_3$)	325-375 ppm[b]
Conductivity	700 µmhos/cm
NH_3-N	0.60 ppm
NO_3-NO_2-N	0.20 ppm
Ortho-Phosphate	0.10 ppm
Aluminum	<0.010 ppm
Arsenic	<0.0005 ppm
Cadmium	<0.002 ppm
Chromium	<0.006 ppm
Cobalt	<0.003 ppm
Copper	<0.002 ppm
Iron	<0.004 ppm
Lead	0.017 ppm
Mercury	0.0008 ppm
Nickel	<0.013 ppm
Silver	<0.0004 ppm
Zinc	0.001 ppm
Measured organophosphorus pesticides	c
Measured organochlorine pesticides plus PCB's	c

[a]After aeration.

[b]Represents seasonal variation, with the monthly range not exceeding 10%.

[c]Below minimum detectable limits.

9.0 REFERENCES

(1) U.S. Congress. 1977. Clean Water Act. Public Law 95-217. Federal Register, Dec. 27, 1977, 91 STAT 1566-1611.

(2) U.S. Environmental Protection Agency. 1983. Effluent Toxicity Testing and Biomonitoring Workshop. Feb. 22-23, 1983, Dallas, Texas.

(3) Peltier, William. 1978. Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms. U.S. Environmental Protection Agency, EPA-660/4-78-012, revised July, 1978. 52 p.

(4) Committee on Methods for Toxicity Tests with Aquatic Organisms. 1975. Methods for Acute Toxicity Tests with Fish, Macroinvertebrates and Amphibians. Environmental Protection Agency, Ecological Research Series EPA-660/3-75-009, April, 1975. 61 p.

(5) American Society for Testing and Materials. 1980. Standard practice for conducting basic acute toxicity tests with fish, macroinvertebrates and amphibians. May, 1980, ASTM Committee E-35.23. 25 p.

(6) American Society for Testing and Materials. 1983. Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates and Amphibians. November, 1983, ASTM Committee E-47.01. 37 p.

(7) American Public Health Association. 1980. Standard Methods for the Examination of Water and Wastewater. 15th ed. Washington, D.C. 1134 p.

(8) (a) U.S. Food and Drug Administration. 1978. Nonclinical Laboratory Studies, Good Laboratory Practice Regulations (21 CFR, Part 58). Federal Register, Vol. 43, No. 247: 59986-60025.

(b) U.S. Environmental Protection Agency. 1983. Pesticide Programs; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 160). Federal Register, Vol 48, No. 230: 53946-53969.

(c) U.S. Environmental Protection Agency. 1983. Toxic Substances Control; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 792). Federal Register, Vol. 48; No. 230:53922-53944.

(d) Organization for Economic Cooperation and Development. 1981. OECD Guidelines for Testing of Chemicals, Principles of Good Laboratory Practice Annex 2, C(81) 30(Final):7-28.

(9) Pennak, R. W. 1978. Fresh-Water Invertebrates of the United States, 2nd Ed. 365 p.

(10) U.S. Environmental Protection Agency. 1979. Static culture methods for sheepshead minnows (Cyprinodon variegatus) and mysid shrimp (Mysidopsis bahia). Unpublished manuscript, U.S. EPA, Athens, GA.

(11) Stephan, C. E., K. A. Busch, R. Smith, J. Burke and R. W. Andrew. 1978. A computer program for calculating an LC_{50}. U. S. Environmental Protection Agency, Duluth, Minnesota, pre-publication manuscript, August, 1978.

(12) Litchfield, J. T., Jr. and F. Wilcoxon. 1949. A Simplified Method of Evaluating Dose-Effect Experiments. Jour. Pharm. Exp. Ther. 96:99-113.

(13) Stephan, C. 1977. Methods for calculating an LC_{50}, p. 65-84. In F. L. Mayer and J. L. Hamelick (eds.). Aquatic Toxicology and Hazard Evaluation. ASTM Special Technical Publication 634. ASTM. Philadelphia.

Assessment of Acute Toxicity of 002
Daphnia Magna

April 1986

ANALYTICAL BIO-CHEMISTRY LABORATORIES, INC.
P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

Static Acute Toxicity Report
#34534

Assessment of the Acute Toxicity of West Diversion Channel to *Daphnia magna*

Submitted To:
Molycorp, Incorporated
Attn: Mr. Fred Martinez
3 miles East of Questa, on State Road 38
P. O. Box 469
Questa, New Mexico 87556

April 21, 1986

ANALYTICAL BIO-CHEMISTRY LABORATORIES, INC.
P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

AMENDMENT TO STUDY PROTOCOL

STUDY TITLE: Assessment of the Acute Toxicity of West Diversion Channel to *Daphnia magna*.

PROTOCOL NUMBER: 8301 AMENDMENT NUMBER: 1

LABORATORY: ABC Laboratories, Inc. ABC STUDY NUMBER: 34534

EFFECTIVE DATE: 4/15/86

Section 5.5 Screen Test

One of the duplicate control chambers was lost during the 24-hour screen test. Since no mortality or abnormal effects were observed in the remaining control chamber, the loss of this replicate did not affect the results of the screen test.

SPONSOR'S STUDY DIRECTOR: Fred [illegible] Date: 04/30/86

STUDY DIRECTOR: [illegible] Date: 4/22/86

SUMMARY

The acute toxicity of West Diversion Channel to Daphnia magna was assessed for U.S. EPA Region 6 NPDES permit requirements using the methods outlined by the Committee on Methods for Toxicity Tests with Aquatic Organisms. Water quality parameters of temperature, dissolved oxygen, conductivity, hardness, alkalinity and pH were measured on receipt of the sample and were within acceptable limits.

The results of the 24-hour static Daphnia magna toxicity screen indicated that the mortality was <20% for West Diversion Channel. In fact, no mortality was observed after the 24-hour exposure period. Therefore, a definitive 48-hour static acute study was not required. All reported values were based upon nominal concentrations.

The no-effect level for West Diversion Channel was estimated to be <100% effluent after 24-hours, since surfacing was observed in one of the 100% effluent replicates.

Submitted By: Analytical Bio-Chemistry Laboratories, Inc.
7200 East ABC Lane
P. O. Box 1097
Columbia, Missouri 65205
(314) 474-8579

Prepared By:

Alan D. Forbis — 4/21/86
Alan D. Forbis — Date
Aquatic Biologist III/Study Director

Scott Frazier — 4/21/86
Scott Frazier — Date
Biologist I

Approved By:

Carl M. Thompson — 4-22-86
Carl M. Thompson — Date
Supervisor, Aquatic Toxicology

Kathy Landholt — 04/22/86
Kathy Landholt — Date
Quality Assurance Officer

Michael Williams — 4/22/86
Michael Williams, Ph.D. — Date
Group Leader, Animal Toxicology

/jh

INTRODUCTION

This static bioassay was performed by the Aquatic Toxicology Division of Analytical Bio-Chemistry Laboratories, Inc., Columbia, Missouri, for Molycorp, Inc., from March 26, 1986 to March 27, 1986. The purpose of this test was to assess toxicity of a 100% solution of West Diversion Channel to Daphnia magna. The assessment was designed to meet the NPDES permit process in U.S. EPA Region 6 (1) requiring biomonitoring of industrial discharges. The screen test was conducted following the procedures outlined in ABC protocol #8301, as approved by Mr. Fred Martinez; Molycorp, Inc. on April 2, 1986.

METHODS AND MATERIALS

The procedures applied for the 24-hour toxicity screen were basically those described in Methods of Measuring the Acute Toxicity of Effluents to Aquatic Organisms (2), Methods for Acute Toxicity Tests with Fish, Macroinvertebrates, and Amphibians (3), Standard Practice for Conducting Acute Toxicity Tests with Fishes, Macroinvertebrates and Amphibians (4), Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates and Amphibians (5), and Standard Methods for the Examination of Water and Wastewater (6). The test lot of Daphnia magna were obtained from an in-house culture at ABC. The taxonomic key presented by Pennak (7) was used to identify to species the test daphnids. All test daphnids were held in a controlled temperature area at 20 ±2°C. The lighting was 50-70 footcandles on a 16-hour daylight photoperiod. During the holding period, they received a suspension of algae (Selenastrum capricornutum) supplemented with a blended fish food suspension at least every 3 days. First-instar daphnids (<24-hours old) were selected for testing and were not fed during the test.

The static Daphnia bioassay was conducted in 250-ml glass beakers containing 200 ml of ABC aged well water with the chemical characteristics listed in Table 1. These vessels were kept at 20 (±2.0) °C in a temperature controlled area. The lighting was maintained at 50-70 footcandles on a 16-hour daylight photoperiod.

A 24-hour screen test, composed of duplicate 100% effluent was performed to estimate the toxicity of the effluent. Also included was a duplicate ABC well water control. A five-six concentration static acute toxicity study would be performed if ≧20% mortality was observed after 24-hours in the screen test.

The West Diversion Channel sample (NPDES Permit Number NM-0022306) was received on March 26, 1986 as a clear liquid and was received at 8°C. The samples were allowed to warm to test temperature before Daphnids were added to test solutions.

LITERATURE CITED

(1) U. S. Environmental Protection Agency. 1983. Effluent Toxicity Testing and biomonitoring Workshop. Feb. 22-23, 1983, Dallas, Texas.

(2) Peltier, William. 1978. Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms. U.S. Environmental Protection Agency, EPA-660/4-78-012, revised July 1978. 52 p.

(3) Committee on Methods for Toxicity Tests with Aquatic Organisms. 1975. Methods of Acute Toxicity Tests with Fish, Macroinvertebrates and Amphibians. Environmental Protection Agency, Ecological Research Series EPA-660/3-75-009, April 1975.

(4) American Society for Testing and Materials. 1980. Standard Practice for Conducting Basic Acute Toxicity Tests with Fish, Macroinvertebrates and Amphibians. May 1980, ASTM Committee E-35.23. 25 p.

(5) American Society for Testing and Materials. 1983. Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates and Amphibians. November 1983, ASTM Committee E-47.01 37 p.

(6) American Public Health Association. 1980. Standard Methods for the Examination of Water and Wastewater. 15th ed. Washington, DC. 1134 p.

(7) Pennak, R. W. 1978. Freshwater Invertebrates of the United States. John Wiley & Sons, New York, 2nd ed. 803 p.

(8) (a) U.S. Food and Drug Administration. 1978. Nonclinical Laboratory Studies, Good Laboratory Practice Regulations (21 CFR, Part 58). Federal Register, Vol. 43, No. 247:59986-60025.

(b) U.S. Environmental Protection Agency. 1983. Pesticide Programs; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 160). Federal Register, Vol. 48, No. 230:53946-53969.

(c) U.S. Environmental Protection Agency. 1983. Toxic Substances Control; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 792). Federal Register, Vol. 48; No. 230:53922-53944.

(d) Organization for Economic Cooperation and Development. 1981. OECD Guidelines for Testing of Chemicals, Principles of Good Laboratory Practice Annex 2, C(81) 30(Final):7-28.

QUALITY ASSURANCE STATEMENT

Quality Assurance Statement for final report #34534 entitled, "Acute Toxicity of West Diversion Channel to *Daphnia magna*," for Mr. Fred Martinez; Molycorp, Inc., Questa, New Mexico.

In accordance with ABC Laboratories' intent that all studies conducted at our facilities are designed and function in conformance with good laboratory practice regulations and the protocols for individual laboratory studies, an inspection of the final report for West Diversion Channel was conducted and found to be in acceptable form by a member of our Quality Assurance Unit. A final inspection of all data and records on April 15, 1986, indicates that the report submitted to you is an accurate reflection of the study as it was conducted by ABC Laboratories.

Should you have any questions relating to the information provided in this statement or the function of our Quality Assurance Unit, please contact the QA Unit at your convenience.

Kathy Landholt 04/22/86

Kathy Landholt Date

Quality Assurance Officer

STUDY COMPLIANCE STATEMENT

Study Compliance Statement for ABC report #34534 entitled, "Acute Toxicity of West Diversion Channel to *Daphnia magna*," for Mr. Fred Martinez; Molycorp, Inc., Questa, New Mexico.

In accordance with ABC Laboratories' intent that all aquatic toxicity tests conducted by our facility follow good laboratory practices, ABC's study director for the above test herein confirms that the study was conducted in compliance with the U.S. E.P.A. Good Laboratory Practice Standards; Toxic Substances Control (40 CFR 792).

All original raw data was sent to Molycorp, Inc., with a copy retained at Analytical Bio-Chemistry Laboratories.

Alan D. Forbis 4/22/86

Alan D. Forbis — Date
ABC Study Director

APPENDIX - RAW DATA

NOTE: Some of the records which appear in this raw data appendix have been provided as photocopies of original records on file at ABC. This has been done by necessity for certain data which are used commonly in several studies at ABC. Such records include reference toxicity test data, standard solution preparations, organism culture logs, and well water chemical screen results.

To be handed over to carrier by Molycorp employee:

DATE: 3/25/86

TIME: 11:45 AM

CARRIER: Fred Martinez

EMPLOYER: Moly Corp Inc union 76

MOLYCORP PERSONNEL: Fred Martinez

Sample(s) delivered to ABC Lab for analysis. Please sign and date this letter upon receipt of sample(s).

Sample received by: ABC Laboratories Inc.
7200 ABC Lane
P.O. Box 1097
Columbia, MO 65205
(314) 474-8579

for the following analyses: Toxicity testing

Received by: Scott Frazier

Date: 3/26/86 10:30 AM

Please return this letter with the analytical results, signed and dated below.

Scott Frazier 4/15/86
Date

EFFLUENT TOXICITY TEST
MORTALITY AND BEHAVIORAL OBSERVATIONS

Effluent Designation: WEST - DIVERSION CHANNEL
Industry: MOLY CORP
Study No.: 34534
ABC Study Director: A. FORBIS

Lab Form No.: 91

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P.O. Box 1097
Columbia, Missouri 65205

☐ Prelim. ☑ Definitive Test Species: Daphnia magna (Lot #86-B-2)
Date Initiated: 3/26/86 3:00 pm Test Terminated: 3/27/86
Dilution Water: NONE No./Vessel: 10 Vessel Size: 250 ml

Effluent Conc. (%)	24 hours Dead	24 hours Obs.	48 hours Dead	48 hours Obs.	72 hours Dead	72 hours Obs.	96 hours Dead	96 hours Obs.
Control A	0	N						
B		*						
100 A	0	1-SUR 9-N						
B	0	N						
Observer	SF 2:30 pm							
Date	3/27/86							

Remarks: N = Normal; LOE = Loss of Equilibrium;
SUR = Surfacing

* CONTROL B REPLICATE LOST, SF, 3/27/86

Prepared By: Scott Sagner Date: 3/26/86
Checked By: A. Forbis Date: 4/5/86

WATER QUALITY FOR EFFLUENT SAMPLES

Test Material: WEST DIVERSION CHANNEL

Study No.: 34534

Protocol No.: 8301

ABC Study Director: A. FORBIS

Lab Form No.: 28b

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane; P.O. Box 1097
Columbia, Missouri 65205

Concentration mg/l	DAY				DAY				DAY				DAY			
	Temp °C	D.O.[a] mg/l	pH[b]	Cond. µMhos	Temp °C	D.O. mg/l	pH	Cond. µMhos	Temp °C	D.O. mg/l	pH	Cond. µMhos	Temp °C	D.O. mg/l	pH	

[a] Dissolved oxygen concentrations - Dissolved Oxygen Probe (YSI Dissolved Oxygen System Model 54).

[b] pH-pH Probe (Corning Model 476182) used with Corning Model 125 pH and mV meter.

[c] 100% Effluent on Receipt	Temp °C	D.O. mg/l	pH	Cond. µMhos	Hard mg/l	Alk. mg/l	~~NH_3 mg/l~~			Date	Int.
	8	9.3	7.3	1000	730	46				3/26/86	SF

Prepared By: Scott Frazier Date: 3/26/86

Checked By: A. Forbis Date: 4/15/86

Remarks:

STUDY# 034534

Pg. 0011 of 0033

PREPARATION OF EFFLUENT CONCENTRATIONS (volume/volume)

Effluent Designation: WEST DIVERSION CHANNEL

Industry: MOLY CORP

Study No.: 34534

ABC Study Director: A. FORBIS

Lab Form No.: 94

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane; P.O. Box 1097
Columbia, MO 65205

☐ PRELIMINARY ☑ DEFINITIVE

Dilution Water Type:

☐ ABC Well Water ☐ ABC Recon. Water

☐ Effluent Receiving Water _____ ☑ Other NONE

Sample No.	Aliquot Volume (ml)	Dilution Volume (ml)	Final Conc. (%)
1	—	200	ABC CONTROL*
2	200	—	100
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			

Remarks: * ABC CONTROL = ABC WELL WATER

Prepared By: Scott Singer Date: 3/26/86

Checked By: A. Forbis Date: 3/26/86

Study Director: Alan D. Forbis Date: 4/15/86

INVERTEBRATE CULTURE RECORD

Species: Daphnia magna

Lot No.: 86-B-2

Date Initiated: 2/26/86

Lab Form No: 52

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P. O. Box 1097
Columbia, MO 65205

Date	No. Adult Alive	Instar Present	Fed & Changed	Study I.D.	Comments	Obs. By:
2/26/86	—	~30	✓		350 ml algae 1 ml AP-100 suspension	SF
3/~~27~~ 2/86	31	0	✓		" "	PC ① ~~SF~~
3/4	30	~80	✓		" "	SF
3/6	30	~80	✓		" "	SF
3/9	reduced to 25	~500	✓		" "	AF
3/11	24	~300	✓		" "	LS
3/13	24	~100	✓		" "	LO
~~3/14~~ ②				~~[illegible]~~ ②		
3/16	24	~300	✓		" "	WAM
3/[illegible]	24	~180	✓		" "	LS
3/19				34343-200		SF
3/20	24	~300	✓		~1 ml AP-100 suspension ~350 ml Algae	SF
3/23	24	~200	✓		" "	LS
3/25	24	~80	✓		" "	SF
3/26				34[illegible]-30 34[illegible]-30 34440-160		SF
3/27	24	~100	✓		~350 ml Algae ~1 ml AP-100 suspension	SF
3-30	23	~250	✓		" "	JHB
4/1	23	~200	✓		" "	LS

Prepared by: Scott Frazier Date: 2/26/86

Checked by: James Sweget Date: 4-3-86

① Entry Error SF 2/27/86
② Entry Error LS 3/14/86

"This is an exact copy of The original document"

By H. Seaton date 4/3/86

This sheet represents a chain of custody for effluent samples collected at out Fall 002 and West Diversion Channel located in Taos County, Questa, New Mexico on 3/25/86

Sample bottles were prepared by Molycorp employees in accordance with guidance set forth by the U.S. Environmental Protection Agency for the existing program permit. Sampling procedures were also followed in accordance with permit requirements.

Sample(s) identification: 002 3/25/86
West Diversion channel 3/25/86

Sample(s) collected at: 10:50 AM 1st. portion *
11:30 ~~AM~~ " " *

* composite sample as required:

By: Fred Martinez
Environmental Water Analyst

DEFINITIVE CONCENTRATION NOTIFICATION

Firm: Molycorp INC
Address: P.O. Box 469
City/State/Zip: Questa, NM 87556
Phone: (505) 586-0212 Ext 206

Lab Form No.: 186
Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P.O. Box 1097
Columbia, MO 65202

Sponsor's Study Director: FRED MARTINEZ
Person notified: same (✓) Other:

Date of Notification: 3/28/86

Notification of preliminary testing results with the following definitive test levels agreed upon:

Test Material	ABC #	Test Species	Test Conc. (mg/l)
WEST DIVERSION CHANNEL	34534	Daphnia magna	N/A

Informed Mr. Fred Martinez that no mortality was observed in the 24 screen test of West Diversion Channel Effluent with Daphnia magna.

Notified By: Scott Hazen Date: 3/28/86

ANALYTICAL BIO-CHEMISTRY LABORATORIES, INC.
P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

Sample Date: April 2, 1985

ANALYSIS RESULTS FOR:

Aquatic Toxicology Division
Analytical Bio-Chemistry Laboratories, Inc.
7200 East ABC Lane
P. O. Box 1097
Columbia, Missouri 65205

Water Analysis Results

ABC Lab No.	33102
Sample I.D.	ABC Aquatic Lab Well Water
NO_3-N	0.74 mg/l
NO_3-NO_2-N	0.74 mg/l
PO_4-P	<0.10 mg/l
Al	<20 µg/l
Co	<4 µg/l
Fe	12 µg/l
As	<0.2 µg/l
Cd	<2 µg/l
Cr	<3 µg/l
Cu	<3 µg/l
Pb	<5 µg/l
Hg	<0.5 µg/l
Ni	<15 µg/l
Ag	<5 µg/l
Zn	11 µg/l

<Indicates less than, if present at all.

Del A. Koch
Chemist III

Please note that the original data for the analysis reported in this table are in ABC's archives.

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P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

Sample Date: April 2, 1985

ANALYSIS RESULTS FOR:

Aquatic Toxicology Division
Analytical Bio-Chemistry Laboratories, Inc.
7200 East ABC Lane
P. O. Box 1097
Columbia, Missouri 65205

Analysis of Aquatic Lab Well Water for Organophosphates

ABC Lab No.	Customer I.D.	Compound	ppb
33103	Aquatic Lab Well Water	Diazinon	<0.10
		Methyl Parathion	<0.10
		Malathion	<0.10
		Ethyl Parathion	<0.10

Randy Ayling 6/25/85
Randy Ayling
Chemist I

Please note that the original data for the analysis reported in this table are in ABC's archives.

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P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

Sample Date: April 2, 1985

ANALYSIS RESULTS FOR:

Aquatic Toxicology Division
Analytical Bio-Chemistry Laboratories, Inc.
7200 East ABC Lane
P. O. Box 1097
Columbia, Missouri 65205

Analysis of Aquatic Lab Well Water for Nonionic
Chlorinated Hydrocarbon Residue

ABC Lab No.	33103
Customer I.D.	ABC Aquatic Lab Well Water
Compound	ppb
αBHC	<0.01
Lindane	<0.01
Heptachlor	<0.01
Heptachlor Epoxide	<0.02
γChlordane	<0.02
αChlordane	<0.02
Aldrin	<0.01
Dieldrin	<0.03
p,p'-DDE	<0.03
o,p'-DDT	<0.05
p,p'-DDD	<0.05
p,p'-DDT	<0.05
Endrin	<0.05
Methoxychlor	<0.20
Toxaphene	<0.50
Aroclor 1242	<0.40
Aroclor 1016	<0.40
Aroclor 1254	<0.40
Aroclor 1260	<0.40
HCB	<0.01
Mirex	<0.10

<indicates less than, if present at all.

Randy Ayling 6/25/85
Randy Ayling
Chemist I

Please note that the original data for the analysis reported in this table are in ABC's archives.

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AMENDMENT TO STUDY PROTOCOL

STUDY TITLE: Assessment of the Acute Toxicity of West Diversion Channel to *Daphnia magna*.

PROTOCOL NUMBER: 8301 AMENDMENT NUMBER: 1

LABORATORY: ABC Laboratories, Inc. ABC STUDY NUMBER: 34534

EFFECTIVE DATE: 4/15/86

Section 5.5 Screen Test

One of the duplicate control chambers was lost during the 24-hour screen test. Since no mortality or abnormal effects were observed in the remaining control chamber, the loss of this replicate did not affect the results of the screen test.

SPONSOR'S STUDY DIRECTOR: _____ Date: _____

STUDY DIRECTOR: [illegible] Date: 4/22/86

ANALYTICAL BIO-CHEMISTRY LABORATORIES, INC.
P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

ABC PROTOCOL NO. 8301

(August 21, 1984)

Effluent Static Bioassay with *Daphnia magna* or Mysid Shrimp (*Mysidopsis bahia*) to Meet EPA Region 6 NPDES Biomonitoring Guidelines

RECEIVED
APR 10 '86
A.B.C. LABS

ABC Study Number 34534

Test Material West Diversion Channel

Test Species *Daphnia magna*

1.0 INTRODUCTION

The use of bioassays in the identification and control of industrial effluents is rapidly increasing. Indeed, aquatic toxicity tests are presently being incorporated into the NPDES guidelines of the Federal Clean Water Act (1). To assist industry in meeting requirements set forth by legislation and regulatory agencies, Analytical Bio-Chemistry Laboratories, Inc. offers a complete range of aquatic bioassay tests of industrial effluents.

As part of the NPDES permit process in U.S. EPA Region 6, a biomonitoring requirement has been imposed for certain permit holders in that area. Discharges subject to the new regulations in Region 6 (Texas, New Mexico, Oklahoma, Arkansas and Louisiana) will be required to submit aquatic toxicity data on their effluents to EPA on a periodic basis.

This ABC protocol discusses the biomonitoring guidelines in Region 6 and describes ABC's technical approach to assist industry in complying with these regulations.

2.0 OBJECTIVES

The primary objective of the toxicity test described herein is to evaluate the acute toxicity of an effluent to freshwater or marine invertebrates under static conditions. The species selected for this testing include *Daphnia magna* (freshwater effluent, <5 ppt salinity) and *Mysidopsis bahia* (saltwater effluent, ≧5 ppt salinity). The effluent will first be tested on a 100% effluent screen basis. If the screen results in ≧20% mortality of test organisms, a 48-hour, five concentration LC_{50} test will be conducted. An LC_{50} is the approximate concentration (percent) of test effluent that produces 50 percent mortality of test organisms after prescribed intervals (24 and 48 hours).

3.0 TESTING FACILITY

The study will be conducted by the Aquatic Toxicology Division of Analytical Bio-Chemistry Laboratories, Inc., 7200 East ABC Lane, P. O. Box 1097, Columbia, Missouri 65205 (314-474-8579).

4.0 REGION 6 BIOMONITORING REQUIREMENT

The following biomonitoring requirement is now being included in the NPDES permit process for many major industrial discharges in EPA Region 6. The permit language given here was that presented by the U.S. EPA at a regional workshop on the matter (2):

> The permittee shall determine if the effluent from this outfall is lethal to twenty (20) percent or greater of the culture of test organisms by use of the "Range-Finding Screening Test," set out in "Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms," EPA-600/4-78-012 (Rev. July, 1978) (3).

Organisms for this test shall be Daphnia sp. if the effluent is less than five (5) parts per thousand salinity or Mysid sp. if the effluent is greater than five (5) parts per thousand salinity. This screening test will be conducted within sixty (60) days of effectiveness of the biomonitoring requirements. Tests will be conducted once each quarter for a duration of two years utilizing a static method for 24 hours and following this dilution scheme only:

Effluent sample* - 100 percent by volume
Dilution water - 0 percent by volume

*24-hour composite; refrigerated after collection

If at any time during the two year testing period a test shows a lethality of twenty (20) percent or more of the test organisms, the permittee shall within twenty-four (24) hours conduct a replacement static 48-hour median lethal concentration (LC_{50}) test on the originally collected sample. Replacement of effluent samples shall be once per 24 hours. Organisms for this test shall be Daphnia sp. if the effluent is less than five (5) parts per thousand salinity and reconstituted fresh water (EPA-600/4-78-012 Section 4) shall be used for dilution. If the effluent contains greater than five (5) parts per thousands salinity, Mysid sp. shall be used as the test organism, and reconstituted seawater will be used as dilution water (EPA-600/4-78-012 Section 4). The remaining LC_{50} methodology is available in EPA-600/4-78-012.

All screening and LC_{50} test results shall be reported with the Discharge Monitoring Reports.

It should be noted by the permittee that slight changes from the above permit language (e.g., pH modification of effluent) may be incorporated into his specific permit. Attention should be given to the permit requirements for each effluent before testing is initiated. ABC requires that a copy of the permit's biomonitoring clause be forwarded to ABC prior to testing so that consideration can be given to pH modification, etc.

5.0 METHODS AND MATERIALS

5.1 Methodology. The methods applied for both the preliminary and definitive phases of this test are basically those described in Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms (3), Methods for Acute Toxicity Tests with Fish, Macroinvertebrates, and Amphibians (4), Standard Practice for Conducting Acute Toxicity Tests with Fishes, Macroinvertebrates, and Amphibians (5), Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates

and Amphibians (6), and Standard Methods for the Examination of Water and Wastewater (7). This protocol has been written to comply with the federal Good Laboratory Practice Regulations (8).

5.2 Test Organisms. The species recommended by the EPA for this testing are Daphnia sp. and Mysid sp. Daphnia sp. are to be used when the effluent salinity is <5 parts per thousand (ppt) and Mysid sp. are to be used when the effluent salinity is ≧5 ppt. The species maintained by ABC, and acceptable by EPA, for this testing are Daphnia magna and Mysidopsis bahia. The test lot of Daphnia magna will be obtained from an in-house culture at ABC. The taxonomic key presented by Pennak (9) will be used to identify to species the test daphnids. All test daphnids will be held in a controlled temperature area at 20 ±2°C. The lighting will be 50-70 footcandles on a 16 hour daylight photoperiod. During the holding period, they will receive a suspension of algae (Selenastrum capricornutum) supplemented with a blended fish food/cerophyl suspension at least every 3 days. First-instar daphnids (<24 hours old) will be selected for testing and will not be fed during the test.

Mysid shrimp (Mysidopsis bahia) will be obtained from a commercial supplier (e.g., Sea Plantations, Inc. or Gulf Breeze Specimens) and maintained in culture at ABC. Mysids will be cultured following the techniques described by the U.S. EPA (10). Test mysids will be selected from a sub-culture of immature individuals.

5.3 Effluent Sample. The sample submitted to ABC for testing must be a 24-hour composite sample, consisting of equal volumes taken every hour (2). The sample should be collected in a glass or polyethylene container, refrigerated after collection, and shipped to arrive at ABC within 24 hours after collection. A one-gallon sub-sample of the 24-hour composite will be satisfactory for the toxicity test(s) required. ABC can provide sample containers and mailing cartons and can assist plant personnel in shipping arrangements so that the sample arrives at ABC in a timely manner.

5.4 Dilution Water. It will not be necessary to collect actual receiving water for effluent dilutions for this test. The biomonitoring regulations recommend the use of reconstituted freshwater or saltwater listed in the EPA effluent methods manual (3). The freshwater dilution water (for Daphnia sp.) will be a hard reconstituted water of 160-180 mg/l hardness (as $CaCO_3$). The reconstituted saltwater (for Mysid sp.) will be prepared from a commercial salt mix similar in composition to that listed in the EPA effluent manual (3) and adjusted to a test salinity of 15-25 ppt.

5.5 Screen Test. The biomonitoring regulations require that a 24-hour 100% effluent screen be conducted before more definitive (LC_{50}) testing is considered. As stated in section 4.0, if ≧20% of the test organisms die in the 100% solution, the permittee shall then conduct a 48-hour static replacement LC_{50} test. This latter test is discussed in section 5.6.

The screen test will be conducted in 250 ml glass beakers containing 200 ml of solution. These test chambers will be held in a controlled temperature area at 20 ±2°C on a 16-hour daylight vs. 8-hour darkness photoperiod with 30-minute dawn/dusk transition periods. The screen test will include duplicate test chambers at 100% vs. duplicate control chambers containing dilution water only. Ten test organisms will be placed in each replicate; therefore, twenty total organisms will be exposed to the 100% effluent and control (dilution) water.

After a 24-hour exposure period, the test chambers will be observed for mortality and abnormal effects. If the combined replicate mortality is ≧20%, the 48-hour LC_{50} test will be initiated.

5.6 48-Hr LC_{50} Test. The definitive test will be conducted by exposing the test organisms to at least 5 effluent concentrations and a dilution water control. The test will be conducted using duplicate beakers per concentration containing 10 test organisms per beaker (=20 per concentration). The test concentrations used will follow one of the logarithmic series presented in Table 1. The most common concentration series used is 10-100% (10, 18, 32, 56 and 100%). Several effluent toxicity tests conducted by ABC have shown that most industrial effluents are toxic within this range.

The test organisms will be placed in the test chambers by stratified random assignment within 30 minutes after solution preparations. The test chambers will be observed for mortality and/or abnormal effects every 24 hours, with a record maintained of mortality and abnormal behavior for each concentration tested.

5.7 Test Procedure - Chemical and Physical. Upon receipt of the test effluent, the following parameters will be measured of the 100% effluent: temperature, dissolved oxygen, pH, conductivity and hardness. These same parameters, except hardness, will be measured at 0 and 48 hours of definitive testing in one replicate of the control, low concentration, middle concentration and high concentration solutions.

5.8 Analysis of Results. The results of the definitive study will be statistically analyzed for 24 and 48 hour LC_{50} values and their corresponding 95 percent confidence limits. These values will be determined by an LC_{50} computer program developed by Stephan et al. (11) or by manual methods described by Litchfield-Wilcoxon (12) and Stephan (13).

5.9 Report. One copy of the report will be submitted. The report will contain all original raw data. Additional copies will be provided, if requested by the sponsor, at a copy rate of $50 per copy. The report will include the following:

5.9.1 Study dates, name and address of test facility.

5.9.2 Objectives and test methods.

5.9.3 Reference to the statistical methods used for data analysis.

5.9.4 Description of test effluent (date of receipt, storage conditions, physical characteristics, and method of preparing test concentrations).

5.9.5 Description of test design.

5.9.6 Summary of the data analysis, mortality observations and test water quality.

5.9.7 Location of raw data.

5.9.8 List and signatures of study personnel.

5.9.9 GLP compliance statement by study director and a statement by ABC's Quality Assurance Unit.

5.9.10 The report appendix will contain the original raw data for mortality observations and water quality, letter of test authorization, letters of authorized protocol changes and a copy of the approved protocol.

5.10 Data Retention. All original raw data generated in the preliminary and definitive studies will be provided to the study sponsor in the appendix to the final report. A copy of the data will be retained in ABC's archives.

6.0 PROTOCOL CHANGES

In the event that modifications of this protocol are deemed necessary, a written statement of any changes and reason(s) proposed by the study sponsor or ABC will be submitted to the other party. All agreed changes will be expressed in writing, signed and dated by the sponsor's study director. The signed changes will be appended to the protocol and included with the final report. This protocol is subject to revision when felt necessary to comply with changes in federal regulations and state of the art methodology. The protocol for a specific study will not be changed while the study is in progress without discussion with the study sponsor.

7.0 SPONSOR AUTHORIZATIONS DURING THE STUDY

Should a problem develop while the study is in progress, ABC will notify the study director within 24 hours. The problem and suggested test modifications will be discussed by telephone. ABC will proceed with the changes felt necessary upon the verbal authorization of the study director. A letter for written authorization will then be submitted by ABC to the study director and handled in the same manner discussed in section 6.0.

8.0 TEST-SPECIFIC INFORMATION

8.1.1 General. The following items will be addressed for each static bioassay. This information is necessary to be in compliance with Good Laboratory Practice Regulations (8). Sections 8.2, 8.3, 8.7 and 8.9 are to be completed by the study sponsor. Sections 8.4, 8.5 and 8.6 will be completed by ABC.

8.1.2 GLP Compliance. To be in compliance with the Federal Good Laboratory Practice regulations, the report of the investigation conducted utilizing this protocol must contain a statement that the study was conducted in accordance with one of the following GLP's.

1. U.S. F.D.A. Good Laboratory Practice Regulations (21 CFR 58).

2. U.S. E.P.A. Good Laboratory Practice Standards; Toxic Substances Control (40 CFR 792).

3. U.S. E.P.A. Good Laboratory Practice Standards; Pesticide Programs (40 CFR 160).

4. OECD Principles of Good Laboratory Practice; Annex 2 C(81) 30(Final).

5. Any state or local GLP which may apply.

The specific GLP regulation, for which this study specific protocol and resultant study must be in compliance with, is to be designated by the study sponsor in section 8.8 of this protocol.

8.2 Study Sponsor:

8.2.1 Company Molycorp, Inc.

8.2.2 Address 3 miles East of Questa on State Rd. 58
P. O. Box 469, Questa, New Mexico 87556

8.2.3 Sponsor's Study Director

Mr. Fred Martinez
Name

Title

8.3 Effluent:

8.3.1 Sample Number Water Diversion Channel

8.3.2 NPDES Permit Number NM-0022306

8.3.3 Physical Description clear liquid

8.3.4 Handling Precautions Normal

8.4 Test Concentrations: To be determined after 24-hour screen of 100% effluent and consultation with study sponsor.

8.4.1 Sponsor's study director notified of screen results:

Date 3/28/86 ABC Biologist Scott Gagner

100% effluent screen = ____ ≧20% mortality ✓ <20% mortality

8.4.2 Definitive test concentrations agreed upon:

8.5 Test Organisms:

8.5.1 Species Daphnia magna

8.5.2 Supplier ABC in-house culture

8.6 Study Dates:

8.6.1 Proposed starting date of definitive study 3/26/86

8.6.2 Proposed completion date of definitive study 4/15/86

8.7 ABC Study Personnel:

8.7.1 Study Director

Alan D. Forbis
Name

Aquatic Biologist III
Title

8.7.2 Aquatic Supervisor

Carl M. Thompson — Aquatic Toxicology Supervisor
Name — Title

8.8 GLP Compliance (check the most appropriate):

EPA-FIFRA____; EPA-TSCA____; FDA____; OECD____; Other NPPES-EPA.

8.9 Protocol Approvals. The following is to be signed by the appropriate study personnel:

8.9.1 Sponsor's Study Director ①

Fred Martinez — Environmental Analyst — 4/2/86
Name — Title — Date

8.9.2 ABC's Study Director

Alan D. Fabi — Aquatic Biologist III — 3-27-86
Name — Title — Date

8.9.3 ABC's Group Leader

Michael J. Miller — Group Leader, Animal Toxicology — 3-27-86
Name — Title — Date

8.10 Authorization to return the test material and sample:

8.10.1 Sponsor's authorized agent to receive the test material and sample.

Mr. Fred Martinez
Name — Title

8.10.2 Shipping Address.

Molycorp, Inc.
Company

3 miles East of Questa Highway 38
Street Address

Questa, — New Mexico — 87556
City — State — Zip Code

505-586-0212
Phone Number

8.10.3 Hazardous shipment information.

Proper Shipping Name

Class of Restricted Article (e.g. flammable, poison, radioactive, etc.)

① Due to the time restrictions for effluent testing following receipt of sample (within 24 hours), this protocol could not be processed for approval

TABLE 1: Guide to selection of experimental concentrations based on progressive bisection of intervals on logarithmic scale.

Col. 1	Col. 2	Col. 3	Col. 4	Col. 5
10.0	10.0	10.0	10.0	10.0
				8.7
			7.5	7.5
				6.5
		5.6	5.6	5.6
				4.9
			4.2	4.2
				3.7
3.2	3.2	3.2	3.2	3.2
				2.8
			2.4	2.4
				2.1
		1.8	1.8	1.8
				1.55
			1.35	1.35
				1.15
1.0	1.0	1.0	1.0	1.0

TABLE 2: Chemical characteristics of well water used by ABC's Aquatic Toxicology Division.

Parameters	Concentration
Temperature	15-20°C[b]
Dissolved Oxygen[a]	9.2-10.1 ppm[b]
pH	7.8-8.3[b]
Hardness ($CaCO_3$)	225-275 ppm[b]
Alkalinity ($CaCO_3$)	325-375 ppm[b]
Conductivity	700 µmhos/cm
NH_3-N	0.60 ppm
NO_3-NO_2-N	0.20 ppm
Ortho-Phosphate	0.10 ppm
Aluminum	<0.010 ppm
Arsenic	<0.0005 ppm
Cadmium	<0.002 ppm
Chromium	<0.006 ppm
Cobalt	<0.003 ppm
Copper	<0.002 ppm
Iron	<0.004 ppm
Lead	0.017 ppm
Mercury	0.0008 ppm
Nickel	<0.013 ppm
Silver	<0.0004 ppm
Zinc	0.001 ppm
Measured organophosphorus pesticides	c
Measured organochlorine pesticides plus PCB's	c

[a]After aeration.

[b]Represents seasonal variation, with the monthly range not exceeding 10%.

[c]Below minimum detectable limits.

9.0 REFERENCES

(1) U.S. Congress. 1977. Clean Water Act. Public Law 95-217. Federal Register, Dec. 27, 1977, 91 STAT 1566-1611.

(2) U.S. Environmental Protection Agency. 1983. Effluent Toxicity Testing and Biomonitoring Workshop. Feb. 22-23, 1983, Dallas, Texas.

(3) Peltier, William. 1978. Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms. U.S. Environmental Protection Agency, EPA-660/4-78-012, revised July, 1978. 52 p.

(4) Committee on Methods for Toxicity Tests with Aquatic Organisms. 1975. Methods for Acute Toxicity Tests with Fish, Macroinvertebrates and Amphibians. Environmental Protection Agency, Ecological Research Series EPA-660/3-75-009, April, 1975. 61 p.

(5) American Society for Testing and Materials. 1980. Standard practice for conducting basic acute toxicity tests with fish, macroinvertebrates and amphibians. May, 1980, ASTM Committee E-35.23. 25 p.

(6) American Society for Testing and Materials. 1983. Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates and Amphibians. November, 1983, ASTM Committee E-47.01. 37 p.

(7) American Public Health Association. 1980. Standard Methods for the Examination of Water and Wastewater. 15th ed. Washington, D.C. 1134 p.

(8) (a) U.S. Food and Drug Administration. 1978. Nonclinical Laboratory Studies, Good Laboratory Practice Regulations (21 CFR, Part 58). Federal Register, Vol. 43, No. 247: 59986-60025.

(b) U.S. Environmental Protection Agency. 1983. Pesticide Programs; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 160). Federal Register, Vol 48, No. 230: 53946-53969.

(c) U.S. Environmental Protection Agency. 1983. Toxic Substances Control; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 792). Federal Register, Vol. 48; No. 230:53922-53944.

(d) Organization for Economic Cooperation and Development. 1981. OECD Guidelines for Testing of Chemicals, Principles of Good Laboratory Practice Annex 2, C(81) 30(Final):7-28.

(9) Pennak, R. W. 1978. Fresh-Water Invertebrates of the United States, 2nd Ed. 365 p.

(10) U.S. Environmental Protection Agency. 1979. Static culture methods for sheepshead minnows (Cyprinodon variegatus) and mysid shrimp (Mysidopsis bahia). Unpublished manuscript, U.S. EPA, Athens, GA.

(11) Stephan, C. E., K. A. Busch, R. Smith, J. Burke and R. W. Andrew. 1978. A computer program for calculating an LC_{50}. U. S. Environmental Protection Agency, Duluth, Minnesota, pre-publication manuscript, August, 1978.

(12) Litchfield, J. T., Jr. and F. Wilcoxon. 1949. A Simplified Method of Evaluating Dose-Effect Experiments. Jour. Pharm. Exp. Ther. 96:99-113.

(13) Stephan, C. 1977. Methods for calculating an LC_{50}, p. 65-84. In F. L. Mayer and J. L. Hamelick (eds.). Aquatic Toxicology and Hazard Evaluation. ASTM Special Technical Publication 634. ASTM. Philadelphia.

Assessment of Acute Toxicity of 002
Daphnia Magna

July 1986

ANALYTICAL BIO-CHEMISTRY LABORATORIES, INC.
P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

Static Acute Toxicity Report
#34867

Assessment of the Acute Toxicity of Outfall 002 to *Daphnia magna*

Submitted To:
Molycorp, Incorporated
Attn: Mr. Fred Martinez
3 miles East of Questa, on State Road 38
P. O. Box 469
Questa, New Mexico 87556

July 15, 1986

Submitted By: Analytical Bio-Chemistry Laboratories, Inc.
7200 East ABC Lane
P. O. Box 1097
Columbia, Missouri 65205
(314) 474-8579

Prepared By:

Scott Frazier 7/17/86
Scott Frazier Date
Biologist I

Approved By:

Alan D. Forbis 7/16/86
Alan D. Forbis Date
Study Director/
Supervisor, Invertebrate and
Biconcentration Investigations

Kathy Landholt 07/17/86
Kathy Landholt Date
Quality Assurance Officer

Carl M. Thompson 7-16-86
Carl M. Thompson Date
Aquatic Toxicology Manager

/lrm

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SUMMARY

The acute toxicity of Outfall 002 to Daphnia magna was assessed for U.S. EPA Region 6 NPDES permit requirements using the methods outlined by the Committee on Methods for Toxicity Tests with Aquatic Organisms. Water quality parameters of temperature, dissolved oxygen, conductivity, hardness, alkalinity and pH were measured on receipt of the sample and were within acceptable limits.

The results of the 24-hour static Daphnia magna toxicity screen indicated that the mortality was <20% for Outfall 002. In fact, no mortality was observed after the 24- hour exposure period. Therefore, a definitive 48-hour static acute study was not required. All reported values were based upon nominal concentrations.

The no-effect level for Outfall 002 was estimated to be <100% effluent after 24-hours, since exposed Daphnids exhibited surfacing at the 100% effluent level.

INTRODUCTION

This static bioassay was performed by the Aquatic Toxicology Division of Analytical Bio-Chemistry Laboratories, Inc., Columbia, Missouri, for Molycorp, Inc., from June 26, 1986 to June 27, 1986. The purpose of this test was to assess toxicity of a 100% solution of Outfall 002 to *Daphnia magna*. The assessment was designed to meet the NPDES permit process in U.S. EPA Region 6 (1) requiring biomonitoring of industrial discharges. The screen test was conducted following the procedures outlined in ABC protocol #8301, as approved by Mr. Fred Martinez; Molycorp, Inc. on July 3, 1986.

METHODS AND MATERIALS

The procedures applied for the 24-hour toxicity screen were basically those described in Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms (2), Methods for Acute Toxicity Tests with Fish, Macroinvertebrates, and Amphibians (3), Standard Practice for Conducting Acute Toxicity Tests with Fishes, Macroinvertebrates and Amphibians (4), Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents and Fishes, Macroinvertebrates and Amphibians (5), and Standard Methods for Examination of Water and Wastewater (6). The test lot of *Daphnia magna* was obtained from an in-house culture at ABC. The taxonomic key presented by Pennak (7) was used to identify the test daphnids to species. All test daphnids were held in a controlled temperature area at 20 (±2)°C. The lighting was 50-70 footcandles on a 16-hour daylight photoperiod. During the holding period, the daphnids were fed a suspension of algae (*Selenastrum capricornutum*) at least every 3 days. First-instar daphnids (<24-hours old) were selected for testing and were not fed during the test.

