SDMS US EPA REGION V -1

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QUALITY ASSURANCE PROJECT PLAN
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
RIVERDALE CHEMICAL COMPANY
CHICAGO HEIGHTS, ILLINOIS

prepared by
IT CORPORATION
October 1, 1985

Approved _____ Date 10/1/85
IT Project Manager

Approved Michael A. Champion Date 10/3/85
Riverdale Chemical Company

Approved James H. Adams, Jr. Date 10/3/85
EPA Region V
Quality Assurance Office

Approved _____ Date 10/3/85
EPA Region V
Remedial Project Manager

IT CORPORATION

Transmittal
QAPP, H & S and Sampling Plans
Riverdale Chemical Company
Chicago Heights, Illinois

RESPONSIVE TO THE NEEDS OF ENVIRONMENTAL MANAGEMENT

October 1, 1985

Project No. 850037

Michael J. Champion, Ph.D.
Vice President
Riverdale Chemical Company
220 East 17th Street
Chicago Heights, IL 60411

Transmittal
QAPP, H & S and Sampling Plans
Riverdale Chemical Company
Chicago Heights, Illinois

Dear Dr. Champion:

Enclosed are five copies of the revised above referenced reports for the Remedial Investigation/Feasibility Study (RI/FS) at the Riverdale Chemical Company plant. The revisions to the report were made in accordance the requests of David Payne of U.S. EPA Region V. Mr. Payne's requests were discussed our telephone conference with Mr. Payne this afternoon. The changes to the plans include:

- Performance Evaluation samples from EMSL-LV will be provided by U.S. EPA Region V. The samples will consist of one blank sample and one spiked sample. Sections 7.1, 8.0, and 9.2.4 of the QAPP and Section 4.0 of the Sampling Plan have been revised to incorporate this change.

- Tentative identifications will be provided for the first 10 volatile and first 20 semivolatile compounds greater than 10 percent of the nearest internal standard. A statement to this effect has been included in Section 7.1 of the QAPP.

- A schedule of milestone dates has been added to Section 9.0 of the Sampling Plan. The schedule defines the number of days after approval of the QAPP that field work will commence (25), analytical results will be submitted to U.S. EPA (70) and the Phase II Scope, if necessary, will be submitted to U.S. EPA (80).

We trust this submittal satisfies your needs at this time. If you have any questions, please contact me at 414-359-2222.

Respectfully submitted,

Joseph D. Adams, Jr. P.E.
Project Manager

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1.0 INTRODUCTION

The purpose of this Quality Assurance Project Plan (QAPP) is to document the procedures that will be undertaken to provide the precision, accuracy, and completeness of the data gathered during the investigation of the Riverdale Chemical Company's (Riverdale) facility in Chicago Heights, Illinois by IT Corporation (IT).

This QAPP has been prepared to document the measures that will be undertaken by IT and its subcontractors so the work performed will be of proper quality to accomplish project objectives and will be responsive to requirements of the U.S. Environmental Protection Agency (U.S. EPA). The plan addresses:

- The QA (quality assurance) objectives of the project;
- Specific QA and QC (quality control) procedures that will be implemented to achieve these objectives; and
- Staff organization and responsibility.

The requirements of the U.S. EPA with regard to quality assurance focus on the acquisition of environmental data of known and acceptable quality. Other aspects of the project, such as engineering analysis and report preparation, will be controlled by the internal requirements of IT Corporation's Quality Assurance Program.

The program is documented in the IT Quality Assurance Manuals for Analytical and Engineering Services. The policies and procedures specified by the manuals serve to define acceptable practices to be employed by personnel engaged in any particular project.

IT Quality Assurance Manuals for Analytical and Engineering Services are composed of controlled documents which are considered proprietary information. IT has included with this submittal to U.S. EPA each document for use on this project. IT requests at the end of this project that these documents be returned.

1.1 PROJECT DESCRIPTION

The Riverdale Plant is located immediately south of East 17th Street in Chicago Heights, Illinois (see Figures 1 and 2). The plant is involved primarily in the formulation of pesticides. There are three large buildings on the plant grounds. Only Building 1 is used in the formulation process (Figure 3). An above ground tank storage area is located in the southwest corner of the site.

One previous sampling and testing study has taken place at the Riverdale site. Ecology and the Environment (E&E) conducted in April 1984 a Field Investigation Team (FIT) study of the plant area as part of the National Dioxin Test Strategy and in response to concerns that contamination may be present on the site grounds with the potential for off-site migration. The FIT investigation consisted of taking 15 surface soil samples (numbered B-1 through B-15 in Figure 3). The samples were analyzed for a variety of organic compounds with the analytical results included in Table 1. The results of the analyses indicated that potentially hazardous materials were present in the surface soils at the facility, including various pesticides, volatile organic compounds, polynuclear aromatic hydrocarbons (PAHs), phenolic compounds (phenolics) and 2,3,7,8-tetrachlorodibenzo-p-dioxin (Dioxin). The Dioxin concentrations were greatest in the soils at the far south end of the plant site.

1.2 PROJECT OBJECTIVES

The overall objective of the Remedial Investigation (RI) recommended for the Riverdale site is to collect the data needed to assess site hazards and evaluate alternatives in the Feasibility Study (FS).

The RI/FS will be conducted using a phased approach. During the Phase I RI, the site geology will be characterized and the vertical and horizontal extent of surface and sub-surface soil contamination will be determined. If the results of Phase I indicate that the contamination has migrated from the surface into the subsurface soils, the scope of the Phase II RI may have to be expanded to include further testing of the samples collected during Phase I, collection and analyses of additional soil samples, drilling of additional soil borings, and/or installation and subsequent sampling of monitoring wells.