The static *Daphnia* bioassay was conducted in 250-ml glass beakers containing 200 ml of sample and/or ABC hard reconstituted water composed of the following compounds in the amounts stated per liter of deionized water:

192 mg $NaHCO_3$
120 mg $CaSO_4 \cdot 2H_2O$
120 mg $MgSO_4$
8 mg KCl

This reconstituted water was prepared to yield a total hardness of 160-180 mg/l (as $CaCO_3$), a total alkalinity of 110-120 mg/l (as $CaCO_3$) and an initial pH of 7.6 to 8.0.

These vessels were kept at 20 (±2.0) °C in a temperature controlled area. The lighting was maintained at 50-70 footcandles on a 16-hour daylight photoperiod.

A 24-hour screen test, composed of duplicate 100% effluent chambers was performed to estimate the toxicity of the effluent. Also included was a duplicate ABC hard reconstituted water control. A five-six concentration static acute toxicity study would be performed if ≧20% mortality was observed after 24-hours in the screen test.

The Outfall 002 sample (NPDES Permit Number NM-0022306) was received on June 26, 1986 as a clear liquid and was received at 11°C. The sample was allowed to warm to test temperature before Daphnids were added to test solutions.

RESULTS

After 24-hours, the results indicated that the mortality of *Daphnia magna* to Outfall 002 was <20%. The no-effect concentration was estimated to be <100% effluent since surfacing was observed in the 100% effluent test chambers. Since mortality was not ≧20% after 24-hours in the screen test, a definitive study was not required per biomonitoring guidelines established for U.S. EPA Region 6.

Table 2 presents the water quality parameters measured on sample receipt. Hardness and alkalinity (as $CaCO_3$) and conductivity for the Outfall 002 sample were 818 mg/l, 156 mg/l and 1400 µMHOS, respectively. Dissolved oxygen and pH for this sample were 8.9 mg/l and 7.5, respectively, and were considered adequate for testing (3).

The study was conducted following the intent of the Good Laboratory Practice Regulations (8) and the final report was reviewed by Analytical Bio-Chemistry Laboratories' Quality Assurance Unit. All original raw data was provided to Molycorp, Inc. with a copy retained at Analytical Bio-Chemistry Laboratories.

TABLE 1: Chemical Characteristics of Well Water Used by ABC's Aquatic Toxicology Division.

Parameters	Concentration
Temperature	15-20°C[b]
Dissolved Oxygen[a]	9.2-10.1 ppm[b]
pH	7.8-8.3[b]
Hardness ($CaCO_3$)	225-275 ppm[b]
Alkalinity ($CaCO_3$)	325-375 ppm[b]
Conductivity	700 μmhos/cm
NO_3-N	0.74 ppm
NO_3-NO_2-N	0.74 ppm
PO_4-P	<0.10 ppm
Aluminum	<20 ppb
Arsenic	<0.2 ppb
Cadmium	<2 ppb
Chromium	<3 ppb
Cobalt	<4 ppb
Copper	<3 ppb
Iron	12 ppb
Lead	<5 ppb
Mercury	<0.5 ppb
Nickel	<15 ppb
Silver	<5 ppb
Zinc	11 ppb
Measured organophosphorus pesticides	c
Measured organochlorine pesticides plus PCB's	c

[a]After aeration.

[b]Represents seasonal variation, with the monthly range not exceeding 10%.

[c]Less than minimum detectable limits for organophosphorus and organochlorine which were <0.10 and <0.50 ppb, respectively.

Sample dates: Trace elements = April 2, 1985, Organophosphate and organochlorine analyses = April 2, 1985.

TABLE 2
Water Quality Measurements of 100%
Outfall 002 Upon Sample Receipt

	100% Outfall 002
Temperature[a]	11*
Dissolved Oxygen[b]	8.9
pH[c]	7.5
Conductivity[d]	1400
Hardness[e]	818
Alkalinity[e]	156

[a]Temperature °C - Monitored with a mercury thermometer.

[b]Dissolved oxygen concentrations (mg/l) - Dissolved Oxygen/BOD Meter (Nester Co. Model 8500). Saturation at 11°C is 11.1 mg/l.

[c]pH - pH Probe (Corning Model 476182) used with a Corning Model 125 pH and mV meter.

[d]Conductivity (μMHOS) - Conductivity Meter (YSI Model 33).

[e]Hardness and alkalinity (mg/l as $CaCO_3$) - HACH Model AL-DT and HAC-DT.

*Test solutions allowed to warm to test temperature (21°C) before addition of daphnids. Dissolved oxygen saturation at 21°C is 9.0 mg/l.

LITERATURE CITED

(1) U. S. Environmental Protection Agency. 1983. Effluent Toxicity Testing and Biomonitoring Workshop. Feb. 22-23, 1983, Dallas, Texas.

(2) Peltier, William. 1978. Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms. U.S. Environmental Protection Agency, EPA-660/4-78-012, revised July 1978. 52 p.

(3) Committee on Methods for Toxicity Tests with Aquatic Organisms. 1975. Methods of Acute Toxicity Tests with Fish, Macroinvertebrates and Amphibians. Environmental Protection Agency, Ecological Research Series EPA-660/3-75-009, April 1975.

(4) American Society for Testing and Materials. 1980. Standard Practice for Conducting Basic Acute Toxicity Tests with Fish, Macroinvertebrates and Amphibians. May 1980, ASTM Committee E-35.23. 25 p.

(5) American Society for Testing and Materials. 1983. Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates and Amphibians. November 1983, ASTM Committee E-47.01 37 p.

(6) American Public Health Association. 1980. Standard Methods for the Examination of Water and Wastewater. 15th ed. Washington, DC. 1134 p.

(7) Pennak, R. W. 1978. Freshwater Invertebrates of the United States. John Wiley & Sons, New York, 2nd ed. 803 p.

(8) (a) U.S. Food and Drug Administration. 1978. Nonclinical Laboratory Studies, Good Laboratory Practice Regulations (21 CFR, Part 58). Federal Register, Vol. 43, No. 247:59986-60025.

(b) U.S. Environmental Protection Agency. 1983. Pesticide Programs; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 160). Federal Register, Vol. 48, No. 230:53946-53969.

(c) U.S. Environmental Protection Agency. 1983. Toxic Substances Control; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 792). Federal Register, Vol. 48; No. 230:53922-53944.

(d) Organization for Economic Cooperation and Development. 1981. OECD Guidelines for Testing of Chemicals, Principles of Good Laboratory Practice Annex 2, C(81) 30(Final):7-28.

QUALITY ASSURANCE STATEMENT

Quality Assurance Statement for final report #34867 entitled, "Assessment of the Acute Toxicity of Outfall 002 to *Daphnia magna*," for Mr. Fred Martinez, Molycorp, Inc.; Questa, New Mexico.

In accordance with ABC Laboratories' intent that all studies conducted at our facilities are designed and function in conformance with good laboratory practice regulations and the protocols for individual laboratory studies, an inspection of the final report for Outfall 002 was conducted and found to be in acceptable form by a member of our Quality Assurance Unit. A final inspection of all data and records on July 14, 1986, indicates that the report submitted to you is an accurate reflection of the study as it was conducted by ABC Laboratories.

Should you have any questions relating to the information provided in this statement or the function of our Quality Assurance Unit, please contact the QA Unit at your convenience.

Kathy Landholt 07/17/86

Kathy Landholt Date
Quality Assurance Officer

STUDY COMPLIANCE STATEMENT

Study Compliance Statement for ABC report #34867 entitled, "Assessment of the Acute Toxicity of Outfall 002 to *Daphnia magna*," for Mr. Fred Martinez, Molycorp, Inc.; Questa, New Mexico.

In accordance with ABC Laboratories' intent that all aquatic toxicity tests conducted by our facility follow good laboratory practices, ABC's study director for the above test herein confirms that the study was conducted in compliance with the U.S. E.P.A. Good Laboratory Practice Standards; Toxic Substances Control (40 CFR 792).

All original raw data were sent to Molycorp, Inc., with a copy retained at Analytical Bio-Chemistry Laboratories.

Alan D. Forbis — Date

Alan D. Forbis
ABC Study Director

APPENDIX - RAW DATA

NOTE: Some of the records which appear in this raw data appendix have been provided as photocopies of original records on file at ABC. This has been done by necessity for certain data which are used commonly in several studies at ABC. Such records include organism culture logs.

EFFLUENT TOXICITY TEST
MORTALITY AND BEHAVIORAL OBSERVATIONS

Effluent Designation: 002 OUTFALL
Industry: MOLYCORP, INC
Study No.: 34867
ABC Study Director: A. FORBIS

Lab Form No.: 91

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P.O. Box 1097
Columbia, Missouri 65205

☑ Prelim. ☐ Definitive Test Species: Daphnia magna (Lot # 86-B-5)
Date Initiated: 6/26/86 1:50 pm Test Terminated: 6/27/86
Dilution Water: N/A No./Vessel: 10 Vessel Size: 250 ml

Effluent Conc. (%)	24 hours Dead	24 hours Obs.	48 hours Dead	48 hours Obs.	72 hours Dead	72 hours Obs.	96 hours Dead	96 hours Obs.
HARD RECON. Control A	0	N						
B	0	N						
100 A	0	2-SUR 8-N						
B	0	5-SUR 5-N						
Observer	SF 12:00							
Date	6/27/86							

Remarks: N = Normal; LOE = Loss of Equilibrium;
SUR = Surfacing

Prepared By: Scott Frazier Date: 6/26/86
Checked By: [illegible] Date: 7/9/86

WATER QUALITY FOR EFFLUENT SAMPLES

Test Material: 002 Outfall

Study No.: 34867

Protocol No.: 8301

ABC Study Director: A. FORBIS

Lab Form No.: 28b

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane; P.O. Box 1097
Columbia, Missouri 65205

Concentration mg/l	DAY Temp °C	DAY D.O.[a] mg/l	DAY pH[b]	DAY Cond. µMhos	DAY Temp °C	DAY D.O. mg/l	DAY pH	DAY Cond. µMhos	DAY Temp °C	DAY D.O. mg/l	DAY pH	DAY Cond. µMhos	DAY Temp °C	DAY D.O. mg/l	DAY pH	

[a] Dissolved oxygen concentrations - ~~Dissolved Oxygen Probe (YSI Dissolved Oxygen System Model 54)~~ DISSOLVED OXYGEN/BOD METER (NESTER COMPANY MODEL 8500). ②

[b] pH-pH Probe (Corning Model 476182) used with Corning Model 125 pH and mV meter.

[c] 100% Effluent on Receipt	Temp °C	D.O. mg/l	pH	Cond. µMhos	Hard mg/l	Alk. mg/l	~~NH_3 mg/l~~			Date	Int.
	11	8.9	7.5	1400	818	156				6/26/86	SF

Prepared By: Scott Jasper Date: 6/26/86

Checked By: Lee T. Sehn Date: 7/9/86

Remarks: ~~Lee T. Sehn~~ ① Entry Error LS 7/9/86
② Labeling change AF 7/14/86

STUDY# 034867

Pg. 0011 of 0031

PREPARATION OF EFFLUENT CONCENTRATIONS (volume/volume)

Effluent Designation: 002 OUTFALL

Industry: MOLYCORP, INC

Study No.: 34867

ABC Study Director: A. FORBIS

Lab Form No.: 94

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane; P.O. Box 1097
Columbia, MO 65205

☑ PRELIMINARY ☐ DEFINITIVE

Dilution Water Type:

☐ ABC Well Water ☑ ABC Recon. Water (HARD)

☐ Effluent Receiving Water _____ ☐ Other _____

Sample No.	Aliquot Volume (ml)	Dilution Volume (ml)	Final Conc. (%)
1	0	200	ABC HARD RECON CONTROL
2	200	0	100 %
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			

Remarks:

Prepared By: Scott Jurgen Date: 6/26/86

Checked By: A. Forbis Date: 7/9/86

Study Director: Alan D. Forbis Date: 7/9/86

OBSERVATIONS AND/OR REMARKS FORM

Test Material: 002 Outfall
Study No.: 34667
Protocol No.: ~~80~~ 8301 ②
ABC Study Director: A. Forbis

Lab Form No.: 55

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P.O. Box 1097
Columbia, Missouri 65205

Date/Initials	Comments
6/26/86 LS	Making Reconstituted H_2O for Control/Dilution Water.

$$\frac{32\,mg\,KCl + 768\,mg\,NaHCO_3 + 480\,mg\,CaSO_4 \cdot 2H_2O + 480\,mg\,MgSO_4}{4\,l\ D.I.\ H_2O}$$

All mixed together then stirred for ½ hr.

① Dating Error LS 6/26/86
② Entry Error LS 6/26/86

Prepared By: [signature] Date: 6/26/86
Checked By: Scott Frazier Date: 7/9/86

INVERTEBRATE CULTURE RECORD

Species: Daphnia Magna

Lot No.: 86-B-5

Date Initiated: 6/9/86

Lab Form No: 52

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P. O. Box 1097
Columbia, MO 65205

Date	No. Adult Alive	Instar Present	Fed & Changed	Study I.D.	Comments	Obs. By:
6/4	—	~30	✓		250 ml algae	LS
6/10	~30	—	✓		~350 ml algae ~5 mg Yeast	SF
6/12	30	—	✓		" "	SF
6/15	30	~20	✓		~350 ml algae	SF
6/16	30	~20	✓		" "	SF
6/17	30	~200	✓		" "	LS
6/18	30	~200	✓		" "	JAM
6/22	30	~250	✓		" "	JAM
6/23	—	—	—	34749-105		LS
6/24	30	~215	✓			JAM
6/25	30	~80	✓		" "	LS
6/26	—	—	—	34867-40 34865-40		SF

"This is an exact copy of The original document"

By [signature] date 6-26-86

Prepared by: [signature] Date: 6/9/86

Checked by: Scott Frazier Date: 7/9/86

MOLYCORP, INC.
QUESTA DIVISION

This sheet represents a chain of custody for effluent samples collected at 002 Outfall and Big Arsenic springs Cerro, N.M. located in Taos County, Questa, New Mexico on 06/25/86

Sample bottles were prepared by Molycorp employees in accordance with guidance set forth by the U.S. Environmental Protection Agency for the existing program permit. Sampling procedures were also followed in accordance with permit requirements.

Sample (s) Identification 002 outfall
Big Arsenic Springs

Sample (s) collected at: 002 outfall 9:00AM last portion of composite
Big Arsenic spring 7:00AM Grab sample

By: J. Moore
Environmental Analyst

Samples handed over to carrier by Molycorp employee

DATE: 06/25/86

TIME: 9:00 AM

CARRIER: Fred Martin to Federal X-press Albuquerque N.M.

EMPLOYER: Moly corp Inc.

MOLYCORP PERNONNEL: Fred Martin

Sample(s) delivered to ABC Lab for analysis. please sign and date this letter upon receipt of sample(s)

Sample received by: ABC Laboratorie Inc.
7200 ABC Lane
P.O. Box 1097
Columbia, MO 65205
(314) 474-8579

for the following analyses: Toxicity testing

Received by: Scott Frazier

DATE: 6/26/86 10:30 AM

Please return this letter with the analytical results, signed and dated below.

Scott Frazier 7/9/86
Date

DEFINITIVE CONCENTRATION NOTIFICATION

Firm: MOLYCORP, INCORPORATED
Address: P.O. BOX 469
City/State/Zip: Questa, N.M. 87556
Phone: (505) 586-0212

Lab Form No.: 186

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P.O. Box 1097
Columbia, MO 65202

Sponsor's Study Director: MR. FRED MARTINEZ

Person notified: same(✓) Other:

Date of Notification: 7/9/86

Notification of preliminary testing results with the following definitive test levels agreed upon:

Test Material	ABC #	Test Species	Test Conc. (mg/l)
Outfall 002	34867	Daphnia magna	N/A

Notified sponsor of <20% mortality in screen test.

Notified By: Scott Frazier Date: 7/9/86

ANALYTICAL BIO-CHEMISTRY LABORATORIES, INC.
P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

RECEIVED
JUL 8 '86
A.B.C. LABS

ABC PROTOCOL NO. 8301
(August 21, 1984)

Effluent Static Bioassay with *Daphnia magna* or Mysid Shrimp (*Mysidopsis bahia*) to Meet EPA Region 6 NPDES Biomonitoring Guidelines

ABC Study Number 34867

Test Material Outfall 002

Test Species Daphnia magna

1.0 INTRODUCTION

The use of bioassays in the identification and control of industrial effluents is rapidly increasing. Indeed, aquatic toxicity tests are presently being incorporated into the NPDES guidelines of the Federal Clean Water Act (1). To assist industry in meeting requirements set forth by legislation and regulatory agencies, Analytical Bio-Chemistry Laboratories, Inc. offers a complete range of aquatic bioassay tests of industrial effluents.

As part of the NPDES permit process in U.S. EPA Region 6, a biomonitoring requirement has been imposed for certain permit holders in that area. Discharges subject to the new regulations in Region 6 (Texas, New Mexico, Oklahoma, Arkansas and Louisiana) will be required to submit aquatic toxicity data on their effluents to EPA on a periodic basis.

This ABC protocol discusses the biomonitoring guidelines in Region 6 and describes ABC's technical approach to assist industry in complying with these regulations.

2.0 OBJECTIVES

The primary objective of the toxicity test described herein is to evaluate the acute toxicity of an effluent to freshwater or marine invertebrates under static conditions. The species selected for this testing include Daphnia magna (freshwater effluent, <5 ppt salinity) and Mysidopsis bahia (saltwater effluent, ≧5 ppt salinity). The effluent will first be tested on a 100% effluent screen basis. If the screen results in ≧20% mortality of test organisms, a 48-hour, five concentration LC_{50} test will be conducted. An LC_{50} is the approximate concentration (percent) of test effluent that produces 50 percent mortality of test organisms after prescribed intervals (24 and 48 hours).

3.0 TESTING FACILITY

The study will be conducted by the Aquatic Toxicology Division of Analytical Bio-Chemistry Laboratories, Inc., 7200 East ABC Lane, P. O. Box 1097, Columbia, Missouri 65205 (314-474-8579).

4.0 REGION 6 BIOMONITORING REQUIREMENT

The following biomonitoring requirement is now being included in the NPDES permit process for many major industrial discharges in EPA Region 6. The permit language given here was that presented by the U.S. EPA at a regional workshop on the matter (2):

> The permittee shall determine if the effluent from this outfall is lethal to twenty (20) percent or greater of the culture of test organisms by use of the "Range-Finding Screening Test," set out in "Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms," EPA-600/4-78-012 (Rev. July, 1978) (3).

Organisms for this test shall be Daphnia sp. if the effluent is less than five (5) parts per thousand salinity or Mysid sp. if the effluent is greater than five (5) parts per thousand salinity. This screening test will be conducted within sixty (60) days of effectiveness of the biomonitoring requirements. Tests will be conducted once each quarter for a duration of two years utilizing a static method for 24 hours and following this dilution scheme only:

Effluent sample* - 100 percent by volume
Dilution water - 0 percent by volume

*24-hour composite; refrigerated after collection

If at any time during the two year testing period a test shows a lethality of twenty (20) percent or more of the test organisms, the permittee shall within twenty-four (24) hours conduct a replacement static 48-hour median lethal concentration (LC_{50}) test on the originally collected sample. Replacement of effluent samples shall be once per 24 hours. Organisms for this test shall be Daphnia sp. if the effluent is less than five (5) parts per thousand salinity and reconstituted fresh water (EPA-600/4-78-012 Section 4) shall be used for dilution. If the effluent contains greater than five (5) parts per thousands salinity, Mysid sp. shall be used as the test organism, and reconstituted seawater will be used as dilution water (EPA-600/4-78-012 Section 4). The remaining LC_{50} methodology is available in EPA-600/4-78-012.

All screening and LC_{50} test results shall be reported with the Discharge Monitoring Reports.

It should be noted by the permittee that slight changes from the above permit language (e.g., pH modification of effluent) may be incorporated into his specific permit. Attention should be given to the permit requirements for each effluent before testing is initiated. ABC requires that a copy of the permit's biomonitoring clause be forwarded to ABC prior to testing so that consideration can be given to pH modification, etc.

5.0 METHODS AND MATERIALS

5.1 Methodology. The methods applied for both the preliminary and definitive phases of this test are basically those described in Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms (3), Methods for Acute Toxicity Tests with Fish, Macroinvertebrates, and Amphibians (4), Standard Practice for Conducting Acute Toxicity Tests with Fishes, Macroinvertebrates, and Amphibians (5), Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates

and Amphibians (6), and Standard Methods for the Examination of Water and Wastewater (7). This protocol has been written to comply with the federal Good Laboratory Practice Regulations (8).

5.2 Test Organisms. The species recommended by the EPA for this testing are Daphnia sp. and Mysid sp. Daphnia sp. are to be used when the effluent salinity is <5 parts per thousand (ppt) and Mysid sp. are to be used when the effluent salinity is ≧5 ppt. The species maintained by ABC, and acceptable by EPA, for this testing are Daphnia magna and Mysidopsis bahia. The test lot of Daphnia magna will be obtained from an in-house culture at ABC. The taxonomic key presented by Pennak (9) will be used to identify to species the test daphnids. All test daphnids will be held in a controlled temperature area at 20 ±2°C. The lighting will be 50-70 footcandles on a 16 hour daylight photoperiod. During the holding period, they will receive a suspension of algae (Selenastrum capricornutum) supplemented with a blended fish food/cerophyl suspension at least every 3 days. First-instar daphnids (<24 hours old) will be selected for testing and will not be fed during the test.

Mysid shrimp (Mysidopsis bahia) will be obtained from a commercial supplier (e.g., Sea Plantations, Inc. or Gulf Breeze Specimens) and maintained in culture at ABC. Mysids will be cultured following the techniques described by the U.S. EPA (10). Test mysids will be selected from a sub-culture of immature individuals.

5.3 Effluent Sample. The sample submitted to ABC for testing must be a 24-hour composite sample, consisting of equal volumes taken every hour (2). The sample should be collected in a glass or polyethylene container, refrigerated after collection, and shipped to arrive at ABC within 24 hours after collection. A one-gallon sub-sample of the 24-hour composite will be satisfactory for the toxicity test(s) required. ABC can provide sample containers and mailing cartons and can assist plant personnel in shipping arrangements so that the sample arrives at ABC in a timely manner.

5.4 Dilution Water. It will not be necessary to collect actual receiving water for effluent dilutions for this test. The biomonitoring regulations recommend the use of reconstituted freshwater or saltwater listed in the EPA effluent methods manual (3). The freshwater dilution water (for Daphnia sp.) will be a hard reconstituted water of 160-180 mg/l hardness (as $CaCO_3$). The reconstituted saltwater (for Mysid sp.) will be prepared from a commercial salt mix similar in composition to that listed in the EPA effluent manual (3) and adjusted to a test salinity of 15-25 ppt.

5.5 Screen Test. The biomonitoring regulations require that a 24-hour 100% effluent screen be conducted before more definitive (LC_{50}) testing is considered. As stated in section 4.0, if ≧20% of the test organisms die in the 100% solution, the permittee shall then conduct a 48-hour static replacement LC_{50} test. This latter test is discussed in section 5.6.

The screen test will be conducted in 250 ml glass beakers containing 200 ml of solution. These test chambers will be held in a controlled temperature area at 20 ±2°C on a 16-hour daylight vs. 8-hour darkness photoperiod with 30-minute dawn/dusk transition periods. The screen test will include duplicate test chambers at 100% vs. duplicate control chambers containing dilution water only. Ten test organisms will be placed in each replicate; therefore, twenty total organisms will be exposed to the 100% effluent and control (dilution) water.

After a 24-hour exposure period, the test chambers will be observed for mortality and abnormal effects. If the combined replicate mortality is ≧20%, the 48-hour LC_{50} test will be initiated.

5.6 48-Hr LC_{50} Test. The definitive test will be conducted by exposing the test organisms to at least 5 effluent concentrations and a dilution water control. The test will be conducted using duplicate beakers per concentration containing 10 test organisms per beaker (=20 per concentration). The test concentrations used will follow one of the logarithmic series presented in Table 1. The most common concentration series used is 10-100% (10, 18, 32, 56 and 100%). Several effluent toxicity tests conducted by ABC have shown that most industrial effluents are toxic within this range.

The test organisms will be placed in the test chambers by stratified random assignment within 30 minutes after solution preparations. The test chambers will be observed for mortality and/or abnormal effects every 24 hours, with a record maintained of mortality and abnormal behavior for each concentration tested.

5.7 Test Procedure - Chemical and Physical. Upon receipt of the test effluent, the following parameters will be measured of the 100% effluent: temperature, dissolved oxygen, pH, conductivity and hardness. These same parameters, except hardness, will be measured at 0 and 48 hours of definitive testing in one replicate of the control, low concentration, middle concentration and high concentration solutions.

5.8 Analysis of Results. The results of the definitive study will be statistically analyzed for 24 and 48 hour LC_{50} values and their corresponding 95 percent confidence limits. These values will be determined by an LC_{50} computer program developed by Stephan et al. (11) or by manual methods described by Litchfield-Wilcoxon (12) and Stephan (13).

5.9 Report. One copy of the report will be submitted. The report will contain all original raw data. Additional copies will be provided, if requested by the sponsor, at a copy rate of $50 per copy. The report will include the following:

5.9.1 Study dates, name and address of test facility.

5.9.2 Objectives and test methods.

5.9.3 Reference to the statistical methods used for data analysis.

5.9.4 Description of test effluent (date of receipt, storage conditions, physical characteristics, and method of preparing test concentrations).

5.9.5 Description of test design.

5.9.6 Summary of the data analysis, mortality observations and test water quality.

5.9.7 Location of raw data.

5.9.8 List and signatures of study personnel.

5.9.9 GLP compliance statement by study director and a statement by ABC's Quality Assurance Unit.

5.9.10 The report appendix will contain the original raw data for mortality observations and water quality, letter of test authorization, letters of authorized protocol changes and a copy of the approved protocol.

5.10 Data Retention. All original raw data generated in the preliminary and definitive studies will be provided to the study sponsor in the appendix to the final report. A copy of the data will be retained in ABC's archives.

6.0 PROTOCOL CHANGES

In the event that modifications of this protocol are deemed necessary, a written statement of any changes and reason(s) proposed by the study sponsor or ABC will be submitted to the other party. All agreed changes will be expressed in writing, signed and dated by the sponsor's study director. The signed changes will be appended to the protocol and included with the final report. This protocol is subject to revision when felt necessary to comply with changes in federal regulations and state of the art methodology. The protocol for a specific study will not be changed while the study is in progress without discussion with the study sponsor.

7.0 SPONSOR AUTHORIZATIONS DURING THE STUDY

Should a problem develop while the study is in progress, ABC will notify the study director within 24 hours. The problem and suggested test modifications will be discussed by telephone. ABC will proceed with the changes felt necessary upon the verbal authorization of the study director. A letter for written authorization will then be submitted by ABC to the study director and handled in the same manner discussed in section 6.0.

8.0 TEST-SPECIFIC INFORMATION

8.1.1 General. The following items will be addressed for each static bioassay. This information is necessary to be in compliance with Good Laboratory Practice Regulations (8). Sections 8.2, 8.3, 8.7 and 8.9 are to be completed by the study sponsor. Sections 8.4, 8.5 and 8.6 will be completed by ABC.

8.1.2 GLP Compliance. To be in compliance with the Federal Good Laboratory Practice regulations, the report of the investigation conducted utilizing this protocol must contain a statement that the study was conducted in accordance with one of the following GLP's.

1. U.S. F.D.A. Good Laboratory Practice Regulations (21 CFR 58).

2. U.S. E.P.A. Good Laboratory Practice Standards; Toxic Substances Control (40 CFR 792).

3. U.S. E.P.A. Good Laboratory Practice Standards; Pesticide Programs (40 CFR 160).

4. OECD Principles of Good Laboratory Practice; Annex 2 C(81) 30(Final).

5. Any state or local GLP which may apply.

The specific GLP regulation, for which this study specific protocol and resultant study must be in compliance with, is to be designated by the study sponsor in section 8.8 of this protocol.

8.2 Study Sponsor:

8.2.1 Company Molycorp, Inc.

8.2.2 Address 38 ① Miles East of Questa on State Road 88 30 ①

P.O. Box 469, Questa, New Mexico 87556

① Typing error AF 7/14/86

8.2.3 Sponsor's Study Director

Mr. Fred Martinez
Name

Title

8.3 Effluent:

8.3.1 Sample Number Outfall 002

8.3.2 NPDES Permit Number NM-0022306

8.3.3 Physical Description Clear Liquid

8.3.4 Handling Precautions Normal precautions

8.4 Test Concentrations: To be determined after 24-hour screen of 100% effluent and consultation with study sponsor.

8.4.1 Sponsor's study director notified of screen results:
Date 7/9/86 ABC Biologist Scott Frazier
100% effluent screen = ____ ≧20% mortality X <20% mortality

8.4.2 Definitive test concentrations agreed upon:
N/A

8.5 Test Organisms:

8.5.1 Species Daphnia magna

8.5.2 Supplier ABC in-house culture

8.6 Study Dates:

8.6.1 Proposed starting date of definitive study 06/23/86

8.6.2 Proposed completion date of definitive study 07/15/86

8.7 ABC Study Personnel:

8.7.1 Study Director

Alan D. Forbis
Name

Supervisor, Invertebrate and Bioconcentration Investigations
Title

8.10.3 Hazardous shipment information.

Proper Shipping Name

Class of Restricted Article (e.g. flammable, poison, radioactive, etc.)

TABLE 1: Guide to selection of experimental concentrations based on progressive bisection of intervals on logarithmic scale.

Col. 1	Col. 2	Col. 3	Col. 4	Col. 5
10.0	10.0	10.0	10.0	10.0
				8.7
			7.5	7.5
				6.5
		5.6	5.6	5.6
				4.9
			4.2	4.2
				3.7
3.2	3.2	3.2	3.2	3.2
				2.8
			2.4	2.4
				2.1
		1.8	1.8	1.8
				1.55
			1.35	1.35
				1.15
1.0	1.0	1.0	1.0	1.0

TABLE 2: Chemical characteristics of well water used by ABC's Aquatic Toxicology Division.

Parameters	Concentration
Temperature	15-20°C[b]
Dissolved Oxygen[a]	9.2-10.1 ppm[b]
pH	7.8-8.3[b]
Hardness ($CaCO_3$)	225-275 ppm[b]
Alkalinity ($CaCO_3$)	325-375 ppm[b]
Conductivity	700 µmhos/cm
NH_3-N	0.60 ppm
NO_3-NO_2-N	0.20 ppm
Ortho-Phosphate	0.10 ppm
Aluminum	<0.010 ppm
Arsenic	<0.0005 ppm
Cadmium	<0.002 ppm
Chromium	<0.006 ppm
Cobalt	<0.003 ppm
Copper	<0.002 ppm
Iron	<0.004 ppm
Lead	0.017 ppm
Mercury	0.0008 ppm
Nickel	<0.013 ppm
Silver	<0.0004 ppm
Zinc	0.001 ppm
Measured organophosphorus pesticides	c
Measured organochlorine pesticides plus PCB's	c

[a]After aeration.

[b]Represents seasonal variation, with the monthly range not exceeding 10%.

[c]Below minimum detectable limits.

9.0 REFERENCES

(1) U.S. Congress. 1977. Clean Water Act. Public Law 95-217. Federal Register, Dec. 27, 1977, 91 STAT 1566-1611.

(2) U.S. Environmental Protection Agency. 1983. Effluent Toxicity Testing and Biomonitoring Workshop. Feb. 22-23, 1983, Dallas, Texas.

(3) Peltier, William. 1978. Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms. U.S. Environmental Protection Agency, EPA-660/4-78-012, revised July, 1978. 52 p.

(4) Committee on Methods for Toxicity Tests with Aquatic Organisms. 1975. Methods for Acute Toxicity Tests with Fish, Macroinvertebrates and Amphibians. Environmental Protection Agency, Ecological Research Series EPA-660/3-75-009, April, 1975. 61 p.

(5) American Society for Testing and Materials. 1980. Standard practice for conducting basic acute toxicity tests with fish, macroinvertebrates and amphibians. May, 1980, ASTM Committee E-35.23. 25 p.

(6) American Society for Testing and Materials. 1983. Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates and Amphibians. November, 1983, ASTM Committee E-47.01. 37 p.

(7) American Public Health Association. 1980. Standard Methods for the Examination of Water and Wastewater. 15th ed. Washington, D.C. 1134 p.

(8) (a) U.S. Food and Drug Administration. 1978. Nonclinical Laboratory Studies, Good Laboratory Practice Regulations (21 CFR, Part 58). Federal Register, Vol. 43, No. 247: 59986-60025.

(b) U.S. Environmental Protection Agency. 1983. Pesticide Programs; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 160). Federal Register, Vol 48, No. 230: 53946-53969.

(c) U.S. Environmental Protection Agency. 1983. Toxic Substances Control; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 792). Federal Register, Vol. 48; No. 230:53922-53944.

(d) Organization for Economic Cooperation and Development. 1981. OECD Guidelines for Testing of Chemicals, Principles of Good Laboratory Practice Annex 2, C(81) 30(Final):7-28.

(9) Pennak, R. W. 1978. Fresh-Water Invertebrates of the United States, 2nd Ed. 365 p.

(10) U.S. Environmental Protection Agency. 1979. Static culture methods for sheepshead minnows (Cyprinodon variegatus) and mysid shrimp (Mysidopsis bahia). Unpublished manuscript, U.S. EPA, Athens, GA.

(11) Stephan, C. E., K. A. Busch, R. Smith, J. Burke and R. W. Andrew. 1978. A computer program for calculating an LC_{50}. U. S. Environmental Protection Agency, Duluth, Minnesota, pre-publication manuscript, August, 1978.

(12) Litchfield, J. T., Jr. and F. Wilcoxon. 1949. A Simplified Method of Evaluating Dose-Effect Experiments. Jour. Pharm. Exp. Ther. 96:99-113.

(13) Stephan, C. 1977. Methods for calculating an LC_{50}, p. 65-84. In F. L. Mayer and J. L. Hamelick (eds.). Aquatic Toxicology and Hazard Evaluation. ASTM Special Technical Publication 634. ASTM. Philadelphia.

Assessment of Acute Toxicity of 002
Daphnia Magna

October 1986

ANALYTICAL BIO-CHEMISTRY LABORATORIES, INC.
P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

Static Acute Toxicity Report
#35134

Assessment of the Acute Toxicity of Outfall 002 to *Daphnia magna*

Submitted To:
Molycorp, Incorporated
Attn: Mr. Fred Martinez
3 Miles East of Questa, on State Road 38
P. O. Box 469
Questa, New Mexico 87556

October 28, 1986

Submitted By: Analytical Bio-Chemistry Laboratories, Inc.
7200 East ABC Lane
P. O. Box 1097
Columbia, Missouri 65205
(314) 474-8579

Prepared By:

Scott Frazier 10/30/86
Scott Frazier Date
Biologist I

Approved By:

Alan D. Forbis 10/30/86
Alan D. Forbis Date
Study Director/
Supervisor, Invertebrate and
Bioconcentration Investigations

Kathy Landholt 10/30/86
Kathy Landholt Date
Quality Assurance Officer

Carl M. Thompson 10-30-86
Carl M. Thompson Date
Aquatic Toxicology Manager

/lrm

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PROJECT PERSONNEL

The study director of the project for Analytical Bio-Chemistry Laboratories, Inc. was Alan D. Forbis; Supervisor, Invertebrate and Bioconcentration Investigations. Supervising the conduct of the study for Molycorp, Incorporated was Mr. Fred Martinez. The following ABC personnel assisted with various phases of the study.

Name	Title	Initials
Alan D. Forbis	Supervisor, Invertebrate and Bioconcentration Investigations	AF (DB)
David Burgess	Biologist II	DB
Scott Frazier	Biologist I	SF
Lee T. Schoen	Biologist I	LS (SF)
James Muckerman	Biological Technician I	JGM

INVERTEBRATE ACUTE TOXICITY COMPENDIUM

Subject: "Assessment of the Acute Toxicity of Outfall 002 to *Daphnia magna*",

ABC Study Number: 35134

Sponsor: Mr. Fred Martinez

Testing Facility: Analytical Bio-Chemistry Laboratories, Inc.
Aquatic Toxicology Division
P.O. Box 1097
Columbia, Missouri 65205 (314-474-8579)

Location of Original Raw Data and Final Report: Molycorp, Incorporated; 3 Miles East of Questa, State Road 38; P.O. Box 469; Questa, New Mexico 87556.

Test Material: Outfall 002

Test Concentrations: Control and 100%

Control Water: 160-180 mg/l (hardness), 110-120 mg/l (alkalinity), NA (pH)

Test Dates: Initiation - October 3, 1986
Termination - October 4, 1986

Length of Study: 24-hours

Results: 24-hour NOEL = estimated to be <100%

Test Species: *Daphnia magna*

Source of Organisms: ABC Laboratories in-house culture

Age of Organisms at Study Initiation: First-instar (≤24-hours old)

SUMMARY

The acute toxicity of Outfall 002 to Daphnia magna was assessed for U.S. EPA Region 6 NPDES permit requirements using the methods outlined by the Committee on Methods for Toxicity Tests with Aquatic Organisms. Water quality parameters of temperature, dissolved oxygen, conductivity, hardness, alkalinity and pH were measured on receipt of the sample and were within acceptable limits.

The results of the 24-hour static Daphnia magna toxicity screen indicated that the mortality was <20% for Outfall 002. In fact, no mortality was observed after the 24- hour exposure period. Therefore, a definitive 48-hour static acute study was not required. All reported values were based upon nominal concentrations.

The no-effect level for Outfall 002 was estimated to be <100% effluent after 24-hours, since some of the exposed Daphnids were observed to surface and lie on the bottom of test chambers at the 100% effluent level.

INTRODUCTION

This static bioassay was performed by the Aquatic Toxicology Division of Analytical Bio-Chemistry Laboratories, Inc., Columbia, Missouri, for Molycorp, Inc., from October 3, 1986 to October 4, 1986. The purpose of this test was to assess toxicity of a 100% solution of Outfall 002 to Daphnia magna. The assessment was designed to meet the NPDES permit process in U.S. EPA Region 6 (1) requiring biomonitoring of industrial discharges. The screen test was conducted following the procedures outlined in ABC protocol 8301, as approved by Mr. Fred Martinez; Molycorp, Inc. on October 10, 1986.

METHODS AND MATERIALS

The procedures applied for the 24-hour toxicity screen were basically those described in Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms (2), Methods for Acute Toxicity Tests with Fish, Macroinvertebrates, and Amphibians (3), Standard Practice for Conducting Acute Toxicity Tests with Fishes, Macroinvertebrates and Amphibians (4), Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents and Fishes, Macroinvertebrates and Amphibians (5), and Standard Methods for Examination of Water and Wastewater (6).

I. Test Organisms

The test lot of Daphnia magna was obtained from an in-house culture at ABC. The taxonomic key presented by Pennak (7) was used to identify the test daphnids to species. All test daphnids were held in a controlled temperature area at 20 (±2)°C. The lighting was 50-70 footcandles on a 16-hour daylight photoperiod. During the holding period, the daphnids were fed algae (Selenastrum capricornutum) supplemented with a suspension of fish food at least every 3 days. First-instar daphnids (<24-hours old) were selected for testing and were not fed during the test.

II. Test Procedure

The static Daphnia bioassay was conducted in 250-ml glass beakers containing 200 ml of sample or ABC hard reconstituted water composed of the following compounds in the amounts stated per liter of deionized water:

192 mg $NaHCO_3$
120 mg $CaSO_4 \cdot 2H_2O$
120 mg $MgSO_4$
8 mg KCl

The reconstituted water was prepared to yield a total hardness of 160-180 mg/l (as $CaCO_3$) and a total alkalinity of 110-120 mg/l (as $CaCO_3$).

These vessels were kept at 20 (±2.0) °C in a temperature controlled area. The lighting was maintained at 50-70 foot-candles on a 16-hour daylight photoperiod.

A 24-hour screen test, composed of duplicate 100% effluent chambers was performed to estimate the toxicity of the effluent. Also included was a duplicate ABC hard reconstituted water control. A five-six concentration static acute toxicity study would be performed if ≧20% mortality was observed after 24-hours in the screen test.

III. Test Compound

The Outfall 002 sample (NPDES Permit Number NM-0022306) was received on October 3, 1986 as a clear liquid and was received at 11°C. The sample was allowed to warm to test temperature before Daphnids were added to test solutions.

IV. Statistics

If a definitive study is necessary, the 24- and 48-hour LC_{50} values and corresponding 95-percent confidence limits are determined by an LC_{50} computer program developed by Stephan et al. (8). This program calculates the LC_{50} statistic and its 95-percent confidence limits using the binomial, moving average angle and probit methods because no one method is appropriate for all possible sets of data. The method of calculation selected is that which gives the narrowest confidence limits for each separate analysis.

RESULTS

After 24-hours, the results indicated that the mortality of *Daphnia magna* to Outfall 002 was <20%. The no-effect concentration was estimated to be <100% effluent since surfacing and a daphnid lying on the bottom of a test vessel were observed in the 100% effluent test chambers. Since mortality was not ≧20% after 24-hours in the screen test, a definitive study was not required per biomonitoring guidelines established for U.S. EPA Region 6.

Table 2 presents the water quality parameters measured on sample receipt. Hardness and alkalinity (as $CaCO_3$) and conductivity for the Outfall 002 sample were 470 mg/l, 153 mg/l and 1300 μMHOS, respectively. Dissolved oxygen and pH for this sample were 9.2 mg/l and 7.4, respectively, and were considered adequate for testing (3).

The study was conducted following the intent of the Good Laboratory Practice Regulations (9) and the final report was reviewed by Analytical Bio-Chemistry Laboratories' Quality Assurance Unit. All original raw data were provided to Molycorp, Incorporated with a copy retained at Analytical Bio-Chemistry Laboratories.

TABLE 1

Water Quality Measurements of 100% Outfall 002 Upon Sample Receipt

	100% Outfall 002
Temperature[a]	11*
Dissolved Oxygen[b]	9.2
pH[c]	7.4
Conductivity[d]	1300
Hardness[e]	470
Alkalinity[e]	153

[a]Temperature °C - Monitored with a mercury thermometer.

[b]Dissolved oxygen concentrations (mg/l) - Dissolved Oxygen/BOD Meter (Nester Co. Model 8500). Saturation at 11°C is 11.1 mg/l.

[c]pH - pH Probe (Corning Model 476182) used with a Corning Model 125 pH and mV meter.

[d]Conductivity (μMHOS) - Conductivity Meter (YSI Model 33).

[e]Hardness and alkalinity (mg/l as $CaCO_3$) - HACH Model AL-DT and HAC-DT.

*Test solutions allowed to warm to test temperature (20°C) before addition of daphnids. Dissolved oxygen saturation at 20°C is 9.2 mg/l.

LITERATURE CITED

(1) U. S. Environmental Protection Agency. 1983. Effluent Toxicity Testing and Biomonitoring Workshop. Feb. 22-23, 1983, Dallas, Texas.

(2) Peltier, William. 1978. Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms. U.S. Environmental Protection Agency, EPA-660/4-78-012, revised July 1978. 52 p.

(3) Committee on Methods for Toxicity Tests with Aquatic Organisms. 1975. Methods of Acute Toxicity Tests with Fish, Macroinvertebrates and Amphibians. Environmental Protection Agency, Ecological Research Series EPA-660/3-75-009, April 1975.

(4) American Society for Testing and Materials. 1980. Standard Practice for Conducting Basic Acute Toxicity Tests with Fish, Macroinvertebrates and Amphibians. May 1980, ASTM Committee E-35.23. 25 p.

(5) American Society for Testing and Materials. 1983. Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates and Amphibians. November 1983, ASTM Committee E-47.01 37 p.

(6) American Public Health Association. 1980. Standard Methods for the Examination of Water and Wastewater. 15th ed. Washington, DC. 1134 p.

(7) Pennak, R. W. 1978. Freshwater Invertebrates of the United States. John Wiley & Sons, New York, 2nd ed. 803 p.

(8) Stephan, C. E., K. A. Busch, R. Smith, J. Burke and R. W. Andrews. 1978. A Computer Program for Calculating an LC_{50}. U. S. Environmental Protection Agency, Duluth, Minnesota, pre-publication manuscript, August, 1978.

(9) (a) U.S. Food and Drug Administration. 1978. Nonclinical Laboratory Studies, Good Laboratory Practice Regulations (21 CFR, Part 58). Federal Register, Vol. 43, No. 247:59986-60025.

(b) U.S. Environmental Protection Agency. 1983. Pesticide Programs; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 160). Federal Register, Vol. 48, No. 230:53946-53969.

(c) U.S. Environmental Protection Agency. 1983. Toxic Substances Control; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 792). Federal Register, Vol. 48; No. 230:53922-53944.

(d) Organization for Economic Cooperation and Development. 1981. OECD Guidelines for Testing of Chemicals, Principles of Good Laboratory Practice Annex 2, C(81) 30(Final):7-28.

QUALITY ASSURANCE STATEMENT

Quality Assurance Statement for final report #35134 entitled, "Assessment of the Acute Toxicity of Outfall 002 to *Daphnia magna*," for Mr. Fred Martinez, Molycorp, Incorporated; Questa, New Mexico.

In accordance with ABC Laboratories' intent that all studies conducted at our facilities are designed and function in conformance with good laboratory practice regulations and the protocols for individual laboratory studies, an inspection of the final report for Outfall 002 was conducted and found to be in acceptable form by a member of our Quality Assurance Unit. A final inspection of all data and records on October 21, 1986, indicates that the report submitted to you is an accurate reflection of the study as it was conducted by ABC Laboratories.

Should you have any questions relating to the information provided in this statement or the function of our Quality Assurance Unit, please contact the QA Unit at your convenience.

Kathy Landholt 10/30/86

Kathy Landholt Date
Quality Assurance Officer

STUDY COMPLIANCE STATEMENT

Study Compliance Statement for ABC report #35134 entitled, "Assessment of the Acute Toxicity of Outfall 002 to *Daphnia magna*," for Mr. Fred Martinez, Molycorp, Inc.; Questa, New Mexico.

In accordance with ABC Laboratories' intent that all aquatic toxicity tests conducted by our facility follow good laboratory practices, ABC's study director for the above test herein confirms that the study was conducted in compliance with the U.S. E.P.A. Good Laboratory Practice Standards; Toxic Substances Control (40 CFR 792).

All original raw data were sent to Molycorp, Incorporated, with a copy retained at Analytical Bio-Chemistry Laboratories.

Alan D. Forbis 10/30/86

Alan D. Forbis — Date
ABC Study Director

APPENDIX - RAW DATA

NOTE: Some of the records which appear in this raw data appendix have been provided as photocopies of original records on file at ABC. This has been done by necessity for certain data which are used commonly in several studies at ABC. Such records include organism culture logs.

EFFLUENT TOXICITY TEST
MORTALITY AND BEHAVIORAL OBSERVATIONS

Effluent Designation: Outfall 002
Industry: Molycorp
Study No.: 35134
ABC Study Director: A. FORBIS

Lab Form No.: 91

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P.O. Box 1097
Columbia, Missouri 65205

☑ Prelim. ☐ Definitive Test Species: Daphnia magna (Lot #86-A-8)
Date Initiated: 10/3/86 Test Terminated: 10/4/86
Dilution Water: N/A No./Vessel: 10 Vessel Size: 250ml

Effluent Conc. (%)	24 hours Dead	24 hours Obs.	48 hours Dead	48 hours Obs.	72 hours Dead	72 hours Obs.	96 hours Dead	96 hours Obs.
Control* A	0	N						
B	0	N						
100 A	0	4-SUR 1-OE 5-N						
B	0	3-SUR 7-N						
Observer Date	SF 10/4/86							

Remarks: N = Normal; LOE = Loss of Equilibrium;
SUR = Surfacing
* CONTROL = HARD RECON. WATER

Prepared By: Scott Frazier Date: 10/3/86
Checked By: [illegible] Date: 10/15/86

WATER QUALITY FOR EFFLUENT SAMPLES

Test Material: outfall 002

Study No.: 35134

Protocol No.: 8301

ABC Study Director: A. Forbis

Lab Form No.: 28b

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane; P.O. Box 1097
Columbia, Missouri 65205

Concentration mg/l	DAY				DAY				DAY				DAY			
	Temp °C	D.O.[a] mg/l	pH[b]	Cond. µMhos	Temp °C	D.O. mg/l	pH	Cond. µMhos	Temp °C	D.O. mg/l	pH	Cond. µMhos	Temp °C	D.O. mg/l	pH	

[a]Dissolved oxygen concentrations - Dissolved Oxygen Probe (YSI Dissolved Oxygen System Model 54).

[b]pH-pH Probe (Corning Model 476182) used with Corning Model 125 pH and mV meter.

[c]	Temp °C	D.O. mg/l	pH	Cond. µMhos	Hard mg/l	Alk. mg/l	~~NH_3 mg/l~~			Date	Int.
100% Effluent on Receipt	11	9.2	7.4	1300	470	153				10/3/86	JCM LJ

Prepared By: [signature] Date: 10-3-86

Checked By: [signature] Date: 10/15/86

Remarks:

STUDY# 035134

pg. 00100f0029

PREPARATION OF EFFLUENT CONCENTRATIONS (volume/volume)

Effluent Designation: Outfall 002

Industry: Molycorp

Study No.: 35134

ABC Study Director: A. FORBIS

Lab Form No.: 94

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane; P.O. Box 1097
Columbia, MO 65205

☑ PRELIMINARY ☐ DEFINITIVE

~~Dilution~~ CONTROL ① Water Type:

☐ ABC Well Water ☑ ABC Recon. Water (HARD)

☐ Effluent Receiving Water _____ ☐ Other _____

Sample No.	Aliquot Volume (ml)	Dilution Volume (ml)	Final Conc. (%)
1	—	200	CONTROL
2	200	—	100
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			

Remarks: ① Labelling change, SF, 10/3/86; ② Date error, SF, 10/3/86

Prepared By: Scott Frazier Date: 10/3/86

Checked By: [illegible] Date: 10/15/86

Study Director: [illegible] Date: 10/15/86

INVERTEBRATE CULTURE RECORD

Species: Daphnia magna

Lot No.: 86-A-8

Date Initiated: 9-18-86

STARTED WITH ~30 INSTAR

Lab Form No: 52

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P. O. Box 1097
Columbia, MO 65205

Date	No. Adult Alive	Instar Present	Fed & Changed	Study I.D.	Comments*	Obs. By:
9/22	30	—	✓		Spirul Algae 0.1 ml Midge Food	JGm
9/23	30	—	✓		" "	JGm
9/26	30	—	✓		" "	LS
9/28	30 ~~29~~ ①	~25	✓		" "	JGm
9/30	30	~400	✓		" "	SF
10/2	30	~50	✓		" "	JGm
10/3	—	—	—	35134 (SCREEN) - 40 35135 (SCREEN) - 40		SF
10/3	30	~400	✓		" "	SF
10/5	30	~150	✓		" "	SF
10/7	29	~250	✓		" "	SF

"This is an exact copy of
The original document"

By [signature] date 10/9/86

Prepared by: Nurit Burger Date: 9/18/86

Checked by: Scott Frazier Date: 10/9/86

* Midge Food is a combination of Alfalfa leaves & tetramin JGm 9/22/86

① Entry Error 9-28-86 JGm

MOLYCORP INC.

QUESTA-DIVISION

UNOCAL-76

Samples handed over to carrier by Molycorp employee

DATE: 10/2/86

TIME: 7:00 am

CARRIER J. Mortner

EMPLOYER: Moly Corp Inc.

MOLYCORP PERSONNEL: J. MD

Sample(s) delivered to ABC lab for analysis. Please sign and date this letter upon receipt of sample(s)

Sample(s) received by: ABC Laboratories Inc.
7200 ABC Lane
P.O. Box 1097
Columbia, MO 65205
(314) 474-8579

for the following analyses: Toxicity testing

Received by: Scott Frazier

DATE: 10/3/86

Please return this letter with the analytical results, signed and dated below.

Scott Frazier 10/29/86
Date

"This is an exact copy of
The original document"

By [illegible] date 11-3-86

MOLYCORP, INC.

QUESTA - DIVISION

UNOCAL- 76

This sheet represents a chain of custody for effluent samples collected at outfall 002 Red River @ Ranger station

Located in Taos County, Questa, New Mexico on 10/2/86

Sample bottles were prepared by Molycorp employees in accordance with guidance set forth by the U.S. Environmental Protection Agency for the existing program permit. Sampling procedures were also followed in accordance with permit requirments.

Sample (s) Identification 002 outfall

Red River @ Ranger station

Sample (s) collected at 002 outfall last portion composite 10:00AM

Red River @ Ranger station Grab 9:30AM

By: [signature]
Environmental Analyst

"This is an exact copy of
The original document"

By [signature] date 11-3-86

DEFINITIVE CONCENTRATION NOTIFICATION

Firm: MOLYCORP INCORPORATED	Lab Form No.: 186
Address: 3 Miles East of Questa, State Rd 38 P.O. Box 469	Analytical Bio-Chemistry Labs
City/State/Zip: Questa N.M. 87556	Aquatic Toxicology Division
Phone: (505) 586-0212	7200 ABC Lane, P.O. Box 1097
	Columbia, MO 65202

Sponsor's Study Director: MR. FRED MARTINEZ

Person notified: same (✓) Other:

Date of Notification: 10/6/86

Notification of preliminary testing results with the following definitive test levels agreed upon:

Test Material	ABC #	Test Species	Test Conc. (mg/l)
Outfall 002	35134	Daphnia magna	N/A

Informed sponsor that no further testing would be required since no mortality observed in 24-Hour screen.

Notified By: Scott Frazier Date: 10/9/86

ANALYTICAL BIO-CHEMISTRY LABORATORIES, INC.
P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

RECEIVED
OCT 14 '81
A. B. C. LABS

ABC PROTOCOL NO. 8301

(August 21, 1984)

Effluent Static Bioassay with *Daphnia magna* or
Mysid Shrimp (*Mysidopsis bahia*) to
Meet EPA Region 6 NPDES Biomonitoring Guidelines

ABC Study Number 35134

Test Material Outfall 002

Test Species Daphnia magna

1.0 INTRODUCTION

The use of bioassays in the identification and control of industrial effluents is rapidly increasing. Indeed, aquatic toxicity tests are presently being incorporated into the NPDES guidelines of the Federal Clean Water Act (1). To assist industry in meeting requirements set forth by legislation and regulatory agencies, Analytical Bio-Chemistry Laboratories, Inc. offers a complete range of aquatic bioassay tests of industrial effluents.

As part of the NPDES permit process in U.S. EPA Region 6, a biomonitoring requirement has been imposed for certain permit holders in that area. Discharges subject to the new regulations in Region 6 (Texas, New Mexico, Oklahoma, Arkansas and Louisiana) will be required to submit aquatic toxicity data on their effluents to EPA on a periodic basis.

This ABC protocol discusses the biomonitoring guidelines in Region 6 and describes ABC's technical approach to assist industry in complying with these regulations.

2.0 OBJECTIVES

The primary objective of the toxicity test described herein is to evaluate the acute toxicity of an effluent to freshwater or marine invertebrates under static conditions. The species selected for this testing include *Daphnia magna* (freshwater effluent, <5 ppt salinity) and *Mysidopsis bahia* (saltwater effluent, ≧5 ppt salinity). The effluent will first be tested on a 100% effluent screen basis. If the screen results in ≧20% mortality of test organisms, a 48-hour, five concentration LC_{50} test will be conducted. An LC_{50} is the approximate concentration (percent) of test effluent that produces 50 percent mortality of test organisms after prescribed intervals (24 and 48 hours).

3.0 TESTING FACILITY

The study will be conducted by the Aquatic Toxicology Division of Analytical Bio-Chemistry Laboratories, Inc., 7200 East ABC Lane, P. O. Box 1097, Columbia, Missouri 65205 (314-474-8579).

4.0 REGION 6 BIOMONITORING REQUIREMENT

The following biomonitoring requirement is now being included in the NPDES permit process for many major industrial discharges in EPA Region 6. The permit language given here was that presented by the U.S. EPA at a regional workshop on the matter (2):

> The permittee shall determine if the effluent from this outfall is lethal to twenty (20) percent or greater of the culture of test organisms by use of the "Range-Finding Screening Test," set out in "Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms," EPA-600/4-78-012 (Rev. July, 1978) (3).

Organisms for this test shall be Daphnia sp. if the effluent is less than five (5) parts per thousand salinity or Mysid sp. if the effluent is greater than five (5) parts per thousand salinity. This screening test will be conducted within sixty (60) days of effectiveness of the biomonitoring requirements. Tests will be conducted once each quarter for a duration of two years utilizing a static method for 24 hours and following this dilution scheme only:

Effluent sample* - 100 percent by volume
Dilution water - 0 percent by volume

*24-hour composite; refrigerated after collection

If at any time during the two year testing period a test shows a lethality of twenty (20) percent or more of the test organisms, the permittee shall within twenty-four (24) hours conduct a replacement static 48-hour median lethal concentration (LC_{50}) test on the originally collected sample. Replacement of effluent samples shall be once per 24 hours. Organisms for this test shall be Daphnia sp. if the effluent is less than five (5) parts per thousand salinity and reconstituted fresh water (EPA-600/4-78-012 Section 4) shall be used for dilution. If the effluent contains greater than five (5) parts per thousands salinity, Mysid sp. shall be used as the test organism, and reconstituted seawater will be used as dilution water (EPA-600/4-78-012 Section 4). The remaining LC_{50} methodology is available in EPA-600/4-78-012.

All screening and LC_{50} test results shall be reported with the Discharge Monitoring Reports.

It should be noted by the permittee that slight changes from the above permit language (e.g., pH modification of effluent) may be incorporated into his specific permit. Attention should be given to the permit requirements for each effluent before testing is initiated. ABC requires that a copy of the permit's biomonitoring clause be forwarded to ABC prior to testing so that consideration can be given to pH modification, etc.

5.0 METHODS AND MATERIALS

5.1 Methodology. The methods applied for both the preliminary and definitive phases of this test are basically those described in Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms (3), Methods for Acute Toxicity Tests with Fish, Macroinvertebrates, and Amphibians (4), Standard Practice for Conducting Acute Toxicity Tests with Fishes, Macroinvertebrates, and Amphibians (5), Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates

and Amphibians (6), and Standard Methods for the Examination of Water and Wastewater (7). This protocol has been written to comply with the federal Good Laboratory Practice Regulations (8).

5.2 Test Organisms. The species recommended by the EPA for this testing are Daphnia sp. and Mysid sp. Daphnia sp. are to be used when the effluent salinity is <5 parts per thousand (ppt) and Mysid sp. are to be used when the effluent salinity is ≥5 ppt. The species maintained by ABC, and acceptable by EPA, for this testing are Daphnia magna and Mysidopsis bahia. The test lot of Daphnia magna will be obtained from an in-house culture at ABC. The taxonomic key presented by Pennak (9) will be used to identify to species the test daphnids. All test daphnids will be held in a controlled temperature area at 20 ±2°C. The lighting will be 50-70 footcandles on a 16 hour daylight photoperiod. During the holding period, they will receive a suspension of algae (Selenastrum capricornutum) supplemented with a blended fish food/cerophyl suspension at least every 3 days. First-instar daphnids (<24 hours old) will be selected for testing and will not be fed during the test.

Mysid shrimp (Mysidopsis bahia) will be obtained from a commercial supplier (e.g., Sea Plantations, Inc. or Gulf Breeze Specimens) and maintained in culture at ABC. Mysids will be cultured following the techniques described by the U.S. EPA (10). Test mysids will be selected from a sub-culture of immature individuals.

5.3 Effluent Sample. The sample submitted to ABC for testing must be a 24-hour composite sample, consisting of equal volumes taken every hour (2). The sample should be collected in a glass or polyethylene container, refrigerated after collection, and shipped to arrive at ABC within 24 hours after collection. A one-gallon sub-sample of the 24-hour composite will be satisfactory for the toxicity test(s) required. ABC can provide sample containers and mailing cartons and can assist plant personnel in shipping arrangements so that the sample arrives at ABC in a timely manner.

5.4 Dilution Water. It will not be necessary to collect actual receiving water for effluent dilutions for this test. The biomonitoring regulations recommend the use of reconstituted freshwater or saltwater listed in the EPA effluent methods manual (3). The freshwater dilution water (for Daphnia sp.) will be a hard reconstituted water of 160-180 mg/l hardness (as $CaCO_3$). The reconstituted saltwater (for Mysid sp.) will be prepared from a commercial salt mix similar in composition to that listed in the EPA effluent manual (3) and adjusted to a test salinity of 15-25 ppt.

5.5 Screen Test. The biomonitoring regulations require that a 24-hour 100% effluent screen be conducted before more definitive (LC_{50}) testing is considered. As stated in section 4.0, if ≥20% of the test organisms die in the 100% solution, the permittee shall then conduct a 48-hour static replacement LC_{50} test. This latter test is discussed in section 5.6.

The screen test will be conducted in 250 ml glass beakers containing 200 ml of solution. These test chambers will be held in a controlled temperature area at 20 ±2°C on a 16-hour daylight vs. 8-hour darkness photoperiod with 30-minute dawn/dusk transition periods. The screen test will include duplicate test chambers at 100% vs. duplicate control chambers containing dilution water only. Ten test organisms will be placed in each replicate; therefore, twenty total organisms will be exposed to the 100% effluent and control (dilution) water.

After a 24-hour exposure period, the test chambers will be observed for mortality and abnormal effects. If the combined replicate mortality is ≧20%, the 48-hour LC_{50} test will be initiated.

5.6 48-Hr LC_{50} Test. The definitive test will be conducted by exposing the test organisms to at least 5 effluent concentrations and a dilution water control. The test will be conducted using duplicate beakers per concentration containing 10 test organisms per beaker (=20 per concentration). The test concentrations used will follow one of the logarithmic series presented in Table 1. The most common concentration series used is 10-100% (10, 18, 32, 56 and 100%). Several effluent toxicity tests conducted by ABC have shown that most industrial effluents are toxic within this range.

The test organisms will be placed in the test chambers by stratified random assignment within 30 minutes after solution preparations. The test chambers will be observed for mortality and/or abnormal effects every 24 hours, with a record maintained of mortality and abnormal behavior for each concentration tested.

5.7 Test Procedure - Chemical and Physical. Upon receipt of the test effluent, the following parameters will be measured of the 100% effluent: temperature, dissolved oxygen, pH, conductivity and hardness. These same parameters, except hardness, will be measured at 0 and 48 hours of definitive testing in one replicate of the control, low concentration, middle concentration and high concentration solutions.

5.8 Analysis of Results. The results of the definitive study will be statistically analyzed for 24 and 48 hour LC_{50} values and their corresponding 95 percent confidence limits. These values will be determined by an LC_{50} computer program developed by Stephan et al. (11) or by manual methods described by Litchfield-Wilcoxon (12) and Stephan (13).

5.9 Report. One copy of the report will be submitted. The report will contain all original raw data. Additional copies will be provided, if requested by the sponsor, at a copy rate of $50 per copy. The report will include the following:

5.9.1 Study dates, name and address of test facility.

5.9.2 Objectives and test methods.

5.9.3 Reference to the statistical methods used for data analysis.

5.9.4 Description of test effluent (date of receipt, storage conditions, physical characteristics, and method of preparing test concentrations).

5.9.5 Description of test design.

5.9.6 Summary of the data analysis, mortality observations and test water quality.

5.9.7 Location of raw data.

5.9.8 List and signatures of study personnel.

5.9.9 GLP compliance statement by study director and a statement by ABC's Quality Assurance Unit.

5.9.10 The report appendix will contain the original raw data for mortality observations and water quality, letter of test authorization, letters of authorized protocol changes and a copy of the approved protocol.

5.10 Data Retention. All original raw data generated in the preliminary and definitive studies will be provided to the study sponsor in the appendix to the final report. A copy of the data will be retained in ABC's archives.

6.0 PROTOCOL CHANGES

In the event that modifications of this protocol are deemed necessary, a written statement of any changes and reason(s) proposed by the study sponsor or ABC will be submitted to the other party. All agreed changes will be expressed in writing, signed and dated by the sponsor's study director. The signed changes will be appended to the protocol and included with the final report. This protocol is subject to revision when felt necessary to comply with changes in federal regulations and state of the art methodology. The protocol for a specific study will not be changed while the study is in progress without discussion with the study sponsor.

7.0 SPONSOR AUTHORIZATIONS DURING THE STUDY

Should a problem develop while the study is in progress, ABC will notify the study director within 24 hours. The problem and suggested test modifications will be discussed by telephone. ABC will proceed with the changes felt necessary upon the verbal authorization of the study director. A letter for written authorization will then be submitted by ABC to the study director and handled in the same manner discussed in section 6.0.

8.0 TEST-SPECIFIC INFORMATION

8.1.1 General. The following items will be addressed for each static bioassay. This information is necessary to be in compliance with Good Laboratory Practice Regulations (8). Sections 8.2, 8.3, 8.7 and 8.9 are to be completed by the study sponsor. Sections 8.4, 8.5 and 8.6 will be completed by ABC.

8.1.2 GLP Compliance. To be in compliance with the Federal Good Laboratory Practice regulations, the report of the investigation conducted utilizing this protocol must contain a statement that the study was conducted in accordance with one of the following GLP's.

1. U.S. F.D.A. Good Laboratory Practice Regulations (21 CFR 58).

2. U.S. E.P.A. Good Laboratory Practice Standards; Toxic Substances Control (40 CFR 792).

3. U.S. E.P.A. Good Laboratory Practice Standards; Pesticide Programs (40 CFR 160).

4. OECD Principles of Good Laboratory Practice; Annex 2 C(81) 30(Final).

5. Any state or local GLP which may apply.

The specific GLP regulation, for which this study specific protocol and resultant study must be in compliance with, is to be designated by the study sponsor in section 8.8 of this protocol.

8.2 Study Sponsor:

8.2.1 Company Molycorp, Inc.

8.2.2 Address 3 Miles East of Questa on State Road 38
P.O. Box 469, Questa, New Mexico 87556

8.2.3 Sponsor's Study Director

Mr. Fred Martinez — Analyst
Name — Title

8.3 Effluent:

8.3.1 Sample Number Outfall 002

8.3.2 NPDES Permit Number NM-0022306

8.3.3 Physical Description Clear Liquid

8.3.4 Handling Precautions Normal precautions

8.4 Test Concentrations: To be determined after 24-hour screen of 100% effluent and consultation with study sponsor.

8.4.1 Sponsor's study director notified of screen results:
Date 10/6/86 ABC Biologist Scott Jasper
100% effluent screen = ____ ≧20% mortality ✓ <20% mortality

8.4.2 Definitive test concentrations agreed upon:
N/A

8.5 Test Organisms:

8.5.1 Species Daphnia magna

8.5.2 Supplier ABC in-house culture

8.6 Study Dates:

8.6.1 Proposed starting date of definitive study 10/03/86

8.6.2 Proposed completion date of definitive study 10/17/86

8.7 ABC Study Personnel:

8.7.1 Study Director

Alan D. Forbis — Supervisor, Invertebrate and Bioconcentration Investigations
Name — Title

8.7.2 Aquatic Supervisor

Alan D. Forbis	Supervisor, Invertebrate and Bioconcentration Investigations
Name	Title

8.8 GLP Compliance (check the most appropriate):

EPA-FIFRA____; EPA-TSCA____; FDA____; OECD____; Other NPDES.

8.9 Protocol Approvals. The following is to be signed by the appropriate study personnel:

8.9.1 Sponsor's Study Director [1]

Fred Martinez	Analyst	10/10/86
Name	Title	Date

8.9.2 ABC's Study Director

Alan D. Forbis	Supervisor, Invertebrate and Bio. Investigations	10/02/86
Name	Title	Date

8.9.3 ABC's Manager

Carl M. Thompson (BBF)	Aquatic Tox. Manager	10/02/86
Name	Title	Date

8.10 Authorization to return the test material and sample:

8.10.1 Sponsor's authorized agent to receive the test material and sample.

Mr. Fred Martinez	Analyst
Name	Title

8.10.2 Shipping Address.

Molycorp, Inc.
Company

3 Miles East of Questa Highway 38
Street Address

Questa	New Mexico	87556
City	State	Zip Code

505-586-0212
Phone Number

[1] Due to the time restrictions for effluent testing following receipt of sample (within 24 hours), this protocol could not be processed for sponsor approval prior to the test.

8.10.3 Hazardous shipment information.

Proper Shipping Name

Class of Restricted Article (e.g. flammable, poison, radioactive, etc.)

TABLE 1: Guide to selection of experimental concentrations based on progressive bisection of intervals on logarithmic scale.

Col. 1	Col. 2	Col. 3	Col. 4	Col. 5
10.0	10.0	10.0	10.0	10.0
				8.7
			7.5	7.5
				6.5
		5.6	5.6	5.6
				4.9
			4.2	4.2
				3.7
3.2	3.2	3.2	3.2	3.2
				2.8
			2.4	2.4
				2.1
		1.8	1.8	1.8
				1.55
			1.35	1.35
				1.15
1.0	1.0	1.0	1.0	1.0

TABLE 2: Chemical Characteristics of Well Water Used by ABC's Aquatic Toxicology Division.

Parameters	Concentration
Temperature	15-20°C[b]
Dissolved Oxygen[a]	9.2-10.1 ppm[b]
pH	7.8-8.3[b]
Hardness ($CaCO_3$)	225-275 ppm[b]
Alkalinity ($CaCO_3$)	325-375 ppm[b]
Conductivity	700 µmhos/cm
NO_3-N	0.58 ppm
NO_3-NO_2-N	0.69 ppm
PO_4-P	<0.50 ppm
Aluminum	<20 ppb
Cadmium	<3 ppb
Chromium	<5 ppb
Cobalt	<5 ppb
Copper	<5 ppb
Iron	<5 ppb
Lead	<20 ppb
Nickel	<10 ppb
Silver	<5 ppb
Zinc	<1 ppb
Measured organophosphorus pesticides	<0.10 ppb
Measured organochlorine pesticides plus PCB's	<0.50 ppb

[a]After aeration.

[b]Represents seasonal variation, with the monthly range not exceeding 10%.

Sample dates: Trace elements = May 12, 1986, organophosphate and organochlorine analyses = May 12, 1986.

9.0 REFERENCES

(1) U.S. Congress. 1977. Clean Water Act. Public Law 95-217. Federal Register, Dec. 27, 1977, 91 STAT 1566-1611.

(2) U.S. Environmental Protection Agency. 1983. Effluent Toxicity Testing and Biomonitoring Workshop. Feb. 22-23, 1983, Dallas, Texas.

(3) Peltier, William. 1978. Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms. U.S. Environmental Protection Agency, EPA-660/4-78-012, revised July, 1978. 52 p.

(4) Committee on Methods for Toxicity Tests with Aquatic Organisms. 1975. Methods for Acute Toxicity Tests with Fish, Macroinvertebrates and Amphibians. Environmental Protection Agency, Ecological Research Series EPA-660/3-75-009, April, 1975. 61 p.

(5) American Society for Testing and Materials. 1980. Standard practice for conducting basic acute toxicity tests with fish, macroinvertebrates and amphibians. May, 1980, ASTM Committee E-35.23. 25 p.

(6) American Society for Testing and Materials. 1983. Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates and Amphibians. November, 1983, ASTM Committee E-47.01. 37 p.

(7) American Public Health Association. 1980. Standard Methods for the Examination of Water and Wastewater. 15th ed. Washington, D.C. 1134 p.

(8) (a) U.S. Food and Drug Administration. 1978. Nonclinical Laboratory Studies, Good Laboratory Practice Regulations (21 CFR, Part 58). Federal Register, Vol. 43, No. 247: 59986-60025.

(b) U.S. Environmental Protection Agency. 1983. Pesticide Programs; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 160). Federal Register, Vol 48, No. 230: 53946-53969.

(c) U.S. Environmental Protection Agency. 1983. Toxic Substances Control; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 792). Federal Register, Vol. 48; No. 230:53922-53944.

(d) Organization for Economic Cooperation and Development. 1981. OECD Guidelines for Testing of Chemicals, Principles of Good Laboratory Practice Annex 2, C(81) 30(Final):7-28.

(9) Pennak, R. W. 1978. Fresh-Water Invertebrates of the United States, 2nd Ed. 365 p.

(10) U.S. Environmental Protection Agency. 1979. Static culture methods for sheepshead minnows (Cyprinodon variegatus) and mysid shrimp (Mysidopsis bahia). Unpublished manuscript, U.S. EPA, Athens, GA.

(11) Stephan, C. E., K. A. Busch, R. Smith, J. Burke and R. W. Andrew. 1978. A computer program for calculating an LC_{50}. U. S. Environmental Protection Agency, Duluth, Minnesota, pre-publication manuscript, August, 1978.

(12) Litchfield, J. T., Jr. and F. Wilcoxon. 1949. A Simplified Method of Evaluating Dose-Effect Experiments. Jour. Pharm. Exp. Ther. 96:99-113.

(13) Stephan, C. 1977. Methods for calculating an LC_{50}, p. 65-84. In F. L. Mayer and J. L. Hamelick (eds.). Aquatic Toxicology and Hazard Evaluation. ASTM Special Technical Publication 634. ASTM. Philadelphia.

Assessment of Acute Toxicity of 002
Daphnia Magna

January 1987

RESULTS

After 24-hours, the results indicated that the mortality of *Daphnia magna* to West Diversion Channel was <20%. The no-effect concentration was estimated to be <100% effluent since surfacing was observed in one of the 100% effluent test replicates. Since mortality was not ≧20% after 24-hours in the screen test, a definitive study was not required per biomonitoring guidelines established for U.S. EPA Region 6.

Table 2 presents the water quality parameters measured on sample receipt. Hardness, alkalinity (as $CaCO_3$) and conductivity for the West Diversion Channel sample were 730 and 46 mg/l and 1000 μMHOS, respectively. Dissolved oxygen and pH for this sample were 9.3 mg/l and 7.3, respectively, and were considered adequate for testing (3).

The study was conducted following the intent of the Good Laboratory Practice Regulations (8) and the final report was reviewed by Analytical Bio-Chemistry Laboratories' Quality Assurance Unit. All original raw data was provided to Molycorp, Inc. with a copy retained at Analytical Bio-Chemistry Laboratories.

TABLE 1: Chemical Characteristics of Well Water Used by ABC's Aquatic Toxicology Division.

Parameters	Concentration
Temperature	15-20°C[b]
Dissolved Oxygen[a]	9.2-10.1 ppm[b]
pH	7.8-8.3[b]
Hardness ($CaCO_3$)	225-275 ppm[b]
Alkalinity ($CaCO_3$)	325-375 ppm[b]
Conductivity	700 µmhos/cm
NO_3-N	0.74 ppm
NO_3-NO_2-N	0.74 ppm
PO_4-P	<0.10 ppm
Aluminum	<20 ppb
Arsenic	<0.2 ppb
Cadmium	<2 ppb
Chromium	<3 ppb
Cobalt	<4 ppb
Copper	<3 ppb
Iron	12 ppb
Lead	<5 ppb
Mercury	<0.5 ppb
Nickel	<15 ppb
Silver	<5 ppb
Zinc	11 ppb
Measured organophosphorus pesticides	c
Measured organochlorine pesticides plus PCB's	c

[a]After aeration.

[b]Represents seasonal variation, with the monthly range not exceeding 10%.

[c]Less than minimum detectable limits for organophosphorus and organochlorine which were <0.10 and <0.50 ppb, respectively.

Sample dates: Trace elements = April 2, 1985, Organophosphate and organochlorine analyses = April 2, 1985.

TABLE 2
Water Quality Measurements of 100%
West Diversion Channel Upon Sample Receipt

	100% West Diversion Channel
Temperature[a]	8*
Dissolved Oxygen[b]	9.3
pH[c]	7.3
Conductivity[d]	1000
Hardness[e]	730
Alkalinity[e]	46

[a]Temperature °C - Monitored with a mercury thermometer.

[b]Dissolved oxygen concentrations (mg/l) - Dissolved Oxygen System (YSI Model 54). Saturation at 8°C is 11.9 mg/l.

[c]pH - pH Probe (Corning Model 476182) used with a Corning Model 125 pH and mV meter.

[d]Conductivity (μMHOS) - Conductivity Meter (YSI Model 33).

[e]Hardness and alkalinity (mg/l as $CaCO_3$) - HACH Model AL-DT and HAC-DT.

*Test solutions allowed to warm to test temperature (19°C) before addition of daphnids. Dissolved oxygen saturation at 19°C is 9.3 mg/l.

ANALYTICAL BIO-CHEMISTRY LABORATORIES, INC.
P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

SPONSOR COMPANY

Molycorp Incorporated
Attn: Mr. Fred Martinez
3 Miles East of Questa, on State Road 38
P.O. Box 469
Questa, New Mexico 87556

STUDY TITLE

Assessment of the Acute Toxicity of Outfall 002 to *Daphnia magna*

DATA REQUIREMENT

U.S. EPA-TSCA, 40 CFR, Part 797
Guideline NPDES

AUTHOR

Alan D. Forbis
Supervisor, Invertebrate and
Bioconcentration Investigations

STUDY COMPLETED ON

January 9, 1987

PERFORMING LABORATORY

Analytical Bio-Chemistry Laboratories, Inc.
Aquatic Toxicology Division
7200 East ABC Lane
P.O. Box 1097
Columbia, Missouri 65205

ABC LABORATORY PROJECT ID

#35404

Submitted By: Analytical Bio-Chemistry Laboratories, Inc.
7200 East ABC Lane
P. O. Box 1097
Columbia, Missouri 65205
(314) 474-8579

Prepared By:

Scott Frazier — 1/27/87
Scott Frazier — Date
Biologist I

Michael Ruddy — 1-27-87
Michael Ruddy — Date
Biological Technician I

Approved By:

Alan D. Forbis — 1/27/87
Alan D. Forbis — Date
Study Director/
Supervisor, Invertebrate and
Bioconcentration Investigations

Phillip M. Buckler — 1/27/87
Phillip M. Buckler — Date
Quality Assurance Supervisor

Carl M. Thompson — 1-27-87
Carl M. Thompson — Date
Aquatic Toxicology Manager

/keh

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PROJECT PERSONNEL

The study director of the project for Analytical Bio-Chemistry Laboratories, Inc. was Alan D. Forbis, Supervisor, Invertebrate and Bioconcentration Investigations. Supervising the conduct of the study for Molycorp, Incorporated was Mr. Fred Martinez. The following ABC personnel assisted with various phases of the study.

Name	Title	Initials
Alan D. Forbis	Supervisor, Invertebrate and Bioconcentration Investigations	AF
Scott Frazier	Biologist I	SF
Michael Ruddy	Biological Technician I	MR
Jennifer Wirth	Biological Aide	JW (by SF)
James G. Muckerman	Biological Technician I	JGM

INVERTEBRATE ACUTE TOXICITY COMPENDIUM

Subject: "Assessment of the Acute Toxicity of Outfall 002 to *Daphnia magna*",

ABC Study Number: 35404

Sponsor: Molycorp, Incorporated

Testing Facility: Analytical Bio-Chemistry Laboratories, Inc.
Aquatic Toxicology Division
P.O. Box 1097
Columbia, Missouri 65205 (314-474-8579)

Location of Original Raw Data and Final Report: Molycorp, Incorporated; 3 Miles East of Questa, State Road 38; P.O. Box 469; Questa, New Mexico 87556.

Test Material: Outfall 002

Test Concentrations: Control and 100%

Control Water: ~176 mg/l as $CaCO_3$ (hardness), 118-120 mg/l as $CaCO_3$ (alkalinity), 7.8 (pH)

Test Dates: Initiation - January 8, 1987
Termination - January 9, 1987

Length of Study: 24-hours

Results: 24-hour NOEL = 100%

Test Species: *Daphnia magna*

Source of Organisms: ABC Laboratories in-house culture

Age of Organisms at Study Initiation: First-instar (≦24-hours old)

SUMMARY

The acute toxicity of Outfall 002 to *Daphnia magna* was assessed for U.S. EPA Region 6 NPDES permit requirements using the methods outlined by the Committee on Methods for Toxicity Tests with Aquatic Organisms. Water quality parameters of temperature, dissolved oxygen, conductivity, hardness, alkalinity and pH were measured on receipt of the sample and were within acceptable limits.

The results of the 24-hour static *Daphnia magna* toxicity screen indicated that no mortality was observed after the 24-hour exposure period. Therefore, a definitive 48-hour static acute study was not required. All reported values were based upon nominal concentrations.

The no-effect level for Outfall 002 was 100% effluent after 24-hours, based on the lack of mortality and abnormal effects.

INTRODUCTION

This static bioassay was performed by the Aquatic Toxicology Division of Analytical Bio-Chemistry Laboratories, Inc., Columbia, Missouri, for Molycorp, Inc., from January 8, 1987 to January 9, 1987. The purpose of this test was to assess toxicity of a 100% solution of Outfall 002 to *Daphnia magna*. The assessment was designed to meet the NPDES permit process in U.S. EPA Region 6 (1) requiring biomonitoring of industrial discharges. The screen test was conducted following the procedures outlined in ABC protocol 8301, as approved by Mr. Fred Martinez; Molycorp, Inc. on January 15, 1987.

METHODS AND MATERIALS

The procedures applied for the 24-hour toxicity screen were basically those described in *Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms* (2), *Methods for Acute Toxicity Tests with Fish, Macroinvertebrates, and Amphibians* (3), *Standard Practice for Conducting Acute Toxicity Tests with Fishes, Macroinvertebrates and Amphibians* (4), *Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents and Fishes, Macroinvertebrates and Amphibians* (5), and *Standard Methods for Examination of Water and Wastewater* (6).

I. Test Organisms

The test lot of *Daphnia magna* was obtained from an in-house culture at ABC. The taxonomic key presented by Pennak (7) was used to identify the test daphnids to species. All test daphnids were held in a controlled temperature area at 20 (±2)°C. The lighting was 50-70 footcandles on a 16-hour daylight photoperiod. During the holding period, the daphnids were fed algae (*Selenastrum capricornutum*) supplemented with a suspension of Tetramin®, cereal leaves and yeast at least every 3 days. First-instar daphnids (<24-hours old) were selected for testing and were not fed during the test.

II. Test Procedure

The static *Daphnia* bioassay was conducted in 250-ml glass beakers containing 200 ml of sample or ABC hard reconstituted water prepared from the following compounds in the amounts stated per liter of deionized water:

192 mg $NaHCO_3$
120 mg $CaSO_4 \cdot 2H_2O$
120 mg $MgSO_4$
8 mg KCl

The reconstituted water was prepared to yield a total hardness of 160-180 mg/l (as $CaCO_3$) a total alkalinity of 110-120 mg/l (as $CaCO_3$), and a pH of 7.6-8.0.

These vessels were kept at 20 (±2.0) °C in a temperature controlled area. The lighting was maintained at 50-70 foot-candles on a 16-hour daylight photoperiod.

A 24-hour screen test, composed of duplicate 100% effluent chambers was performed to estimate the toxicity of the effluent. Also included was a duplicate ABC hard reconstituted water control. A five-six concentration static acute toxicity study would be performed if ≧20% mortality was observed after 24-hours in the screen test.

III. Test Material

The Outfall 002 sample (NPDES Permit Number NM-0022306) was received on January 8, 1987 as a clear liquid and was received at 5°C. The sample was allowed to warm to test temperature before daphnids were added to test solutions.

IV. Statistics

If a definitive study is necessary, the 24- and 48-hour LC_{50} values and corresponding 95-percent confidence limits are determined by an LC_{50} computer program developed by Stephan et al. (8). This program calculates the LC_{50} statistic and its 95-percent confidence limits using the binomial, moving average angle and probit methods because no one method is appropriate for all possible sets of data. The method of calculation selected is that which gives the narrowest confidence limits for each separate analysis.

RESULTS

After 24-hours, the results indicated no mortality of *Daphnia magna* to Outfall 002. The no-effect concentration was 100% effluent based on the lack of mortality and abnormal effects. Since no mortality was observed after 24-hours in the screen test, a definitive study was not required per biomonitoring guidelines established for U.S. EPA Region 6.

Table 1 presents the water quality parameters measured on sample receipt. Hardness and alkalinity (as $CaCO_3$) and conductivity for the Outfall 002 sample were 922 mg/l, 163 mg/l and 1350 µMhos/cm, respectively. Dissolved oxygen and pH for this sample were 8.7 mg/l and 7.4, respectively, and were considered adequate for testing (3).

The study was conducted following the intent of the Good Laboratory Practice Regulations (9) and the final report was reviewed by Analytical Bio-Chemistry Laboratories' Quality Assurance Unit. All original raw data were provided to Molycorp, Incorporated with a copy retained at Analytical Bio-Chemistry Laboratories.

TABLE 1

Water Quality Measurements of 100% Outfall 002 Upon Sample Receipt

	100% Outfall 002
Temperature[a]	5*
Dissolved Oxygen[b]	8.7
pH[c]	7.4
Conductivity[d]	1350
Hardness[e]	922
Alkalinity[e]	163

[a]Temperature °C - Monitored with a mercury thermometer.

[b]Dissolved oxygen concentrations (mg/l) - Dissolved Oxygen Probe (YSI Dissolved Oxygen System Model 54). Measured at 7°C. Saturation at 7°C is 12.2mg/l.

[c]pH - pH Probe (Corning Model 476182) used with a Corning Model 125 pH and mV meter.

[d]Conductivity (μMhos/cm) - Conductivity Meter (YSI Model 33).

[e]Hardness and alkalinity (mg/l as $CaCO_3$) - HACH Model AL-DT and HAC-DT.

*Test solutions allowed to warm to test temperature (19°C) before addition of daphnids. Dissolved oxygen saturation at 19°C is 9.3 mg/l.

LITERATURE CITED

(1) U. S. Environmental Protection Agency. 1983. Effluent Toxicity Testing and Biomonitoring Workshop. Feb. 22-23, 1983, Dallas, Texas.

(2) Peltier, William. 1978. Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms. U.S. Environmental Protection Agency, EPA-660/4-78-012, revised July 1978. 52 p.

(3) Committee on Methods for Toxicity Tests with Aquatic Organisms. 1975. Methods of Acute Toxicity Tests with Fish, Macroinvertebrates and Amphibians. Environmental Protection Agency, Ecological Research Series EPA-660/3-75-009, April 1975.

(4) American Society for Testing and Materials. 1980. Standard Practice for Conducting Basic Acute Toxicity Tests with Fish, Macroinvertebrates and Amphibians. May 1980, ASTM Committee E-35.23. 25 p.

(5) American Society for Testing and Materials. 1983. Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates and Amphibians. November 1983, ASTM Committee E-47.01 37 p.

(6) American Public Health Association. 1980. Standard Methods for the Examination of Water and Wastewater. 15th ed. Washington, DC. 1134 p.

(7) Pennak, R. W. 1978. Freshwater Invertebrates of the United States. John Wiley & Sons, New York, 2nd ed. 803 p.

(8) Stephan, C. E., K. A. Busch, R. Smith, J. Burke and R. W. Andrews. 1978. A Computer Program for Calculating an LC_{50}. U. S. Environmental Protection Agency, Duluth, Minnesota, pre-publication manuscript, August, 1978.

(9) (a) U.S. Food and Drug Administration. 1978. Nonclinical Laboratory Studies, Good Laboratory Practice Regulations (21 CFR, Part 58). Federal Register, Vol. 43, No. 247:59986-60025.

(b) U.S. Environmental Protection Agency. 1983. Pesticide Programs; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 160). Federal Register, Vol. 48, No. 230:53946-53969.

(c) U.S. Environmental Protection Agency. 1983. Toxic Substances Control; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 792). Federal Register, Vol. 48; No. 230:53922-53944.

(d) Organization for Economic Cooperation and Development. 1981. OECD Guidelines for Testing of Chemicals, Principles of Good Laboratory Practice Annex 2, C(81) 30(Final):7-28.

QUALITY ASSURANCE STATEMENT

Quality Assurance Statement for final report #35404 entitled, "Assessment of the Acute Toxicity of Outfall 002 to *Daphnia magna*," for Mr. Fred Martinez, Molycorp, Incorporated; Questa, New Mexico.

In accordance with ABC Laboratories' intent that all studies conducted at our facilities are designed and function in conformance with good laboratory practice regulations and the protocols for individual laboratory studies, an inspection of the final report for Outfall 002 was conducted and found to be in acceptable form by a member of our Quality Assurance Unit. A final inspection of all data and records on January 25, 1987, indicates that the report submitted to you is an accurate reflection of the study as it was conducted by ABC Laboratories.

Should you have any questions relating to the information provided in this statement or the function of our Quality Assurance Unit, please contact the QA Unit at your convenience.

Phillip M. Buckler 1/27/87

Phillip M. Buckler Date
Quality Assurance Supervisor

STUDY COMPLIANCE STATEMENT

Study Compliance Statement for ABC report #35404 entitled, "Assessment of the Acute Toxicity of Outfall 002 to *Daphnia magna*," for Mr. Fred Martinez, Molycorp, Inc.; Questa, New Mexico.

In accordance with ABC Laboratories' intent that all aquatic toxicity tests conducted by our facility follow good laboratory practices, ABC's study director for the above test herein confirms that the study was conducted in compliance with the U.S. E.P.A. Good Laboratory Practice Standards; Toxic Substances Control (40 CFR 792).

All original raw data were sent to Molycorp, Incorporated, with a copy retained at Analytical Bio-Chemistry Laboratories.

Alan D. Forbis 1/27/87

Alan D. Forbis — Date
ABC Study Director

APPENDIX - RAW DATA

NOTE: Some of the records which appear in this raw data appendix have been provided as photocopies of original records on file at ABC. This has been done by necessity for certain data which are used commonly in several studies at ABC. Such records include organism culture logs.

EFFLUENT TOXICITY TEST
MORTALITY AND BEHAVIORAL OBSERVATIONS

Effluent Designation: OUTFALL 002
Industry: Moly Corp INC.
Study No.: 35494
ABC Study Director: A. Forbes

Lab Form No.: 91

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P.O. Box 1097
Columbia, Missouri 65205

☑ Prelim. ☐ Definitive Test Species: DAPHNIA MAGNA (Lot #86-E-8)
Date Initiated: 1-8-87 (1:50PM) Test Terminated: 1/9/87
Dilution Water: HARD-DECHL No./Vessel: 10 Vessel Size: 250ml

Effluent * Conc. (%)	24 hours Dead	24 hours Obs.	48 hours Dead	48 hours Obs.	72 hours Dead	72 hours Obs.	96 hours Dead	96 hours Obs.
Control								
A	0	N						
B	0	N						
100%								
A	0	N						
B	0	N						
Observer	SF 1:00pm							
Date	1/9/87							

Remarks: N = Normal; LOE = Loss of Equilibrium;
SUR = Surfacing
*TEST SOLUTIONS @ 19°C AT TEST INITIATION MR 1-8-87

Prepared By: [signature] Date: 1-8-87
Checked By: [signature] Date: 1/9/87

WATER QUALITY FOR EFFLUENT SAMPLES

Test Material: Outfall 002

Study No.: 35404

Protocol No.: ~~8301~~ ① 8301

ABC Study Director: A Forbis

Lab Form No.: 28b

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane; P.O. Box 1097
Columbia, Missouri 65205

Concentration mg/1	DAY				DAY				DAY				DAY			
	Temp °C	D.O.[a] mg/1	pH[b]	Cond. µMhos	Temp °C	D.O. mg/1	pH	Cond. µMhos	Temp °C	D.O. mg/1	pH	Cond. µMhos	Temp °C	D.O. mg/1	pH	

[a]Dissolved oxygen concentrations - Dissolved Oxygen Probe (YSI Dissolved Oxygen System Model 54).

[b]pH-pH Probe (Corning Model 476182) used with Corning Model 125 pH and mV meter.