TABLE 1
ORGANIC ANALYSES OF SOILS AT RIVERDALE CHEMICAL COMPANY
CONDUCTED BY ECOLOGY AND ENVIRONMENT, APRIL 1984
RIVERDALE CHEMICAL COMPANY
CHICAGO, HEIGHTS, ILLINOIS
RESULTS ARE IN PARTS PER BILLION - PPB

Parameter	B-1	B-2	B-3	B-4	B-5	B-6	B-7	B-8	B-9	B-10	Duplicate B-10	B-11	B-12	B-13	B-14	Background B-15
2,3,7,8 TCDD	ND	3.6	1.3	1.1	31.6	5.3	364	12.9	3.6	13.9	14.2*	38.3	0.76	1.68	36.4	ND
2,4,6-trichlorophenol	LT						4,500		LT	2,600		1,100				
2,4-dichlorophenol		LT			LT		22,000									
phenol		LT														
2,4,5-trichlorophenol							9,500									
1,2,4-trichlorobenzene										870						
hexachlorobenzene									LT							
fluoranthene	LT	630	840	1,800			120	2,200	550	1,400		410				
isophorone										LT						
naphthalene							1,300	LT	1,400	750					6,400	
bis(2-ethylhexyl)phthalate		LT	903											1,100		
di-n-octyl phthalate													5,100			
benzo(a)anthracene	LT	560	LT	1,000			2,700	1,200	420	460						
benzo(a)pyrene		LT		LT				890		LT						
benzo(b)fluoranthene		870	1,800	LT			810	3,700	LT	1,000						
benzo(k)fluoranthene		LT	1,800	LT				3,700	LT	1,000						
chrysene	LT	700	LT	1,300			650	1,100	470	800		LT				
acenaphthylene							LT		470							
anthracene								430								
benzo(ghi)perylene									850	LT						
phenanthrene	LT	460	LT	1,700			400	1,700	LT	970		LT				
indeno(1,2,3-c,d)pyrene									LT	LT						
pyrene	500	1,600	703	2,200	LT		460	2,100	550	860		LT	LT			
4-chloroaniline					36,000											
2-methylnaphthalene					LT		4,400		6,400	1,900					19,000	
1,1,2,2-tetrachloroethane		LT				LT										
chloroform				LT		14.0										
ethylbenzene						LT									25.0	
methylene chloride	22	40			120			51.0		10.0		26.0				
fluorotrichloromethane	LT	5.5		2.9	4.1	5.3	2.4	LT	3.7	3.3				LT		LT
tetrachloroethane					4.3											
toluene															4.6	
4-methyl-2-pentanone						LT										
styrene		LT														
xylene		LT													94.0	
aldrin		5,400	3,800	25,000	320	2,700	71,000	250,000	11,000	14,000		2,100	300	300	35,000	
dieldrin		31,000	78,000	7,900	210	1,500	72,000	79,000	12,000	19,000		8,300	860	530	12,000	45
chlordane	3,200		310,000	5,000		1,900		1,100,000	29,000	48,000			2,300	1,200		78
4,4'-DDT				610		450			1,400				210	580		
4,4'-DDE			9,300	190			2,200	17,000	800			4,700				
4,4'-DDD									990				57.0	360		
heptachlor		190,000	34,000	460		16,000	440	130,000	3,100			1,400	220	610		
heptachlor epoxide																
toxaphene	160,000															

* - Only 2,3,7,8 TCDD analyses were duplicated.
LT - Detected but below detection limits.