[c]	Temp °C	D.O.* mg/1	pH	Cond. µMhos	Hard mg/1	Alk. mg/1	NH_3 mg/1			Date	Int.
100% Effluent on Receipt	5	~~8~~ 8.7	7.4	1350	922	163	X			1-8-87 ~~1-8-87~~	MR

Prepared By: [signature] Date: 1-8-87

Checked By: [signature] Date: 1/9/87

Remarks: * D.O. TESTED @ 7°C MR 1-8-87

① ENTRY ERROR 1-13-87 MR

ABC LABS #035404

pg 001 of 0036

OBSERVATIONS AND/OR REMARKS FORM

Test Material: *

Study No.: 35404 & 35405

Protocol No.: 8301

ABC Study Director: A. Forbis

Lab Form No.: 55

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P.O. Box 1097
Columbia, Missouri 65205

Date/Initials	Comments

* OUTFALL 002 AND RED RIVER UPSTREAM SAMPLE

4 LITERS OF RECONSTITUTED WATER WERE PREPARED WITH DEIONIZED WATER AND THE FOLLOWING AMOUNTS OF COMPOUNDS; $NaHCO_3$ (.768g), $CaSO_4 \cdot 2H_2O$ (.480g), $MgSO_4$ (.480g) AND KCl (.032g).

** ALKALINITY AND HARDNESS TESTS ON THE RECONSTITUTED WATER RESULTED IN VALUES OF (118-120), AND ~176 RESPECTIVELY.

** ALKALINITY AND HARDNESS (mg/l as $CaCO_3$) HACH Model AL-DT and HAC-DT.

Prepared By: [signature] Date: 1-7-87

Checked By: [signature] Date: 1/8/87

OBSERVATIONS AND/OR REMARKS FORM

Test Material: Outfall 002
Study No.: 35404
Protocol No.: 8301
ABC Study Director: A. FORBIS

Lab Form No.: 55

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P.O. Box 1097
Columbia, Missouri 65205

Date/Initials	Comments
1/9/87 SF	An analysis of pH in control water at 24 Hours yielded a value of 7.8. (CORNING Model 125 pH and mv meter / CORNING Model 476182 pH probe).

Prepared By: Scott Frazier Date: 1/9/87
Checked By: [illegible] Date: 1/26/87

PREPARATION OF EFFLUENT CONCENTRATIONS (volume/volume)

Effluent Designation: Outfall 002
Industry: Molycorp INC
Study No.: 35404
ABC Study Director: A. FORBIS

Lab Form No.: 94
Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane; P.O. Box 1097
Columbia, MO 65205

☑ PRELIMINARY (SCREEN) ☐ DEFINITIVE

Dilution Water Type:

☐ ABC Well Water ☑ ABC Recon. Water (HARD)

☐ Effluent Receiving Water _____ ☐ Other _____

Sample No.	Aliquot Volume (ml)	Dilution Volume (ml)	Final Conc. (%)
1	0	200	CONTROL
2	200	0	100
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			

Remarks:

Prepared By: Scott Frazier Date: 1/8/87
Checked By: Alan D. Forbis Date: 1/9/87
Study Director: Alan D. Forbis Date: 1/9/87

INVERTEBRATE CULTURE RECORD

Species: Daphnia Magna

Lot No.: 86-E-8

Date Initiated: 12/23/86

Lab Form No: 52

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P. O. Box 1097
Columbia, MO 65205

Date	No. Adult Alive	Instar Present	Fed & Changed	Study I.D.	Comments	Obs. By:
12/23	—	~30	✓		~200 ml algae + ~0.1 ml midgi food	JB.
12/28	30	—	✓		" "	JGm
12/30	29	—	✓		" "	MR
1/1/87	29	~75	✓		" "	MR
1/4/87	29	~300	✓		" "	SF
1/6	29	~300	✓		" "	gw
1/7	29	~30	✓		" "	gw
1/8	—	—	—	35404 — 40 35405 — 40		MR
1/8	29	~400	✓		" "	gw
1/11	29	~200	✓		" "	JGm

"This is an exact copy of The original document"

By Jennifer Wirth date 1/13/87

Prepared by: Jennifer Wirth Date: 12/23/86

Checked by: Scott Mayer Date: 1/13/87

*Midgi food is yeast, Tetramin®, cereal leaves in water, gw 12/23/86

DEFINITIVE CONCENTRATION NOTIFICATION

Firm: Molycorp, Inc
Address: P.O. Box 469
City/State/Zip: Questa, NM 87556
Phone: (505) 586-0212

Lab Form No.: 186
Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P.O. Box 1097
Columbia, MO 65202

Sponsor's Study Director: MR. FRED MARTINEZ
Person notified: same(✓) Other:

Date of Notification: 1/9/87

Notification of preliminary testing results with the following definitive test levels agreed upon:

Test Material	ABC #	Test Species	Test Conc. (mg/l)
Outfall 002	35404	Daphnia magna	N/A

Informed sponsor that screen test mortality was 0%.

Notified By: Scott Frazier Date: 1/9/87

ANALYTICAL BIO-CHEMISTRY LABORATORIES, INC.
P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

RECEIVED
JAN 20 '87
ABC LABS

ABC PROTOCOL NO. 8301

(August 21, 1984)

Effluent Static Bioassay with *Daphnia magna* or Mysid Shrimp (*Mysidopsis bahia*) to Meet EPA Region 6 NPDES Biomonitoring Guidelines

ABC Study Number 35404

Test Material Outfall 002

Test Species Daphnia magna

1.0 INTRODUCTION

The use of bioassays in the identification and control of industrial effluents is rapidly increasing. Indeed, aquatic toxicity tests are presently being incorporated into the NPDES guidelines of the Federal Clean Water Act (1). To assist industry in meeting requirements set forth by legislation and regulatory agencies, Analytical Bio-Chemistry Laboratories, Inc. offers a complete range of aquatic bioassay tests of industrial effluents.

As part of the NPDES permit process in U.S. EPA Region 6, a biomonitoring requirement has been imposed for certain permit holders in that area. Discharges subject to the new regulations in Region 6 (Texas, New Mexico, Oklahoma, Arkansas and Louisiana) will be required to submit aquatic toxicity data on their effluents to EPA on a periodic basis.

This ABC protocol discusses the biomonitoring guidelines in Region 6 and describes ABC's technical approach to assist industry in complying with these regulations.

2.0 OBJECTIVES

The primary objective of the toxicity test described herein is to evaluate the acute toxicity of an effluent to freshwater or marine invertebrates under static conditions. The species selected for this testing include *Daphnia magna* (freshwater effluent, <5 ppt salinity) and *Mysidopsis bahia* (saltwater effluent, ≥5 ppt salinity). The effluent will first be tested on a 100% effluent screen basis. If the screen results in ≥20% mortality of test organisms, a 48-hour, five concentration LC_{50} test will be conducted. An LC_{50} is the approximate concentration (percent) of test effluent that produces 50 percent mortality of test organisms after prescribed intervals (24 and 48 hours).

3.0 TESTING FACILITY

The study will be conducted by the Aquatic Toxicology Division of Analytical Bio-Chemistry Laboratories, Inc., 7200 East ABC Lane, P. O. Box 1097, Columbia, Missouri 65205 (314-474-8579).

4.0 REGION 6 BIOMONITORING REQUIREMENT

The following biomonitoring requirement is now being included in the NPDES permit process for many major industrial discharges in EPA Region 6. The permit language given here was that presented by the U.S. EPA at a regional workshop on the matter (2):

> The permittee shall determine if the effluent from this outfall is lethal to twenty (20) percent or greater of the culture of test organisms by use of the "Range-Finding Screening Test," set out in "Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms," EPA-600/4-78-012 (Rev. July, 1978) (3).

Organisms for this test shall be Daphnia sp. if the effluent is less than five (5) parts per thousand salinity or Mysid sp. if the effluent is greater than five (5) parts per thousand salinity. This screening test will be conducted within sixty (60) days of effectiveness of the biomonitoring requirements. Tests will be conducted once each quarter for a duration of two years utilizing a static method for 24 hours and following this dilution scheme only:

Effluent sample* - 100 percent by volume
Dilution water - 0 percent by volume

*24-hour composite; refrigerated after collection

If at any time during the two year testing period a test shows a lethality of twenty (20) percent or more of the test organisms, the permittee shall within twenty-four (24) hours conduct a replacement static 48-hour median lethal concentration (LC_{50}) test on the originally collected sample. Replacement of effluent samples shall be once per 24 hours. Organisms for this test shall be Daphnia sp. if the effluent is less than five (5) parts per thousand salinity and reconstituted fresh water (EPA-600/4-78-012 Section 4) shall be used for dilution. If the effluent contains greater than five (5) parts per thousands salinity, Mysid sp. shall be used as the test organism, and reconstituted seawater will be used as dilution water (EPA-600/4-78-012 Section 4). The remaining LC_{50} methodology is available in EPA-600/4-78-012.

All screening and LC_{50} test results shall be reported with the Discharge Monitoring Reports.

It should be noted by the permittee that slight changes from the above permit language (e.g., pH modification of effluent) may be incorporated into his specific permit. Attention should be given to the permit requirements for each effluent before testing is initiated. ABC requires that a copy of the permit's biomonitoring clause be forwarded to ABC prior to testing so that consideration can be given to pH modification, etc.

5.0 METHODS AND MATERIALS

5.1 Methodology. The methods applied for both the preliminary and definitive phases of this test are basically those described in Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms (3), Methods for Acute Toxicity Tests with Fish, Macroinvertebrates, and Amphibians (4), Standard Practice for Conducting Acute Toxicity Tests with Fishes, Macroinvertebrates, and Amphibians (5), Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates

and Amphibians (6), and Standard Methods for the Examination of Water and Wastewater (7). This protocol has been written to comply with the federal Good Laboratory Practice Regulations (8).

5.2 Test Organisms. The species recommended by the EPA for this testing are Daphnia sp. and Mysid sp. Daphnia sp. are to be used when the effluent salinity is <5 parts per thousand (ppt) and Mysid sp. are to be used when the effluent salinity is ≧5 ppt. The species maintained by ABC, and acceptable by EPA, for this testing are Daphnia magna and Mysidopsis bahia. The test lot of Daphnia magna will be obtained from an in-house culture at ABC. The taxonomic key presented by Pennak (9) will be used to identify to species the test daphnids. All test daphnids will be held in a controlled temperature area at 20 ±2°C. The lighting will be 50-70 footcandles on a 16 hour daylight photoperiod. During the holding period, they will receive a suspension of algae (Selenastrum capricornutum) supplemented with a blended fish food/cerophyl suspension at least every 3 days. First-instar daphnids (<24 hours old) will be selected for testing and will not be fed during the test.

Mysid shrimp (Mysidopsis bahia) will be obtained from a commercial supplier (e.g., Sea Plantations, Inc. or Gulf Breeze Specimens) and maintained in culture at ABC. Mysids will be cultured following the techniques described by the U.S. EPA (10). Test mysids will be selected from a sub-culture of immature individuals.

5.3 Effluent Sample. The sample submitted to ABC for testing must be a 24-hour composite sample, consisting of equal volumes taken every hour (2). The sample should be collected in a glass or polyethylene container, refrigerated after collection, and shipped to arrive at ABC within 24 hours after collection. A one-gallon sub-sample of the 24-hour composite will be satisfactory for the toxicity test(s) required. ABC can provide sample containers and mailing cartons and can assist plant personnel in shipping arrangements so that the sample arrives at ABC in a timely manner.

5.4 Dilution Water. It will not be necessary to collect actual receiving water for effluent dilutions for this test. The biomonitoring regulations recommend the use of reconstituted freshwater or saltwater listed in the EPA effluent methods manual (3). The freshwater dilution water (for Daphnia sp.) will be a hard reconstituted water of 160-180 mg/l hardness (as $CaCO_3$). The reconstituted saltwater (for Mysid sp.) will be prepared from a commercial salt mix similar in composition to that listed in the EPA effluent manual (3) and adjusted to a test salinity of 15-25 ppt.

5.5 Screen Test. The biomonitoring regulations require that a 24-hour 100% effluent screen be conducted before more definitive (LC_{50}) testing is considered. As stated in section 4.0, if ≧20% of the test organisms die in the 100% solution, the permittee shall then conduct a 48-hour static replacement LC_{50} test. This latter test is discussed in section 5.6.

The screen test will be conducted in 250 ml glass beakers containing 200 ml of solution. These test chambers will be held in a controlled temperature area at 20 ±2°C on a 16-hour daylight vs. 8-hour darkness photoperiod with 30-minute dawn/dusk transition periods. The screen test will include duplicate test chambers at 100% vs. duplicate control chambers containing dilution water only. Ten test organisms will be placed in each replicate; therefore, twenty total organisms will be exposed to the 100% effluent and control (dilution) water.

After a 24-hour exposure period, the test chambers will be observed for mortality and abnormal effects. If the combined replicate mortality is ≧20%, the 48-hour LC_{50} test will be initiated.

5.6 48-Hr LC_{50} Test. The definitive test will be conducted by exposing the test organisms to at least 5 effluent concentrations and a dilution water control. The test will be conducted using duplicate beakers per concentration containing 10 test organisms per beaker (=20 per concentration). The test concentrations used will follow one of the logarithmic series presented in Table 1. The most common concentration series used is 10-100% (10, 18, 32, 56 and 100%). Several effluent toxicity tests conducted by ABC have shown that most industrial effluents are toxic within this range.

The test organisms will be placed in the test chambers by stratified random assignment within 30 minutes after solution preparations. The test chambers will be observed for mortality and/or abnormal effects every 24 hours, with a record maintained of mortality and abnormal behavior for each concentration tested.

5.7 Test Procedure - Chemical and Physical. Upon receipt of the test effluent, the following parameters will be measured of the 100% effluent: temperature, dissolved oxygen, pH, conductivity and hardness. These same parameters, except hardness, will be measured at 0 and 48 hours of definitive testing in one replicate of the control, low concentration, middle concentration and high concentration solutions.

5.8 Analysis of Results. The results of the definitive study will be statistically analyzed for 24 and 48 hour LC_{50} values and their corresponding 95 percent confidence limits. These values will be determined by an LC_{50} computer program developed by Stephan et al. (11) or by manual methods described by Litchfield-Wilcoxon (12) and Stephan (13).

5.9 Report. One copy of the report will be submitted. The report will contain all original raw data. Additional copies will be provided, if requested by the sponsor, at a copy rate of $50 per copy. The report will include the following:

5.9.1 Study dates, name and address of test facility.

5.9.2 Objectives and test methods.

5.9.3 Reference to the statistical methods used for data analysis.

5.9.4 Description of test effluent (date of receipt, storage conditions, physical characteristics, and method of preparing test concentrations).

5.9.5 Description of test design.

5.9.6 Summary of the data analysis, mortality observations and test water quality.

5.9.7 Location of raw data.

5.9.8 List and signatures of study personnel.

5.9.9 GLP compliance statement by study director and a statement by ABC's Quality Assurance Unit.

5.9.10 The report appendix will contain the original raw data for mortality observations and water quality, letter of test authorization, letters of authorized protocol changes and a copy of the approved protocol.

5.10 Data Retention. All original raw data generated in the preliminary and definitive studies will be provided to the study sponsor in the appendix to the final report. A copy of the data will be retained in ABC's archives.

6.0 PROTOCOL CHANGES

In the event that modifications of this protocol are deemed necessary, a written statement of any changes and reason(s) proposed by the study sponsor or ABC will be submitted to the other party. All agreed changes will be expressed in writing, signed and dated by the sponsor's study director. The signed changes will be appended to the protocol and included with the final report. This protocol is subject to revision when felt necessary to comply with changes in federal regulations and state of the art methodology. The protocol for a specific study will not be changed while the study is in progress without discussion with the study sponsor.

7.0 SPONSOR AUTHORIZATIONS DURING THE STUDY

Should a problem develop while the study is in progress, ABC will notify the study director within 24 hours. The problem and suggested test modifications will be discussed by telephone. ABC will proceed with the changes felt necessary upon the verbal authorization of the study director. A letter for written authorization will then be submitted by ABC to the study director and handled in the same manner discussed in section 6.0.

8.0 TEST-SPECIFIC INFORMATION

8.1.1 General. The following items will be addressed for each static bioassay. This information is necessary to be in compliance with Good Laboratory Practice Regulations (8). Sections 8.2, 8.3, 8.7 and 8.9 are to be completed by the study sponsor. Sections 8.4, 8.5 and 8.6 will be completed by ABC.

8.1.2 GLP Compliance. To be in compliance with the Federal Good Laboratory Practice regulations, the report of the investigation conducted utilizing this protocol must contain a statement that the study was conducted in accordance with one of the following GLP's.

1. U.S. F.D.A. Good Laboratory Practice Regulations (21 CFR 58).

2. U.S. E.P.A. Good Laboratory Practice Standards; Toxic Substances Control (40 CFR 792).

3. U.S. E.P.A. Good Laboratory Practice Standards; Pesticide Programs (40 CFR 160).

4. OECD Principles of Good Laboratory Practice; Annex 2 C(81) 30(Final).

5. Any state or local GLP which may apply.

The specific GLP regulation, for which this study specific protocol and resultant study must be in compliance with, is to be designated by the study sponsor in section 8.8 of this protocol.

8.2 Study Sponsor:

8.2.1 Company Molycorp, Incorporated

8.2.2 Address 3 Miles East of Questa on State Road 38
P.O. Box 469, Questa, New Mexico 87556

8.2.3 Sponsor's Study Director

Mr. Fred Martinez — Name
Analyst — Title

8.3 Effluent:

8.3.1 Sample Number Outfall 002

8.3.2 NPDES Permit Number NM-0022306

8.3.3 Physical Description Clear Liquid

8.3.4 Handling Precautions Normal Precautions

8.4 Test Concentrations: To be determined after 24-hour screen of 100% effluent and consultation with study sponsor.

8.4.1 Sponsor's study director notified of screen results:
Date 1/9/87 ABC Biologist Scott Jeffer
100% effluent screen = ____ ≧20% mortality ✓ <20% mortality

8.4.2 Definitive test concentrations agreed upon:
N/A

8.5 Test Organisms:

8.5.1 Species Daphnia magna

8.5.2 Supplier ABC in-house culture

8.6 Study Dates:

8.6.1 Proposed starting date of definitive study 1/8/87

8.6.2 Proposed completion date of definitive study 1/22/87

8.7 ABC Study Personnel:

8.7.1 Study Director

Alan D. Forbis — Name
Supervisor, Invertebrate and Bioconcentration Investigations — Title

8.7.2 Aquatic Manager

Carl M. Thompson	Aquatic Toxicology Manager
Name	Title

8.8 GLP Compliance (check the most appropriate):

EPA-FIFRA____; EPA-TSCA____; FDA____; OECD____; Other NPDES.

8.9 Protocol Approvals. The following is to be signed by the appropriate study personnel:

8.9.1 Sponsor's Study Director

Fred Mortner	Analyst	1/15/87
Name	Title	Date

8.9.2 ABC's Study Director

	Supervisor, Invertebrate and Bio. Investigations	1/6/87
Name	Title	Date

8.9.3 ABC's Manager

Carl M. Thompson	Aquatic Tox. Manager	1/6/87
Name	Title	Date

8.10 Authorization to return the test material and sample:

8.10.1 Sponsor's authorized agent to receive the test material and sample.

Fred Mortner	Analyst
Name	Title

8.10.2 Shipping Address.

Molycorp, Incorporated
Company

3 Miles East of Questa Highway 38
Street Address

Questa	New Mexico	87556
City	State	Zip Code

505-586-0212
Phone Number

8.10.3 Hazardous shipment information.

Proper Shipping Name

Class of Restricted Article (e.g. flammable, poison, radioactive, etc.)

TABLE 1: Guide to selection of experimental concentrations based on progressive bisection of intervals on logarithmic scale.

Col. 1	Col. 2	Col. 3	Col. 4	Col. 5
10.0	10.0	10.0	10.0	10.0
				8.7
			7.5	7.5
				6.5
		5.6	5.6	5.6
				4.9
			4.2	4.2
				3.7
3.2	3.2	3.2	3.2	3.2
				2.8
			2.4	2.4
				2.1
		1.8	1.8	1.8
				1.55
			1.35	1.35
				1.15
1.0	1.0	1.0	1.0	1.0

TABLE 2: Chemical Characteristics of Well Water Used by ABC's Aquatic Toxicology Division.

Parameters	Concentration
Temperature	15-20°C[b]
Dissolved Oxygen[a]	9.2-10.1 ppm[b]
pH	7.8-8.3[b]
Hardness ($CaCO_3$)	225-275 ppm[b]
Alkalinity ($CaCO_3$)	325-375 ppm[b]
Conductivity	700 µmhos/cm
NO_3-N	0.58 ppm
NO_3-NO_2-N	0.69 ppm
PO_4-P	<0.50 ppm
Aluminum	<20 ppb
Cadmium	<3 ppb
Chromium	<5 ppb
Cobalt	<5 ppb
Copper	<5 ppb
Iron	<5 ppb
Lead	<20 ppb
Nickel	<10 ppb
Silver	<5 ppb
Zinc	<1 ppb
Measured organophosphorus pesticides	<0.10 ppb
Measured organochlorine pesticides plus PCB's	<0.50 ppb

[a]After aeration.

[b]Represents seasonal variation, with the monthly range not exceeding 10%.

Sample dates: Trace elements = May 12, 1986, organophosphate and organochlorine analyses = May 12, 1986.

9.0 REFERENCES

(1) U.S. Congress. 1977. Clean Water Act. Public Law 95-217. Federal Register, Dec. 27, 1977, 91 STAT 1566-1611.

(2) U.S. Environmental Protection Agency. 1983. Effluent Toxicity Testing and Biomonitoring Workshop. Feb. 22-23, 1983, Dallas, Texas.

(3) Peltier, William. 1978. Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms. U.S. Environmental Protection Agency, EPA-660/4-78-012, revised July, 1978. 52 p.

(4) Committee on Methods for Toxicity Tests with Aquatic Organisms. 1975. Methods for Acute Toxicity Tests with Fish, Macroinvertebrates and Amphibians. Environmental Protection Agency, Ecological Research Series EPA-660/3-75-009, April, 1975. 61 p.

(5) American Society for Testing and Materials. 1980. Standard practice for conducting basic acute toxicity tests with fish, macroinvertebrates and amphibians. May, 1980, ASTM Committee E-35.23. 25 p.

(6) American Society for Testing and Materials. 1983. Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates and Amphibians. November, 1983, ASTM Committee E-47.01. 37 p.

(7) American Public Health Association. 1980. Standard Methods for the Examination of Water and Wastewater. 15th ed. Washington, D.C. 1134 p.

(8) (a) U.S. Food and Drug Administration. 1978. Nonclinical Laboratory Studies, Good Laboratory Practice Regulations (21 CFR, Part 58). Federal Register, Vol. 43, No. 247: 59986-60025.

(b) U.S. Environmental Protection Agency. 1983. Pesticide Programs; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 160). Federal Register, Vol 48, No. 230: 53946-53969.

(c) U.S. Environmental Protection Agency. 1983. Toxic Substances Control; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 792). Federal Register, Vol. 48; No. 230:53922-53944.

(d) Organization for Economic Cooperation and Development. 1981. OECD Guidelines for Testing of Chemicals, Principles of Good Laboratory Practice Annex 2, C(81) 30(Final):7-28.

(9) Pennak, R. W. 1978. Fresh-Water Invertebrates of the United States, 2nd Ed. 365 p.

(10) U.S. Environmental Protection Agency. 1979. Static culture methods for sheepshead minnows (Cyprinodon variegatus) and mysid shrimp (Mysidopsis bahia). Unpublished manuscript, U.S. EPA, Athens, GA.

(11) Stephan, C. E., K. A. Busch, R. Smith, J. Burke and R. W. Andrew. 1978. A computer program for calculating an LC_{50}. U. S. Environmental Protection Agency, Duluth, Minnesota, pre-publication manuscript, August, 1978.

(12) Litchfield, J. T., Jr. and F. Wilcoxon. 1949. A Simplified Method of Evaluating Dose-Effect Experiments. Jour. Pharm. Exp. Ther. 96:99-113.

(13) Stephan, C. 1977. Methods for calculating an LC_{50}, p. 65-84. In F. L. Mayer and J. L. Hamelick (eds.). Aquatic Toxicology and Hazard Evaluation. ASTM Special Technical Publication 634. ASTM. Philadelphia.

Assessment of Acute Toxicity of 002
Daphnia Magna

April 1987

ANALYTICAL BIO-CHEMISTRY LABORATORIES, INC.
P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

April 27, 1987

Molycorp, Incorporated
Attn: Mr. Fred Martinez
3 Miles East of Questa, on State Road 38
P.O. Box 469
Questa, New Mexico 87556

RE: Assessment of the Acute Toxicity of Outfall 002 to Daphnia magna, ABC Study Number 35725.

Dear Mr. Martinez:

Please find enclosed Analytical Bio-Chemistry Laboratories' final static report for the above cited study. We hope you find the report to your satisfaction.

If you have any questions, please do not hesitate to give us a call.

Sincerely,

Scott Frazier
Biologist I

SF/rml

Enclosures

Submitted By: Analytical Bio-Chemistry Laboratories, Inc.
7200 East ABC Lane
P. O. Box 1097
Columbia, Missouri 65205
(314) 474-8579

Prepared By:

Scott Frazier 4/27/87
Scott Frazier — Date
Biologist I

David Burgess 4-27-87
David Burgess — Date
Biologist II

Approved By:

Alan D. Forbis 4/27/87
Alan D. Forbis — Date
Supervisor, Invertebrate/Bioconcentration
Investigations/Study Director

Phillip M. Buckler 4/27/87
Phillip M. Buckler — Date
Quality Assurance Supervisor

Carl M. Thompson 4-27-87
Carl M. Thompson — Date
Aquatic Toxicology Manager

:rml14

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PROJECT PERSONNEL

The Study Director of the project for Analytical Bio-Chemistry Laboratories, Inc. was Alan D. Forbis, Supervisor, Invertebrate/ Bioconcentration Investigations. Supervising the conduct of the study for Molycorp, Incorporated was Mr. Fred Martinez. The following ABC personnel assisted with various phases of the study.

Name	Title	Initials
Alan D. Forbis	Supervisor, Invertebrate/ Bioconcentration Investigations	AF
Scott Frazier	Biologist I	SF
Michael Ruddy	Biological Technician I	MR
James G. Muckerman	Biological Technician I	JGM
David Burgess	Biologist II	DB
Jeffrey Latham	Biological Technician I	JL

INVERTEBRATE ACUTE TOXICITY COMPENDIUM

Subject: "Assessment of the Acute Toxicity of Outfall 002 to *Daphnia magna*," ABC Study Number 35725.

Sponsor: Molycorp, Incorporated

Testing Facility: Analytical Bio-Chemistry Laboratories, Inc.
Aquatic Toxicology Division
P.O. Box 1097
Columbia, Missouri 65205 (314-474-8579)

Location of Original Raw Data and Final Report:

Molycorp, Incorporated
3 Miles East of Questa, on State Road 38
Questa, New Mexico 87556

Test Material: Outfall 002

Test Concentrations: Control and 100%

Dilution Water: Hard reconstituted water.

Test Dates: Initiation - April 9, 1987

Termination - April 10, 1987

Length of Study: 24-hours

Results: 24-hour mortality at 100% effluent = 0%

Test Species: *Daphnia magna*

Source of Organisms: ABC Laboratories in-house culture.

Age of Organisms at Study Initiation: First-instar (≤24-hours old)

SUMMARY

The acute toxicity of Outfall 002 to Daphnia magna was assessed for U.S. EPA Region 6 NPDES permit requirements using the methods outlined by the Committee on Methods for Toxicity Tests with Aquatic Organisms. Water quality parameters of temperature, dissolved oxygen, conductivity, hardness, alkalinity and pH were measured on receipt of the sample and were within acceptable limits.

The results of the 24-hour static Daphnia magna toxicity screen indicated that no mortality was observed after the 24-hour exposure period. Therefore, a definitive 48-hour static acute study was not required. All reported values were based upon nominal concentrations.

The no-effect level for Outfall 002 was <100% effluent after 24-hours, based on the lack of mortality and abnormal effects. The surfacing in both the control and 100% concentration were judged to be a result of daphnid molting and not an abnormal effect.

INTRODUCTION

This static bioassay was performed by the Aquatic Toxicology Division of Analytical Bio-Chemistry Laboratories, Inc., Columbia, Missouri, for Molycorp, Inc., from April 9, 1987 to April 10, 1987. The purpose of this test was to assess toxicity of a 100% solution of Outfall 002 to *Daphnia magna*. The assessment was designed to meet the NPDES permit process in U.S. EPA Region 6 (1) requiring biomonitoring of industrial discharges. The screen test was conducted following the procedures outlined in ABC protocol 8301, as approved by Mr. Fred Martinez; Molycorp, Inc. on April 16, 1987.

METHODS AND MATERIALS

The procedures applied for the 24-hour toxicity screen were basically those described in Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms (2), Methods for Acute Toxicity Tests with Fish, Macroinvertebrates and Amphibians (3), Standard Practice for Conducting Acute Toxicity Tests with Fishes, Macroinvertebrates and Amphibians (4), Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents and Fishes, Macroinvertebrates and Amphibians (5), and Standard Methods for Examination of Water and Wastewater (6).

I. Test Organisms

The test lot of *Daphnia magna* was obtained from an in-house culture at ABC. The taxonomic key presented by Pennak (7) was used to identify the test daphnids to species. All test daphnids were held in a controlled temperature area at 20 (±2)°C. The lighting was 50-70 footcandles on a 16-hour daylight photoperiod. During the holding period, the daphnids were fed algae (*Selenastrum capricornutum*) supplemented with a suspension of Tetramin®, cereal leaves and yeast at least every 3 days. First-instar daphnids (<24-hours old) were selected for testing and were not fed during the test.

II. Test Procedure

The static *Daphnia* bioassay was conducted in 250-ml glass beakers containing 200 ml of sample or hard reconstituted water prepared from the following compounds in the amounts stated per liter of deionized water:

192 mg $NaHCO_3$

120 mg $CaSO_4 \cdot 2H_2O$

120 mg $MgSO_4$

8 mg KCl

The reconstituted water was prepared to yield a total hardness of 160-180 mg/l (as $CaCO_3$) a total alkalinity of 110-120 mg/l (as $CaCO_3$), and a pH of 7.6-8.0 (3).

These vessels were kept at 20 (±2.0) °C in a temperature controlled area. The lighting was maintained at 50-70 footcandles on a 16-hour daylight photoperiod.

A 24-hour screen test, composed of duplicate 100% effluent chambers was performed to estimate the toxicity of the effluent. Also included was a duplicate ABC hard reconstituted water control. A five-six concentration static acute toxicity study would be performed if ≥20% mortality was observed after 24-hours in the screen test.

III. Test Material

The Outfall 002 sample (NPDES Permit Number NM-0022306) was received on April 9, 1987 as a clear liquid and was received at 6°C. The sample was allowed to warm to test temperature (22°C) before daphnids were added to test solutions.

RESULTS

After 24-hours, the results indicated no mortality of *Daphnia magna* to Outfall 002. The no effect concentration was <100% effluent based on the lack of mortality and abnormal effects. The surfacing in both the control and 100% concentration were judged to be a result of daphnid molting and not an abnormal effect. Since no mortality was observed after 24-hours in the screen test, a definitive study was not required per biomonitoring guidelines established for U.S. EPA Region 6.

Table 1 presents the water quality parameters measured on sample receipt. Hardness and alkalinity (as $CaCO_3$) and conductivity for the Outfall 002 sample were 1000-1082 mg/l, 152-166 mg/l and 1200 µMhos/cm, respectively. Dissolved oxygen and pH for this sample were 9.1 mg/l and 7.4, respectively, and were considered adequate for testing (3).

The study was conducted following the intent of the Good Laboratory Practice Regulations (8) and the final report was reviewed by Analytical Bio-Chemistry Laboratories' Quality Assurance Unit. All original raw data were provided to Molycorp, Incorporated with a copy retained at Analytical Bio-Chemistry Laboratories.

TABLE 1

Water Quality Measurements of 100%
Outfall 002 Upon Sample Receipt

	100% Outfall 002
Temperature[a]	6°C*
Dissolved Oxygen[b]	9.1
pH[c]	7.4
Conductivity[d]	1200
Hardness[e]	1000-1082
Alkalinity[e]	152-166

[a]Temperature °C - Monitored with a mercury thermometer.

[b]Dissolved oxygen concentrations (mg/l) - Dissolved Oxygen Probe (YSI Dissolved Oxygen System Model 54). Dissolved oxygen saturation at 6°C is 12.5 mg/l.

[c]pH - pH Probe (Corning Model 476182) used with a Corning Model 125 pH and mV meter.

[d]Conductivity (µMhos/cm) - Conductivity Meter (YSI Model 33).

[e]Hardness and alkalinity (mg/l as $CaCO_3$) - HACH Model AL-DT and HAC-DT.

*Test solutions allowed to warm to test temperature (22°C) before addition of daphnids. Dissolved oxygen saturation at 22°C is 8.8 mg/l.

LITERATURE CITED

(1) U.S. Environmental Protection Agency. 1983. Effluent Toxicity Testing and Biomonitoring Workshop. Feb. 22-23, 1983, Dallas, Texas.

(2) Peltier, William. 1978. Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms. U.S. Environmental Protection Agency, EPA-660/4-78-012, revised July 1978. 52 p.

(3) Committee on Methods for Toxicity Tests with Aquatic Organisms. 1975. Methods of Acute Toxicity Tests with Fish, Macroinvertebrates and Amphibians. Environmental Protection Agency, Ecological Research Series EPA-660/3-75-009, April 1975.

(4) American Society for Testing and Materials. 1980. Standard Practice for Conducting Basic Acute Toxicity Tests with Fish, Macroinvertebrates and Amphibians. May 1980, ASTM Committee E-35.23. 25 p.

(5) American Society for Testing and Materials. 1983. Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates and Amphibians. November 1983, ASTM Committee E-47.01. 37 p.

(6) American Public Health Association. 1980. Standard Methods for the Examination of Water and Wastewater. 15th ed. Washington, DC. 1134 p.

(7) Pennak, R. W. 1978. Freshwater Invertebrates of the United States. John Wiley & Sons, New York, 2nd ed. 803 p.

(8) U.S. Environmental Protection Agency. 1983. Toxic Substances Control; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 792). Federal Register, Vol. 48; No. 230:53922-53944.

QUALITY ASSURANCE STATEMENT

Quality Assurance Statement for Report #35725 entitled, "Assessment of the Acute Toxicity of Outfall 002 to *Daphnia magna*," for Mr. Fred Martinez, Molycorp, Incorporated; Questa, New Mexico.

In accordance with ABC Laboratories' intent that all studies conducted at our facilities are designed and function in conformance with good laboratory practice regulations and the protocols for individual laboratory studies, an inspection of the final report for Outfall 002 was conducted and found to be in an acceptable form by a member of our Quality Assurance Unit. A final inspection of all data and records on April 23, 1987, indicated that the report submitted to you is an accurate reflection of the study as it was conducted by ABC Laboratories.

Should you have any questions relating to the information provided in this statement or the function of our Quality Assurance Unit, please contact the Quality Assurance Unit at your convenience.

Phillip M. Buckler 4/27/87

Phillip M. Buckler Date
Quality Assurance Supervisor

STUDY COMPLIANCE STATEMENT

Study Compliance Statement for ABC Report #35725 entitled, "Assessment of the Acute Toxicity of Outfall 002 to *Daphnia magna*," for Mr. Fred Martinez, Molycorp, Inc.; Questa, New Mexico.

In accordance with ABC Laboratories' intent that all aquatic toxicity tests conducted by our facility follow good laboratory practices, ABC's study director for the above test herein confirms that the study was conducted in compliance with the U.S. E.P.A. Good Laboratory Practice Standards; Toxic Substances Control (40 CFR 792).

All original raw data were provided to Molycorp, Incorporated, with a copy retained at Analytical Bio-Chemistry Laboratories.

Alan D. Forbis 4/27/87

Alan D. Forbis — Date

ABC Study Director

APPENDIX - RAW DATA

NOTE: Some of the records which appear in this raw data appendix have been provided as photocopies of original records on file at ABC. This has been done by necessity for certain data which are used commonly in several studies at ABC. Such records may include organism culture logs.

EFFLUENT TOXICITY TEST MORTALITY AND BEHAVIORAL OBSERVATIONS

Effluent Designation: Outfall 002

Industry: Molycorp Study No.: 35725

ABC Study Director: A. FORBIS

Form No.: 91 (4/8/87)

Analytical Bio-Chemistry Labs
P. O. Box 1097, 7200 ABC Lane
Columbia, Missouri 65205

☑ Prelim. ☐ Definitive Test Species: Daphnia magna

Dilution Water: Hard Reconstituted No./Vessel: 10 (Lot #: 87-B-2)

Date Initiated: 4/9/87 Test Terminated: 4/10/87

Effluent Concentration (%)		0 Hr. Soln.	24 Hours Dead	24 Hours Obs.	48 Hours Dead	48 Hours Obs.	Dead	Obs.
Control	A	CLR	0	N				
	B	"	0	2-SUR 8-N				
100	A	"	0	N				
	B	"	0	1-SUR 9-N				
Observer Date		SF 4/9/87	SF	4/10/87				

REMARKS: N = Normal, SUR = Surfacing, C = Clumping, ERR = Erratic Movement

TR = Trailing_____, CT = Coated with extraneous material,

Q = Quiescent; OB = On Bottom; CLR = Clear, P = Precipitate,

CLD = Cloudy/colored solution, SF = Surface Film

NOTE: TEST SOLUTIONS AT 22°C AT ZERO HOUR, SF 4/9/87.

Prepared By: Scott Frazier Date: 4/9/87

Checked By: David Bunzen Date: 4-10-87

WATER QUALITY FOR EFFLUENT SAMPLES

Test Material: Outfall 002

Study No.: 35725 Protocol No.: 8301

ABC Study Director: A. FORBIS

Form No.: 348 (2/3/87)

Analytical Bio-Chemistry Labs
7200 ABC Lane, P.O. Box 1097
Columbia, MO 65201

Concentration %	DAY				DAY				DAY				DAY			
	Temp °C	D.O. [a] mg/l	pH [b]	Cond. [c] uMhos/cm	Temp °C	D.O. mg/l	pH	Cond. uMhos/cm	Temp °C	D.O. mg/l	pH	Cond. uMhos/cm	Temp °C	D.O. mg/l	pH	Cond. uMhos/cm

100% Effluent on Receipt	Temp °C	D.O. [a] mg/l	pH [b]	Cond. [c] uMhos/cm	Hard [d] mg/l	Alk. [d] mg/l		Date	Init.
	6	9.1	7.4	1200	1000-1082	152-166		4/9/87	SF/MR

[a] Dissolved oxygen concentrations - Dissolved Oxygen Probe (YSI Dissolved Oxygen System Model 54)
[b] pH Probe (Corning Model 476182) used with Corning Model 125 pH and mV meter
[c] Salinity/conductivity/temperature meter (YSI Model 33)
[d] mg/l as $CaCO_3$; Hach Models HAC-DT and AL-DT, respectively.

Remarks:

Prepared By: Scott Frazier Date: 4/9/87

Checked By: David Burgess Date: 4-10-87

ABC LABS #035725

PREPARATION OF EFFLUENT CONCENTRATIONS (volume/volume)

Effluent Designation: Outfall 002

Industry: Moly Corp

Study No.: 35725

ABC Study Director: A. Forbis

Lab Form No.: 94

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane; P.O. Box 1097
Columbia, MO 65205

☑ PRELIMINARY ☐ DEFINITIVE

Dilution Water Type:

☐ ABC Well Water ☑ ABC Recon. Water (HARD)

☐ Effluent Receiving Water _____ ☐ Other _____

Sample No.	Aliquot Volume (ml)	Dilution Volume (ml)	Final Conc.* (%)
1	0	200	Control
2	200	0	100
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			

Remarks: *prepared by: SF/JL 4/9/87 in duplicate SF 4/9/87

FORM Prepared By: [signature] Date: 4-9-87

Checked By: [signature] Date: 4-10-87

Study Director: [signature] Date: 4/23/87

OBSERVATIONS AND/OR REMARKS FORM

Test Material: Outfall 002
Study No.: 35725
Protocol No.: 8301
ABC Study Director: A. Forbis

Lab Form No.: 55

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P.O. Box 1097
Columbia, Missouri 65205

Date/Initials		Comments
4-9-87	JL	Reconstituted water was prepared in the following manner: 192 mg $NaHCO_3$, 120 mg $CaSO_4 \cdot 2H_2O$, 120 mg $MgSO_4$, and 8 mg KCl, were weighed and put in a 1000 ml volumetric flask of deionized water. This was stirred and sonicated to aid the compounds to go into solution.

Prepared By: Jeffrey E. Latham Date: 4-9-87
Checked By: [illegible] Date: 4/9/87

MOLYCORP INC.

QUESTA-DIVISION

UNOCAL-76

Samples handed over to carrier by Molycorp employee

DATE: 4/8/87

TIME: 7:00 am

CARRIER Fred Martinez

EMPLOYER: Molycorp Inc.

MOLYCORP PERSONNEL: F. Martinez

Sample(s) delivered to ABC lab for analysis. Please sign and date this letter upon receipt of sample(s)

Sample(s) received by: ABC Laboratories Inc.
7200 ABC Lane
P.O. Box 1097
Columbia, MO 65205
(314) 474-8579

for the following analyses: Toxicity testing

Received by: Scott Frazier

DATE: 4/9/87

Please return this letter with the analytical results, signed and dated below.

Scott Frazier 4/23/87
Date

MOLYCORP

QUESTA - DIVISION

UNOCAL- 76

This sheet represents a chain of custody for effluent samples collected at outfall 002

Located in Taos County, Questa, New Mexico on 4/8/87

Sample bottles were prepared by Molycorp employees in accordance with guidance set forth by the U.S. Environmental Protection Agency for the existing program permit. Sampling procedures were also followed in accordance with permit requirments.

Sample (s) Identification 002 outfall last portion of composite 7:00am.

Sample (s) Identification

By: Fred Moeller

INVERTEBRATE CULTURE RECORD

Species: DAPHNIA [illegible]

Lot No.: 87-B-[illegible]

Date Initiated: 2-26-87

Lab Form No: 52

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P. O. Box 1097
Columbia, MO 65205

Date	No. Adult Alive	Instar Present	Fed & Changed	Study I.D.	Comments*	Obs. By:
2/26	—	~30	✓		FED ~ 200 ml Algae + 2.1 ml [illegible] Feed	MR
3/1	30	—	✓		" "	MR
3/3	30	—	✓		" "	JL
3/5	30	—	✓		" "	MR
3/8	30	~100	✓		" "	SF
3/10	30	~350	✓		" "	MR
3/12	30	~50	✓		"	MR
3/13	—	—	—	35565-140		SF
3/15	30	~500	✓		"	DB
3/17	30	~400	✓		"	MR
3/18	30	0	✓		"	MR
3/19	30	~175	✓		"	MR
3/20	—	—	—	35608-120		SF
3/22	29	~200		✓	"	JGM
3/24	29	~350	✓		"	MR
3/26	29	~300	✓		"	MR
3/29	28	~500	✓		" "	JGM
3/31	27	~250	✓		" "	WR

"This is an exact copy of The original document"
By D. BURGESS date 4-10-87

Prepared by: [signature] Date: 2-26-87

Checked by: [signature] Date: 4/1/87

*Mirage Feed is Tetramin, Cereal Leaves & Yeast MR 2-26-87

INVERTEBRATE CULTURE RECORD

Species: DAPHNIA MAGNA

Lot No.: 87-B-2

Date Initiated: 2-26-87

Lab Form No: 52

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P. O. Box 1097
Columbia, MO 65205

Date	No. Adult Alive	Instar Present	Fed & Changed	Study I.D.	Comments*	Obs. By:
4-1	—	—	—	35672	130 INSTAR FOR TEST	MR
4-1	—	—	—	35679	60 INSTAR FOR TEST	MR
4-2	26	~200	—		FED ALGAE (200 mL) + 0.10 mL MIRAGE FOOD	MR
4/5	26	~400	✓		" "	SF
4/6	26	~200	✓		" "	JGM
4-8	26	~300	✓		" "	MR
4/9	—	—	—	35642	210 instar for test	SF
4/9	—	—	—	35725	~~30~~ 40 ① INSTAR FOR TEST	JL
4/9	—	—	—	35693	210 INSTAR FOR TEST	JL
4/9	26	~100	✓		" "	JGM

"This is an exact copy of The original document"

By D. BURGESS date 4-10-87

Prepared by: William C. Riddle Date: 4-1-87

Checked by: Darrel Burgess Date: 4-10-87

* MIRAGE FOOD IS TETRAMIN & CEREAL LEAVES & YEAST MR 4-1-87

① ENTRY ERROR JL 4-9-87

DEFINITIVE CONCENTRATION NOTIFICATION

Firm: Molycorp, INC
Address: 3 Miles E of Questa, St Rd 38
City/State/Zip: Questa N.M.
Phone: ( )

Lab Form No.: 186
Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P.O. Box 1097
Columbia, MO 65202

Sponsor's Study Director: Mr. Fred Martinez
Person notified: same (✓) Other:

Date of Notification: 4/14/87

Notification of preliminary testing results with the following definitive test levels agreed upon:

Test Material	ABC #	Test Species	Test Conc. (mg/l)
Outfall 002	35725	Daphnia magna	N/A

Sponsor called and was informed of 0% mortality in screen test.

Notified By: Scott Frazier Date: 4/14/87

ANALYTICAL BIO-CHEMISTRY LABORATORIES, INC.
P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

RECEIVED
FEB 20 '87

ABC PROTOCOL NO. 8301

(August 21, 1984)

Effluent Static Bioassay with Daphnia magna or
Mysid Shrimp (Mysidopsis bahia) to
Meet EPA Region 6 NPDES Biomonitoring Guidelines

ABC Study Number 35725

Test Material Outfall 002

Test Species Daphnia magna

1.0 INTRODUCTION

The use of bioassays in the identification and control of industrial effluents is rapidly increasing. Indeed, aquatic toxicity tests are presently being incorporated into the NPDES guidelines of the Federal Clean Water Act (1). To assist industry in meeting requirements set forth by legislation and regulatory agencies, Analytical Bio-Chemistry Laboratories, Inc., offers a complete range of aquatic bioassay tests of industrial effluents.

As part of the NPDES permit process in U.S. EPA Region 6, a biomonitoring requirement has been imposed for certain permit holders in that area. Discharges subject to the new regulations in Region 6 (Texas, New Mexico, Oklahoma, Arkansas and Louisiana) will be required to submit aquatic toxicity data on their effluents to EPA on a periodic basis.

This ABC protocol discusses the biomonitoring guidelines in Region 6 and describes ABC's technical approach to assist industry in complying with these regulations.

2.0 OBJECTIVES

The primary objective of the toxicity test described herein is to evaluate the acute toxicity of an effluent to freshwater or marine invertebrates under static conditions. The species selected for this testing include Daphnia magna (freshwater effluent, <5 ppt salinity) and Mysidopsis bahia (saltwater effluent, ≥5 ppt salinity). The effluent will first be tested on a 100% effluent screen basis. If the screen results in ≥20% mortality of test organisms, a 48-hour, five concentration LC_{50} test will be conducted. An LC_{50} is the approximate concentration (percent) of test effluent that produces 50 percent mortality of test organisms after prescribed intervals (24 and 48 hours).

3.0 TESTING FACILITY

The study will be conducted by the Aquatic Toxicology Division of Analytical Bio-Chemistry Laboratories, Inc., 7200 East ABC Lane, P. O. Box 1097, Columbia, Missouri 65205 (314-474-8579).

4.0 REGION 6 BIOMONITORING REQUIREMENT

The following biomonitoring requirement is now being included in the NPDES permit process for many major industrial discharges in EPA Region 6. The permit language given here was that presented by the U.S. EPA at a regional workshop on the matter (2):

> The permittee shall determine if the effluent from this outfall is lethal to twenty (20) percent or greater of the culture of test organisms by use of the "Range-Finding Screening Test," set out in "Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms," EPA-600/4-78-012 (Revised July, 1978) (3).

Organisms for this test shall be Daphnia sp. if the effluent is less than five (5) parts per thousand salinity or Mysid sp. if the effluent is greater than five (5) parts per thousand salinity. This screening test will be conducted within sixty (60) days of effectiveness of the biomonitoring requirements. Tests will be conducted once each quarter for a duration of two years utilizing a static method for 24 hours and following this dilution scheme only:

Effluent samples*	-	100 percent by volume
Dilution water	-	0 percent by volume

*24-hour composite; refrigerated after collection

If at any time during the two year testing period a test shows a lethality of twenty (20) percent or more of the test organisms, the permittee shall within twenty-four (24) hours conduct a replacement static 48-hour median lethal concentration (LC_{50}) test on the originally collected sample. Replacement of effluent samples shall be once per 24 hours. Organisms for this test shall be Daphnia sp. if the effluent is less than five (5) parts per thousand salinity and reconstituted fresh water (EPA-600/4-78-012 Section 4) shall be used for dilution. If the effluent contains greater than five (5) parts per thousands salinity, Mysid sp. shall be used as the test organism, and reconstituted seawater will be used as dilution water (EPA-600/4-78-012 Section 4). The remaining LC_{50} methodology is available in EPA-600/4-78-012.

All screening and LC_{50} test results shall be reported with the Discharge Monitoring Reports.

It should be noted by the permittee that slight changes from the above permit language (e.g., pH modification of effluent) may be incorporated into his specific permit. Attention should be given to the permit requirements for each effluent before testing is initiated. ABC requires that a copy of the permit's biomonitoring clause be forwarded to ABC prior to testing so that consideration can be give to pH modification, etc.

5.0 METHODS AND MATERIALS

5.1 Methodology. The methods applied for both the preliminary and definitive phases of this test are basically those described in Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms (3), Methods for Acute Toxicity Tests with Fish, Macroinvertebrates, and Amphibians (4), Standard Practice for Conducting Acute Toxicity Tests with Fishes, Macroinvertebrates, and Amphibians (5), Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates and Amphibians (6), and Standard Methods for the Examination of Water and Wastewater (7). This protocol has been written to comply with the federal Good Laboratory Practice Regulations (8).

5.2 Test Organisms. The species recommended by the EPA for this testing are Daphnia sp. and Mysid sp. Daphnia sp. are to be used when the effluent salinity is <5 parts per thousand (ppt) and Mysid sp. are to be used when the effluent salinity ≥5 ppt. The species maintained by ABC, and acceptable by EPA, for this testing are Daphnia magna and Mysidopsis bahia. The test lot of Daphnia magna will be obtained from an in-house culture at ABC. The taxonomic key presented by Pennak (9) will be used to identify to species the test daphnids. All test daphnids will be held in a controlled temperature area at 20 ±2°C. The lighting will be 50-70 footcandles on a 16 hour daylight photoperiod. During the holding period, they will receive a suspension of algae (Selenastrum capricornutum) supplemented with a blended fish food/cerophyl suspension at least every 3 days. First-instar daphnids (<24 hours old) will be selected for testing and will not be fed during the test.

Mysid shrimp (Mysidopsis bahia) will be obtained from a commercial supplier (e.g., Sea Plantations, Inc. or Gulf Breeze Specimens) and maintained in culture at ABC. Mysids will be cultured following the techniques described by the U.S. EPA (10). Test mysids will be selected from a sub-culture of immature individuals.

5.3 Effluent Sample. The sample submitted to ABC for testing must be a 24-hour composite sample, consisting of equal volumes taken every hour (2). The sample should be collected in a glass or polyethylene container, refrigerated after collection, and shipped to arrive at ABC within 24 hours after collection. A one-gallon sub-sample of the 24-hour composite will be satisfactory for the toxicity test(s) required. ABC can provide sample containers and mailing cartons and can assist plant personnel in shipping arrangements so that the sample arrives at ABC in a timely manner.

5.4 Dilution Water. It will not be necessary to collect actual receiving water for effluent dilutions for this test. The biomonitoring regulations recommend the use of reconstituted freshwater or saltwater listed in the EPA effluent methods manual (3). The freshwater dilution water (for Daphnia sp.) will be a hard reconstituted water of 160-180 mg/l hardness (as $CaCO_3$). The reconstituted saltwater (for Mysid sp.) will be prepared from a commercial salt mix similar in composition to that listed in the EPA effluent manual (3) and adjusted to a test salinity of 15-25 ppt.

5.5 Screen Test. The biomonitoring regulations require that a 24-hour 100% effluent screen be conducted before more definitive (LC_{50}) testing is considered. As stated in section 4.0, if ≥20% of the test organisms die in the 100% solution, the permittee shall then conduct a 48-hour static replacement LC_{50} test. This latter test is discussed in section 5.6.

The screen test will be conducted in 250 ml glass beakers containing 200 ml of solution. These test chambers will be held in a controlled temperature area at 20 ±2°C on a 16-hour daylight vs.

8-hour darkness photoperiod with 30-minute dawn/dusk transition periods. The screen test will include duplicate test chambers at 100% vs. duplicate control chambers containing dilution water only. Ten test organisms will be placed in each replicate; therefore, twenty total organisms will be exposed to the 100% effluent and control (dilution) water.

After a 24-hour exposure period, the test chambers will be observed for mortality and abnormal effects. If the combined replicate mortality is ≥20%, the 48-hour LC_{50} test will be initiated.

5.6 48-Hr LC_{50} Test. The definitive test will be conducted by exposing the test organisms to at least 5 effluent concentrations and a dilution water control. The test will be conducted using duplicate beakers per concentration containing 10 test organisms per beaker (=20 per concentration). The test concentrations used will follow one of the logarithmic series presented in Table 1. The most common concentration series used is 10-100% (10, 18, 32, 56 and 100%). Several effluent toxicity tests conducted by ABC have shown that most industrial effluents are toxic within this range.

The test organisms will be placed in the test chambers by stratified random assignment within 30 minutes after solution preparations. The test chambers will be observed for mortality and/or abnormal effects every 24 hours, with a record maintained of mortality and abnormal behavior for each concentration tested.

5.7 Test Procedure - Chemical and Physical. Upon receipt of the test effluent, the following parameters will be measured of the 100% effluent: temperature, dissolved oxygen, pH, conductivity and hardness. These same parameters, except hardness, will be measured at 0 and 48 hours of definitive testing in one replicate of the control, low concentration, middle concentration and high concentration solutions.

5.8 Analysis of Results. The results of the definitive study will be statistically analyzed for 24 and 48 hour LC_{50} values and their corresponding 95 percent confidence limits. These values will be determined by an LC_{50} computer program developed by Stephan et al. (11) or by manual methods described by Litchfield-Wilcoxon (12) and Stephan (13).

5.9 Report. One copy of the report will be submitted. The report will contain all original raw data. Additional copies will be provided, if requested by the sponsor, at a copy rate of $50 per copy. The report will include the following:

5.9.1 Study dates, name and address of test facility.

5.9.2 Objectives and test methods.

5.9.3 Reference to the statistical methods used for data analysis.

5.9.4 Description of test effluent (date of receipt, storage conditions, physical characteristics, and method of preparing test concentrations).

5.9.5 Description of test design.

5.9.6 Summary of the data analysis, mortality observations and test water quality.

5.9.7 Location of raw data.

5.9.8 List and signatures of study personnel.

5.9.9 GLP compliance statement by study director and a statement by ABC's Quality Assurance Unit.

5.9.10 The report appendix will contain the original raw data for mortality observations and water quality, letter of test authorization, letters of authorized protocol changes and a copy of the approved protocol.

5.10 Data Retention. All original raw data generated in the preliminary and definitive studies will be provided to the study sponsor in the appendix to the final report. A copy of the data will be retained in ABC's archives.

6.0 PROTOCOL CHANGES

In the event that modifications of this protocol are deemed necessary, a writtens statement of any changes and reason(s) proposed by the study sponsor or ABC will be submitted to the other party. All agreed changes will be expressed in writing, signed and dated by the sponsor's study director. The signed changes will be appended to the protocol and included with the final report. This protocol is subject to revision when felt necessary to comply with changes in federal regulations and state of the art methodology. The protocol for a specific study will not be changed while the study is in progress without discussion with the study sponsor.

7.0 SPONSOR AUTHORIZATIONS DURING THE STUDY

Should a problem develop while the study is in progress, ABC will notify the study director within 24 hours. The problem and suggested test modifications will be discussed by telephone. ABC will proceed with the changes felt necessary upon the verbal authorization of the study director. A letter for written authorization will then be submitted by ABC to the study director and handled in the same manner discussed in section 6.0.

8.0 TEST-SPECIFIC INFORMATION

8.1.1 General. The following items will be addressed for each static bioassay. This information is necessary to be in compliance with Good Laboratory Practice Regulations (8). Sections 8.2, 8.3, 8.7 and 8.9 are to be completed by the study sponsor. Sections 8.4, 8.5 and 8.6 will be completed by ABC.

8.1.2 GLP Compliance. To be in compliance with the Federal Good Laboratory Practice regulations, the report of the investigation conducted utilizing this protocol must contain a statement that the study was conducted in accordance with one of the following GLP's.

1. U.S. F.D.A. Good Laboratory Practice Regulations (21 CFR 58).

2. U.S. E.P.A. Good Laboratory Practice Standards; Toxic Substances Control (40 CFR 792).

3. U.S. E.P.A. Good Laboratory Practice Standards; Pesticide Programs (40 CFR 160).

4. OECD Principles of Good Laboratory Practice; Annex 2 C(81) 30(Final).

5. Any state or local GLP which may apply.

The specific GLP regulation, for which this study specific protocol and resultant study must be in compliance with, is to be designated by the study sponsor in section 8.8 of this protocol.

8.2 Study Sponsor:

8.2.1 Company Molycorp, Incorporated

8.2.2 Address 3 Miles East of Questa on State Road 38
P.O. Box 469, Questa, New Mexico 87556

8.2.3 Sponsor's Study Director

Mr. Fred Martinez — Name
Analyst — Title

8.3 Effluent:

8.3.1 Sample Number Outfall 002

8.3.2 NPDES Permit Number NM-0022306

8.3.3 Physical Description Clear Liquid

8.3.4 Handling Precautions Normal Precautions

8.4 Test Concentrations: To be determined after 24-hour screen of 100% effluent and consultation with study sponsor.

8.4.1 Sponsor's Study Director notified of screen results:
Date 4/14/87 ABC Biologist [illegible] (4/21/87)
100% effluent screen = _____ ≥20% mortality X <20% mortality

8.4.2 Definitive test concentrations agreed upon:
N/A

8.5 Test Organisms:

8.5.1 Species Daphnia magna

8.5.2 Supplier ABC in-house culture

8.6 Study Dates:

8.6.1 Proposed starting date of definitive study 4/8/87

8.6.2 Proposed completion date of definitive study 4/15/87

8.7 ABC Study Personnel:

8.7.1 Study Director

Alan D. Forbis — Name
Supervisor, Invertebrate and Bioconcentration Investigations — Title

8.7.2 Aquatic Manager

Carl M. Thompson	Aquatic Toxicology Manager
Name	Title

8.8 GLP Compliance (check the most appropriate):

EPA-FIFRA ____; EPA-TSCA ____; FDA ____; OECD ____; Other NPDES .

8.9 Protocol Approvals. The following is to be signed by the appropriate study personnel:

8.9.1 Sponsor's Study Director

Fred Martinez	Analyst	04/16/87
Name	Title	Date

8.9.2 ABC's Study Director

[illegible]	Supervisor, Invertebrate and Bio. Investigations	4/9/87
Name	Title	Date

8.9.3 ABC's Manager

Carl M. Thompson	Aquatic Tox. Manager	4/8/87
Name	Title	Date

8.10 Authorization to return the test material and sample:

8.10.1 Sponsor's authorized agent to receive the test material and sample.

Fred Martinez	Analyst
Name	Title

8.10.2 Shipping Address

Molycorp, Incorporated
Company

3 Miles East of Questa on Highway 38
Street Address

Questa	New Mexico	87556
City	State	Zip Code

505-586-0212
Phone Number

8.10.3 Hazardous Shipment Information

Proper Shipping Name

Class of Restricted Article (e.g. flammable, poison, radioactive, etc.)

TABLE 1

Guide to Selection of Experimental Concentrations Based on Progressive Bisection of Intervals on Logarithmic Scale.

Col. 1	Col. 2	Col. 3	Col. 4	Col. 5
10.0	10.0	10.0	10.0	10.0
				8.7
			7.5	7.5
				6.5
		5.6	5.6	5.6
				4.9
			4.2	4.2
				3.7
3.2	3.2	3.2	3.2	3.2
				2.8
			2.4	2.4
				2.1
		1.8	1.8	1.8
				1.55
			1.35	1.35
				1.15
1.0	1.0	1.0	1.0	1.0

TABLE 2

Chemical Characteristics of Well Water Used by ABC's Aquatic Toxicology Division.

Parameter	Concentration
Temperature	15-20°C[b]
Dissolved Oxygen[a]	9.2-10.1 ppm[b]
pH	7.8-8.3[b]
Hardness ($CaCO_3$)	225-275 ppm[b]
Alkalinity ($CaCO_3$)	325-375 ppm[b]
Conductivity	700 µmhos/cm
NO_3-N	0.58 ppm
NO_3-NO_2-N	0.69 ppm
PO_4-P	<0.50 ppm
Aluminum	<20 ppb
Cadmium	<3 ppb
Chromium	<5 ppb
Cobalt	<5 ppb
Copper	<5 ppb
Iron	<5 ppb
Lead	<20 ppb
Nickel	<10 ppb
Silver	<5 ppb
Zinc	<1 ppb
Measured organophosphorus pesticides	<0.10 ppb
Measured organochlorine pesticides plus PCB's	<0.50 ppb

[a]After aeration.

[b]Represents seasonal variation, with the monthly range not exceeding 10%.

Sample dates: Trace elements = May 12, 1986, organophosphate and organochlorine analyses = May 12, 1986.

9.0 REFERENCES

(1) U.S. Congress. 1977. Clean Water Act of 1977. Public Law 95-217. Federal Register, December 27, 1977, 91 STAT 1566-1611.

(2) U.S. Environmental Protection Agency. 1983. Effluent Toxicity Testing and Biomonitoring Workshop. Feb. 22-23, 1983, Dallas, Texas.

(3) Peltier, William. 1978. Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms. U.S. Environmental Protection Agency, EPA-660/4-78-012, revised July, 1978. 52 p.

(4) Committee on Methods for Toxicity Tests with Aquatic Organisms. 1975. Methods for Acute Toxicity Tests with Fish, Macroinvertebrates and Amphibians. Environmental Protection Agency, Ecological Research Series EPA-660/3-75-009, April, 1975. 61 p.

(5) American Society for Testing and Materials. 1980. Standard practice for conducting basic acute toxicity tests with fish, macroinvertebrates and amphibians. May, 1980, ASTM Committee E-35.23. 25 p.

(6) American Society for Testing and Materials. 1983. Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates and Amphibians. November, 1983, ASTM Committee E-47.01. 37 p.

(7) American Public Health Association. 1980. Standard Methods for the Examination of Water and Wastewater. 15th ed. Washington, D.C. 1134 p.

(8) (a) U.S. Food and Drug Administration. 1978. Nonclinical Laboratory Studies, Good Laboratory Practice Regulations (21 CFR, Part 58). Federal Register, Vol. 43, No. 247: 59986-60025.

(b) U.S. Environmental Protection Agency. 1983. Pesticide Programs; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 160). Federal Register, Vol. 48, No. 230: 53946-53969.

(c) U.S. Environmental Protection Agency. 1983. Toxic Substances Control; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 792). Federal Register, Vol. 48; No. 230:53922-53944.

(d) Organization for Economic Cooperation and Development. 1981. OECD Guidelines for Testing of Chemicals, Principles of Good Laboratory Practice Annex 2, C(81) 30 (Final):7-28.

(9) Pennak, R. W. 1978. Fresh-Water Invertebrates of the United States, 2nd Ed. 365 p.

(10) U.S. Environmental Protection Agency. 1979. Static culture methods for sheepshead minnows (Cyprinodon variegatus) and mysid shrimp (Mysidopsis bahia). Unpublished manuscript, U.S. EPA, Athens, GA.

(11) Stephan, C. E., K. A. Busch, R. Smith, J. Burke and R. W. Andrew. 1978. A computer program for calculating an LC_{50}. U. S. Environmental Protection Agency, Duluth, Minnesota, pre-publication manuscript, August, 1978.

(12) Litchfield, J. T., Jr. and F. Wilcoxon. 1949. A Simplified Method of Evaluating Dose-Effect Experiments. Jour. Pharm. Exp. Ther. 96:99-113.

(13) Stephan, C. 1977. Methods for calculating an LC_{50}, p. 65-84. In F. L. Mayer and J. L. Hamelick (eds.). Aquatic Toxicology and Hazard Evaluation. ASTM Special Technical Publication 634. ASTM. Philadelphia.

Assessment of Acute Toxicity of 002
Daphnia Magna

June 1987

ANALYTICAL BIO-CHEMISTRY LABORATORIES, INC.
P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

SPONSOR COMPANY

Molycorp Incorporated
Attn: Mr. Fred Martinez
3 Miles East of Questa, on State Road 38
P.O. Box 469
Questa, New Mexico 87556

STUDY TITLE

Assessment of the Acute Toxicity of Outfall 002 to *Daphnia magna*

DATA REQUIREMENT

U.S. EPA-TSCA, 40 CFR, Part 797
Guideline NPDES

AUTHOR

Alan D. Forbis
Supervisor, Invertebrate and
Bioconcentration Investigations

STUDY COMPLETED ON

June 26, 1987

PERFORMING LABORATORY

Analytical Bio-Chemistry Laboratories, Inc.
Aquatic Toxicology Division
7200 East ABC Lane
P.O. Box 1097
Columbia, Missouri 65205

ABC LABORATORY PROJECT ID

#36035

Submitted By: Analytical Bio-Chemistry Laboratories, Inc.
7200 East ABC Lane
P.O. Box 1097
Columbia, Missouri 65205
(314) 474-8579

Prepared By:

Scott Frazier 7/22/87
Scott Frazier Date
Biologist I

Approved By:

Alan Forbis (CT) 7-24-87
Alan D. Forbis Date
Study Director/
Supervisor, Invertebrate and
Bioconcentration Investigations

Kent Ediger 7/22/87
Kent Ediger Date
Quality Assurance

Carl M. Thompson 7-24-87
Carl M. Thompson Date
Aquatic Toxicology Manager

/r119

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PROJECT PERSONNEL

The study director of the project for Analytical Bio-Chemistry Laboratories, Inc. was Alan D. Forbis, Supervisor, Invertebrate and Bioconcentration Investigations. Supervising the conduct of the study for Molycorp, Incorporated was Mr. Fred Martinez. The following ABC personnel assisted with various phases of the study.

Name	Title	Initials
Alan D. Forbis	Supervisor, Invertebrate and Bioconcentration Investigations	AF(CT)
Scott Frazier	Biologist I	SF
Michael Ruddy	Biological Technician I	MR
Jeff Latham	Biological Technician I	JL

INVERTEBRATE ACUTE TOXICITY COMPENDIUM

Subject: "Assessment of the Acute Toxicity of Outfall 002 to Daphnia magna",

ABC Study Number: 36035

Sponsor: Molycorp, Incorporated

Testing Facility: Analytical Bio-Chemistry Laboratories, Inc.
Aquatic Toxicology Division
P.O. Box 1097
Columbia, Missouri 65205 (314-474-8579)

Location of Original Raw Data and Final Report:

Molycorp, Incorporated
3 Miles East of Questa, State Road 38
P.O. Box 469
Questa, New Mexico 87556.

Test Material: Outfall 002

Test Concentrations: Control and 100%

Control Water: 160 mg/l as $CaCO_3$ (hardness), 114 mg/l as $CaCO_3$ (alkalinity), 8.2 (pH)

Test Dates: Initiation - June 25, 1987

Termination - June 26, 1987

Length of Study: 24-hours

Results: 24-hour NOEL = 100%

Test Species: Daphnia magna

Source of Organisms: ABC Laboratories in-house culture

Age of Organisms at Study Initiation: First-instar (≤24-hours old)

SUMMARY

The acute toxicity of Outfall 002 to Daphnia magna was assessed for U.S. EPA Region 6 NPDES permit requirements using the methods outlined by the Committee on Methods for Toxicity Tests with Aquatic Organisms. Water quality parameters of temperature, dissolved oxygen, conductivity, hardness, alkalinity and pH were measured on receipt of the sample and were within acceptable limits.

The results of the 24-hour static Daphnia magna toxicity screen indicated that no mortality was observed after the 24-hour exposure period. Therefore, a definitive 48-hour static acute study was not required. All reported values were based upon nominal concentrations.

The no-effect level for Outfall 002 was 100% effluent after 24-hours, based on the lack of mortality and abnormal effects.

INTRODUCTION

This static bioassay was performed by the Aquatic Toxicology Division of Analytical Bio-Chemistry Laboratories, Inc., Columbia, Missouri, for Molycorp, Inc., from June 25, 1987 to June 26, 1987. The purpose of this test was to assess toxicity of a 100% solution of Outfall 002 to *Daphnia magna*. The assessment was designed to meet the NPDES permit process in U.S. EPA Region 6 (1) requiring biomonitoring of industrial discharges. The screen test was conducted following the procedures outlined in ABC protocol 8301, as approved by Mr. Fred Martinez; Molycorp, Inc. on July 1, 1987.

METHODS AND MATERIALS

The procedures applied for the 24-hour toxicity screen were basically those described in Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms (2), Methods for Acute Toxicity Tests with Fish, Macroinvertebrates, and Amphibians (3), Standard Practice for Conducting Acute Toxicity Tests with Fishes, Macroinvertebrates and Amphibians (4), Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents and Fishes, Macroinvertebrates and Amphibians (5), and Standard Methods for Examination of Water and Wastewater (6).

I. Test Organisms

The test lot of *Daphnia magna* was obtained from an in-house culture at ABC. The taxonomic key presented by Pennak (7) was used to identify the test daphnids to species. All test daphnids were held in a controlled temperature area at 20 (±2)°C. The lighting was 50-70 footcandles on a 16-hour daylight photoperiod. During the holding period, the daphnids were fed algae (*Selenastrum capricornutum*) supplemented with a suspension of Tetramin®, cereal leaves and yeast at least every 3 days. First-instar daphnids (<24-hours old) were selected for testing and were not fed during the test.

II. Test Procedure

The static *Daphnia* bioassay was conducted in 250-ml glass beakers containing 200 ml of sample or ABC hard reconstituted water prepared from the following compounds in the amounts stated per liter of deionized water:

192 mg $NaHCO_3$
120 mg $CaSO_4 \cdot 2H_2O$
120 mg $MgSO_4$
8 mg KCl

The reconstituted water was prepared to yield a total hardness of 160-180 mg/l (as $CaCO_3$) and a total alkalinity of 110-120 mg/l (as $CaCO_3$).

These vessels were kept at 20 (±2.0) °C in a temperature controlled area. The lighting was maintained at 50-70 foot-candles on a 16-hour daylight photoperiod.

A 24-hour screen test, composed of duplicate 100% effluent chambers was performed to estimate the toxicity of the effluent. Also included was a duplicate ABC hard reconsituted water control. A five-six concentration static acute toxicity study would be performed if ≥20% mortality was observed after 24-hours in the screen test.

III. Test Material

The Outfall 002 sample (NPDES Permit Number NM-0022306) was received on June 25, 1987 as a clear liquid and was received at 12°C. The sample was allowed to warm to test temperature (20 ±2°C) before daphnids were added to test solutions.

RESULTS

After 24-hours, the results indicated no mortality of Daphnia magna to Outfall 002. The no-effect concentration was 100% effluent based on the lack of mortality and abnormal effects. Since no mortality was observed after 24-hours in the screen test, a definitive study was not required per biomonitoring guidelines established for U.S. EPA Region 6.

Table 1 presents the water quality parameters measured on sample receipt. Hardness and alkalinity (mg/l as $CaCO_3$) and conductivity for the Outfall 002 sample were 900, 162 and 1320 µMhos/cm, respectively. Dissolved oxygen and pH for this sample were 7.9 mg/l and 7.4, respectively, and were considered adequate for testing (3).

The study was conducted following the intent of the Good Laboratory Practice Regulations (8) and the final report was reviewed by Analytical Bio-Chemistry Laboratories' Quality Assurance Unit. All original raw data were provided to Molycorp, Incorporated with a copy retained at Analytical Bio-Chemistry Laboratories.

TABLE 1

Water Quality Measurements of 100% Outfall 002 Upon Sample Receipt

	100% Outfall 002
Temperature[a]	12*
Dissolved Oxygen[b]	7.9
pH[c]	7.4
Conductivity[d]	1320
Hardness[e]	900
Alkalinity[e]	162

[a]Temperature °C - Monitored with a mercury thermometer.

[b]Dissolved oxygen concentrations (mg/l) - Dissolved Oxygen Probe (YSI Dissolved Oxygen System Model 54). Dissolved Oxygen saturation at 12°C is 10.8 mg/l.

[c]pH - pH Probe (Corning Model 476182) used with a Corning Model 125 pH and mV meter.

[d]Conductivity (μMhos/cm) - Conductivity Meter (YSI Model 33).

[e]Hardness and alkalinity (mg/l as $CaCO_3$) - Colorimetric titration procedure adapted from standard methods.

*Test solutions allowed to warm to test temperature (22°C) before addition of daphnids. Dissolved oxygen saturation at 22°C is 8.8 mg/l.

LITERATURE CITED

(1) U. S. Environmental Protection Agency. 1983. Effluent Toxicity Testing and Biomonitoring Workshop. Feb. 22-23, 1983, Dallas, Texas.

(2) Peltier, William. 1978. Methods of Measuring the Acute Toxicity of Effluents to Aquatic Organisms. U.S. Environmental Protection Agency, EPA-660/4-78-012, revised July 1978. 52 p.

(3) Committee on Methods for Toxicity Tests with Aquatic Organisms. 1975. Methods of Acute Toxicity Tests with Fish, Macroinvertebrates and Amphibians. Environmental Protection Agency, Ecological Research Series EPA-660/3-75-009, April 1975.

(4) American Society for Testing and Materials. 1980. Standard Practice for Conducting Basic Acute Toxicity Tests with Fish, Macroinvertebrates and Amphibians. May 1980, ASTM Committee E-35.23. 25 p.

(5) American Society for Testing and Materials. 1983. Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates and Amphibians. November 1983, ASTM Committee E-47.01 37 p.

(6) American Public Health Association. 1980. Standard Methods for the Examination of Water and Wastewater. 15th ed. Washington, DC. 1134 p.

(7) Pennak, R. W. 1978. Freshwater Invertebrates of the United States. John Wiley & Sons, New York, 2nd ed. 803 p.

(8) U.S. Environmental Protection Agency. 1983. Toxic Substances Control; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 792). Federal Register, Vol. 48; No. 230:53922-53944.

QUALITY ASSURANCE STATEMENT

Quality Assurance Statement for final report #36035 entitled, "Assessment of the Acute Toxicity of Outfall 002 to *Daphnia magna*," for Mr. Fred Martinez, Molycorp, Incorporated; Questa, New Mexico.

In accordance with ABC Laboratories' intent that all studies conducted at our facilities are designed and function in conformance with good laboratory practice regulations and the protocols for individual laboratory studies, an inspection of the final report for Outfall 002 was conducted and found to be in acceptable from by a member of our Quality Assurance Unit. A final inspection of all data and records on July 13, 1987, indicates that the report submitted to you is an accurate reflection of the study as it was conducted by ABC Laboratories.

Should you have any questions relating to the information provided in this statement or the function of our Quality Assurance Unit, please contact the QA Unit at your convenience.

Kent Ediger 7/22/87

Kent Ediger/ Date
Quality Assurance
Officer

STUDY COMPLIANCE STATEMENT

Study Compliance Statement for ABC report #36035 entitled, "Assessment of the Acute Toxicity of Outfall 002 to *Daphnia magna*," for Mr. Fred Martinez, Molycorp, Inc.; Questa, New Mexico.

In accordance with ABC Laboratories' intent that all aquatic toxicity tests conducted by our facility follow good laboratory practices, ABC's study director for the above test herein confirms that the study was conducted in compliance with the U.S. E.P.A. Good Laboratory Practice Standards; Toxic Substances Control (40 CFR 792).

All original raw data were sent to Molycorp, Incorporated, with a copy retained at Analytical Bio-Chemistry Laboratories.

Alan Forbis (T) 7-27-87

Alan D. Forbis Date
ABC Study Director

APPENDIX - RAW DATA

EFFLUENT TOXICITY TEST MORTALITY AND BEHAVIORAL OBSERVATIONS

Effluent Designation: OUTFALL 002

Protocol No.: 8301 Study No.: 36035

ABC Study Director: A. FORBIS

Form No.: 91 (4/8/87)

Analytical Bio-Chemistry Labs
P. O. Box 1097, 7200 ABC Lane
Columbia, Missouri 65205

☑ Prelim. ☐ Definitive Test Species: Daphnia magna

CONTROL/ Dilution Water: HARD RECONSTITUTED No./Vessel: 10 (Lot #: 87-B-4)

Date Initiated: 6-25-87 (12:00pm) Test Terminated: 6/26/87

Effluent Concentration (%)		0 Hr. Soln.	24 Hours Dead	24 Hours Obs.	48 Hours Dead	48 Hours Obs.	Dead	Obs.
Control	A	CLR	0	N				
	B	"	0	N				
100	A	"	0	N				
	B	"	0	N				
Observer Date		SF 6/25/87	SF 6/26/87					

REMARKS: N = Normal, SUR = Surfacing, C = Clumping, ERR = Erratic Movement
TR = Trailing_____, CT = Coated with extraneous material,
Q = Quiescent; OB = On Bottom; CLR = Clear, P = Precipitate,
CLD = Cloudy/colored solution, SF = Surface Film

Prepared By: Scott Frazier Date: 6/24/87

Checked By: Alan Q. Forbis Date: 7/8/87

WATER QUALITY FOR EFFLUENT SAMPLES

Test Material: OUTFALL 002

Study No.: 36035 Protocol No.: 8301

ABC Study Director: A FORBIS

Form No.: 348 (2/3/87)

Analytical Bio-Chemistry Labs
7200 ABC Lane, P.O. Box 1097
Columbia, MO 65201

Concentration %	6-25-87 JL DAY 0 24HR SCREEN				6-26-87 JL DAY 1 24HR SCREEN				DAY				DAY			
	Temp °C	D.O.[a] mg/l	pH[b]	Cond.[c] uMhos/cm	Temp °C	D.O. mg/l	pH	Cond. uMhos/cm	Temp °C	D.O. mg/l	pH	Cond. uMhos/cm	Temp °C	D.O. mg/l	pH	Cond. uMhos/cm
RECON CONTROL	22	9.1	8.2	① ~~2~~ 470	21	8.8	8.3									
100	22	7.8	7.6	1250	21	8.8	8.0									

100% Effluent on Receipt	Temp °C	D.O.[a] mg/l	pH[b]	Cond.[c] uMhos/cm	Hard[d] mg/l	Alk.[d] mg/l		Date	Init.
	12	7.9	7.4	1320	900	162		6-25-87	JL

[a] Dissolved oxygen concentrations - Dissolved Oxygen Probe (YSI Dissolved Oxygen System Model 54)
[b] pH Probe (Corning Model 476182) used with Corning Model 125 pH and mV meter
[c] Salinity/conductivity/temperature meter (YSI Model 33)
[d] mg/l as $CaCO_3$; Hach Models HAC-DT and AL-DT, respectively.*

Prepared By: Scott Frazier Date: 6/24/87

Checked By: [illegible] Date: 7/8/87

Remarks: ① ENTRY ERROR JL 6-25-87

*Method used was a colormetric titration procedure adapted from standard methods, SF 06/25/87.

ABC LABS #036035

Pg 001 of 0037

OBSERVATIONS AND/OR REMARKS FORM

Test Material: OUTFALL 002
Study No.: 360035
Protocol No.: 8301
ABC Study Director: A. FORBES

Lab Form No.: 55

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P.O. Box 1097
Columbia, Missouri 65205

Date/Initials	Comments
MR 6-23-87	FOUR LITERS OF ABC HARD RECONSTITUTED WATER WERE PREPARED IN DE-IONIZED WATER WITH THE FOLLOWING AMOUNTS OF COMPOUNDS; $NaHCO_3$ - 0.768g, $CaSO_4 \cdot 2H_2O$ - 0.480g, $MgSO_4$ - 0.480g AND KCl - 0.032g. THIS RECONSTITUTED WATER YIELDED VALUES OF 160 mg/l HARDNESS AND 114 mg/l ALKALINITY.

Prepared By: [signature] Date: 6-23-87
Checked By: [signature] Date: 6/26/87

PREPARATION OF EFFLUENT CONCENTRATIONS (Volume/Volume)

Effluent Designation: OUTFALL 002
Protocol Number: 8301
Study Number: 36035
ABC Study Director: A. FORBIS

Form No.: 94 (6/11/87)

Analytical Bio-Chemistry Labs
P. O. Box 1097; 7200 ABC Lane
Columbia, Missouri 65201

☑ Preliminary ☐ Definitive

Control and/or Dilution Water Type:

☐ ABC Well Water ☑ ABC HARD Recon. Water
☐ Effluent Receiving Water _____ ☐ Other _____

Sample Number	Aliquot Volume (ml)	Dilution Volume (ml)	Final Concentration (%)
1	0	200	CONTROL
2	200	0	100
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			

Remarks:
Prepared as: ☐ Single rep. ☑ Duplicate rep. ☐ triplicate rep.

Prepared by: Scott Frazier Date: 6/25/87
Checked by: [illegible] Date: 7/8/87
Study Director: [illegible] Date: 7/8/87

INVERTEBRATE CULTURE RECORD

Species: DAPHNIA MAGNA

Lot No.: 87-B-4

Date Initiated: 5-19-87

Lab Form No: 52

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P. O. Box 1097
Columbia, MO 65205

Date	No. Adult Alive	Instar Present	Fed & Changed	Study I.D.	Comments*	Obs. By:
5-19	70	30	✓		FED 200 ml ALGAE + 0.10 ml MIDGE FOOD	MR
5-21	30	—	✓		" "	MR
5/25	30	—	✓		" "	SF
5-27	30	~175	—		" "	MR
5-31	30	~250	✓		" "	JGM
6-1	29	~40	✓		" "	JW
6/2	—	—	—	35732	-120 INSTAR COMBINED WITH ANOTHER EQUAL LOT FOR THIS STUDY	SF
6/2	28	~100	✓		FED 200 ml algae + 0.10 ml midge food	JW
6-4	28	~150	✓		" "	MR
6-7	27	~100	✓		" "	JGM
6-8	27 ②	~20	✓		" "	JL
6-10	27	~200	✓		" "	MR
6-12	27	~150	✓		" "	JL
6-14	26	~300	✓		" "	MR
6/15	26	~30	✓		" "	JW
6/16	—	—	—	35961	- 250 INSTAR FOR STUDY	JL
6/17	26	~400	✓		FED. 200 ml algae + 0.10 ml midge food	JL
6/18	26	~150	✓		" "	JW

"This is an exact copy of The original document" By [illegible] date 6/29/87

Prepared by: [signature] Date: 5-19-87

Checked by: [signature] Date: 6/19/87

*MIDGE FOOD IS TETRAMIN ① CEREAL LEAVES AND YEAST MR 5-19-87
① ENTRY ERROR MR 5-21-87 ② ENTRY ERROR, SF, 6/5/87

INVERTEBRATE CULTURE RECORD

Species: Daphnia magna

Lot No.: 87-B-4

Date Initiated: 5-19-87

Lab Form No: 52

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P. O. Box 1097
Columbia, MO 65205

Date	No. Adult Alive	Instar Present	Fed & Changed	Study I.D.	Comments	Obs. By:
6-21	26	~500	✓			MR
6/23	26	~50	✓			MR
6/24	—	—	—	35494	280 INSTAR FOR STUDY	JL
6/24	—	—	—	36037	140 INSTAR FOR STUDY	SF
6/24	—	—	—	35876	120 INSTAR FOR STUDY	JL
6/24	26	~100	✓			JL
6/25	—	—	—	36035	40 INSTAR FOR STUDY	SF
6/25	25	—	✓			JW.
6/28	25	~600	✓		Fed ~ 20ml concentrated ALGAC & 0.10ml midge Food	JGM

"This is an exact copy of
The original document"

By J. Wirth date June 25, 87

Prepared by: James A. Mickman Date: 6-19-87

Checked by: Scott Frazier Date: 6/29/87

MOLYCORP INC.

QUESTA-DIVISION

UNOCAL-76

Samples handed over to carrier by Molycorp employee

DATE: 06/23/87

TIME: 7:30AM

CARRIER J.M.B.

EMPLOYER: Molycorp Inc.

MOLYCORP PERSONNEL: JMB

Sample(s) delivered to ABC lab for analysis. Please sign and date this letter upon receipt of sample(s)

Sample(s) received by: ABC Laboratories Inc.
7200 ABC Lane
P.O. Box 1097
Columbia, MO 65205
(314) 474-8579

for the following analyses: Toxicity testing

Received by: Scott Snyder

DATE: 6/25/87

Please return this letter with the analytical results, signed and dated below.

Scott Snyder 7/8/87
Date

MOLYCORP, INC.

QUESTA - DIVISION

UNOCAL- 76

This sheet represents a chain of custody for effluent samples collected at outfall 002 Molycorp Inc

Located in Taos County, Questa, New Mexico on 06/23/87 last portion of composite 7:32 AM.

Sample bottles were prepared by Molycorp employees in accordance with guidance set forth by the U.S. Environmental Protection Agency for the existing program permit. Sampling procedures were also followed in accordance with permit requirments.

Sample (s) Identification 002

Sample (s) Identification

By: J. Martinez

DEFINITIVE CONCENTRATION NOTIFICATION

Firm: Molycorp, Inc.
Address: P.O. Box 469
City/State/Zip: Questa, NM 87556
Phone: 505) 586-0212

Lab Form No.: 186
Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P.O. Box 1097
Columbia, MO 65202

Sponsor's Study Director: MR. FRED MARTINEZ
Person notified: same (✓) Other:

Date of Notification: 6/26/87

Notification of preliminary testing results with the following definitive test levels agreed upon:

Test Material	ABC #	Test Species	Test Conc. (mg/l)
Outfall 002	~~36037~~ ① 36035	~~Daphnia~~ magna	N/A *

* No mortality in 24 hr. screen test therefore no further testing required.

① Entry Error, SF, 7/13/87

Notified By: Scott Frazier Date: 6/26/87

ANALYTICAL BIO-CHEMISTRY LABORATORIES, INC.
P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

RECEIVED
JUL 7 '87
A.B.C. LABS

ABC PROTOCOL NO. 8301

(August 21, 1984)

Effluent Static Bioassay with *Daphnia magna* or Mysid Shrimp (*Mysidopsis bahia*) to Meet EPA Region 6 NPDES Biomonitoring Guidelines

ABC Study Number 36035

Test Material Outfall 002

Test Species *Daphnia magna*

1.0 INTRODUCTION

The use of bioassays in the identification and control of industrial effluents is rapidly increasing. Indeed, aquatic toxicity tests are presently being incorporated into the NPDES guidelines of the Federal Clean Water Act (1). To assist industry in meeting requirements set forth by legislation and regulatory agencies, Analytical Bio-Chemistry Laboratories, Inc., offers a complete range of aquatic bioassay tests of industrial effluents.

As part of the NPDES permit process in U.S. EPA Region 6, a biomonitoring requirement has been imposed for certain permit holders in that area. Discharges subject to the new regulations in Region 6 (Texas, New Mexico, Oklahoma, Arkansas and Louisiana) will be required to submit aquatic toxicity data on their effluents to EPA on a periodic basis.

This ABC protocol discusses the biomonitoring guidelines in REgion 6 and describes ABC's technical approach to assist industry in complying with these regulations.

2.0 OBJECTIVES

The primary objective of the toxicity test described herein is to evaluate the acute toxicity of an effluent to freshwater or marine invertebrates under static conditions. The species selected for this testing include *Daphnia magna* (freshwater effluent, <5 ppt salinity) and *Mysidopsis bahia* (saltwater effluent, ≥5 ppt salinity). The effluent will first be tested on a 100% effluent screen basis. If the screen results in ≥20% mortality of test organisms, a 48-hour, five concentration LC_{50} test will be conducted. An LC_{50} is the approximate concentration (percent) of test effluent that produces 50 percent mortality of test organisms after prescribed intervals (24 and 48 hours).

3.0 TESTING FACILITY

The study will be conducted by the Aquatic Toxicology Division of Analytical Bio-Chemistry Laboratories, Inc., 7200 East ABC Lane, P. O. Box 1097, Columbia, Missouri 65205, 314-474-8579.

4.0 REGION 6 BIOMONITORING REQUIREMENT

The following biomonitoring requirement is now being included in the NPDES permit process for many major industrial discharges in EPA Region 6. The permit language given here was that presented by the U.S. EPA at a regional workshop on the matter (2):

> The permittee shall determine if the effluent from this outfall is lethal to twenty (20) percent or greater of the culture of test organisms by use of the "Range-Finding Screening Test," set out in "Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms," EPA-600/4-78-012 (Revised July, 1978) (3).

Organisms for this test shall be Daphnia sp. if the effluent is less than five (5) parts per thousand salinity or Mysid sp. if the effluent is greater than five (5) parts per thousand salinity. This screening test will be conducted within sixty (60) days of effectiveness of the biomonitoring requirements. Tests will be conducted once each quarter for a duration of two years utilizing a static method for 24 hours and following this dilution scheme only:

Effluent samples*	-	100 percent by volume
Dilution water	-	0 percent by volume

*24-hour composite; refrigerated after collection

If at any time during the two year testing period a test shows a lethality of twenty (20) percent or more of the test organisms, the permittee shall within twenty-four (24) hours conduct a replacement static 48-hour median lethal concentration (LC_{50}) test on the originally collected sample. Replacement of effluent is less than five (5) parts per thousand salinity and reconstituted fresh water (EPA-600/4-78-012 Section 4) shall be used for dilution. If the effluent contains greater than five (5) parts per thousands salinity, Mysid sp. shall be used as the test organism, and reconstituted seawater will be used as dilution water (EPA-600/4-78-012 Section 4). The remaining LC_{50} methodology is available in EPA-600/4-78-012.

All screening and LC_{50} test results shall be reported with the Discharge Monitoring Reports.

It should be noted by the permittee that slight changes from the above permit language (e.g., pH modification of effluent) may be incorporated into his specific permit. Attention should be given to the permit requirements for each effluent before testing is initiated. ABC requires that a copy of the permit's biomonitoring clause be forwarded to ABC prior to testing so that consideration can be give to pH modification, et.

5.0 METHODS AND MATERIALS

5.1 Methodology. The methods applied for both the preliminary and definitive phases of this test are basically those described in Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms (3), Methods for Acute Toxicity Tests with Fish, Macroinvertebrates, and Amphibians (4), Standard Practice for Conducting Acute Toxicity Tests with Fishes, Macroinvertebrates, and Amphibians (5), Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates and Amphibians (6), and Standard Methods for the Examination of Water and Wastewater (7). This protocol has been written to comply with the federal Good Laboratory Practice Regulations (8).

5.2 Test Organisms. The species recommended by the EPA for this testing are Daphnia sp. and Mysid sp. Daphnia sp. are to used when the effluent salinity is <5 parts per thousand (ppt) and Mysid sp. are to be used when the effluent salinity ≥5 ppt. The species maintained by ABC, and acceptable by EPA, for this testing are Daphnia magna and Mysidopsis bahia. The test lot of Daphnia magna will be obtained from an in-house culture at ABC. The taxonomic key presented by Pennak (9) will be used to identify to species the test daphnids. All test daphnids will be held in a controlled temperature area at 20 ±2°C. The lighting will be 50-70 foot-candles on a 16 hour daylight photoperiod. During the holding period, they will be fed algae (Selenastrum capricornutum) supplemented with a blended fish food/cerophyl suspension at least every 3 days. First-instar daphnids (<24 hours old) will be selected for testing and will not be fed during the test.