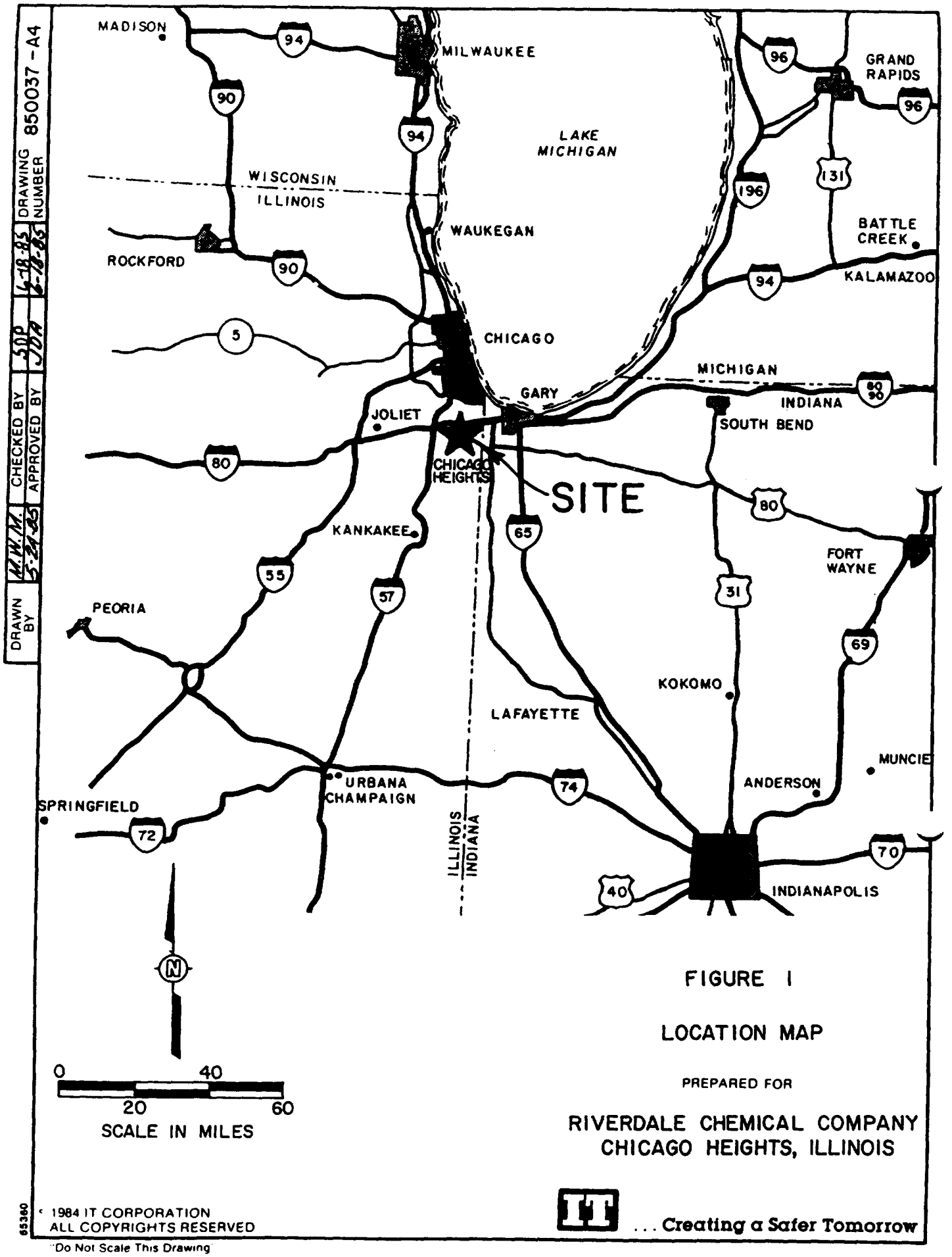

FIGURE 1

LOCATION MAP

PREPARED FOR

RIVERDALE CHEMICAL COMPANY
CHICAGO HEIGHTS, ILLINOIS

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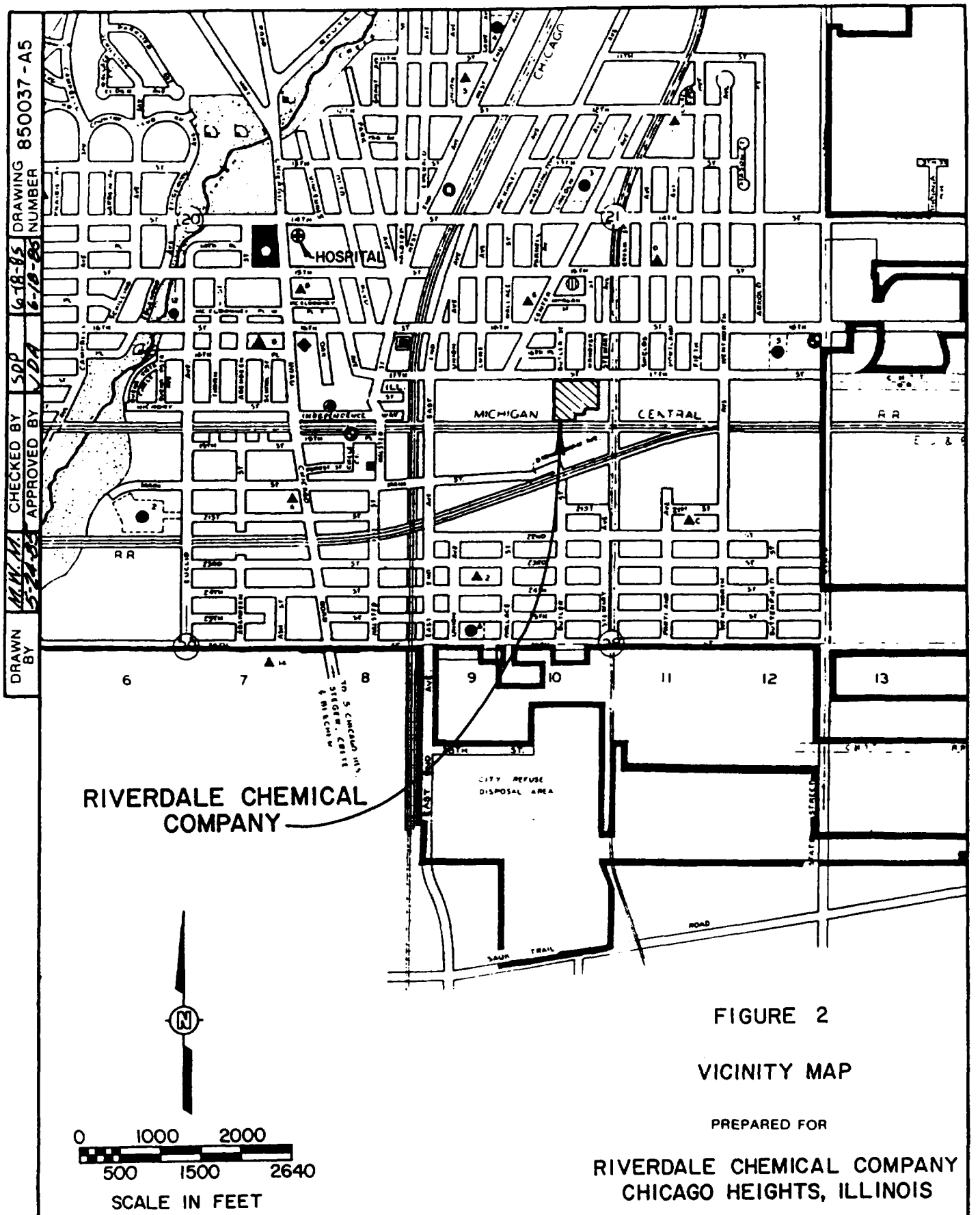

FIGURE 2

VICINITY MAP

PREPARED FOR

RIVERDALE CHEMICAL COMPANY
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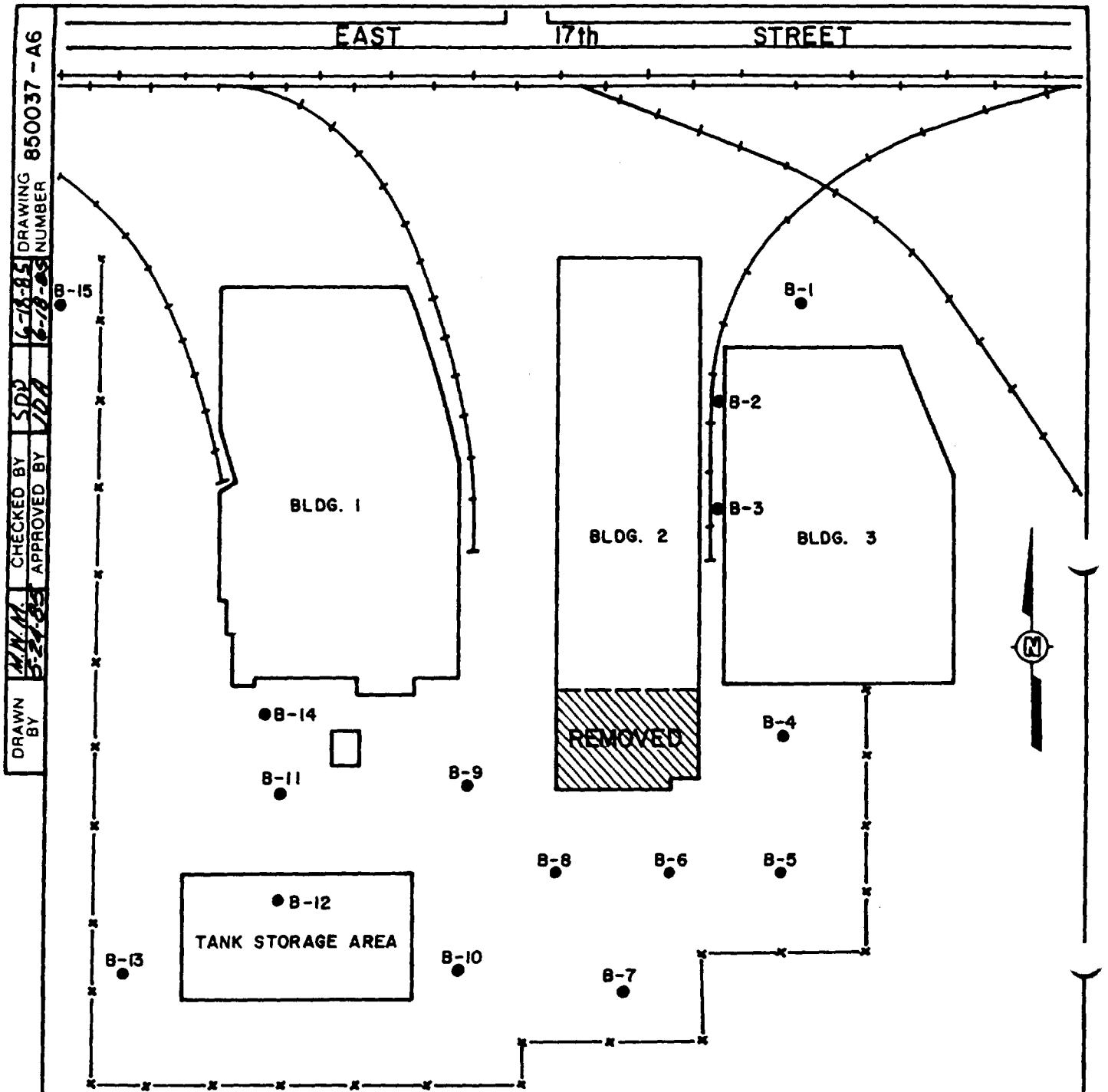

FIGURE 3

SITE MAP
ECOLOGY & ENVIRONMENT
SAMPLING LOCATIONS

PREPARED FOR

RIVERDALE CHEMICAL COMPANY
CHICAGO HEIGHTS, ILLINOIS

LEGEND

B-11● SAMPLE LOCATIONS

RAILROAD

FENCE LINE

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The scope of this QAPP includes only the Phase I RI. The Phase I RI at the Riverdale site will consist of the following:

- Nine soil borings will be drilled to a depth of 10-15 feet or as necessary to define the near surface geology. Sixteen samples from the borings will be selected for analysis of Dioxin. Four composite samples will be selected and tested for Hazardous Substances List (HSL) organics and HSL pesticides as listed in Table 2. The boring locations are shown in Figure 4.

- Approximately 20 surface soil samples will be collected from both on-site and off-site locations (if access is permitted). Twelve samples collected south of Buildings 1 and 3 will be tested for Dioxin. The other eight samples will be composited into two samples for Dioxin analysis. The 20 samples will be composited into six samples for analyses of HSL organics and HSL pesticides as listed in Table 2.

- All samples will be screened with an Organic Vapor Analyzer (OVA) or a HNu which will aid in selecting the samples to be tested.