Mysid shrimp (Mysidopsis bahia) will be obtained from a commercial supplier (e.g., Sea Plantations, Inc. or Gulf Breeze Specimens) and maintained in culture at ABC. Mysids will be cultured following the techniques described by the U.S. EPA (10). Test mysids will be selected from a sub-culture of immature individuals.

5.3 Effluent Sample. The sample submitted to ABC for testing must be a 24-hour composite sample, consisting of equal volumes taken every hour (2). The sample should be collected in a glass or polyethylene container, refrigerated after collection, and shipped to arrive at ABC within 24 hours after collection. A one-gallon sub-samples of the 240hour composite will be satisfactory for the toxicity test(s) required. ABC can provide sample containers and mailing cartons and can assist plant personnel in shipping arrangements so that the sample arrives at ABC in a timely manner.

5.4 Dilution Water. It will not be necessary to collect actual receiving water for effluent dilutions for this test. The biomonitoring regulations recommend the use of reconstituted freshwater or saltwater listed in the EPA effluent methods manual (3). The freshwater dilution water (for Daphnia sp.) will be a hard reconstituted water of 160-180 mg/l hardness (as $CaCO_3$). The reconstituted saltwater (for Mysid sp.) will be prepared from a commercial salt mix similar in composition to that listed in the EPA effluent manual (3) and adjusted to a test salinity of 15-25 ppt.

5.5 Screen Test. The biomonitoring regulations require that a 24-hour 100% effluent screen be conducted before more definitive (LC_{50}) testing is considered. As stated in section 4.0, if ≥20% of the test organisms die in the 100% solution, the permittee shall then conduct a 48-hour static replacement LC_{50} test. This latter test is discussed in section 5.6.

The screen test will be conducted in 250 ml glass beakers containing 200 ml of solution. These test vessels will be held in a controlled temperature area at 20 ±2°C on a 16-hour daylight vs.

8-hour darkness photoperiod with 30-minute dawn/dusk transition periods. The screen test will include duplicate test chambers at 100% vs. duplicate control chambers containing dilution water only. Ten test organisms will be placed in each replicate; therefore, twenty total organisms will be exposed to the 100% effluent and control (dilution) water.

After a 24-hour exposure period, the test chambers will be observed for mortality and abnormal effects. If the combined replicate mortality is ≥20%, the 48-hour LC_{50} test will be initiated.

5.6 48-Hr LC_{50} Test. The definitive test will be conducted by exposing the test organisms to at least 5 effluent concentrations and a dilution water control. The test will be conducted using duplicate beakers per concentration containing 10 test organisms per beaker (=20 per concentration). The test concentrations used will follow one of the logarithmic series presented in Table 1. The most common concentration series used is 10-100% (10, 18, 32, 56 and 100%). Several effluent toxicity tests conducted by ABC have shown that most industrial effluents are toxic within this range.

The test organisms will be placed in the test chambers by stratified random assignment within 30 minutes after solution preparation. The test chambers will be observed for mortality and/or abnormal effects every 24 hours, with a record maintained of mortality and abnormal behavior for each concentration tested.

5.7 Test Procedure - Chemical and Physical. Upon receipt of the test effluent, the following parameters will be measured of the 100% effluent: temperature, dissolved oxygen, pH, conductivity and hardness. These same parameters, except hardness, will be measured at 0 and 48 hours of definitive testing in one replicate of the control, low concentration, middle concentration and high concentration solutions.

5.8 Analysis of Results. The results of the definitive study will be statistically analyzed for 24 and 48 hour LC_{50} values and their corresponding 95 percent confidence limits. These values will be determined by an LC_{50} computer program developed by Stephan et al. (11) or by manual methods described by Litchfield-Wilcoxon (12) and Stephan (13).

5.9 Report. One copy of the report will be submitted. The report will contain all original raw data. Additional copies will be provided, if requested by the sponsor, at a copy rate of $50 per copy. The report will include the following:

5.9.1 Study dates, name and address of test facility.

5.9.2 Objectives and test methods.

5.9.3 Reference to the statistical methods used for data analysis.

5.9.4 Description of test effluent (date of receipt, storage conditions, physical characteristics, and method of preparing test concentrations).

5.9.5 Description of test design.

5.9.6 Summary of the data analysis, mortality observations and test water quality.

5.9.7 Location of raw data.

5.9.8 List and signatures of study personnel.

5.9.9 GLP compliance statement by study director and a statement by ABC's Quality Assurance Unit.

5.9.10 The report appendix will contain the original raw data for mortality observations and water quality, letter of test authorization, letters of authorized protocol changes and a copy of the approved protocol.

5.10 Data Retention. All original raw data generated in the preliminary and definitive studies will be provided to the study sponsor in the appendix to the final report. A copy of the data will be retained in ABC's archives.

6.0 PROTOCOL CHANGES

In the event that modifications of this protocol are deemed necessary, a writtens statement of any changes and reason(s) proposed by the study sponsor or ABC will be submitted to the other party. All agreed changes will be expressed in writing, signed and dated by the sponsor's study director. The signed changes will be appended to the protocol and included with the final report. This protocol is subject to revision when felt necessary to comply with changes in federal regulations and state of the art methodology. The protocol for a specific study will not be changed while the study is in progress without discussion with the study sponsor.

7.0 SPONSOR AUTHORIZATIONS DURING THE STUDY

Should a problem develop while the study is in progress, ABC will notify the study director within 24 hours. The problem and suggested test modifications will be discussed by telephone. ABC will proceed with the changes felt necessary upon the verbal authorization of the study director. A letter for written authorization will then be submitted by ABC to the study director and handled in the same manner discussed in section 6.0.

8.0 TEST-SPECIFIC INFORMATION

8.1.1 General. The following items will be addressed for each static bioassay. This information is necessary to be in compliance with Good Laboratory Practice Regulations (8). Sections 8.2, 8.3, 8.7 and 8.9 are to be completed by the study sponsor. Sections 8.4, 8.5 and 8.6 will be completed by ABC.

8.1.2 GLP Compliance. To be in compliance with the Federal Good Laboratory Practice regulations, the report of the investigation conducted utilizing this protocol must contain a statement that the study was conducted in accordance with one of the following GLP's.

1. U.S. F.D.A. Good Laboratory Practice Regulations (21 CFR 58).

2. U.S. E.P.A. Good Laboratory Practice Standards; Toxic Substances Control (40 CFR 792).

3. U.S. E.P.A. Good Laboratory Practice Standards; Pesticide Programs (40 CFR 160).

4. OECD Principles of Good Laboratory Practice; Annex 2 C(81) 30(Final).

5. Any state or local GLP which may apply.

The specific GLP regulation, for which this study specific protocol and resultant study must be in compliance with, is to be designated by the study sponsor in section 8.8 of this protocol.

8.2 Study Sponsor:

8.2.1 Company Molycorp, Incorporated

8.2.2 Address 3 Miles East of Questa on State Road 38
P.O. Box 469, Questa, New Mexico 87556

8.2.3 Sponsor's Study Director

Name	Title
Mr. Fred Martinez	Analyst

8.3 Effluent:

8.3.1 Sample Number Outfall 002

8.3.2 NPDES Permit Number NM-0022306

8.3.3 Physical Description Clear Liquid

8.3.4 Handling Precautions Normal Precautions

8.4 Test Concentrations: To be determined after 24-hour screen of 100% effluent and consultation with study sponsor.

8.4.1 Sponsor's Study Director notified of screen results:
Date 6/26/87 ABC Biologist Scott Frazier
100% effluent screen = _____ ≥20% mortality X <20% mortality

8.4.2 Definitive test concentrations agreed upon:

8.5 Test Organisms:

8.5.1 Species Daphnia magna

8.5.2 Supplier ABC in-house culture

8.6 Study Dates:

8.6.1 Proposed starting date of definitive study 6/25/87

8.6.2 Proposed completion date of definitive study 7/7/87

8.7 ABC Study Personnel:

8.7.1 Study Director

Name	Title
Alan D. Forbis	Supervisor, Invertebrate and Bioconcentration Investigations

8.7.2 Aquatic Manager

Name	Title
Carl M. Thompson	Aquatic Toxicology Manager

8.8 GLP Compliance (check the most appropriate):

EPA-FIFRA ____; EPA-TSCA ____; FDA ____; OECD ____; Other NPDES ✓.

8.9 Protocol Approvals. The following is to be signed by the appropriate study personnel:

8.9.1 Sponsor's Study Director[1]

Name	Title	Date
Fred Martinez	Analyst	07/01/87

8.9.2 ABC's Study Director

Name	Title	Date
Ken D. Fortis	Supervisor, Invertebrate and Bio. Investigations	6/23/87

8.9.3 ABC's Manager

Name	Title	Date
Carl M. Thompson	Aquatic Tox. Manager	6-23-87

8.10 Authorization to return the test material and sample:

8.10.1 Sponsor's authorized agent to receive the test material and sample.

Name	Title
Fred Martinez	Environmental - Analyst

[1] Due to the time restrictions for effluent testing following receipt of sample (within 24 hours), this protocol may not be processed for sponsor approval prior to the test.

8.10.2 Shipping Address

Molycorp, Incorporated
Company

3 Miles East of Questa Highway 38
Street Address

Questa	New Mexico	87556
City	State	Zip Code

505-586-0212
Phone Number

8.10.3 Hazardous Shipment Information

N/A
Proper Shipping Name

Class of Restricted Article (e.g. flammable, poison, radioactive, etc.)

TABLE 1

Guide to Selection of Experimental Concentrations Based on Progressive Bisection of Intervals on Logarithmic Scale

Col. 1	Col. 2	Col. 3	Col. 4	Col. 5
10.0	10.0	10.0	10.0	10.0
				8.7
			7.5	7.5
				6.5
		5.6	5.6	5.6
				4.9
			4.2	4.2
				3.7
3.2	3.2	3.2	3.2	3.2
				2.8
			2.4	2.4
				2.1
		1.8	1.8	1.8
				1.55
			1.35	1.35
				1.15
1.0	1.0	1.0	1.0	1.0

TABLE 2

Chemical Characteristics of Well Water Used by ABC's Aquatic Toxicology Division

Parameter	Concentration
Temperature	15-20°C[b]
Dissolved Oxygen[a]	9.2-10.1 ppm[b]
pH	7.8-8.3[b]
Hardness ($CaCO_3$)	225-275 ppm[b]
Alkalinity ($CaCO_3$)	325-375 ppm[b]
Conductivity	700 µmhos/cm
NO_3-N	0.58 ppm
NO_3-NO_2-N	0.69 ppm
PO_4-P	<0.50 ppm
Aluminum	<20 ppb
Cadmium	<3 ppb
Chromium	<5 ppb
Cobalt	<5 ppb
Copper	<5 ppb
Iron	<5 ppb
Lead	<20 ppb
Nickel	<10 ppb
Silver	<5 ppb
Zinc	<1 ppb
Measured organophosphorus pesticides	<0.10 ppb
Measured organochlorine pesticides plus PCB's	<0.50 ppb

[a]After aeration.

[b]Represents seasonal variation, with the monthly range not exceeding 10%.

Sample dates: Trace elements = May 12, 1986, organophosphate and organochlorine analyses = May 12, 1986.

9.0 REFERENCES

(1) U.S. Congress. 1977. Clean Water Act of 1977. Public Law 95-217. Federal Register, December 27, 1977: 1566-1611.

(2) U.S. Environmental Protection Agency. 1983. Effluent Toxicity Testing and Biomonitoring Workshop. Feb. 22-23, 1983, Dallas, Texas.

(3) Peltier, William. 1978. Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms. U.S. Environmental Protection Agency, EPA-660/4-78-012, revised July, 1978. 52 p.

(4) Committee on Methods for Toxicity Tests with Aquatic Organisms. 1975. Methods for Acute Toxicity Tests with Fish, Macroinvertebrates and Amphibians. Environmental Protection Agency, Ecological Research Series EPA-660/3-75-009, April, 1975. 61 p.

(5) American Society for Testing and Materials. 1980. Standard practice for conducting basic acute toxicity tests with fish, macroinvertebrates and amphibians. May, 1980, ASTM Committee E-35.23. 25 p.

(6) American Society for Testing and Materials. 1983. Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates and Amphibians. November, 1983, ASTM Committee E-47.01. 37 p.

(7) American Public Health Association. 1980. Standard Methods for the Examination of Water and Wastewater. 15th ed. Washington, D.C. 1134 p.

(8) (a) U.S. Food and Drug Administration. 1978. Nonclinical Laboratory Studies, Good Laboratory Practice Regulations (21 CFR, Part 58). Federal Register, Vol. 43, No. 247: 59986-60025.

(b) U.S. Environmental Protection Agency. 1983. Pesticide Programs; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 160). Federal Register, Vol. 48, No. 230: 53946-53969.

(c) U.S. Environmental Protection Agency. 1983. Toxic Substances Control; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 792). Federal Register, Vol. 48; No. 230:53922-53944.

(d) Organization for Economic Cooperation and Development. 1981. OECD Guidelines for Testing of Chemicals, Principles of Good Laboratory Practice Annex 2, C(81) 30 (Final):7-28.

(9) Pennak, R. W. 1978. Fresh-Water Invertebrates of the United States, 2nd Ed. 365 p.

(10) U.S. Environmental Protection Agency. 1979. Static culture methods for sheepshead minnows (Cyprinodon variegatus) and mysid shrimp (Mysidopsis bahia). Unpublished manuscript, U.S. EPA, Athens, GA.

(11) Stephan, C. E., K. A. Busch, R. Smith, J. Burke and R. W. Andrew. 1978. A computer program for calculating an LC_{50}. U. S. Environmental Protection Agency, Duluth, Minnesota, pre-publication manuscript, August, 1978.

(12) Litchfield, J. T., Jr. and F. Wilcoxon. 1949. A Simplified Method of Evaluating Dose-Effect Experiments. Jour. Pharm. Exp. Ther. 96:99-113.

(13) Stephan, C. 1977. Methods for calculating an LC_{50}, p. 65-84. In F. L. Mayer and J. L. Hamelick (eds.). Aquatic Toxicology and Hazard Evaluation. ASTM Special Technical Publication 634. ASTM. Philadelphia.

Assessment of Acute Toxicity of 002
Daphnia Magna

June 1987

ANALYTICAL BIO-CHEMISTRY LABORATORIES, INC.
P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

SPONSOR COMPANY

Molycorp, Incorporated
Attn: Mr. Fred Martinez
3 Miles East of Questa, on State Road 38
P.O. Box 469
Questa, New Mexico 87556

STUDY TITLE

Assessment of the Acute Toxicity of Red River Upstream Sample to *Daphnia magna*

DATA REQUIREMENT

U.S. EPA-TSCA, 40 CFR, Part 797
Guideline NPDES

AUTHOR

Alan D. Forbis
Supervisor, Invertebrate and
Bioconcentration Investigations

STUDY COMPLETED ON

January 9, 1987

PERFORMING LABORATORY

Analytical Bio-Chemistry Laboratories, Inc.
Aquatic Toxicology Division
7200 East ABC Lane
P.O. Box 1097
Columbia, Missouri 65205

ABC LABORATORY PROJECT ID

#35405

Submitted By: Analytical Bio-Chemistry Laboratories, Inc.
7200 East ABC Lane
P. O. Box 1097
Columbia, Missouri 65205
(314) 474-8579

Prepared By:

Scott Frazier — 1/27/87
Scott Frazier — Date
Biologist I

Michael Ruddy — 1-27-87
Michael Ruddy — Date
Biological Technician I

Approved By:

Alan D. Forbis — 1/27/87
Alan D. Forbis — Date
Study Director/
Supervisor, Invertebrate and
Bioconcentration Investigations

Phillip M. Buckler — 1/27/87
Phillip M. Buckler — Date
Quality Assurance Supervisor

Carl M. Thompson — 1-27-87
Carl M. Thompson — Date
Aquatic Toxicology Manager

/keh

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PROJECT PERSONNEL

The study director of the project for Analytical Bio-Chemistry Laboratories, Inc. was Alan D. Forbis, Supervisor, Invertebrate and Bioconcentration Investigations. Supervising the conduct of the study for Molycorp, Incorporated was Mr. Fred Martinez. The following ABC personnel assisted with various phases of the study.

Name	Title	Initials
Alan D. Forbis	Supervisor, Invertebrate and Bioconcentration Investigations	AF
Scott Frazier	Biologist I	SF
Michael Ruddy	Biological Technician I	MR
Jennifer Wirth	Biological Aide	JW (by SF)
James G. Muckerman	Biological Technician I	JGM

INVERTEBRATE ACUTE TOXICITY COMPENDIUM

Subject: "Assessment of the Acute Toxicity of Red River Upstream Sample to *Daphnia magna*",

ABC Study Number: 35405

Sponsor: Molycorp, Incorporated

Testing Facility: Analytical Bio-Chemistry Laboratories, Inc.
Aquatic Toxicology Division
P.O. Box 1097
Columbia, Missouri 65205 (314-474-8579)

Location of Original Raw Data and Final Report: Molycorp, Incorporated; 3 Miles East of Questa, State Road 38; P.O. Box 469; Questa, New Mexico 87556.

Test Material: Red River Upstream Sample

Test Concentrations: Control and 100%

Control Water: ~176 mg/l as $CaCO_3$ (hardness), 118-120 mg/l as $CaCO_3$ (alkalinity), 7.8 (pH)

Test Dates: Initiation - January 8, 1987
Termination - January 9, 1987

Length of Study: 24-hours

Results: 24-hour NOEL = 100%

Test Species: *Daphnia magna*

Source of Organisms: ABC Laboratories in-house culture

Age of Organisms at Study Initiation: First-instar (≦24-hours old)

SUMMARY

The acute toxicity of Red River Upstream Sample to Daphnia magna was assessed for U.S. EPA Region 6 NPDES permit requirements using the methods outlined by the Committee on Methods for Toxicity Tests with Aquatic Organisms. Water quality parameters of temperature, dissolved oxygen, conductivity, hardness, alkalinity and pH were measured on receipt of the sample and were within acceptable limits.

The results of the 24-hour static Daphnia magna toxicity screen indicated that no mortality was observed after the 24-hour exposure period. Therefore, a definitive 48-hour static acute study was not required. All reported values were based upon nominal concentrations.

The no-effect level for Red River Upstream Sample was 100% effluent after 24-hours, based on the lack of mortality and abnormal effects.

INTRODUCTION

This static bioassay was performed by the Aquatic Toxicology Division of Analytical Bio-Chemistry Laboratories, Inc., Columbia, Missouri, for Molycorp, Inc., from January 8, 1987 to January 9, 1987. The purpose of this test was to assess toxicity of a 100% solution of Red River Upstream Sample to Daphnia magna. The assessment was designed to meet the NPDES permit process in U.S. EPA Region 6 (1) requiring biomonitoring of industrial discharges. The screen test was conducted following the procedures outlined in ABC protocol 8301, as approved by Mr. Fred Martinez; Molycorp, Inc. on January 15, 1987.

METHODS AND MATERIALS

The procedures applied for the 24-hour toxicity screen were basically those described in Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms (2), Methods for Acute Toxicity Tests with Fish, Macroinvertebrates, and Amphibians (3), Standard Practice for Conducting Acute Toxicity Tests with Fishes, Macroinvertebrates and Amphibians (4), Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents and Fishes, Macroinvertebrates and Amphibians (5), and Standard Methods for Examination of Water and Wastewater (6).

I. Test Organisms

The test lot of Daphnia magna was obtained from an in-house culture at ABC. The taxonomic key presented by Pennak (7) was used to identify the test daphnids to species. All test daphnids were held in a controlled temperature area at 20 (±2)°C. The lighting was 50-70 footcandles on a 16-hour daylight photoperiod. During the holding period, the daphnids were fed algae (Selenastrum capricornutum) supplemented with a suspension of Tetramin®, cereal leaves and yeast at least every 3 days. First-instar daphnids (<24-hours old) were selected for testing and were not fed during the test.

II. Test Procedure

The static Daphnia bioassay was conducted in 250-ml glass beakers containing 200 ml of sample or ABC hard reconstituted water prepared from the following compounds in the amounts stated per liter of deionized water:

192 mg $NaHCO_3$
120 mg $CaSO_4 \cdot 2H_2O$
120 mg $MgSO_4$
8 mg KCl

The reconstituted water was prepared to yield a total hardness of 160-180 mg/l (as $CaCO_3$) a total alkalinity of 110-120 mg/l (as $CaCO_3$), and a pH of 7.6-8.0.

These vessels were kept at 20 (±2.0) °C in a temperature controlled area. The lighting was maintained at 50-70 foot-candles on a 16-hour daylight photoperiod.

A 24-hour screen test, composed of duplicate 100% effluent chambers was performed to estimate the toxicity of the effluent. Also included was a duplicate ABC hard reconstituted water control. A five-six concentration static acute toxicity study would be performed if ≧20% mortality was observed after 24-hours in the screen test.

III. Test Material

The Red River Upstream Sample was received on January 8, 1987 as a clear liquid and was received at 5°C. The sample was allowed to warm to test temperature before daphnids were added to test solutions.

IV. Statistics

If a definitive study is necessary, the 24- and 48-hour LC_{50} values and corresponding 95-percent confidence limits are determined by an LC_{50} computer program developed by Stephan et al. (8). This program calculates the LC_{50} statistic and its 95-percent confidence limits using the binomial, moving average angle and probit methods because no one method is appropriate for all possible sets of data. The method of calculation selected is that which gives the narrowest confidence limits for each separate analysis.

RESULTS

After 24-hours, the results indicated no mortality of *Daphnia magna* to Red River Upstream Sample. The no-effect concentration was 100% effluent, based on the lack of mortality and abnormal effects. Since no mortality was observed in the screen test, a definitive study was not required per biomonitoring guidelines established for U.S. EPA Region 6.

Table 1 presents the water quality parameters measured on sample receipt. Hardness and alkalinity (as $CaCO_3$) and conductivity for the Red River Upstream Sample were 176 mg/l, 48 mg/l and 250 µMhos/cm, respectively. Dissolved oxygen and pH for this sample were 9.9 mg/l and 7.0, respectively, and were considered adequate for testing (3).

The study was conducted following the intent of the Good Laboratory Practice Regulations (9) and the final report was reviewed by Analytical Bio-Chemistry Laboratories' Quality Assurance Unit. All original raw data were provided to Molycorp, Incorporated with a copy retained at Analytical Bio-Chemistry Laboratories.

TABLE 1

Water Quality Measurements of 100%
Red River Upstream Sample Upon Sample Receipt

	100% Effluent
Temperature[a]	5*
Dissolved Oxygen[b]	9.9
pH[c]	7.0
Conductivity[d]	250
Hardness[e]	176
Alkalinity[e]	48

[a]Temperature °C - Monitored with a mercury thermometer.

[b]Dissolved oxygen concentrations (mg/l) - Dissolved Oxygen Probe (YSI Dissolved Oxygen System Model 54). Measured at 7°C. Saturation at 7°C is 12.2mg/l.

[c]pH - pH Probe (Corning Model 476182) used with a Corning Model 125 pH and mV meter.

[d]Conductivity (μMhos/cm) - Conductivity Meter (YSI Model 33).

[e]Hardness and alkalinity (mg/l as $CaCO_3$) - HACH Model AL-DT and HAC-DT.

*Test solutions allowed to warm to test temperature (19°C) before addition of daphnids. Dissolved oxygen saturation at 19°C is 9.3 mg/l.

LITERATURE CITED

(1) U. S. Environmental Protection Agency. 1983. Effluent Toxicity Testing and Biomonitoring Workshop. Feb. 22-23, 1983, Dallas, Texas.

(2) Peltier, William. 1978. Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms. U.S. Environmental Protection Agency, EPA-660/4-78-012, revised July 1978. 52 p.

(3) Committee on Methods for Toxicity Tests with Aquatic Organisms. 1975. Methods of Acute Toxicity Tests with Fish, Macroinvertebrates and Amphibians. Environmental Protection Agency, Ecological Research Series EPA-660/3-75-009, April 1975.

(4) American Society for Testing and Materials. 1980. Standard Practice for Conducting Basic Acute Toxicity Tests with Fish, Macroinvertebrates and Amphibians. May 1980, ASTM Committee E-35.23. 25 p.

(5) American Society for Testing and Materials. 1983. Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates and Amphibians. November 1983, ASTM Committee E-47.01 37 p.

(6) American Public Health Association. 1980. Standard Methods for the Examination of Water and Wastewater. 15th ed. Washington, DC. 1134 p.

(7) Pennak, R. W. 1978. Freshwater Invertebrates of the United States. John Wiley & Sons, New York, 2nd ed. 803 p.

(8) Stephan, C. E., K. A. Busch, R. Smith, J. Burke and R. W. Andrews. 1978. A Computer Program for Calculating an LC_{50}. U. S. Environmental Protection Agency, Duluth, Minnesota, pre-publication manuscript, August, 1978.

(9) (a) U.S. Food and Drug Administration. 1978. Nonclinical Laboratory Studies, Good Laboratory Practice Regulations (21 CFR, Part 58). Federal Register, Vol. 43, No. 247:59986-60025.

(b) U.S. Environmental Protection Agency. 1983. Pesticide Programs; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 160). Federal Register, Vol. 48, No. 230:53946-53969.

(c) U.S. Environmental Protection Agency. 1983. Toxic Substances Control; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 792). Federal Register, Vol. 48; No. 230:53922-53944.

(d) Organization for Economic Cooperation and Development. 1981. OECD Guidelines for Testing of Chemicals, Principles of Good Laboratory Practice Annex 2, C(81) 30(Final):7-28.

QUALITY ASSURANCE STATEMENT

Quality Assurance Statement for final report #35405 entitled, "Assessment of the Acute Toxicity of Red River Upstream Sample to *Daphnia magna*," for Mr. Fred Martinez, Molycorp, Incorporated; Questa, New Mexico.

In accordance with ABC Laboratories' intent that all studies conducted at our facilities are designed and function in conformance with good laboratory practice regulations and the protocols for individual laboratory studies, an inspection of the final report for Red River Upstream Sample was conducted and found to be in acceptable form by a member of our Quality Assurance Unit. A final inspection of all data and records on January 25, 1987, indicates that the report submitted to you is an accurate reflection of the study as it was conducted by ABC Laboratories.

Should you have any questions relating to the information provided in this statement or the function of our Quality Assurance Unit, please contact the QA Unit at your convenience.

Phillip M. Buckler 1/27/87

Phillip M. Buckler Date
Quality Assurance Supervisor

STUDY COMPLIANCE STATEMENT

Study Compliance Statement for ABC report #35405 entitled, "Assessment of the Acute Toxicity of Red River Upstream Sample to *Daphnia magna*," for Mr. Fred Martinez, Molycorp, Inc.; Questa, New Mexico.

In accordance with ABC Laboratories' intent that all aquatic toxicity tests conducted by our facility follow good laboratory practices, ABC's study director for the above test herein confirms that the study was conducted in compliance with the U.S. E.P.A. Good Laboratory Practice Standards; Toxic Substances Control (40 CFR 792).

All original raw data were sent to Molycorp, Incorporated, with a copy retained at Analytical Bio-Chemistry Laboratories.

Alan D. Forbis 1/27/87

Alan D. Forbis — Date
ABC Study Director

APPENDIX - RAW DATA

NOTE: Some of the records which appear in this raw data appendix have been provided as photocopies of original records on file at ABC. This has been done by necessity for certain data which are used commonly in several studies at ABC. Such records include organism culture logs.

EFFLUENT TOXICITY TEST
MORTALITY AND BEHAVIORAL OBSERVATIONS

Effluent Designation: Red River Upstream
Industry: Molly Corp Inc.
Study No.: 35405
ABC Study Director: A. Forbis

Lab Form No.: 91

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P.O. Box 1097
Columbia, Missouri 65205

☑ Prelim. ☐ Definitive Test Species: Daphnia magna (Lot # 86-E-8)
Date Initiated: 1-8-87 (1:50 pm) Test Terminated: 1/9/87
Dilution Water: H2O-recon No./Vessel: 10 Vessel Size: 250 ml

Effluent * Conc. (%)	24 hours Dead	24 hours Obs.	48 hours Dead	48 hours Obs.	72 hours Dead	72 hours Obs.	96 hours Dead	96 hours Obs.
Control								
A	0	N						
B	0	N						
100%								
A	0	N						
B	0	N						
Observer	SF (1:00 pm)							
Date	1/9/87							

Remarks: N = Normal; LOE = Loss of Equilibrium; SUR = Surfacing
* TEST SOLUTIONS @ 19°C AT TEST INITIATION MR 1-8-87

Prepared By: [signature] Date: 1-8-87
Checked By: [signature] Date: 1/9/87

WATER QUALITY FOR EFFLUENT SAMPLES

Test Material: Red River Upstream Sample

Study No.: 35405

Protocol No.: ~~8304~~ ① 8301

ABC Study Director: A. Forbis

Lab Form No.: 28b

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane; P.O. Box 1097
Columbia, Missouri 65205

Concentration mg/1	DAY				DAY				DAY				DAY			
	Temp °C	D.O.[a] mg/1	pH[b]	Cond. µMhos	Temp °C	D.O. mg/1	pH	Cond. µMhos	Temp °C	D.O. mg/1	pH	Cond. µMhos	Temp °C	D.O. mg/1	pH	

[a]Dissolved oxygen concentrations - Dissolved Oxygen Probe (YSI Dissolved Oxygen System Model 54).

[b]pH-pH Probe (Corning Model 476182) used with Corning Model 125 pH and mV meter.

[c] 100% Effluent on Receipt	Temp °C	D.O.* mg/1	pH	Cond. µMhos	Hard mg/1	Alk. mg/1	NH_3 mg/1			Date	Int.
	5	9.9	7.0	2500	176	48	X			1-8-87	MR

Prepared By: [signature] Date: 1-8-87

Checked By: [signature] Date: 1/9/87

Remarks: *D.O. reread @ 7°C MR 1-8-87

① ENTRY ERROR MR 1-13-87

ABC LABS #035405

pg 001 of 003b

OBSERVATIONS AND/OR REMARKS FORM

Test Material: *	Lab Form No.: 55
Study No.: 35404 & 35405	Analytical Bio-Chemistry Labs Aquatic Toxicology Division 7200 ABC Lane, P.O. Box 1097 Columbia, Missouri 65205
Protocol No.: 8301	
ABC Study Director: A. Forbis	

Date/Initials	Comments

* OUTFALL 002 AND RED RIVER UPSTREAM SAMPLE

4 LITERS OF RECONSTITUTED WATER WERE PREPARED WITH DEIONIZED WATER AND THE FOLLOWING AMOUNTS OF COMPOUNDS; $NaHCO_3$ (.768g), $CaSO_4 \cdot 2H_2O$ (.480g), $MgSO_4$ (.480g) AND KCl (.032g).

** ALKALINITY AND HARDNESS TESTS ON THE RECONSTITUTED WATER RESULTED IN VALUES OF (118-120), AND ~176 RESPECTIVELY.

"This is an exact copy of The original document"

By [signature] date 1/13/87

** ALKALINITY AND HARDNESS (mg/l AS $CaCO_3$) HACH Model AL-DT and HAC-DT.

Prepared By: [signature] Date: 1-7-87

Checked By: [signature] Date: 1/8/87

OBSERVATIONS AND/OR REMARKS FORM

Test Material: RED RIVER UPSTREAM SAMPLE

Study No.: 35405

Protocol No.: 8301

ABC Study Director: A. FORBIS

Lab Form No.: 55

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P.O. Box 1097
Columbia, Missouri 65205

Date/Initials	Comments
1/9/87 SF	An analysis of pH in control water at 24 hours yielded a value of 7.8. (CORNING Model 125 pH and mV meter / CORNING Model 476182 pH probe)

Prepared By: Scott Frazier Date: 1/9/87

Checked By: [illegible] Date: 1/26/87

PREPARATION OF EFFLUENT CONCENTRATIONS (volume/volume)

Effluent Designation: Red River Upstream Sample
Industry: Molycorp INC
Study No.: 35405
ABC Study Director: A. FORBIS

Lab Form No.: 94
Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane; P.O. Box 1097
Columbia, MO 65205

☑ PRELIMINARY (SCREEN) ☐ DEFINITIVE

Dilution Water Type:

☐ ABC Well Water ☑ ABC Recon. Water (HARD)

☐ Effluent Receiving Water _____ ☐ Other _____

Sample No.	Aliquot Volume (ml)	Dilution Volume (ml)	Final Conc. (%)
1	0	200	CONTROL
2	200	0	100
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			

Remarks:

Prepared By: [signature] Date: 1/8/87
Checked By: [signature] Date: 1/9/87
Study Director: [signature] Date: 1/9/87

INVERTEBRATE CULTURE RECORD

Species: Daphnia Magna

Lot No.: 86-E-8

Date Initiated: 12/23/86

Lab Form No: 52

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P. O. Box 1097
Columbia, MO 65205

Date	No. Adult Alive	Instar Present	Fed & Changed	Study I.D.	Comments	Obs. By:
12/23	—	~30	✓		~200 ml algae + ~0.1 ml midge food	JW.
12/28	30	—	✓		" "	JGm
12/30	29	—	✓		" "	MR
1/1/87	29	~75	✓		" "	MR
1/4/87	29	~300	✓		" "	SF
1/6	29	~300	✓		" "	JW
1/7	29	~30	✓		" "	JW
1/8	—	—	—	3540.4 — 40 3540.5 — 40		MR
1/8	29	~400	✓		" "	JW
1/11	29	~200	✓		" "	JGm

"This is an exact copy of The original document"

By Jennifer Wirth date 1/13/87

Prepared by: Jennifer Wirth Date: 12/23/86

Checked by: Scott Frazier Date: 1/13/87

*midge food is yeast, Tetramin® & cereal leaves in water JW 12/23/86

MOLYCORP, INC.

QUESTA - DIVISION

UNOCAL- 76

This sheet represents a chain of custody for effluent samples collected at outfall 002 Red River @ Capulin

Located in Taos County, Questa, New Mexico on 1/7/87 above

Sample bottles were prepared by Molycorp employees in accordance with guidance set forth by the U.S. Environmental Protection Agency for the existing program permit. Sampling procedures were also followed in accordance with permit requirments.

Sample (s) Identification 002 outfall

Red River above Capulin Canyon

Sample (s) ~~Identification~~ collected at 002 outfall last portion of composite 7:30 am

Red River above Capulin Canyon

Grab 7:00 am

By: Fred Parker

Environmental Analyst

"This is an exact copy of The original document"

By Jennifer Dilth date 1/13/87

ABC LABS #035405

MOLYCORP INC.

QUESTA-DIVISION

UNOCAL-76

Les handed over to carrier by Molycorp employee

DATE: 1/7/87

TIME: 7:30 AM

CARRIER: J. Martinez

EMPLOYER: Molycorp Inc.

MOLYCORP PERSONNEL: J. Martinez

Sample(s) delivered to ABC lab for analysis. Please sign and date this letter upon receipt of sample(s)

Sample(s) received by: ABC Laboratories Inc.
7200 ABC Lane
P.O. Box 1097
Columbia, MO 65205
(314) 474-8579

for the following analyses: Toxicity testing

Received by: Scott Frazier ABC LABS

DATE: 1/8/87 10:30 AM

Please return this letter with the analytical results, signed and dated below.

Scott Frazier 1/13/~~86~~ 87
Date

"This is an exact copy of
The original document"
By Scott Frazier date 1/13/87

@ Dating Error, SF, 1/13/87

ABC LABS #035405

DEFINITIVE CONCENTRATION NOTIFICATION

Firm: Molycorp, Inc.
Address: P.O. 469
City/State/Zip: Questa, NM 87556
Phone: (505) 586-0212

Lab Form No.: 186
Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P.O. Box 1097
Columbia, MO 65202

Sponsor's Study Director: MR. FRED MARTINEZ
Person notified: same (✓) Other:

Date of Notification: 1/9/87

Notification of preliminary testing results with the following definitive test levels agreed upon:

Test Material	ABC #	Test Species	Test Conc. (mg/l)
Red River Upstream Sample	35405	Daphnia magna	N/A

Informed sponsor that screen test mortality was 0%.

Notified By: Scott Sanger Date: 1/9/87

ANALYTICAL BIO-CHEMISTRY LABORATORIES, INC.
P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

RECEIVED
JAN 20 '87
A.B.C. LABS

ABC PROTOCOL NO. 8301

(August 21, 1984)

Effluent Static Bioassay with *Daphnia magna* or
Mysid Shrimp (*Mysidopsis bahia*) to
Meet EPA Region 6 NPDES Biomonitoring Guidelines

ABC Study Number 35405

Test Material Red River Upstream Sample

Test Species *Daphnia magna*

1.0 INTRODUCTION

The use of bioassays in the identification and control of industrial effluents is rapidly increasing. Indeed, aquatic toxicity tests are presently being incorporated into the NPDES guidelines of the Federal Clean Water Act (1). To assist industry in meeting requirements set forth by legislation and regulatory agencies, Analytical Bio-Chemistry Laboratories, Inc. offers a complete range of aquatic bioassay tests of industrial effluents.

As part of the NPDES permit process in U.S. EPA Region 6, a biomonitoring requirement has been imposed for certain permit holders in that area. Discharges subject to the new regulations in Region 6 (Texas, New Mexico, Oklahoma, Arkansas and Louisiana) will be required to submit aquatic toxicity data on their effluents to EPA on a periodic basis.

This ABC protocol discusses the biomonitoring guidelines in Region 6 and describes ABC's technical approach to assist industry in complying with these regulations.

2.0 OBJECTIVES

The primary objective of the toxicity test described herein is to evaluate the acute toxicity of an effluent to freshwater or marine invertebrates under static conditions. The species selected for this testing include *Daphnia magna* (freshwater effluent, <5 ppt salinity) and *Mysidopsis bahia* (saltwater effluent, ≥5 ppt salinity). The effluent will first be tested on a 100% effluent screen basis. If the screen results in ≥20% mortality of test organisms, a 48-hour, five concentration LC_{50} test will be conducted. An LC_{50} is the approximate concentration (percent) of test effluent that produces 50 percent mortality of test organisms after prescribed intervals (24 and 48 hours).

3.0 TESTING FACILITY

The study will be conducted by the Aquatic Toxicology Division of Analytical Bio-Chemistry Laboratories, Inc., 7200 East ABC Lane, P. O. Box 1097, Columbia, Missouri 65205 (314-474-8579).

4.0 REGION 6 BIOMONITORING REQUIREMENT

The following biomonitoring requirement is now being included in the NPDES permit process for many major industrial discharges in EPA Region 6. The permit language given here was that presented by the U.S. EPA at a regional workshop on the matter (2):

> The permittee shall determine if the effluent from this outfall is lethal to twenty (20) percent or greater of the culture of test organisms by use of the "Range-Finding Screening Test," set out in "Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms," EPA-600/4-78-012 (Rev. July, 1978) (3).

Organisms for this test shall be Daphnia sp. if the effluent is less than five (5) parts per thousand salinity or Mysid sp. if the effluent is greater than five (5) parts per thousand salinity. This screening test will be conducted within sixty (60) days of effectiveness of the biomonitoring requirements. Tests will be conducted once each quarter for a duration of two years utilizing a static method for 24 hours and following this dilution scheme only:

Effluent sample* - 100 percent by volume
Dilution water - 0 percent by volume

*24-hour composite; refrigerated after collection

If at any time during the two year testing period a test shows a lethality of twenty (20) percent or more of the test organisms, the permittee shall within twenty-four (24) hours conduct a replacement static 48-hour median lethal concentration (LC_{50}) test on the originally collected sample. Replacement of effluent samples shall be once per 24 hours. Organisms for this test shall be Daphnia sp. if the effluent is less than five (5) parts per thousand salinity and reconstituted fresh water (EPA-600/4-78-012 Section 4) shall be used for dilution. If the effluent contains greater than five (5) parts per thousands salinity, Mysid sp. shall be used as the test organism, and reconstituted seawater will be used as dilution water (EPA-600/4-78-012 Section 4). The remaining LC_{50} methodology is available in EPA-600/4-78-012.

All screening and LC_{50} test results shall be reported with the Discharge Monitoring Reports.

It should be noted by the permittee that slight changes from the above permit language (e.g., pH modification of effluent) may be incorporated into his specific permit. Attention should be given to the permit requirements for each effluent before testing is initiated. ABC requires that a copy of the permit's biomonitoring clause be forwarded to ABC prior to testing so that consideration can be given to pH modification, etc.

5.0 METHODS AND MATERIALS

5.1 Methodology. The methods applied for both the preliminary and definitive phases of this test are basically those described in Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms (3), Methods for Acute Toxicity Tests with Fish, Macroinvertebrates, and Amphibians (4), Standard Practice for Conducting Acute Toxicity Tests with Fishes, Macroinvertebrates, and Amphibians (5), Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates

and Amphibians (6), and Standard Methods for the Examination of Water and Wastewater (7). This protocol has been written to comply with the federal Good Laboratory Practice Regulations (8).

5.2 Test Organisms. The species recommended by the EPA for this testing are Daphnia sp. and Mysid sp. Daphnia sp. are to be used when the effluent salinity is <5 parts per thousand (ppt) and Mysid sp. are to be used when the effluent salinity is ≥5 ppt. The species maintained by ABC, and acceptable by EPA, for this testing are Daphnia magna and Mysidopsis bahia. The test lot of Daphnia magna will be obtained from an in-house culture at ABC. The taxonomic key presented by Pennak (9) will be used to identify to species the test daphnids. All test daphnids will be held in a controlled temperature area at 20 ±2°C. The lighting will be 50-70 footcandles on a 16 hour daylight photoperiod. During the holding period, they will receive a suspension of algae (Selenastrum capricornutum) supplemented with a blended fish food/cerophyl suspension at least every 3 days. First-instar daphnids (<24 hours old) will be selected for testing and will not be fed during the test.

Mysid shrimp (Mysidopsis bahia) will be obtained from a commercial supplier (e.g., Sea Plantations, Inc. or Gulf Breeze Specimens) and maintained in culture at ABC. Mysids will be cultured following the techniques described by the U.S. EPA (10). Test mysids will be selected from a sub-culture of immature individuals.

5.3 Effluent Sample. The sample submitted to ABC for testing must be a 24-hour composite sample, consisting of equal volumes taken every hour (2). The sample should be collected in a glass or polyethylene container, refrigerated after collection, and shipped to arrive at ABC within 24 hours after collection. A one-gallon sub-sample of the 24-hour composite will be satisfactory for the toxicity test(s) required. ABC can provide sample containers and mailing cartons and can assist plant personnel in shipping arrangements so that the sample arrives at ABC in a timely manner.

5.4 Dilution Water. It will not be necessary to collect actual receiving water for effluent dilutions for this test. The biomonitoring regulations recommend the use of reconstituted freshwater or saltwater listed in the EPA effluent methods manual (3). The freshwater dilution water (for Daphnia sp.) will be a hard reconstituted water of 160-180 mg/l hardness (as $CaCO_3$). The reconstituted saltwater (for Mysid sp.) will be prepared from a commercial salt mix similar in composition to that listed in the EPA effluent manual (3) and adjusted to a test salinity of 15-25 ppt.

5.5 Screen Test. The biomonitoring regulations require that a 24-hour 100% effluent screen be conducted before more definitive (LC_{50}) testing is considered. As stated in section 4.0, if ≥20% of the test organisms die in the 100% solution, the permittee shall then conduct a 48-hour static replacement LC_{50} test. This latter test is discussed in section 5.6.

The screen test will be conducted in 250 ml glass beakers containing 200 ml of solution. These test chambers will be held in a controlled temperature area at 20 ±2°C on a 16-hour daylight vs. 8-hour darkness photoperiod with 30-minute dawn/dusk transition periods. The screen test will include duplicate test chambers at 100% vs. duplicate control chambers containing dilution water only. Ten test organisms will be placed in each replicate; therefore, twenty total organisms will be exposed to the 100% effluent and control (dilution) water.

After a 24-hour exposure period, the test chambers will be observed for mortality and abnormal effects. If the combined replicate mortality is ≧20%, the 48-hour LC_{50} test will be initiated.

5.6 48-Hr LC_{50} Test. The definitive test will be conducted by exposing the test organisms to at least 5 effluent concentrations and a dilution water control. The test will be conducted using duplicate beakers per concentration containing 10 test organisms per beaker (=20 per concentration). The test concentrations used will follow one of the logarithmic series presented in Table 1. The most common concentration series used is 10-100% (10, 18, 32, 56 and 100%). Several effluent toxicity tests conducted by ABC have shown that most industrial effluents are toxic within this range.

The test organisms will be placed in the test chambers by stratified random assignment within 30 minutes after solution preparations. The test chambers will be observed for mortality and/or abnormal effects every 24 hours, with a record maintained of mortality and abnormal behavior for each concentration tested.

5.7 Test Procedure - Chemical and Physical. Upon receipt of the test effluent, the following parameters will be measured of the 100% effluent: temperature, dissolved oxygen, pH, conductivity and hardness. These same parameters, except hardness, will be measured at 0 and 48 hours of definitive testing in one replicate of the control, low concentration, middle concentration and high concentration solutions.

5.8 Analysis of Results. The results of the definitive study will be statistically analyzed for 24 and 48 hour LC_{50} values and their corresponding 95 percent confidence limits. These values will be determined by an LC_{50} computer program developed by Stephan et al. (11) or by manual methods described by Litchfield-Wilcoxon (12) and Stephan (13).

5.9 Report. One copy of the report will be submitted. The report will contain all original raw data. Additional copies will be provided, if requested by the sponsor, at a copy rate of $50 per copy. The report will include the following:

5.9.1 Study dates, name and address of test facility.

5.9.2 Objectives and test methods.

5.9.3 Reference to the statistical methods used for data analysis.

5.9.4 Description of test effluent (date of receipt, storage conditions, physical characteristics, and method of preparing test concentrations).