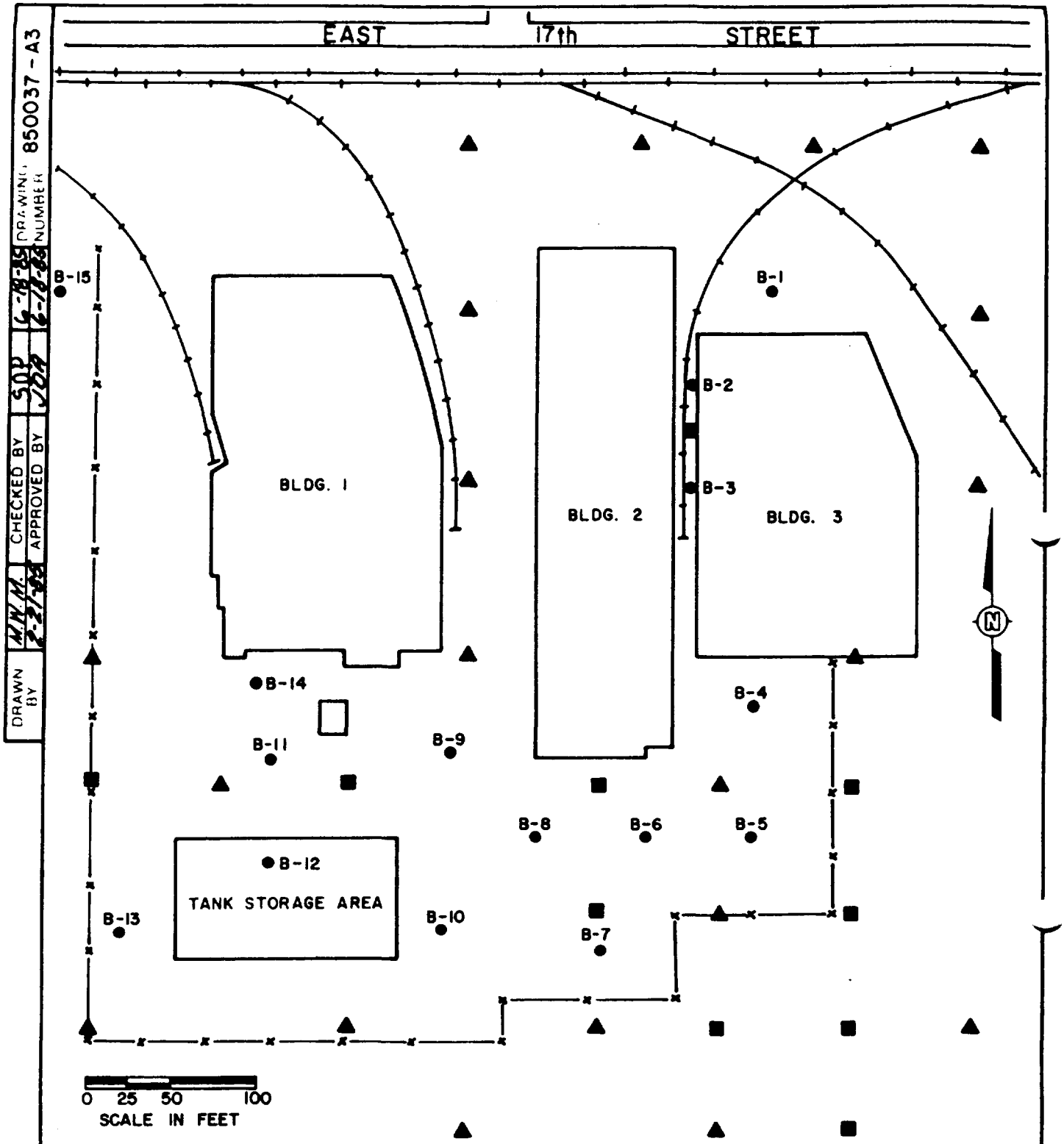
TABLE 2
HAZARDOUS SUBSTANCE LIST (HSL) AND
CONTRACT REQUIRED DETECTION LIMITS (CRDL)[a]
RIVERDALE CHEMICAL COMPANY
CHICAGO HEIGHTS, ILLINOIS

COMPOUNDS	CAS NUMBER	DETECTION LIMITS[b] LOW WATER (ug/L)	DETECTION LIMITS[b] LOW SOIL/SEDIMENT (ug/Kg)
VOLATILES		(c)	(d)
1. Chloromethane	74-87-3	10	10
2. Bromoethane	74-83-9	10	10
3. Vinyl Chloride	75-01-4	10	10
4. Chloroethane	75-00-3	10	10
5. Methylene Chloride	75-09-2	5	5
6. Acetone	67-64-1	10	10
7. Carbon Disulfide	75-15-0	5	5
8. 1,1-Dichloroethene	75-35-4	5	5
9. 1,1-Dichloroethane	75-35-3	5	5
10. trans-1,2-Dichloroethene	156-60-5	5	5
11. Chloroform	67-66-3	5	5
12. 1,2-Dichloroethane	107-06-2	5	5
13. 2-Butanone	78-93-3	10	10
14. 1,1,1-Trichloroethane	71-55-6	5	5
15. Carbon Tetrachloride	56-23-5	5	5
16. Vinyl Acetate	108-05-4	10	10
17. Bromodichloromethane	75-27-4	5	5
18. 1,1,2,2-Tetrachloroethane	79-34-5	5	5
19. 1,2-Dichloropropane	78-87-5	5	5
20. trans-1,3-Dichloropropene	10061-02-6	5	5
21. Trichloroethene	79-01-6	5	5
22. Dibromochloromethane	124-48-1	5	5
23. 1,1,2-Trichloroethane	79-00-5	5	5
24. Benzene	71-43-2	5	5
25. cis-1,3-Dichloropropene	10061-01-5	5	5
26. 2-Chloroethyl Vinyl Ether	110-75-8	10	10
27. Bromoform	75-25-2	5	5
28. 2-Hexanone	591-78-6	10	10
29. 4-Methyl-2-pentanone	108-10-1	10	10
30. Tetrachloroethene	127-18-4	5	5
31. Toluene	108-88-3	5	5
32. Chlorobenzene	108-90-7	5	5
33. Ethyl Benzene	100-41-4	5	5
34. Styrene	100-42-5	5	5
35. Total Xylenes		5	5
SEMI-VOLATILES		(e)	(f)
36. N-Nitrosodimethylamine	62-75-9	10	330
37. Phenol	108-95-2	10	330
38. Aniline	62-53-3	10	330
39. bis(2-Chloroethyl) ether	111-44-4	10	330
40. 2-Chlorophenol	95-57-8	10	330
41. 1,3-Dichlorobenzene	541-73-1	10	330
42. 1,4-Dichlorobenzene	106-46-7	10	330
43. Benzyl Alcohol	100-51-6	10	330
44. 1,2-Dichlorobenzene	95-50-1	10	330
45. 2-Methylphenol	95-48-7	10	330
46. bis(2-Chloroisopropyl) ether	39638-32-9	10	330
47. 4-Methylphenol	106-44-5	10	330
48. N-Nitroso-Dipropylamine	621-64-7	10	330
49. Hexachloroethane	67-72-1	10	330
50. Nitrobenzene	98-95-3	10	330
51. Isophorone	78-59-1	10	330
52. 2-Nitrophenol	88-75-5	10	330
53. 2,4-Dimethylphenol	105-67-9	10	330
54. Benzoic Acid	65-85-0	50	1600
55. bis(2-Chloroethoxy) methane	111-91-1	10	330
56. 2,4-Dichlorophenol	120-83-2	10	330
57. 1,2,4-Trichlorobenzene	120-82-1	10	330
58. Naphthalene	91-20-3	10	330
59. 4-Chloroaniline	106-47-8	10	330
60. Hexachlorobutadiene	87-68-3	10	330
61. 4-Chloro-3-methylphenol (para-chloro-meta-cresol)	59-50-7	10	330
62. 2-Methylnaphthalene	91-57-6	10	330
63. Hexachlorocyclopentadiene	77-47-4	10	330
64. 2,4,6-Trichlorophenol	88-06-2	10	330
65. 2,4,5-Trichlorophenol	95-95-4	50	1600
66. 2-Chloronaphthalene	91-58-7	10	330
67. 2-Nitroaniline	88-74-4	50	1600
68. Dimethyl Phthalate	131-11-3	10	330
69. Acenaphthylene	208-96-8	10	330
70. 3-Nitroaniline	99-09-2	50	1600
71. Acenaphthene	83-32-9	10	330
72. 2,4-Dinitrophenol	51-28-5	50	1600
73. 4-Nitrophenol	100-02-7	50	1600
74. Dibenzofuran	132-64-9	10	330
75. 2,4-Dinitrotoluene	121-14-2	10	330

TABLE 2
HAZARDOUS SUBSTANCE LIST (HSL) AND
CONTRACT REQUIRED DETECTION LIMITS (CRDL)[a]
RIVERDALE CHEMICAL COMPANY
CHICAGO HEIGHTS, ILLINOIS
(Continued)

COMPOUNDS	CAS NUMBER	DETECTION LIMITS[b] LOW WATER (ug/L)	DETECTION LIMITS[b] LOW SOIL/SEDIMENT (ug/Kg)
SEMI-VOLATILES		(e)	(f)
76. 2,6-Dinitrotoluene	606-20-2	10	330
77. Diethylphthalate	84-66-2	10	330
78. 4-Chlorophenyl Phenyl ether	7005-72-3	10	330
79. Fluorene	86-73-7	10	330
80. 4-Nitroaniline	100-01-6	50	1600
81. 4,6-Dinitro-2-methylphenol	534-52-1	50	1600
82. N-nitrosodiphenylamine	86-30-6	10	330
83. 4-Bromophenyl Phenyl ether	101-55-3	10	330
84. Hexachlorobenzene	118-74-1	10	330
85. Pentachlorophenol	87-86-5	50	1600
86. Phenanthrene	85-01-8	10	330
87. Anthracene	120-12-7	10	330
88. Di-n-butylphthalate	84-74-2	10	330
89. Fluoranthene	206-44-0	10	330
90. Benzidine	92-87-5	80	2600
91. Pyrene	129-00-0	10	330
92. Butyl Benzyl Phthalate	85-68-7	10	330
93. 3,3'-Dichlorobenzidine	91-94-1	20	660
94. Benzo(a)anthracene	56-55-3	10	330
95. bis(2-ethylhexyl)phthalate	117-81-7	10	330
96. Chrysene	218-01-9	10	330
97. Di-n-octyl Phthalate	117-84-0	10	330
98. Benzo(b)fluoranthene	205-99-2	10	330
99. Benzo(k) fluoranthene	207-08-9	10	330
100. Benzo(a)pyrene	50-32-8	10	330
101. Indeno(1,2,3-cd)pyrene	193-39-5	10	330
102. Dibenz(a,h)anthracene	53-70-3	10	330
103. Benzo(g,h,i)perylene	191-24-2	10	330
PESTICIDES		(g)	(h)
104. alpha-BHC	319-84-6	0.05	8.0
105. beta-BHC	319-85-7	0.05	8.0
106. delta-BHC	319-86-8	0.05	8.0
107. gamma-BHC (Lindane)	58-89-9	0.05	8.0
108. Heptachlor	76-44-8	0.05	8.0
109. Aldrin	309-00-2	0.05	8.0
110. Heptachlor Epoxide	1024-57-3	0.05	8.0
111. Endosulfan I	959-98-8	0.05	8.0
112. Dieldrin	60-57-1	0.10	16.0
113. 4,4'-DDE	72-55-9	0.10	16.0
114. Endrin	72-20-8	0.10	16.0
115. Endosulfan II	33213-65-9	0.10	16.0
116. 4,4'-DDD	72-54-8	0.10	16.0
117. Endrin Aldehyde	7421-93-4	0.10	16.0
118. Endosulfan Sulfate	1031-07-8	0.10	16.0
119. 4,4'-DDT	50-29-3	0.10	16.0
120. Endrin Ketone	53494-70-5	0.10	16.0
121. Methoxychlor	72-43-5	0.5	80.0
122. Chlrodane	57-74-9	0.5	80.0
123. Toxaphene	8001-35-2	1.0	160.0
124. AROCLOR-1016	12674-11-2	0.5	80.0
125. AROCLOR-1221	11104-28-2	0.5	80.0
126. AROCLOR-1232	11141-16-5	0.5	80.0
127. AROCLOR-1242	53469-21-9	0.5	80.0
128. AROCLOR-1248	12672-29-6	0.5	80.0
129. AROCLOR-1254	11097-69-1	1.0	160.0
130. AROCLOR-1260	11096-82-5	1.0	160.0
DIOXIN			
2,2,7,8-TCDD		0.01[i]	1.0

[a] Specific detection limits are highly matrix dependent. The detection limits listed herein are provided for guidance and may not always be achievable.
[b] Detection limits listed for soil/sediment are based on wet weight. The detection limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, as required by the contract, will be higher.
[c] Medium Water Contract Required Detection Limits (CRDL) for Volatile HSL Compounds are 100 times the individual Low Water CRDL.
[d] Medium Soil/Sediment Contract Required Detection Limits (CRDL) for Volatile HSL Compounds are 100 times the individual Low Soil/Sediment CRDL.
[e] Medium Water Contract Required Detection Limits (CRDL) for Semi-Volatile HSL Compounds are 100 times the individual Low Water CRDL.
[f] Medium Soil/Sediment Contract Required Detection Limits (CRDL) for Semi-Volatile HSL Compounds are 60 times the individual Low Soil/Sediment CRDL.
[g] Medium Water Contract Required Detection Limits (CRDL) for Pesticide HSL Compounds are 100 times the individual Low Water CRDL.
[h] Medium Soil/Sediment Contract Required Detection Limits (CRDL) for Pesticide HSL Compounds are 15 times the individual Low Soil/Sediment CRDL.
[i] Routine detection limit, 10 ppt, not addressed by the IFB.