5.9.5 Description of test design.

5.9.6 Summary of the data analysis, mortality observations and test water quality.

5.9.7 Location of raw data.

5.9.8 List and signatures of study personnel.

5.9.9 GLP compliance statement by study director and a statement by ABC's Quality Assurance Unit.

5.9.10 The report appendix will contain the original raw data for mortality observations and water quality, letter of test authorization, letters of authorized protocol changes and a copy of the approved protocol.

5.10 Data Retention. All original raw data generated in the preliminary and definitive studies will be provided to the study sponsor in the appendix to the final report. A copy of the data will be retained in ABC's archives.

6.0 PROTOCOL CHANGES

In the event that modifications of this protocol are deemed necessary, a written statement of any changes and reason(s) proposed by the study sponsor or ABC will be submitted to the other party. All agreed changes will be expressed in writing, signed and dated by the sponsor's study director. The signed changes will be appended to the protocol and included with the final report. This protocol is subject to revision when felt necessary to comply with changes in federal regulations and state of the art methodology. The protocol for a specific study will not be changed while the study is in progress without discussion with the study sponsor.

7.0 SPONSOR AUTHORIZATIONS DURING THE STUDY

Should a problem develop while the study is in progress, ABC will notify the study director within 24 hours. The problem and suggested test modifications will be discussed by telephone. ABC will proceed with the changes felt necessary upon the verbal authorization of the study director. A letter for written authorization will then be submitted by ABC to the study director and handled in the same manner discussed in section 6.0.

8.0 TEST-SPECIFIC INFORMATION

8.1.1 General. The following items will be addressed for each static bioassay. This information is necessary to be in compliance with Good Laboratory Practice Regulations (8). Sections 8.2, 8.3, 8.7 and 8.9 are to be completed by the study sponsor. Sections 8.4, 8.5 and 8.6 will be completed by ABC.

8.1.2 GLP Compliance. To be in compliance with the Federal Good Laboratory Practice regulations, the report of the investigation conducted utilizing this protocol must contain a statement that the study was conducted in accordance with one of the following GLP's.

1. U.S. F.D.A. Good Laboratory Practice Regulations (21 CFR 58).

2. U.S. E.P.A. Good Laboratory Practice Standards; Toxic Substances Control (40 CFR 792).

3. U.S. E.P.A. Good Laboratory Practice Standards; Pesticide Programs (40 CFR 160).

4. OECD Principles of Good Laboratory Practice; Annex 2 C(81) 30(Final).

5. Any state or local GLP which may apply.

The specific GLP regulation, for which this study specific protocol and resultant study must be in compliance with, is to be designated by the study sponsor in section 8.8 of this protocol.

8.2 Study Sponsor:

8.2.1 Company Molycorp, Incorporated

8.2.2 Address 3 Miles East of Questa on State Road 38
P.O. Box 469, Questa, New Mexico 87556

8.2.3 Sponsor's Study Director

Mr. Fred Martinez — Name
Analyst — Title

8.3 Effluent:

8.3.1 Sample Number Red River Upstream Sample

8.3.2 NPDES Permit Number NM-0022306

8.3.3 Physical Description Clear Liquid

8.3.4 Handling Precautions Normal Precautions

8.4 Test Concentrations: To be determined after 24-hour screen of 100% effluent and consultation with study sponsor.

8.4.1 Sponsor's study director notified of screen results:
Date 1/9/87 ABC Biologist [signature]
100% effluent screen = ____ ≥20% mortality ✓ <20% mortality

8.4.2 Definitive test concentrations agreed upon:
N/A

8.5 Test Organisms:

8.5.1 Species Daphnia magna

8.5.2 Supplier ABC in-house culture

8.6 Study Dates:

8.6.1 Proposed starting date of definitive study 1/8/87

8.6.2 Proposed completion date of definitive study 1/22/87

8.7 ABC Study Personnel:

8.7.1 Study Director

Alan D. Forbis — Name
Supervisor, Invertebrate and Bioconcentration Investigations — Title

8.7.2 Aquatic Manager

Carl M. Thompson — Aquatic Toxicology Manager
Name — Title

8.8 GLP Compliance (check the most appropriate):

EPA-FIFRA____; EPA-TSCA____; FDA____; OECD____; Other NPDES.

8.9 Protocol Approvals. The following is to be signed by the appropriate study personnel:

8.9.1 Sponsor's Study Director

Fred Martinez — Analyst — 1/15/87
Name — Title — Date

8.9.2 ABC's Study Director

Alan D. Fuller — Supervisor, Invertebrate and Bio. Investigations — 1/6/87
Name — Title — Date

8.9.3 ABC's Manager

Carly Thomson — Aquatic Tox. Manager — _____
Name — Title — Date

8.10 Authorization to return the test material and sample:

8.10.1 Sponsor's authorized agent to receive the test material and sample.

Fred Martinez — Analyst
Name — Title

8.10.2 Shipping Address.

Molycorp, Incorporated
Company

3 Miles East of Questa Highway 38
Street Address

Questa — New Mexico — 87556
City — State — Zip Code

505-586-0212
Phone Number

8.10.3 Hazardous shipment information.

_____
Proper Shipping Name

_____
Class of Restricted Articles (e.g. flammable, poison, radioactive, etc.)

TABLE 1: Guide to selection of experimental concentrations based on progressive bisection of intervals on logarithmic scale.

Col. 1	Col. 2	Col. 3	Col. 4	Col. 5
10.0	10.0	10.0	10.0	10.0
				8.7
			7.5	7.5
				6.5
		5.6	5.6	5.6
				4.9
			4.2	4.2
				3.7
3.2	3.2	3.2	3.2	3.2
				2.8
			2.4	2.4
				2.1
		1.8	1.8	1.8
				1.55
			1.35	1.35
				1.15
1.0	1.0	1.0	1.0	1.0

TABLE 2: Chemical Characteristics of Well Water Used by ABC's Aquatic Toxicology Division.

Parameters	Concentration
Temperature	15-20°C[b]
Dissolved Oxygen[a]	9.2-10.1 ppm[b]
pH	7.8-8.3[b]
Hardness ($CaCO_3$)	225-275 ppm[b]
Alkalinity ($CaCO_3$)	325-375 ppm[b]
Conductivity	700 µmhos/cm
NO_3-N	0.58 ppm
NO_3-NO_2-N	0.69 ppm
PO_4-P	<0.50 ppm
Aluminum	<20 ppb
Cadmium	<3 ppb
Chromium	<5 ppb
Cobalt	<5 ppb
Copper	<5 ppb
Iron	<5 ppb
Lead	<20 ppb
Nickel	<10 ppb
Silver	<5 ppb
Zinc	<1 ppb
Measured organophosphorus pesticides	<0.10 ppb
Measured organochlorine pesticides plus PCB's	<0.50 ppb

[a]After aeration.

[b]Represents seasonal variation, with the monthly range not exceeding 10%.

Sample dates: Trace elements = May 12, 1986, organophosphate and organochlorine analyses = May 12, 1986.

9.0 REFERENCES

(1) U.S. Congress. 1977. Clean Water Act. Public Law 95-217. Federal Register, Dec. 27, 1977, 91 STAT 1566-1611.

(2) U.S. Environmental Protection Agency. 1983. Effluent Toxicity Testing and Biomonitoring Workshop. Feb. 22-23, 1983, Dallas, Texas.

(3) Peltier, William. 1978. Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms. U.S. Environmental Protection Agency, EPA-660/4-78-012, revised July, 1978. 52 p.

(4) Committee on Methods for Toxicity Tests with Aquatic Organisms. 1975. Methods for Acute Toxicity Tests with Fish, Macroinvertebrates and Amphibians. Environmental Protection Agency, Ecological Research Series EPA-660/3-75-009, April, 1975. 61 p.

(5) American Society for Testing and Materials. 1980. Standard practice for conducting basic acute toxicity tests with fish, macroinvertebrates and amphibians. May, 1980, ASTM Committee E-35.23. 25 p.

(6) American Society for Testing and Materials. 1983. Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates and Amphibians. November, 1983, ASTM Committee E-47.01. 37 p.

(7) American Public Health Association. 1980. Standard Methods for the Examination of Water and Wastewater. 15th ed. Washington, D.C. 1134 p.

(8) (a) U.S. Food and Drug Administration. 1978. Nonclinical Laboratory Studies, Good Laboratory Practice Regulations (21 CFR, Part 58). Federal Register, Vol. 43, No. 247: 59986-60025.

(b) U.S. Environmental Protection Agency. 1983. Pesticide Programs; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 160). Federal Register, Vol 48, No. 230: 53946-53969.

(c) U.S. Environmental Protection Agency. 1983. Toxic Substances Control; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 792). Federal Register, Vol. 48; No. 230:53922-53944.

(d) Organization for Economic Cooperation and Development. 1981. OECD Guidelines for Testing of Chemicals, Principles of Good Laboratory Practice Annex 2, C(81) 30(Final):7-28.

(9) Pennak, R. W. 1978. Fresh-Water Invertebrates of the United States, 2nd Ed. 365 p.

(10) U.S. Environmental Protection Agency. 1979. Static culture methods for sheepshead minnows (Cyprinodon variegatus) and mysid shrimp (Mysidopsis bahia). Unpublished manuscript, U.S. EPA, Athens, GA.

(11) Stephan, C. E., K. A. Busch, R. Smith, J. Burke and R. W. Andrew. 1978. A computer program for calculating an LC_{50}. U. S. Environmental Protection Agency, Duluth, Minnesota, pre-publication manuscript, August, 1978.

(12) Litchfield, J. T., Jr. and F. Wilcoxon. 1949. A Simplified Method of Evaluating Dose-Effect Experiments. Jour. Pharm. Exp. Ther. 96:99-113.

(13) Stephan, C. 1977. Methods for calculating an LC_{50}, p. 65-84. In F. L. Mayer and J. L. Hamelick (eds.). Aquatic Toxicology and Hazard Evaluation. ASTM Special Technical Publication 634. ASTM. Philadelphia.

Assessment of Acute Toxicity of West Diversion Channel

December 1985

ANALYTICAL BIO-CHEMISTRY LABORATORIES, INC.
P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

Static Acute Toxicity Report
#34191

Assessment of the Acute Toxicity of West Diversion Channel to *Daphnia magna*

Submitted To:

Molycorp, Incorporated
Attn: Mr. John Stribling
3 miles East of Questa, on State Road 38
P.O. Box 469
Questa, New Mexico 87556

December 27, 1985

Submitted By: Analytical Bio-Chemistry Laboratories, Inc.
7200 East ABC Lane
P. O. Box 1097
Columbia, Missouri 65205
(314) 474-8579

Prepared By:

Alan D. Forbis 12/31/85
Alan D. Forbis Date
Aquatic Biologist III/Study Director

David Burgess 12/30/85
David Burgess Date
Biologist II

Approved By:

Carl M. Thompson 1-2-86
Carl M. Thompson Date
Supervisor, Aquatic Toxicology

Kathy Landholt 01/02/86
Kathy Landholt Date
Quality Assurance Officer

Michael Williams 1/2/86
Michael Williams, Ph.D. Date
Group Leader, Animal Toxicology

/bsb

SUMMARY

The acute toxicity of West Diversion Channel to *Daphnia magna* was assessed for U.S. EPA Region 6 NPDES permit requirements using the methods outlined by the Committee on Methods for Toxicity Tests with Aquatic Organisms. Water quality parameters of temperature, dissolved oxygen, conductivity, hardness and pH were measured on receipt of the sample and were within acceptable limits.

The results of the 24-hour static *Daphnia magna* toxicity screen indicated that the mortality was <20% for West Diversion Channel. In fact, no mortality was observed after the 24-hour exposure period. Therefore, a definitive 48-hour static acute study was not required. All reported values were based upon nominal concentrations.

The no-effect level observed for West Diversion Channel was 100% of effluent after 24-hours, which was based on the lack of mortality and abnormal effects.

INTRODUCTION

This static bioassay was performed by the Aquatic Toxicology Division of Analytical Bio-Chemistry Laboratories, Inc., Columbia, Missouri, for Molycorp, Inc., from December 11, 1985 to December 12, 1985. The purpose of this test was to assess toxicity of a 100% solution of West Diversion Channel to Daphnia magna. The assessment was designed to meet the NPDES permit process in U.S. EPA Region 6 (1) requiring biomonitoring of industrial charges. The screen test was conducted following the procedures outlined in ABC protocol #8301, as approved by Mr. John Stribling; Molycorp, Inc. on December 13, 1985.

METHODS AND MATERIALS

The procedures applied for the 24-hour toxicity screen were basically those described in Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms (2), Methods for Acute Toxicity Tests with Fish, Macroinvertebrates, and Amphibians (3), Standard Practice for Conducting Acute Toxicity Tests with Fishes, Macroinvertebrates, and Amphibians (4), Proposed Standard Practice for Con-ducting Acute Toxicity Test on Aqueous Effluents with Fishes, Macroinvertebrates and Amphibians (5), and Standard Methods for the Examination of Water and Wastewater (6). The test lot of Daphnia magna were obtained from a in-house culture at ABC. The taxonomic key presented by Pennak (7) was used to identify to species the test daphnids. All test daphnids were held in a controlled temperature area at 20 ±2°C. The lighting was 50-70 footcandles on a 16 hour daylight photoperiod. During the holding period, they received a suspension of algae (Selenastrum capricornutum) supplemented with a blended fish food/cerophyl suspension at least every 3 days. First-instar daphnids (<24-hours old) were selected for testing and were not fed during the test.

The static Daphnid bioassay was conducted in 250-ml glass beakers containing 200 ml of sample and/or ABC well water. The ABC well water control contained water with chemical characteristics listed in Table 1. These vessels were kept at 20 (±2.0) °C in a temperature controlled area. The lighting was maintained at 50-70 footcandles on a 16-hour daylight photoperiod.

A 24-hour screen test, composed of duplicate 100% effluent was performed to estimate the toxicity of the effluent. Also included was a duplicate ABC well water control. A five-six concentration static acute toxicity study would be performed if ≧20% mortality was observed after 24-hours in the screen test.

The West Diversion Channel was received on December 11, 1985 as a clear liquid and was received at 2°C. The samples were allowed to warm to test temperature before addition to Daphnia.

RESULTS

After 24-hour, the results indicated that the mortality of *Daphnia magna* to West Diversion Channel was <20%. The no-effect concentration based on the lack of mortality and abnormal effects was 100%. No abnormal effects, such as mortality, surfacing, and daphnids lying on the bottom were observed after 24-hours. Since mortality was not required per biomonitoring guidelines established for U.S. EPA Region 6.

Table 2 presents the water quality parameters measured on sample receipt. Hardness, alkalinity (as $CaCO_3$) and conductivity for the West Diversion Channel sample were 472 and 176 mg/l and 1200 µMHOS, respectively. Dissolved oxygen and pH for this sample were 9.8 mg/l and 7.3, respectively, and were considered adequate for testing (3).

The study was conducted following the intent of the Good Laboratory Practice Regulations (8) and the final report was reviewed by Analytical Bio-Chemistry Laboratories' Quality Assurance Unit. All orginal raw data was provided to Molycorp, Inc. with a copy retained at Analytical Bio-Chemistry Laboratories.

TABLE 1: Chemical Characteristics of Well Water Used by ABC's Aquatic Toxicology Division.

Parameters	Concentration
Temperature	15-20°C[b]
Dissolved Oxygen[a]	9.2-10.1 ppm[b]
pH	7.8-8.3[b]
Hardness ($CaCO_3$)	225-275 ppm[b]
Alkalinity ($CaCO_3$)	325-375 ppm[b]
Conductivity	700 μmhos/cm
NO_3-N	0.74 ppm
NO_3-NO_2-N	0.74 ppm
PO_4-P	<0.10 ppm
Aluminum	<20 ppb
Arsenic	<0.2 ppb
Cadmium	<2 ppb
Chromium	<3 ppb
Cobalt	<4 ppb
Copper	<3 ppb
Iron	12 ppb
Lead	<5 ppb
Mercury	<0.5 ppb
Nickel	<15 ppb
Silver	<5 ppb
Zinc	11 ppb
Measured organophosphorus pesticides	c
Measured organochlorine pesticides plus PCB's	c

[a]After aeration.

[b]Represents seasonal variation, with the monthly range not exceeding 10%.

[c]Less than minimum detectable limits.

TABLE 2

Water Quality Measurements During the Acute Toxicity Screen Test of West Diversion Channel to *Daphnia magna*

	ABC Well Water		100% West Diversion Channel		
	0 hr.	24 hr.	On Receipt	0 hr.	24 hr
Temperature [a]	20	20	2	20	20
Dissolved Oxygen [b]	8.5	8.5	9.8	9.6	9.7
pH [c]	8.1	8.4	7.3	7.2	8.1
Conductivity [d]	350	400	1200	1250	1500
Hardness [e]	---	---	472	---	---
Alkalinity [e]	---	---	176	---	---

[a]Temperature °C - Monitored with a mercury thermometer.

[b]Dissolved oxygen concentrations (mg/l) - Dissolved Oxygen System (YSI Model 54). Saturation at 20°C is 9.2 mg/l.

[c]pH - pH Probe (Corning Model 476182) used with a Corning Model 125 pH and mV meter.

[d]Conductivity (μMHOS) - Conductivity Meter (YSI Model 33).

[e]Hardness and alkalinity (mg/l as $CaCO_3$) - HACH Model AL-DT and HAC-DT.

LITERATURE CITED

(1) U.S. Environmental Protection Agency. 1983. Effluent Toxicity Testing and Biomonitoring Workshop. Feb. 22-23, 1983, Dallas, Texas.

(2) Peltier, William. 1978. Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms. U.S. Environmental Protection Agency, EPA-660/4-78-012, revised July 1978. 52 p.

(3) Committee on Methods for Toxicity Tests with Aquatic Organisms. 1975. Methods for Acute Toxicity Tests with Fish, Macroinvertebrates and Amphibians. Environmental Protection Agency, Ecological Research Series EPA-660/3-75-009, April 1975. 61 p.

(4) American Society for Testing and Materials. 1980. Standard practice for conducting basic acute toxicity tests with fish, macroinvertebrates and amphibians. May 1980, ASTM Committee E-35.23. 25 p.

(5) American Society for Testing and Materials. 1983. Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates and Amphibians. November 1983, ASTM Committee E-47.01. 37 p.

(6) American Public Health Association. 1980. Standard Methods for the examination of Water and Wastewater. 15th ed. Washington, DC. 1134 p.

(7) Pennak, R. W. 1978. Freshwater Invertebrates of the United States. John Wiley & Sons, New York, 2nd ed. 803 p.

(8) (a) U.S. Food and Drug Administration. 1978. Nonclinical Laboratory Studies, Good Laboratory Practice Regulations (21 CFR, Part 58). Federal Register, Vol. 43, No. 247:59986-60025.

(b) U.S. Environmental Protection Agency. 1983. Pesticide Programs; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 160) Federal Register, Vol. 48, No. 230:53946-53969

(c) U.S. Environmental Protection Agency. 1983. Toxic Substances Control; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 792). Federal Register, Vol. 48; No. 230:53922-53944

(d) Organization for Economic Cooperation and Development. 1981. OECD Guidelines for Testing of Chemicals, Principles of Good Laboratory Practice Annex 2, C(81) 30(Final):7-28.

QUALITY ASSURANCE STATEMENT

Quality Assurance Statement for final report #34191 entitled, "Acute Toxicity of West Diversion Channel to *Daphnia magna*," for John Stribling; Molycorp, Inc., Questa, New Mexico.

In accordance with ABC Laboratories' intent that all studies conducted at our facilities are designed and function in conformance with good laboratory practice regulations and the protocols for individual laboratory studies, an inspection of the final report for West Diversion Channel was conducted and found to be in acceptable form by a member of our Quality Assurance Unit. A final inspection of all data and records on December 20, 1985, indicates that the report submitted to you is an accurate reflection of the study as it was conducted by ABC Laboratories.

Should you have any questions relating to the information provided in this statement or the function of our Quality Assurance Unit, please contact the QA Unit at your convenience.

Kathy Landholt 01/02/86

Kathy Landholt Date
Quality Assurance Officer

STUDY COMPLIANCE STATEMENT

Study Compliance Statement for ABC report #34191 entitled, "Acute Toxicity of West Diversion Channel to *Daphnia magna*," for John Stribling; Molycorp, Inc., Questa, New Mexico.

In accordance with ABC Laboratories' intent that all aquatic toxicity tests conducted by our facility follow good laboratory practices, ABC's study director for the above test herein confirms that the study was conducted in compliance with the U.S. E.P.A. Good Laboratory Practice Standard; Toxic Substances Control (40 CFR 792).

All original raw data was sent to Molycorp, Inc., with a copy retained at Analytical Bio-Chemistry Laboratories.

Alan D. Forbis 12/31/85

Alan D. Forbis — Date
ABC Study Director

STUDY COMPLIANCE STATEMENT

Study Compliance Statement for ABC report #34191 entit
Toxicity of West Diversion Channel to Daphnia magna,
Stribling; Molycorp, Inc., Questa, New Mexico.

In accordance with ABC Laboratories' intent that
toxicity tests conducted by our facility follow good
practices, ABC's study director for the above test herein c
the study was conducted in compliance with the U.S.
Laboratory Practice Standard; Toxic Substances Control (4

All original raw data was sent to Molycorp, Inc.,
retained at Analytical Bio-Chemistry Laboratories.

Alan D. Forbis

Alan D. Forbis
ABC Study Director

APPENDIX - RAW DATA

NOTE: Some of the records which appear in this raw data appendix have been provided as photocopies of original records on file at ABC. This has been done by necessity for certain data which are used commonly in several studies at ABC. Such records include reference toxicity test data, standard solution preparations, organism culture logs, and well water chemical screen results.

EFFLUENT TOXICITY TEST
MORTALITY AND BEHAVIORAL OBSERVATIONS

Effluent Designation: WEST DIVERSION CHANNEL
Industry: MOLYCORP
Study No.: 34191
ABC Study Director: A. FORBIS

Lab Form No.: 91

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P.O. Box 1097
Columbia, Missouri 65205

☐ Prelim. ☑ Definitive Test Species: D. magna (Lot #85-C-9)
Date Initiated: 12-11-85 (4:00 AM) Test Terminated: 12-12-85
Dilution Water: NONE No./Vessel: 10 Vessel Size: 250ml

Effluent Conc. (%)	24 hours Dead	24 hours Obs.	48 hours Dead	48 hours Obs.	72 hours Dead	72 hours Obs.	96 hours Dead	96 hours Obs.
Control A	0	N						
B	0	N						
100 A	0	N						
B	0	N						
Observer Date	DB 12/12/85 3:40PM							

Remarks: N = Normal; LOE = Loss of Equilibrium;
SUR = Surfacing

Prepared By: Daniel Burgess Date: 12/11/85
Checked By: Alan D. Forbis Date: 12/12/85

WATER QUALITY FOR EFFLUENT SAMPLES

Test Material: WEST DIVERSION CHANNEL

Study No.: 34191

Protocol No.: 8301

ABC Study Director: A. FORBIS

Lab Form No.: 28b

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane; P.O. Box 1097
Columbia, Missouri 65205

Concentration ~~mg/l~~ %	12/11/85 DAY 0 DB				12/12/85 DAY 1 DB				DAY				DAY			
	Temp °C	D.O.[a] mg/l	pH[b]	Cond. µMhos	Temp °C	D.O. mg/l	pH	Cond. µMhos	Temp °C	D.O. mg/l	pH	Cond. µMhos	Temp °C	D.O. mg/l	pH	
CONTROL	20	8.5	8.1	350	20	8.5	8.4	400								
100 (A)	20	9.6	7.2	1250	20	9.7	8.1	1500								

[a]Dissolved oxygen concentrations - Dissolved Oxygen Probe (YSI Dissolved Oxygen System Model 54).

[b]pH-pH Probe (Corning Model 476182) used with Corning Model 125 pH and mV meter.

[c] 100% Effluent on Receipt	Temp °C	D.O. mg/l	pH	Cond. µMhos	Hard mg/l	Alk. mg/l	~~NH_3 mg/l~~			Date	Int.
	2	9.8	7.3	1200	472	176				12-11-85	DB

Prepared By: Darrel Bunzer Date: 12-11-85

Checked By: A. Forbis Date: 12/11/85

Remarks:

STUDY# 034191

PREPARATION OF EFFLUENT CONCENTRATIONS (volume/volume)

Effluent Designation: WEST DIVERSION CHANNEL

Industry: MOLYCORP

Study No.: 34191

ABC Study Director: A. FORBIS

Lab Form No.: 94

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane; P.O. Box 1097
Columbia, MO 65205

☐ PRELIMINARY ☑ DEFINITIVE

Dilution Water Type:

☐ ABC Well Water ☐ ABC Recon. Water

☐ Effluent Receiving Water _____ ☑ Other NONE

Sample No.	Aliquot Volume (ml)	Dilution Volume (ml)	Final Conc. (%)
1	—	200	CONTROL
2	200	—	100
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			

Remarks:

Prepared By: David Burgess Date: 12/11/85

Checked By: A. Forbis Date: 12/11/85

Study Director: A. Forbis Date: 12/11/85

INVERTEBRATE CULTURE RECORD

Species: Daphnia magna

Lot No.: 85-C-9

Date Initiated: 11-19-85

Lab Form No: 52

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P. O. Box 1097
Columbia, MO 65205

Date	No. Adult Alive	Instar Present	Fed & Changed	Study I.D.	Comments	Obs. By:
11/19	—	~25	✓		300 ml ALGAE 2ml MIDGE FOOD	DB
11/21	—	~25	✓		" "	SF
11/24	25	—	✓		" "	DB
11/26	23	—	✓		" "	SF
11/29	23	~150	✓		" "	SF
12/1	23	~500	✓		" "	Wam
12/3	23	~40	✓		" "	SF
12/5	23	900	✓		" "	DB
12-8	23	~600	✓		" "	DB
12-9				34029 — 160		SF
12-10	22	~100	✓		" "	SF
12/11				33847 — 120 34190 — 40 34191 — 40		DB
12/11	22	700	✓		" "	DB

"This is an exact copy of The original document"

By C. Wilhelm date 12-12-85

Prepared by: Darrel Burgess Date: 11/19/85

Checked by: A. Forbes Date: 12/16/85

ANALYTICAL BIO-CHEMISTRY LABORATORIES, INC.
P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

Sample Date: April 2, 1985

ANALYSIS RESULTS FOR:

Aquatic Toxicology Division
Analytical Bio-Chemistry Laboratories, Inc.
7200 East ABC Lane
P. O. Box 1097
Columbia, Missouri 65205

Water Analysis Results

ABC Lab No.	33102
Sample I.D.	ABC Aquatic Lab Well Water
NO_3-N	0.74 mg/1
NO_3-NO_2-N	0.74 mg/1
PO_4-P	<0.10 mg/1
Al	<20 µg/1
Co	<4 µg/1
Fe	12 µg/1
As	<0.2 µg/1
Cd	<2 µg/1
Cr	<3 µg/1
Cu	<3 µg/1
Pb	<5 µg/1
Hg	<0.5 µg/1
Ni	<15 µg/1
Ag	<5 µg/1
Zn	11 µg/1

<Indicates less than, if present at all.

Del A. Koch
Chemist III

Please note that the original data for the analysis reported in this table are in ABC's archives.

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Sample Date: April 2, 1985

ANALYSIS RESULTS FOR:

Aquatic Toxicology Division
Analytical Bio-Chemistry Laboratories, Inc.
7200 East ABC Lane
P. O. Box 1097
Columbia, Missouri 65205

Analysis of Aquatic Lab Well Water for Organophosphates

ABC Lab No.	Customer I.D.	Compound	ppb
33103	Aquatic Lab Well Water	Diazinon	<0.10
		Methyl Parathion	<0.10
		Malathion	<0.10
		Ethyl Parathion	<0.10

Randy Ayling 6/25/85

Randy Ayling
Chemist I

Please note that the original data for the analysis reported in this table are in ABC's archives.

ANALYTICAL BIO-CHEMISTRY LABORATORIES, INC.
P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

Sample Date: April 2, 1985

ANALYSIS RESULTS FOR:

Aquatic Toxicology Division
Analytical Bio-Chemistry Laboratories, Inc.
7200 East ABC Lane
P. O. Box 1097
Columbia, Missouri 65205

Analysis of Aquatic Lab Well Water for Nonionic
Chlorinated Hydrocarbon Residue

ABC Lab No.	33103
Customer I.D.	ABC Aquatic Lab Well Water
Compound	ppb
αBHC	<0.01
Lindane	<0.01
Heptachlor	<0.01
Heptachlor Epoxide	<0.02
γChlordane	<0.02
αChlordane	<0.02
Aldrin	<0.01
Dieldrin	<0.03
p,p'-DDE	<0.03
o,p'-DDT	<0.05
p,p'-DDD	<0.05
p,p'-DDT	<0.05
Endrin	<0.05
Methoxychlor	<0.20
Toxaphene	<0.50
Aroclor 1242	<0.40
Aroclor 1016	<0.40
Aroclor 1254	<0.40
Aroclor 1260	<0.40
HCB	<0.01
Mirex	<0.10

<indicates less than, if present at all.

Randy Ayling 6/25/85
Randy Ayling
Chemist I

Please note that the original data for the analysis reported in this table are in ABC's archives.

MOLYCORP, INC.
QUESTA DIVISION

This sheet represents a chain of custody for effluent samples collected at OUTFALL 002 and WEST DIVERSION CHANNEL located in Taos County, Questa, New Mexico on 12-10-85.

Sample bottles were prepared by Molycorp employees in accordance with guidance set forth by the U.S. Environmental Protection Agency for the existing program permit. Sampling procedures were also followed in accordance with permit requirements.

Sample(s) Identification: 1. 002-12-10-85
2. WEST DIVERSION CHANNEL 12-10-8.

Sample(s) collected at: 1. 10:11 AM LAST PORTION
2. 10:13 AM " " "

By: John Stratton
Environmental ~~Technician~~ ENG.

"This is an exact copy of
The original document"
By B. Blacklock date 12-16-85

Sample(s) handed over to carrier by Molycorp employee:

DATE: 12-10-85

TIME: 10:45 AM

CARRIER: Timothy L Seeger

EMPLOYER: Garrett Inc.

MOLYCORP PERSONNEL: John [illegible]

Sample(s) delivered to ABC Lab for analysis. Please sign and date this letter upon receipt of sample(s).

Sample received by: ABC Laboratories Inc.
7200 ABC Lane
P.O. Box 1097
Columbia, MO 65205
(314) 474-8579

for the following analyses: Toxicity testing

Received by: David Burgess
ABC Laboratories Inc.

Date: 12-11-85 10:30 AM

Please return this letter with the analytical results, signed and dated below.

David Burgess
ABC Laboratories Inc.

12-16-85
Date

"This is an exact copy of
The original document"

By B. Blacklock date 12-16-85

DEFINITIVE CONCENTRATION NOTIFICATION

Firm: MOLYCORP
Address: P.O. Box 469
City/State/Zip: Questa, NM 87556
Phone: ( )

Lab Form No.: 186

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P.O. Box 1097
Columbia, MO 65202

Sponsor's Study Director: JOHN STRIBLING
Person notified: same (✓) Other:

Date of Notification: 12/12/85

Notification of preliminary testing results with the following definitive test levels agreed upon:

Test Material	ABC #	Test Species	Test Conc. ~~(mg/l)~~ %
WEST DIVERSION CHANNEL	34191	D. magna	100

NOTIFIED JOHN STRIBLING OF THE RESULTS OF THE 24 HR TOX SCREEN. MORTALITY WAS <20%, SO A DEFINITIVE TEST WAS NOT NEEDED.

Notified By: Janet Bunger Date: 12/12/85

ANALYTICAL BIO-CHEMISTRY LABORATORIES, INC.
P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

ABC PROTOCOL NO. 8301

(August 21, 1984)

Effluent Static Bioassay with *Daphnia magna* or Mysid Shrimp (*Mysidopsis bahia*) to Meet EPA Region 6 NPDES Biomonitoring Guidelines

ABC Study Number 34191

Test Material West Diversion Channel

Test Species *Daphnia magna*

ABC LABS
DEC 20 1985
RECEIVED

1.0 INTRODUCTION

The use of bioassays in the identification and control of industrial effluents is rapidly increasing. Indeed, aquatic toxicity tests are presently being incorporated into the NPDES guidelines of the Federal Clean Water Act (1). To assist industry in meeting requirements set forth by legislation and regulatory agencies, Analytical Bio-Chemistry Laboratories, Inc. offers a complete range of aquatic bioassay tests of industrial effluents.

As part of the NPDES permit process in U.S. EPA Region 6, a biomonitoring requirement has been imposed for certain permit holders in that area. Discharges subject to the new regulations in Region 6 (Texas, New Mexico, Oklahoma, Arkansas and Louisiana) will be required to submit aquatic toxicity data on their effluents to EPA on a periodic basis.

This ABC protocol discusses the biomonitoring guidelines in Region 6 and describes ABC's technical approach to assist industry in complying with these regulations.

2.0 OBJECTIVES

The primary objective of the toxicity test described herein is to evaluate the acute toxicity of an effluent to freshwater or marine invertebrates under static conditions. The species selected for this testing include *Daphnia magna* (freshwater effluent, <5 ppt salinity) and *Mysidopsis bahia* (saltwater effluent, ≧5 ppt salinity). The effluent will first be tested on a 100% effluent screen basis. If the screen results in ≧20% mortality of test organisms, a 48-hour, five concentration LC_{50} test will be conducted. An LC_{50} is the approximate concentration (percent) of test effluent that produces 50 percent mortality of test organisms after prescribed intervals (24 and 48 hours).

3.0 TESTING FACILITY

The study will be conducted by the Aquatic Toxicology Division of Analytical Bio-Chemistry Laboratories, Inc., 7200 East ABC Lane, P. O. Box 1097, Columbia, Missouri 65205 (314-474-8579).

4.0 REGION 6 BIOMONITORING REQUIREMENT

The following biomonitoring requirement is now being included in the NPDES permit process for many major industrial discharges in EPA Region 6. The permit language given here was that presented by the U.S. EPA at a regional workshop on the matter (2):

> The permittee shall determine if the effluent from this outfall is lethal to twenty (20) percent or greater of the culture of test organisms by use of the "Range-Finding Screening Test," set out in "Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms," EPA-600/4-78-012 (Rev. July, 1978) (3).

Organisms for this test shall be Daphnia sp. if the effluent is less than five (5) parts per thousand salinity or Mysid sp. if the effluent is greater than five (5) parts per thousand salinity. This screening test will be conducted within sixty (60) days of effectiveness of the biomonitoring requirements. Tests will be conducted once each quarter for a duration of two years utilizing a static method for 24 hours and following this dilution scheme only:

Effluent sample* - 100 percent by volume
Dilution water - 0 percent by volume

*24-hour composite; refrigerated after collection

If at any time during the two year testing period a test shows a lethality of twenty (20) percent or more of the test organisms, the permittee shall within twenty-four (24) hours conduct a replacement static 48-hour median lethal concentration (LC_{50}) test on the originally collected sample. Replacement of effluent samples shall be once per 24 hours. Organisms for this test shall be Daphnia sp. if the effluent is less than five (5) parts per thousand salinity and reconstituted fresh water (EPA-600/4-78-012 Section 4) shall be used for dilution. If the effluent contains greater than five (5) parts per thousands salinity, Mysid sp. shall be used as the test organism, and reconstituted seawater will be used as dilution water (EPA-600/4-78-012 Section 4). The remaining LC_{50} methodology is available in EPA-600/4-78-012.

All screening and LC_{50} test results shall be reported with the Discharge Monitoring Reports.

It should be noted by the permittee that slight changes from the above permit language (e.g., pH modification of effluent) may be incorporated into his specific permit. Attention should be given to the permit requirements for each effluent before testing is initiated. ABC requires that a copy of the permit's biomonitoring clause be forwarded to ABC prior to testing so that consideration can be given to pH modification, etc.

5.0 METHODS AND MATERIALS

5.1 Methodology. The methods applied for both the preliminary and definitive phases of this test are basically those described in Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms (3), Methods for Acute Toxicity Tests with Fish, Macroinvertebrates, and Amphibians (4), Standard Practice for Conducting Acute Toxicity Tests with Fishes, Macroinvertebrates, and Amphibians (5), Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates

and Amphibians (6), and Standard Methods for the Examination of Water and Wastewater (7). This protocol has been written to comply with the federal Good Laboratory Practice Regulations (8).

5.2 Test Organisms. The species recommended by the EPA for this testing are Daphnia sp. and Mysid sp. Daphnia sp. are to be used when the effluent salinity is <5 parts per thousand (ppt) and Mysid sp. are to be used when the effluent salinity is ≧5 ppt. The species maintained by ABC, and acceptable by EPA, for this testing are Daphnia magna and Mysidopsis bahia. The test lot of Daphnia magna will be obtained from an in-house culture at ABC. The taxonomic key presented by Pennak (9) will be used to identify to species the test daphnids. All test daphnids will be held in a controlled temperature area at 20 ±2°C. The lighting will be 50-70 footcandles on a 16 hour daylight photoperiod. During the holding period, they will receive a suspension of algae (Selenastrum capricornutum) supplemented with a blended fish food/cerophyl suspension at least every 3 days. First-instar daphnids (<24 hours old) will be selected for testing and will not be fed during the test.

Mysid shrimp (Mysidopsis bahia) will be obtained from a commercial supplier (e.g., Sea Plantations, Inc. or Gulf Breeze Specimens) and maintained in culture at ABC. Mysids will be cultured following the techniques described by the U.S. EPA (10). Test mysids will be selected from a sub-culture of immature individuals.

5.3 Effluent Sample. The sample submitted to ABC for testing must be a 24-hour composite sample, consisting of equal volumes taken every hour (2). The sample should be collected in a glass or polyethylene container, refrigerated after collection, and shipped to arrive at ABC within 24 hours after collection. A one-gallon sub-sample of the 24-hour composite will be satisfactory for the toxicity test(s) required. ABC can provide sample containers and mailing cartons and can assist plant personnel in shipping arrangements so that the sample arrives at ABC in a timely manner.

5.4 Dilution Water. It will not be necessary to collect actual receiving water for effluent dilutions for this test. The biomonitoring regulations recommend the use of reconstituted freshwater or saltwater listed in the EPA effluent methods manual (3). The freshwater dilution water (for Daphnia sp.) will be a hard reconstituted water of 160-180 mg/l hardness (as $CaCO_3$). The reconstituted saltwater (for Mysid sp.) will be prepared from a commercial salt mix similar in composition to that listed in the EPA effluent manual (3) and adjusted to a test salinity of 15-25 ppt.

5.5 Screen Test. The biomonitoring regulations require that a 24-hour 100% effluent screen be conducted before more definitive (LC_{50}) testing is considered. As stated in section 4.0, if ≧20% of the test organisms die in the 100% solution, the permittee shall then conduct a 48-hour static replacement LC_{50} test. This latter test is discussed in section 5.6.

The screen test will be conducted in 250 ml glass beakers containing 200 ml of solution. These test chambers will be held in a controlled temperature area at 20 ±2°C on a 16-hour daylight vs. 8-hour darkness photoperiod with 30-minute dawn/dusk transition periods. The screen test will include duplicate test chambers at 100% vs. duplicate control chambers containing dilution water only. Ten test organisms will be placed in each replicate; therefore, twenty total organisms will be exposed to the 100% effluent and control (dilution) water.

After a 24-hour exposure period, the test chambers will be observed for mortality and abnormal effects. If the combined replicate mortality is ≧20%, the 48-hour LC_{50} test will be initiated.

5.6 48-Hr LC_{50} Test. The definitive test will be conducted by exposing the test organisms to at least 5 effluent concentrations and a dilution water control. The test will be conducted using duplicate beakers per concentration containing 10 test organisms per beaker (=20 per concentration). The test concentrations used will follow one of the logarithmic series presented in Table 1. The most common concentration series used is 10-100% (10, 18, 32, 56 and 100%). Several effluent toxicity tests conducted by ABC have shown that most industrial effluents are toxic within this range.

The test organisms will be placed in the test chambers by stratified random assignment within 30 minutes after solution preparations. The test chambers will be observed for mortality and/or abnormal effects every 24 hours, with a record maintained of mortality and abnormal behavior for each concentration tested.

5.7 Test Procedure - Chemical and Physical. Upon receipt of the test effluent, the following parameters will be measured of the 100% effluent: temperature, dissolved oxygen, pH, conductivity and hardness. These same parameters, except hardness, will be measured at 0 and 48 hours of definitive testing in one replicate of the control, low concentration, middle concentration and high concentration solutions.

5.8 Analysis of Results. The results of the definitive study will be statistically analyzed for 24 and 48 hour LC_{50} values and their corresponding 95 percent confidence limits. These values will be determined by an LC_{50} computer program developed by Stephan et al. (11) or by manual methods described by Litchfield-Wilcoxon (12) and Stephan (13).

5.9 Report. One copy of the report will be submitted. The report will contain all original raw data. Additional copies will be provided, if requested by the sponsor, at a copy rate of $50 per copy. The report will include the following:

5.9.1 Study dates, name and address of test facility.

5.9.2 Objectives and test methods.

5.9.3 Reference to the statistical methods used for data analysis.

5.9.4 Description of test effluent (date of receipt, storage conditions, physical characteristics, and method of preparing test concentrations).

5.9.5 Description of test design.

5.9.6 Summary of the data analysis, mortality observations and test water quality.

5.9.7 Location of raw data.

5.9.8 List and signatures of study personnel.

5.9.9 GLP compliance statement by study director and a statement by ABC's Quality Assurance Unit.

5.9.10 The report appendix will contain the original raw data for mortality observations and water quality, letter of test authorization, letters of authorized protocol changes and a copy of the approved protocol.

5.10 Data Retention. All original raw data generated in the preliminary and definitive studies will be provided to the study sponsor in the appendix to the final report. A copy of the data will be retained in ABC's archives.

6.0 PROTOCOL CHANGES

In the event that modifications of this protocol are deemed necessary, a written statement of any changes and reason(s) proposed by the study sponsor or ABC will be submitted to the other party. All agreed changes will be expressed in writing, signed and dated by the sponsor's study director. The signed changes will be appended to the protocol and included with the final report. This protocol is subject to revision when felt necessary to comply with changes in federal regulations and state of the art methodology. The protocol for a specific study will not be changed while the study is in progress without discussion with the study sponsor.

7.0 SPONSOR AUTHORIZATIONS DURING THE STUDY

Should a problem develop while the study is in progress, ABC will notify the study director within 24 hours. The problem and suggested test modifications will be discussed by telephone. ABC will proceed with the changes felt necessary upon the verbal authorization of the study director. A letter for written authorization will then be submitted by ABC to the study director and handled in the same manner discussed in section 6.0.

8.0 TEST-SPECIFIC INFORMATION

8.1.1 General. The following items will be addressed for each static bioassay. This information is necessary to be in compliance with Good Laboratory Practice Regulations (8). Sections 8.2, 8.3, 8.7 and 8.9 are to be completed by the study sponsor. Sections 8.4, 8.5 and 8.6 will be completed by ABC.

8.1.2 GLP Compliance. To be in compliance with the Federal Good Laboratory Practice regulations, the report of the investigation conducted utilizing this protocol must contain a statement that the study was conducted in accordance with one of the following GLP's.

1. U.S. F.D.A. Good Laboratory Practice Regulations (21 CFR 58).

2. U.S. E.P.A. Good Laboratory Practice Standards; Toxic Substances Control (40 CFR 792).

3. U.S. E.P.A. Good Laboratory Practice Standards; Pesticide Programs (40 CFR 160).

4. OECD Principles of Good Laboratory Practice; Annex 2 C(81) 30(Final).

5. Any state or local GLP which may apply.

The specific GLP regulation, for which this study specific protocol and resultant study must be in compliance with, is to be designated by the study sponsor in section 8.8 of this protocol.

8.2 Study Sponsor:

8.2.1 Company Molycorp, Inc.

8.2.2 Address 3 miles East of Questa on State Road 58
P.O. Box 469, Questa, New Mexico 87556

8.2.3 Sponsor's Study Director

Mr. John Stribling — Environmental Eng.
Name — Title

8.3 Effluent:

8.3.1 Sample Number West Diversion Channel

8.3.2 NPDES Permit Number NM - 0022306

8.3.3 Physical Description clear liquid

8.3.4 Handling Precautions Normal

8.4 Test Concentrations: To be determined after 24-hour screen of 100% effluent and consultation with study sponsor.

8.4.1 Sponsor's study director notified of screen results:
Date 12-12-85 ABC Biologist David Bunger
100% effluent screen = ____ ≧20% mortality ✓ <20% mortality

8.4.2 Definitive test concentrations agreed upon:
NO DEFINITIVE TEST

8.5 Test Organisms:

8.5.1 Species Daphnia magna

8.5.2 Supplier ABC in-house culture

8.6 Study Dates:

8.6.1 Proposed starting date of definitive study 12-11-85

8.6.2 Proposed completion date of definitive study 12-31-85

8.7 ABC Study Personnel:

8.7.1 Study Director

Alan D. Forbis — Aquatic Biologist III
Name — Title

8.7.2 Aquatic Supervisor

Carl M. Thompson	Aquatic Toxicology Supervisor
Name	Title

8.8 GLP Compliance (check the most appropriate):

EPA-FIFRA____; EPA-TSCA____; FDA____; OECD____; Other NPDES-EPA

8.9 Protocol Approvals. The following is to be signed by the appropriate study personnel:

8.9.1 Sponsor's Study Director

[signature]	Environmental Engineer	12-13-85 ①
Name	Title	Date

8.9.2 ABC's Study Director

[signature]	Aquatic Biologist III	12-10-85
Name	Title	Date

8.9.3 ABC's Group Leader

[signature]	Group Leader, Animal Toxicology	12-10-85
Name	Title	Date

8.10 Authorization to return the test material and sample:

8.10.1 Sponsor's authorized agent to receive the test material and sample.

[signature]	Environmental Eng.
Name	Title

8.10.2 Shipping Address.

Polycorp Inc.
Company

3 miles East of Questa Hwy. 38
Street Address

Questa	N.M.	87556
City	State	Zip Code

505-586-0212
Phone Number

8.10.3 Hazardous shipment information.

Proper Shipping Name

Class of Restricted Article (e.g. flammable, poison, radioactive, etc.)