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CHECKED BY SDP 6-18-85
APPROVED BY JDA 6-18-85
DRAWING NUMBER 850037-A3

LEGEND

- B-11 ● SAMPLE LOCATION BY OTHERS
- ■ PROPOSED BORING LOCATION
- ▲ PROPOSED SURFACE SAMPLING LOCATION
- RAILROAD
- FENCE LINE

FIGURE 4

SAMPLING LOCATIONS

PREPARED FOR

RIVERDALE CHEMICAL COMPANY

CHICAGO HEIGHTS, ILLINOIS

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Do Not Scale This Drawing

2.0 PROJECT ORGANIZATION AND RESPONSIBILITY

The principal IT personnel assigned to the investigation of the Riverdale facility are Joseph Adams (Project Manager), William Thayer (Project Executive), Lee Kettren (Quality Assurance Manager), Mark Hinchey (Project Hydrogeologist/Engineer), Jack Hall (Laboratory Director), and Gary Woody and Linda Pocratsky (QC Coordinators). Other personnel will be assigned as deemed necessary. Their responsibilities are described in the following sections.

2.1 PROJECT MANAGER

The Project Manager (PM) will be the prime point of contact with Riverdale and will have primary responsibility for technical, financial, and scheduling matters. His duties will include:

- Assignment of duties to the project staff and orientation of the staff to the needs and requirements of the project
- Supervision of the performance of project team members
- Budget and schedule control
- Review of subcontractor work and approval of subcontract invoices
- Review of interim reports on all task and subtask activities
- Establishment of a project record keeping system
- Review of all major project deliverables for technical accuracy and completeness
- Coordination of the QAPP activities including the Performance Audits and System Audits for Field and Laboratory as required by EPA Region V.
- Project closeout.

2.2 PROJECT EXECUTIVE

The Project Executive's responsibilities will include:

- Providing sufficient resources to the project team in order for it to respond fully to the requirements of the investigation
- Providing direction and guidance to the PM as appropriate
- Reviewing the quality of the data gathered during the course of the project and the final project report.

2.3 QUALITY ASSURANCE MANAGER

The Quality Assurance Manager (QAM) is in charge of audits and monitors adherence to the project QA objectives. The QAM reports directly to the PM.

the QAM is responsible for ensuring all project work undergoes adequate quality review. The QAM's responsibilities will include:

- Contacting the analytical laboratories receiving samples to determine if samples are properly prepared, packaged, and identified
- Conducting field audits of sampling episodes to provide that sample identification and chain-of-custody procedures are being followed
- Contacting the PM to provide that personnel assigned to field sampling episodes are properly trained in sample identification and chain-of-custody procedures
- Review work products.

2.4 PROJECT HYDROGEOLOGIST/ENGINEER

The Project Hydrogeologist/Engineer will be responsible for all field activities and data evaluation, which are as follows:

- Providing direction and supervision to the drilling contractor during the drilling of soil borings
- Maintaining a log for each borehole
- Supervising the collection of all soil samples and providing for their proper handling and shipping
- Monitoring all drilling and sampling operations to provide that the drilling contractor and sampling team members adhere to the QAPP

- Coordinating activities with the PM
- Processing and evaluating the results of the chemical analysis of the samples prior to preparation of the RI report.

2.5 SITE SAFETY OFFICER

The Site Safety Officer (SSO) will be responsible for providing that all team members adhere to the site safety requirements. Additional responsibilities are as follows:

- Updating equipment or procedures based upon new information gathered during the site inspection
- Modifying the levels of protection based upon site observations
- Enforcing the "buddy system"
- Determining and posting locations and routes to medical facilities, including poison control centers; and arranging for emergency transportation to medical facilities
- Notifying local public emergency officers, i.e., police and fire departments, of the nature of the team's operations and posting their telephone numbers
- Examining work party members for symptoms of exposure or stress
- Providing emergency medical care and first aid as necessary on-site. The SSO has the ultimate responsibility to stop any operation that threatens the health or safety of the team or surrounding populace.

The Project Hydrogeologist/Engineer may also assume the role of SSO.

2.6 LABORATORY DIRECTOR (IT Analytical Services, Knoxville, TN)

Responsibilities of the Laboratory Director shall, as appropriate, include:

- General supervision of the laboratory, including QA/QC procedures
- Collaboration with the Project Group in establishing sampling and testing programs

- Schedule and execution of testing programs
- Serving as liaison between the Laboratory Staff and other personnel
- Serving as the "collection point" for Laboratory Staff reporting of nonconformances and changes in laboratory activities
- Notification of the Laboratory and Quality Assurance Groups of specific laboratory nonconformances and changes
- Maintenance of laboratory data
- Release of testing data and results.

2.7 QC COORDINATORS

Responsibilities of the QC Coordinators shall, as appropriate, include:

- Supervising sample receipt and coding to ensure compliance with required chain-of-custody procedures.
- Ensuring that all required QC samples are properly inserted in the sample stream.
- Ensuring that samples are properly preserved and that holding times are not exceeded.
- Reviewing QC data for compliance with project requirements.
- Initiating corrective action when necessary.

3.0 QUALITY ASSURANCE OBJECTIVES

This project will be performed in conformace with IT's Quality Assurance Program requirements, and applicable federal, state and contract requirements. Sample analyses will be performed as specified by EPA Contract Laboratory Program protocol and methods. Project quality assurance objectives are as follows:

- The scientific data generated will be of sufficient or greater quality to stand up to scientific and legal scrutiny
- The data will be gathered or developed in accordance with procedures appropriate for the intended use of the data
- The data will be of known and acceptable precision, accuracy and completeness.