① Due to the time restrictions for effluent testing following receipt of sample (within 24 hours), this protocol could not be processed for sponsor approval prior to the test

0028 of 0032
STUDY# 034194

TABLE 1: Guide to selection of experimental concentrations based on progressive bisection of intervals on logarithmic scale.

Col. 1	Col. 2	Col. 3	Col. 4	Col. 5
10.0	10.0	10.0	10.0	10.0
				8.7
			7.5	7.5
				6.5
		5.6	5.6	5.6
				4.9
			4.2	4.2
				3.7
3.2	3.2	3.2	3.2	3.2
				2.8
			2.4	2.4
				2.1
		1.8	1.8	1.8
				1.55
			1.35	1.35
				1.15
1.0	1.0	1.0	1.0	1.0

TABLE 2: Chemical characteristics of well water used by ABC's Aquatic Toxicology Division.

Parameters	Concentration
Temperature	15-20°C[b]
Dissolved Oxygen[a]	9.2-10.1 ppm[b]
pH	7.8-8.3[b]
Hardness ($CaCO_3$)	225-275 ppm[b]
Alkalinity ($CaCO_3$)	325-375 ppm[b]
Conductivity	700 μmhos/cm
NH_3-N	0.60 ppm
NO_3-NO_2-N	0.20 ppm
Ortho-Phosphate	0.10 ppm
Aluminum	<0.010 ppm
Arsenic	<0.0005 ppm
Cadmium	<0.002 ppm
Chromium	<0.006 ppm
Cobalt	<0.003 ppm
Copper	<0.002 ppm
Iron	<0.004 ppm
Lead	0.017 ppm
Mercury	0.0008 ppm
Nickel	<0.013 ppm
Silver	<0.0004 ppm
Zinc	0.001 ppm
Measured organophosphorus pesticides	c
Measured organochlorine pesticides plus PCB's	c

[a]After aeration.

[b]Represents seasonal variation, with the monthly range not exceeding 10%.

[c]Below minimum detectable limits.

9.0 REFERENCES

(1) U.S. Congress. 1977. Clean Water Act. Public Law 95-217. Federal Register, Dec. 27, 1977, 91 STAT 1566-1611.

(2) U.S. Environmental Protection Agency. 1983. Effluent Toxicity Testing and Biomonitoring Workshop. Feb. 22-23, 1983, Dallas, Texas.

(3) Peltier, William. 1978. Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms. U.S. Environmental Protection Agency, EPA-660/4-78-012, revised July, 1978. 52 p.

(4) Committee on Methods for Toxicity Tests with Aquatic Organisms. 1975. Methods for Acute Toxicity Tests with Fish, Macroinvertebrates and Amphibians. Environmental Protection Agency, Ecological Research Series EPA-660/3-75-009, April, 1975. 61 p.

(5) American Society for Testing and Materials. 1980. Standard practice for conducting basic acute toxicity tests with fish, macroinvertebrates and amphibians. May, 1980, ASTM Committee E-35.23. 25 p.

(6) American Society for Testing and Materials. 1983. Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates and Amphibians. November, 1983, ASTM Committee E-47.01. 37 p.

(7) American Public Health Association. 1980. Standard Methods for the Examination of Water and Wastewater. 15th ed. Washington, D.C. 1134 p.

(8) (a) U.S. Food and Drug Administration. 1978. Nonclinical Laboratory Studies, Good Laboratory Practice Regulations (21 CFR, Part 58). Federal Register, Vol. 43, No. 247: 59986-60025.

(b) U.S. Environmental Protection Agency. 1983. Pesticide Programs; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 160). Federal Register, Vol 48, No. 230: 53946-53969.

(c) U.S. Environmental Protection Agency. 1983. Toxic Substances Control; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 792). Federal Register, Vol. 48; No. 230:53922-53944.

(d) Organization for Economic Cooperation and Development. 1981. OECD Guidelines for Testing of Chemicals, Principles of Good Laboratory Practice Annex 2, C(81) 30(Final):7-28.

(9) Pennak, R. W. 1978. Fresh-Water Invertebrates of the United States, 2nd Ed. 365 p.

(10) U.S. Environmental Protection Agency. 1979. Static culture methods for sheepshead minnows (Cyprinodon variegatus) and mysid shrimp (Mysidopsis bahia). Unpublished manuscript, U.S. EPA, Athens, GA.

(11) Stephan, C. E., K. A. Busch, R. Smith, J. Burke and R. W. Andrew. 1978. A computer program for calculating an LC_{50}. U. S. Environmental Protection Agency, Duluth, Minnesota, pre-publication manuscript, August, 1978.

(12) Litchfield, J. T., Jr. and F. Wilcoxon. 1949. A Simplified Method of Evaluating Dose-Effect Experiments. Jour. Pharm. Exp. Ther. 96:99-113.

(13) Stephan, C. 1977. Methods for calculating an LC_{50}, p. 65-84. In F. L. Mayer and J. L. Hamelick (eds.). Aquatic Toxicology and Hazard Evaluation. ASTM Special Technical Publication 634. ASTM. Philadelphia.

Assessment of Acute Toxicity of West Diversion Channel

April 1986

ANALYTICAL BIO-CHEMISTRY LABORATORIES, INC.
P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

Static Acute Toxicity Report
#34534

Assessment of the Acute Toxicity of West Diversion Channel to *Daphnia magna*

Submitted To:
Molycorp, Incorporated
Attn: Mr. Fred Martinez
3 miles East of Questa, on State Road 38
P. O. Box 469
Questa, New Mexico 87556

April 21, 1986

ANALYTICAL BIO-CHEMISTRY LABORATORIES, INC.
P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

AMENDMENT TO STUDY PROTOCOL

STUDY TITLE: Assessment of the Acute Toxicity of West Diversion Channel to *Daphnia magna*.

PROTOCOL NUMBER: 8301 AMENDMENT NUMBER: 1

LABORATORY: ABC Laboratories, Inc. ABC STUDY NUMBER: 34534

EFFECTIVE DATE: 4/15/86

Section 5.5 Screen Test

One of the duplicate control chambers was lost during the 24-hour screen test. Since no mortality or abnormal effects were observed in the remaining control chamber, the loss of this replicate did not affect the results of the screen test.

SPONSOR'S STUDY DIRECTOR: Fred [illegible] Date: 04/30/86

STUDY DIRECTOR: Allen R. [illegible] Date: 4/22/86

SUMMARY

The acute toxicity of West Diversion Channel to *Daphnia magna* was assessed for U.S. EPA Region 6 NPDES permit requirements using the methods outlined by the Committee on Methods for Toxicity Tests with Aquatic Organisms. Water quality parameters of temperature, dissolved oxygen, conductivity, hardness, alkalinity and pH were measured on receipt of the sample and were within acceptable limits.

The results of the 24-hour static *Daphnia magna* toxicity screen indicated that the mortality was <20% for West Diversion Channel. In fact, no mortality was observed after the 24-hour exposure period. Therefore, a definitive 48-hour static acute study was not required. All reported values were based upon nominal concentrations.

The no-effect level for West Diversion Channel was estimated to be <100% effluent after 24-hours, since surfacing was observed in one of the 100% effluent replicates.

Submitted By: Analytical Bio-Chemistry Laboratories, Inc.
7200 East ABC Lane
P. O. Box 1097
Columbia, Missouri 65205
(314) 474-8579

Prepared By:

Alan D. Forbis — 4/21/86
Alan D. Forbis — Date
Aquatic Biologist III/Study Director

Scott Frazier — 4/21/86
Scott Frazier — Date
Biologist I

Approved By:

Carl M. Thompson — 4-22-86
Carl M. Thompson — Date
Supervisor, Aquatic Toxicology

Kathy Landholt — 04/22/86
Kathy Landholt — Date
Quality Assurance Officer

Michael D. Williams — 4/22/86
Michael Williams, Ph.D. — Date
Group Leader, Animal Toxicology

/jh

INTRODUCTION

This static bioassay was performed by the Aquatic Toxicology Division of Analytical Bio-Chemistry Laboratories, Inc., Columbia, Missouri, for Molycorp, Inc., from March 26, 1986 to March 27, 1986. The purpose of this test was to assess toxicity of a 100% solution of West Diversion Channel to Daphnia magna. The assessment was designed to meet the NPDES permit process in U.S. EPA Region 6 (1) requiring biomonitoring of industrial discharges. The screen test was conducted following the procedures outlined in ABC protocol #8301, as approved by Mr. Fred Martinez; Molycorp, Inc. on April 2, 1986.

METHODS AND MATERIALS

The procedures applied for the 24-hour toxicity screen were basically those described in Methods of Measuring the Acute Toxicity of Effluents to Aquatic Organisms (2), Methods for Acute Toxicity Tests with Fish, Macroinvertebrates, and Amphibians (3), Standard Practice for Conducting Acute Toxicity Tests with Fishes, Macroinvertebrates and Amphibians (4), Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates and Amphibians (5), and Standard Methods for the Examination of Water and Wastewater (6). The test lot of Daphnia magna were obtained from an in-house culture at ABC. The taxonomic key presented by Pennak (7) was used to identify to species the test daphnids. All test daphnids were held in a controlled temperature area at 20 ±2°C. The lighting was 50-70 footcandles on a 16-hour daylight photoperiod. During the holding period, they received a suspension of algae (Selenastrum capricornutum) supplemented with a blended fish food suspension at least every 3 days. First-instar daphnids (<24-hours old) were selected for testing and were not fed during the test.

The static Daphnia bioassay was conducted in 250-ml glass beakers containing 200 ml of ABC aged well water with the chemical characteristics listed in Table 1. These vessels were kept at 20 (±2.0) °C in a temperature controlled area. The lighting was maintained at 50-70 footcandles on a 16-hour daylight photoperiod.

A 24-hour screen test, composed of duplicate 100% effluent was performed to estimate the toxicity of the effluent. Also included was a duplicate ABC well water control. A five-six concentration static acute toxicity study would be performed if ≧20% mortality was observed after 24-hours in the screen test.

The West Diversion Channel sample (NPDES Permit Number NM-0022306) was received on March 26, 1986 as a clear liquid and was received at 8°C. The samples were allowed to warm to test temperature before Daphnids were added to test solutions.

RESULTS

After 24-hours, the results indicated that the mortality of *Daphnia magna* to West Diversion Channel was <20%. The no-effect concentration was estimated to be <100% effluent since surfacing was observed in one of the 100% effluent test replicates. Since mortality was not ≧20% after 24-hours in the screen test, a definitive study was not required per biomonitoring guidelines established for U.S. EPA Region 6.

Table 2 presents the water quality parameters measured on sample receipt. Hardness, alkalinity (as $CaCO_3$) and conductivity for the West Diversion Channel sample were 730 and 46 mg/l and 1000 μMHOS, respectively. Dissolved oxygen and pH for this sample were 9.3 mg/l and 7.3, respectively, and were considered adequate for testing (3).

The study was conducted following the intent of the Good Laboratory Practice Regulations (8) and the final report was reviewed by Analytical Bio-Chemistry Laboratories' Quality Assurance Unit. All original raw data was provided to Molycorp, Inc. with a copy retained at Analytical Bio-Chemistry Laboratories.

TABLE 1: Chemical Characteristics of Well Water Used by ABC's Aquatic Toxicology Division.

Parameters	Concentration
Temperature	15-20°C[b]
Dissolved Oxygen[a]	9.2-10.1 ppm[b]
pH	7.8-8.3[b]
Hardness ($CaCO_3$)	225-275 ppm[b]
Alkalinity ($CaCO_3$)	325-375 ppm[b]
Conductivity	700 µmhos/cm
NO_3-N	0.74 ppm
NO_3-NO_2-N	0.74 ppm
PO_4-P	<0.10 ppm
Aluminum	<20 ppb
Arsenic	<0.2 ppb
Cadmium	<2 ppb
Chromium	<3 ppb
Cobalt	<4 ppb
Copper	<3 ppb
Iron	12 ppb
Lead	<5 ppb
Mercury	<0.5 ppb
Nickel	<15 ppb
Silver	<5 ppb
Zinc	11 ppb
Measured organophosphorus pesticides	c
Measured organochlorine pesticides plus PCB's	c

[a]After aeration.

[b]Represents seasonal variation, with the monthly range not exceeding 10%.

[c]Less than minimum detectable limits for organophosphorus and organochlorine which were <0.10 and <0.50 ppb, respectively.

Sample dates: Trace elements = April 2, 1985, Organophosphate and organochlorine analyses = April 2, 1985.

TABLE 2
Water Quality Measurements of 100%
West Diversion Channel Upon Sample Receipt

	100% West Diversion Channel
Temperature[a]	8*
Dissolved Oxygen[b]	9.3
pH[c]	7.3
Conductivity[d]	1000
Hardness[e]	730
Alkalinity[e]	46

[a]Temperature °C - Monitored with a mercury thermometer.

bDissolved oxygen concentrations (mg/l) - Dissolved Oxygen System (YSI Model 54). Saturation at 8°C is 11.9 mg/l.

[c]pH - pH Probe (Corning Model 476182) used with a Corning Model 125 pH and mV meter.

[d]Conductivity (μMHOS) - Conductivity Meter (YSI Model 33).

[e]Hardness and alkalinity (mg/l as $CaCO_3$) - HACH Model AL-DT and HAC-DT.

*Test solutions allowed to warm to test temperature (19°C) before addition of daphnids. Dissolved oxygen saturation at 19°C is 9.3 mg/l.

LITERATURE CITED

(1) U. S. Environmental Protection Agency. 1983. Effluent Toxicity Testing and biomonitoring Workshop. Feb. 22-23, 1983, Dallas, Texas.

(2) Peltier, William. 1978. Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms. U.S. Environmental Protection Agency, EPA-660/4-78-012, revised July 1978. 52 p.

(3) Committee on Methods for Toxicity Tests with Aquatic Organisms. 1975. Methods of Acute Toxicity Tests with Fish, Macroinvertebrates and Amphibians. Environmental Protection Agency, Ecological Research Series EPA-660/3-75-009, April 1975.

(4) American Society for Testing and Materials. 1980. Standard Practice for Conducting Basic Acute Toxicity Tests with Fish, Macroinvertebrates and Amphibians. May 1980, ASTM Committee E-35.23. 25 p.

(5) American Society for Testing and Materials. 1983. Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates and Amphibians. November 1983, ASTM Committee E-47.01 37 p.

(6) American Public Health Association. 1980. Standard Methods for the Examination of Water and Wastewater. 15th ed. Washington, DC. 1134 p.

(7) Pennak, R. W. 1978. Freshwater Invertebrates of the United States. John Wiley & Sons, New York, 2nd ed. 803 p.

(8) (a) U.S. Food and Drug Administration. 1978. Nonclinical Laboratory Studies, Good Laboratory Practice Regulations (21 CFR, Part 58). Federal Register, Vol. 43, No. 247:59986-60025.

(b) U.S. Environmental Protection Agency. 1983. Pesticide Programs; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 160). Federal Register, Vol. 48, No. 230:53946-53969.

(c) U.S. Environmental Protection Agency. 1983. Toxic Substances Control; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 792). Federal Register, Vol. 48; No. 230:53922-53944.

(d) Organization for Economic Cooperation and Development. 1981. OECD Guidelines for Testing of Chemicals, Principles of Good Laboratory Practice Annex 2, C(81) 30(Final):7-28.

QUALITY ASSURANCE STATEMENT

Quality Assurance Statement for final report #34534 entitled, "Acute Toxicity of West Diversion Channel to *Daphnia magna*," for Mr. Fred Martinez; Molycorp, Inc., Questa, New Mexico.

In accordance with ABC Laboratories' intent that all studies conducted at our facilities are designed and function in conformance with good laboratory practice regulations and the protocols for individual laboratory studies, an inspection of the final report for West Diversion Channel was conducted and found to be in acceptable form by a member of our Quality Assurance Unit. A final inspection of all data and records on April 15, 1986, indicates that the report submitted to you is an accurate reflection of the study as it was conducted by ABC Laboratories.

Should you have any questions relating to the information provided in this statement or the function of our Quality Assurance Unit, please contact the QA Unit at your convenience.

Kathy Landholt 04/22/86

Kathy Landholt Date

Quality Assurance Officer

STUDY COMPLIANCE STATEMENT

Study Compliance Statement for ABC report #34534 entitled, "Acute Toxicity of West Diversion Channel to *Daphnia magna*," for Mr. Fred Martinez; Molycorp, Inc., Questa, New Mexico.

In accordance with ABC Laboratories' intent that all aquatic toxicity tests conducted by our facility follow good laboratory practices, ABC's study director for the above test herein confirms that the study was conducted in compliance with the U.S. E.P.A. Good Laboratory Practice Standards; Toxic Substances Control (40 CFR 792).

All original raw data was sent to Molycorp, Inc., with a copy retained at Analytical Bio-Chemistry Laboratories.

Alan D. Forbis 4/22/86

Alan D. Forbis — Date

ABC Study Director

APPENDIX - RAW DATA

NOTE: Some of the records which appear in this raw data appendix have been provided as photocopies of original records on file at ABC. This has been done by necessity for certain data which are used commonly in several studies at ABC. Such records include reference toxicity test data, standard solution preparations, organism culture logs, and well water chemical screen results.

[illegible] handed over to carrier by Molycorp employee:

DATE: 3/25/86

TIME: 11:45 AM

CARRIER: Fred Martinez

EMPLOYER: Moly Corp Inc UNION 76

MOLYCORP PERSONNEL: Fred Martinez

Sample(s) delivered to ABC Lab for analysis. Please sign and date this letter upon receipt of sample(s).

Sample received by: ABC Laboratories Inc.
7200 ABC Lane
P.O. Box 1097
Columbia, MO 65205
(314) 474-8579

for the following analyses: Toxicity testing

Received by: Scott Frazier

Date: 3/26/86 10:30 AM

Please return this letter with the analytical results, signed and dated below.

Scott Frazier 4/15/86
Date

EFFLUENT TOXICITY TEST
MORTALITY AND BEHAVIORAL OBSERVATIONS

Effluent Designation: WEST - DIVERSION CHANNEL
Industry: MOLY CORP
Study No.: 34534
ABC Study Director: A. FORBIS

Lab Form No.: 91

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P.O. Box 1097
Columbia, Missouri 65205

☐ Prelim. ☑ Definitive Test Species: Daphnia magna (Lot #86-B-2)
Date Initiated: 3/26/86 3:00 pm Test Terminated: 3/27/86
Dilution Water: NONE No./Vessel: 10 Vessel Size: 250 ml

Effluent Conc. (%)		24 hours Dead	24 hours Obs.	48 hours Dead	48 hours Obs.	72 hours Dead	72 hours Obs.	96 hours Dead	96 hours Obs.
Control	A	0	N						
	B		*						
100	A	0	1-SUR 9-N						
	B	0	N						
Observer		SF 2:30 pm							
Date		3/27/86							

Remarks: N = Normal; LOE = Loss of Equilibrium;
SUR = Surfacing
* CONTROL B REPLICATE LOST, SF, 3/27/86

Prepared By: [signature] Date: 3/26/86
Checked By: [signature] Date: 4/15/86

WATER QUALITY FOR EFFLUENT SAMPLES

Test Material: WEST DIVERSION CHANNEL

Study No.: 34534

Protocol No.: 8301

ABC Study Director: A. FORBIS

Lab Form No.: 28b

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane; P.O. Box 1097
Columbia, Missouri 65205

Concentration mg/1	DAY				DAY				DAY				DAY			
	Temp °C	D.O.[a] mg/1	pH[b]	Cond. µMhos	Temp °C	D.O. mg/1	pH	Cond. µMhos	Temp °C	D.O. mg/1	pH	Cond. µMhos	Temp °C	D.O. mg/1	pH	

[a]Dissolved oxygen concentrations - Dissolved Oxygen Probe (YSI Dissolved Oxygen System Model 54).

[b]pH-pH Probe (Corning Model 476182) used with Corning Model 125 pH and mV meter.

[c]	Temp °C	D.O. mg/1	pH	Cond. µMhos	Hard mg/1	Alk. mg/1	NH_3 mg/1			Date	Int.
100% Effluent on Receipt	8	9.3	7.3	1000	730	46	/			3/26/86	SF

Prepared By: Scott Frazier Date: 3/26/86

Checked By: R. Forbis Date: 4/15/86

Remarks:

STUDY# 034534

PREPARATION OF EFFLUENT CONCENTRATIONS (volume/volume)

Effluent Designation: WEST DIVERSION CHANNEL

Industry: MOLY CORP

Study No.: 34534

ABC Study Director: A. FORBIS

Lab Form No.: 94

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane; P.O. Box 1097
Columbia, MO 65205

☐ PRELIMINARY ☑ DEFINITIVE

Dilution Water Type:

☐ ABC Well Water ☐ ABC Recon. Water

☐ Effluent Receiving Water _____ ☑ Other NONE

Sample No.	Aliquot Volume (ml)	Dilution Volume (ml)	Final Conc. (%)
1	—	200	ABC CONTROL*
2	200	—	100
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			

Remarks: * ABC CONTROL = ABC WELL WATER

Prepared By: [signature] Date: 3/26/86

Checked By: [signature] Date: 3/26/86

Study Director: [signature] Date: 4/15/86

INVERTEBRATE CULTURE RECORD

Species: Daphnia magna

Lot No.: 86-B-2

Date Initiated: 2/26/86

Lab Form No: 52

Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P. O. Box 1097
Columbia, MO 65205

Date	No. Adult Alive	Instar Present	Fed & Changed	Study I.D.	Comments	Obs. By:
2/26/86	—	~30	✓		350 ml algae 1 ml AP-100 suspension	SF
3/~~27~~ 2/86	31	0	✓		" "	PC ~~SF~~ ①
3/4	30	~80	✓		" "	SF
3/6	30	~80	✓		" "	SF
3/9	reduced to 25	~500	✓		" "	AF
3/11	24	~300	✓		" "	LS
3/13	24	~100	✓		" "	LO
~~3/14~~ ②				~~[illegible]~~ ②		
3/16	24	~300	✓		" "	WAM
3/18	24	~180	✓		" "	LS
3/19				34343 - 200		SF
3/20	24	~300	✓		~1 ml AP-100 suspension ~350 ml Algae	SF
3/23	24	~200	✓		" "	LS
3/25	24	~80	✓		" "	SF
3/26				34[illegible] - 30 34[illegible] - 30 34440 - 160		SF
3/27	24	~100	✓		~350 ml Algae ~1 ml AP-100 suspension	SF
3-30	23	~250	✓		" "	JHB
4/1	23	~200	✓		" "	LS

Prepared by: Scott Frazier Date: 2/26/86

Checked by: James Sweet Date: 4-3-86

① Entry Error SF 2/27/86
② Entry Error LS 3/14/86

"This is an exact copy of The original document"
By H. Seaton date 4/3/86

This sheet represents a chain of custody for effluent samples collected at out Fall 002 and West Diversion Channel located in Taos County, Questa, New Mexico on 3/25/86

Sample bottles were prepared by Molycorp employees in accordance with guidance set forth by the U.S. Environmental Protection Agency for the existing program permit. Sampling procedures were also followed in accordance with permit requirements.

Sample(s) identification: 002 3/25/86
West Diversion Channel 3/25/86

Sample(s) collected at: 10:50 AM last portion *
11:30 AM " " *

* composite sample as required.

By: Fred Martinez
Environmental Water Analyst

DEFINITIVE CONCENTRATION NOTIFICATION

Firm: Molycorp INC
Address: P.O. Box 469
City/State/Zip: Questa, NM 87556
Phone: (505) 586-0212 Ext 206

Lab Form No.: 186
Analytical Bio-Chemistry Labs
Aquatic Toxicology Division
7200 ABC Lane, P.O. Box 1097
Columbia, MO 65202

Sponsor's Study Director: FRED MARTINEZ
Person notified: same (✓) Other:

Date of Notification: 3/28/86

Notification of preliminary testing results with the following definitive test levels agreed upon:

Test Material	ABC #	Test Species	Test Conc. (mg/l)
WEST DIVERSION CHANNEL	34534	*Daphnia magna*	N/A

Informed Mr. Fred Martinez that no mortality was observed in the 24 screen test of West Diversion Channel Effluent with *Daphnia magna*.

Notified By: Scott Frazier Date: 3/28/86

ANALYTICAL BIO-CHEMISTRY LABORATORIES, INC.
P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

Sample Date: April 2, 1985

ANALYSIS RESULTS FOR:

Aquatic Toxicology Division
Analytical Bio-Chemistry Laboratories, Inc.
7200 East ABC Lane
P. O. Box 1097
Columbia, Missouri 65205

Water Analysis Results

ABC Lab No.	33102
Sample I.D.	ABC Aquatic Lab Well Water
NO_3-N	0.74 mg/l
NO_3-NO_2-N	0.74 mg/l
PO_4-P	<0.10 mg/l
Al	<20 µg/l
Co	<4 µg/l
Fe	12 µg/l
As	<0.2 µg/l
Cd	<2 µg/l
Cr	<3 µg/l
Cu	<3 µg/l
Pb	<5 µg/l
Hg	<0.5 µg/l
Ni	<15 µg/l
Ag	<5 µg/l
Zn	11 µg/l

<Indicates less than, if present at all.

Del A. Koch
Chemist III

Please note that the original data for the analysis reported in this table are in ABC's archives.

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P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

Sample Date: April 2, 1985

ANALYSIS RESULTS FOR:

Aquatic Toxicology Division
Analytical Bio-Chemistry Laboratories, Inc.
7200 East ABC Lane
P. O. Box 1097
Columbia, Missouri 65205

Analysis of Aquatic Lab Well Water for Organophosphates

ABC Lab No.	Customer I.D.	Compound	ppb
33103	Aquatic Lab Well Water	Diazinon	<0.10
		Methyl Parathion	<0.10
		Malathion	<0.10
		Ethyl Parathion	<0.10

Randy Ayling 6/25/85
Randy Ayling
Chemist I

Please note that the original data for the analysis reported in this table are in ABC's archives.

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P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

Sample Date: April 2, 1985

ANALYSIS RESULTS FOR:

Aquatic Toxicology Division
Analytical Bio-Chemistry Laboratories, Inc.
7200 East ABC Lane
P. O. Box 1097
Columbia, Missouri 65205

Analysis of Aquatic Lab Well Water for Nonionic
Chlorinated Hydrocarbon Residue

ABC Lab No.	33103
Customer I.D.	ABC Aquatic Lab Well Water
Compound	ppb
αBHC	<0.01
Lindane	<0.01
Heptachlor	<0.01
Heptachlor Epoxide	<0.02
γChlordane	<0.02
αChlordane	<0.02
Aldrin	<0.01
Dieldrin	<0.03
p,p'-DDE	<0.03
o,p'-DDT	<0.05
p,p'-DDD	<0.05
p,p'-DDT	<0.05
Endrin	<0.05
Methoxychlor	<0.20
Toxaphene	<0.50
Aroclor 1242	<0.40
Aroclor 1016	<0.40
Aroclor 1254	<0.40
Aroclor 1260	<0.40
HCB	<0.01
Mirex	<0.10

<indicates less than, if present at all.

Randy Ayling 6/25/85
Randy Ayling
Chemist I

Please note that the original data for the analysis reported in this table are in ABC's archives.

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P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

AMENDMENT TO STUDY PROTOCOL

STUDY TITLE: Assessment of the Acute Toxicity of West Diversion Channel to *Daphnia magna*.

PROTOCOL NUMBER: 8301 AMENDMENT NUMBER: 1

LABORATORY: ABC Laboratories, Inc. ABC STUDY NUMBER: 34534

EFFECTIVE DATE: 4/15/86

Section 5.5 Screen Test

One of the duplicate control chambers was lost during the 24-hour screen test. Since no mortality or abnormal effects were observed in the remaining control chamber, the loss of this replicate did not affect the results of the screen test.

SPONSOR'S STUDY DIRECTOR: _____ Date: _____

STUDY DIRECTOR: Alan D. Fish Date: 4/22/86

ANALYTICAL BIO-CHEMISTRY LABORATORIES, INC.
P.O. Box 1097 • Columbia, MO 65205
Shipping Address: 7200 East ABC Lane, Columbia, MO 65202
(314) 474-8579 • Telex 821 814 • Answer Back (ABCLAB UD)

ABC PROTOCOL NO. 8301

(August 21, 1984)

Effluent Static Bioassay with Daphnia magna or
Mysid Shrimp (Mysidopsis bahia) to
Meet EPA Region 6 NPDES Biomonitoring Guidelines

RECEIVED
APR 10 '86
A. B. C. LABS

ABC Study Number 34534

Test Material West Diversion Channel

Test Species Daphnia magna

1.0 INTRODUCTION

The use of bioassays in the identification and control of industrial effluents is rapidly increasing. Indeed, aquatic toxicity tests are presently being incorporated into the NPDES guidelines of the Federal Clean Water Act (1). To assist industry in meeting requirements set forth by legislation and regulatory agencies, Analytical Bio-Chemistry Laboratories, Inc. offers a complete range of aquatic bioassay tests of industrial effluents.

As part of the NPDES permit process in U.S. EPA Region 6, a biomonitoring requirement has been imposed for certain permit holders in that area. Discharges subject to the new regulations in Region 6 (Texas, New Mexico, Oklahoma, Arkansas and Louisiana) will be required to submit aquatic toxicity data on their effluents to EPA on a periodic basis.

This ABC protocol discusses the biomonitoring guidelines in Region 6 and describes ABC's technical approach to assist industry in complying with these regulations.

2.0 OBJECTIVES

The primary objective of the toxicity test described herein is to evaluate the acute toxicity of an effluent to freshwater or marine invertebrates under static conditions. The species selected for this testing include *Daphnia magna* (freshwater effluent, <5 ppt salinity) and *Mysidopsis bahia* (saltwater effluent, ≧5 ppt salinity). The effluent will first be tested on a 100% effluent screen basis. If the screen results in ≧20% mortality of test organisms, a 48-hour, five concentration LC_{50} test will be conducted. An LC_{50} is the approximate concentration (percent) of test effluent that produces 50 percent mortality of test organisms after prescribed intervals (24 and 48 hours).

3.0 TESTING FACILITY

The study will be conducted by the Aquatic Toxicology Division of Analytical Bio-Chemistry Laboratories, Inc., 7200 East ABC Lane, P. O. Box 1097, Columbia, Missouri 65205 (314-474-8579).

4.0 REGION 6 BIOMONITORING REQUIREMENT

The following biomonitoring requirement is now being included in the NPDES permit process for many major industrial discharges in EPA Region 6. The permit language given here was that presented by the U.S. EPA at a regional workshop on the matter (2):

> The permittee shall determine if the effluent from this outfall is lethal to twenty (20) percent or greater of the culture of test organisms by use of the "Range-Finding Screening Test," set out in "Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms," EPA-600/4-78-012 (Rev. July, 1978) (3).

Organisms for this test shall be Daphnia sp. if the effluent is less than five (5) parts per thousand salinity or Mysid sp. if the effluent is greater than five (5) parts per thousand salinity. This screening test will be conducted within sixty (60) days of effectiveness of the biomonitoring requirements. Tests will be conducted once each quarter for a duration of two years utilizing a static method for 24 hours and following this dilution scheme only:

Effluent sample* - 100 percent by volume
Dilution water - 0 percent by volume

*24-hour composite; refrigerated after collection

If at any time during the two year testing period a test shows a lethality of twenty (20) percent or more of the test organisms, the permittee shall within twenty-four (24) hours conduct a replacement static 48-hour median lethal concentration (LC_{50}) test on the originally collected sample. Replacement of effluent samples shall be once per 24 hours. Organisms for this test shall be Daphnia sp. if the effluent is less than five (5) parts per thousand salinity and reconstituted fresh water (EPA-600/4-78-012 Section 4) shall be used for dilution. If the effluent contains greater than five (5) parts per thousands salinity, Mysid sp. shall be used as the test organism, and reconstituted seawater will be used as dilution water (EPA-600/4-78-012 Section 4). The remaining LC_{50} methodology is available in EPA-600/4-78-012.

All screening and LC_{50} test results shall be reported with the Discharge Monitoring Reports.

It should be noted by the permittee that slight changes from the above permit language (e.g., pH modification of effluent) may be incorporated into his specific permit. Attention should be given to the permit requirements for each effluent before testing is initiated. ABC requires that a copy of the permit's biomonitoring clause be forwarded to ABC prior to testing so that consideration can be given to pH modification, etc.

5.0 METHODS AND MATERIALS

5.1 Methodology. The methods applied for both the preliminary and definitive phases of this test are basically those described in Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms (3), Methods for Acute Toxicity Tests with Fish, Macroinvertebrates, and Amphibians (4), Standard Practice for Conducting Acute Toxicity Tests with Fishes, Macroinvertebrates, and Amphibians (5), Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates

and Amphibians (6), and Standard Methods for the Examination of Water and Wastewater (7). This protocol has been written to comply with the federal Good Laboratory Practice Regulations (8).

5.2 Test Organisms. The species recommended by the EPA for this testing are Daphnia sp. and Mysid sp. Daphnia sp. are to be used when the effluent salinity is <5 parts per thousand (ppt) and Mysid sp. are to be used when the effluent salinity is ≧5 ppt. The species maintained by ABC, and acceptable by EPA, for this testing are Daphnia magna and Mysidopsis bahia. The test lot of Daphnia magna will be obtained from an in-house culture at ABC. The taxonomic key presented by Pennak (9) will be used to identify to species the test daphnids. All test daphnids will be held in a controlled temperature area at 20 ±2°C. The lighting will be 50-70 footcandles on a 16 hour daylight photoperiod. During the holding period, they will receive a suspension of algae (Selenastrum capricornutum) supplemented with a blended fish food/cerophyl suspension at least every 3 days. First-instar daphnids (<24 hours old) will be selected for testing and will not be fed during the test.

Mysid shrimp (Mysidopsis bahia) will be obtained from a commercial supplier (e.g., Sea Plantations, Inc. or Gulf Breeze Specimens) and maintained in culture at ABC. Mysids will be cultured following the techniques described by the U.S. EPA (10). Test mysids will be selected from a sub-culture of immature individuals.

5.3 Effluent Sample. The sample submitted to ABC for testing must be a 24-hour composite sample, consisting of equal volumes taken every hour (2). The sample should be collected in a glass or polyethylene container, refrigerated after collection, and shipped to arrive at ABC within 24 hours after collection. A one-gallon sub-sample of the 24-hour composite will be satisfactory for the toxicity test(s) required. ABC can provide sample containers and mailing cartons and can assist plant personnel in shipping arrangements so that the sample arrives at ABC in a timely manner.

5.4 Dilution Water. It will not be necessary to collect actual receiving water for effluent dilutions for this test. The biomonitoring regulations recommend the use of reconstituted freshwater or saltwater listed in the EPA effluent methods manual (3). The freshwater dilution water (for Daphnia sp.) will be a hard reconstituted water of 160-180 mg/l hardness (as $CaCO_3$). The reconstituted saltwater (for Mysid sp.) will be prepared from a commercial salt mix similar in composition to that listed in the EPA effluent manual (3) and adjusted to a test salinity of 15-25 ppt.

5.5 Screen Test. The biomonitoring regulations require that a 24-hour 100% effluent screen be conducted before more definitive (LC_{50}) testing is considered. As stated in section 4.0, if ≧20% of the test organisms die in the 100% solution, the permittee shall then conduct a 48-hour static replacement LC_{50} test. This latter test is discussed in section 5.6.

The screen test will be conducted in 250 ml glass beakers containing 200 ml of solution. These test chambers will be held in a controlled temperature area at 20 ±2°C on a 16-hour daylight vs. 8-hour darkness photoperiod with 30-minute dawn/dusk transition periods. The screen test will include duplicate test chambers at 100% vs. duplicate control chambers containing dilution water only. Ten test organisms will be placed in each replicate; therefore, twenty total organisms will be exposed to the 100% effluent and control (dilution) water.

After a 24-hour exposure period, the test chambers will be observed for mortality and abnormal effects. If the combined replicate mortality is ≧20%, the 48-hour LC_{50} test will be initiated.

5.6 48-Hr LC_{50} Test. The definitive test will be conducted by exposing the test organisms to at least 5 effluent concentrations and a dilution water control. The test will be conducted using duplicate beakers per concentration containing 10 test organisms per beaker (=20 per concentration). The test concentrations used will follow one of the logarithmic series presented in Table 1. The most common concentration series used is 10-100% (10, 18, 32, 56 and 100%). Several effluent toxicity tests conducted by ABC have shown that most industrial effluents are toxic within this range.

The test organisms will be placed in the test chambers by stratified random assignment within 30 minutes after solution preparations. The test chambers will be observed for mortality and/or abnormal effects every 24 hours, with a record maintained of mortality and abnormal behavior for each concentration tested.

5.7 Test Procedure - Chemical and Physical. Upon receipt of the test effluent, the following parameters will be measured of the 100% effluent: temperature, dissolved oxygen, pH, conductivity and hardness. These same parameters, except hardness, will be measured at 0 and 48 hours of definitive testing in one replicate of the control, low concentration, middle concentration and high concentration solutions.

5.8 Analysis of Results. The results of the definitive study will be statistically analyzed for 24 and 48 hour LC_{50} values and their corresponding 95 percent confidence limits. These values will be determined by an LC_{50} computer program developed by Stephan et al. (11) or by manual methods described by Litchfield-Wilcoxon (12) and Stephan (13).

5.9 Report. One copy of the report will be submitted. The report will contain all original raw data. Additional copies will be provided, if requested by the sponsor, at a copy rate of $50 per copy. The report will include the following:

5.9.1 Study dates, name and address of test facility.

5.9.2 Objectives and test methods.

5.9.3 Reference to the statistical methods used for data analysis.

5.9.4 Description of test effluent (date of receipt, storage conditions, physical characteristics, and method of preparing test concentrations).

5.9.5 Description of test design.

5.9.6 Summary of the data analysis, mortality observations and test water quality.

5.9.7 Location of raw data.

5.9.8 List and signatures of study personnel.

5.9.9 GLP compliance statement by study director and a statement by ABC's Quality Assurance Unit.

5.9.10 The report appendix will contain the original raw data for mortality observations and water quality, letter of test authorization, letters of authorized protocol changes and a copy of the approved protocol.

5.10 Data Retention. All original raw data generated in the preliminary and definitive studies will be provided to the study sponsor in the appendix to the final report. A copy of the data will be retained in ABC's archives.

6.0 PROTOCOL CHANGES

In the event that modifications of this protocol are deemed necessary, a written statement of any changes and reason(s) proposed by the study sponsor or ABC will be submitted to the other party. All agreed changes will be expressed in writing, signed and dated by the sponsor's study director. The signed changes will be appended to the protocol and included with the final report. This protocol is subject to revision when felt necessary to comply with changes in federal regulations and state of the art methodology. The protocol for a specific study will not be changed while the study is in progress without discussion with the study sponsor.

7.0 SPONSOR AUTHORIZATIONS DURING THE STUDY

Should a problem develop while the study is in progress, ABC will notify the study director within 24 hours. The problem and suggested test modifications will be discussed by telephone. ABC will proceed with the changes felt necessary upon the verbal authorization of the study director. A letter for written authorization will then be submitted by ABC to the study director and handled in the same manner discussed in section 6.0.

8.0 TEST-SPECIFIC INFORMATION

8.1.1 General. The following items will be addressed for each static bioassay. This information is necessary to be in compliance with Good Laboratory Practice Regulations (8). Sections 8.2, 8.3, 8.7 and 8.9 are to be completed by the study sponsor. Sections 8.4, 8.5 and 8.6 will be completed by ABC.

8.1.2 GLP Compliance. To be in compliance with the Federal Good Laboratory Practice regulations, the report of the investigation conducted utilizing this protocol must contain a statement that the study was conducted in accordance with one of the following GLP's.

1. U.S. F.D.A. Good Laboratory Practice Regulations (21 CFR 58).

2. U.S. E.P.A. Good Laboratory Practice Standards; Toxic Substances Control (40 CFR 792).

3. U.S. E.P.A. Good Laboratory Practice Standards; Pesticide Programs (40 CFR 160).

4. OECD Principles of Good Laboratory Practice; Annex 2 C(81) 30(Final).

5. Any state or local GLP which may apply.

The specific GLP regulation, for which this study specific protocol and resultant study must be in compliance with, is to be designated by the study sponsor in section 8.8 of this protocol.

8.2 Study Sponsor:

8.2.1 Company Molycorp, Inc.

8.2.2 Address 3 miles East of Questa on State Rd. 58
P. O. Box 469, Questa, New Mexico 87556

8.2.3 Sponsor's Study Director

Mr. Fred Martinez
Name

Title

8.3 Effluent:

8.3.1 Sample Number Water Diversion Channel

8.3.2 NPDES Permit Number NM-0022306

8.3.3 Physical Description clear liquid

8.3.4 Handling Precautions Normal

8.4 Test Concentrations: To be determined after 24-hour screen of 100% effluent and consultation with study sponsor.

8.4.1 Sponsor's study director notified of screen results:

Date 3/28/86 ABC Biologist Scott [illegible]

100% effluent screen = _____ ≧20% mortality ✓ <20% mortality

8.4.2 Definitive test concentrations agreed upon:

8.5 Test Organisms:

8.5.1 Species Daphnia magna

8.5.2 Supplier ABC in-house culture

8.6 Study Dates:

8.6.1 Proposed starting date of definitive study 3/26/86

8.6.2 Proposed completion date of definitive study 4/15/86

8.7 ABC Study Personnel:

8.7.1 Study Director

Alan D. Forbis
Name

Aquatic Biologist III
Title

8.7.2 Aquatic Supervisor

Carl M. Thompson — Aquatic Toxicology Supervisor
Name — Title

8.8 GLP Compliance (check the most appropriate):

EPA-FIFRA____; EPA-TSCA____; FDA____; OECD____; Other NPPES-EPA____.

8.9 Protocol Approvals. The following is to be signed by the appropriate study personnel:

8.9.1 Sponsor's Study Director ①

Fred Martinez — Environmental Analyst — 4/2/86
Name — Title — Date

8.9.2 ABC's Study Director

[signature] — Aquatic Biologist III — 3-27-86
Name — Title — Date

8.9.3 ABC's Group Leader

[signature] — Group Leader, Animal Toxicology — 3-27-86
Name — Title — Date

8.10 Authorization to return the test material and sample:

8.10.1 Sponsor's authorized agent to receive the test material and sample.

Mr. Fred Martinez — ____
Name — Title

8.10.2 Shipping Address.

Molycorp, Inc.
Company

3 miles East of Questa Highway 38
Street Address

Questa, — New Mexico — 87556
City — State — Zip Code

505-586-0212
Phone Number

8.10.3 Hazardous shipment information.

____
Proper Shipping Name

____
Class of Restricted Article (e.g. flammable, poison, radioactive, etc.)

① Due to the time restrictions for effluent testing following receipt of sample (within 24 hours), this protocol could not be processed for sponsor

TABLE 1: Guide to selection of experimental concentrations based on progressive bisection of intervals on logarithmic scale.

Col. 1	Col. 2	Col. 3	Col. 4	Col. 5
10.0	10.0	10.0	10.0	10.0
				8.7
			7.5	7.5
				6.5
		5.6	5.6	5.6
				4.9
			4.2	4.2
				3.7
3.2	3.2	3.2	3.2	3.2
				2.8
			2.4	2.4
				2.1
		1.8	1.8	1.8
				1.55
			1.35	1.35
				1.15
1.0	1.0	1.0	1.0	1.0

TABLE 2: Chemical characteristics of well water used by ABC's Aquatic Toxicology Division.

Parameters	Concentration
Temperature	15-20°C[b]
Dissolved Oxygen[a]	9.2-10.1 ppm[b]
pH	7.8-8.3[b]
Hardness ($CaCO_3$)	225-275 ppm[b]
Alkalinity ($CaCO_3$)	325-375 ppm[b]
Conductivity	700 µmhos/cm
NH_3-N	0.60 ppm
NO_3-NO_2-N	0.20 ppm
Ortho-Phosphate	0.10 ppm
Aluminum	<0.010 ppm
Arsenic	<0.0005 ppm
Cadmium	<0.002 ppm
Chromium	<0.006 ppm
Cobalt	<0.003 ppm
Copper	<0.002 ppm
Iron	<0.004 ppm
Lead	0.017 ppm
Mercury	0.0008 ppm
Nickel	<0.013 ppm
Silver	<0.0004 ppm
Zinc	0.001 ppm
Measured organophosphorus pesticides	c
Measured organochlorine pesticides plus PCB's	c

[a]After aeration.

[b]Represents seasonal variation, with the monthly range not exceeding 10%.

[c]Below minimum detectable limits.

9.0 REFERENCES

(1) U.S. Congress. 1977. Clean Water Act. Public Law 95-217. Federal Register, Dec. 27, 1977, 91 STAT 1566-1611.

(2) U.S. Environmental Protection Agency. 1983. Effluent Toxicity Testing and Biomonitoring Workshop. Feb. 22-23, 1983, Dallas, Texas.

(3) Peltier, William. 1978. Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms. U.S. Environmental Protection Agency, EPA-660/4-78-012, revised July, 1978. 52 p.

(4) Committee on Methods for Toxicity Tests with Aquatic Organisms. 1975. Methods for Acute Toxicity Tests with Fish, Macroinvertebrates and Amphibians. Environmental Protection Agency, Ecological Research Series EPA-660/3-75-009, April, 1975. 61 p.

(5) American Society for Testing and Materials. 1980. Standard practice for conducting basic acute toxicity tests with fish, macroinvertebrates and amphibians. May, 1980, ASTM Committee E-35.23. 25 p.

(6) American Society for Testing and Materials. 1983. Proposed Standard Practice for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates and Amphibians. November, 1983, ASTM Committee E-47.01. 37 p.

(7) American Public Health Association. 1980. Standard Methods for the Examination of Water and Wastewater. 15th ed. Washington, D.C. 1134 p.

(8) (a) U.S. Food and Drug Administration. 1978. Nonclinical Laboratory Studies, Good Laboratory Practice Regulations (21 CFR, Part 58). Federal Register, Vol. 43, No. 247: 59986-60025.

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