This QAPP has been prepared in direct response to these goals. This plan describes the Quality Assurance Program to be implemented and the quality control procedures to be followed by IT and its subcontractors during the course of the project. These procedures will:

- Maintain the necessary level of quality of each aspect of the analytical program by providing the appropriate level of verification testing, checking, and statistical analysis of laboratory program procedures
- Assist in the early recognition of factors which may adversely affect the quality of data, and provide for the implementation of procedures to correct these adverse effects
- Enhance the utility of all data produced by the laboratory for decision-making purposes by requiring sufficient documentation of the testing process. This provides information on the limitations of the analytical results.

In this regard, the QAPP will provide for the definition and evaluation of the following parameters:

- o Detection Limits
- o Data Precision
- o Data Accuracy
- o Completeness of Data.

3.1 DETECTION LIMITS

The detection limit for a given parameter is determined by procedures specified in the CLP methods. Table 2 provides a listing of the CLP Contract Required Detection Limits. Per CLP protocol, samples are screened to determine if they are categorized as low or medium level.

3.2 DATA PRECISION AND EVALUATION

Precision is a measure of the mutual agreement among individual measurements of the same property, usually under prescribed similar conditions. The Relative Percent Difference (RPD) parameter will be used to define the precision between replicate analyses. This parameter is defined in Section 12.0. The precision objectives for the organic analyses will be the same as those established by EPA CLP protocol. Non-homogenous constituents in the soil samples may produce poor precision in the results.

3.3 DATA ACCURACY AND EVALUATION

Accuracy is defined as the degree of agreement of a measurement with an accepted reference or true value. The Percent Recovery (%R), determined by sample spiking, is typically used to define the accuracy of an analytical procedure. This parameter is defined in Section 12.0. The accuracy objectives for the organic analyses will be the same as those established by the U.S. EPA for its CLP. Non-homogenous constituents in the soil samples may also affect the percent recovery results, if the native analytes in the spiked and unspiked aliquots have different concentrations.

3.4 COMPLETENESS OF DATA

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct normal conditions. Over 90 percent of all data obtained on this project should be valid based upon evaluation of the quality control data.

4.0 SAMPLING PROCEDURES

Any sample obtained during the course of a field investigation should be representative of the site and free of contaminants from sources other than the immediate environment being sampled. The equipment and the techniques that will be employed to obtain representative, unbiased samples will be in accordance with IT Standard Operating Procedures and the following U.S. EPA monographs:

15 -- Purposes and Objectives of Sampling
17 -- Soil and Sediment Sampling Methods
19 -- Methods for Collecting Hazardous Samples
22 -- Sampling Handling, Packaging and Shipping Procedures

The Manual of Practice titled, "Subsurface Investigation", associated with the IT Engineering Services Quality Assurance Manual provides information on the advancement of geotechnical borings and geotechnical sampling. This chapter of the Manual will be used to supplement this plan, as necessary.

An internal technical procedure entitled "Visual Classification of Subsurface Materials, Classification Log Legend," which describes the manner in which information obtained from subsurface borings is recorded, shall also be used on this project.

Information obtained from site exploration activities shall be recorded and documented in accordance with Chapter 5 of the IT Engineering Services Quality Assurance Manual. Required documentation of field investigation and testing includes a daily log of project activities, the appropriate subsurface logs and test data forms. Examples of this documentation are shown in Figures 5 through 7.

The Sampling Plan (Appendix A) describes the numbers and types of samples to be collected; sampling equipment, procedures, and locations; sample containers; methods of sample preservation; decontamination procedures; shipping and packaging methods; analytical tests to be performed; sampling personnel; and sampling schedule.

IT CORPORATION

FIGURE 5
RIVERDALE CHEMICAL COMPANY
CHICAGO HEIGHTS, ILLINOIS

DAILY LOG				
	DATE			
	NO.			
	SHEET		OF	

FIELD ACTIVITY DAILY LOG

PROJECT NAME	PROJECT NUMBER
FIELD ACTIVITY SUBJECT :	
DESCRIPTION OF DAILY ACTIVITIES AND EVENTS :	SKETCH

CHANGES FROM PLANS AND SPECIFICATIONS:	PERSONNEL ON SITE:
WEATHER CONDITIONS:	IMPORTANT TELEPHONE CALLS:
PREPARED BY:	(FIELD ENGINEER) DATE:

FIGURE 6
VISUAL CLASSIFICATION OF SOILS
RIVERDALE CHEMICAL COMPANY
CHICAGO HEIGHTS, ILLINOIS

IT CORPORATION

VISUAL CLASSIFICATION OF SOILS

PROJECT NUMBER:	PROJECT NAME:	
BORING NUMBER:	COORDINATES:	DATE:
ELEVATION:	GWL: Depth Date/Time	DATE STARTED:
ENGINEER/GEOLOGIST:	Depth Date/Time	DATE COMPLETED:
DRILLING METHODS:		PAGE OF

DEPTH ( )	SAMPLE TYPE & NO.	BLOWS ON SAMPLER PER ( )	RECOVERY ( )	DESCRIPTION	USCS SYMBOL	MEASURED CONSISTENCY (TSF)	REMARKS

NOTES

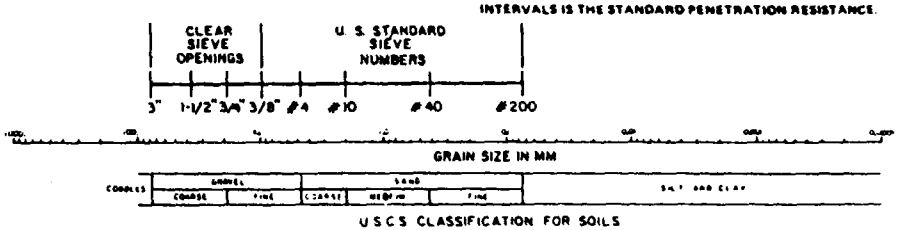
CONSISTENCY OF COHESIVE SOILS

CONSISTENCY	UNCONFINED COMPRESSIVE STRENGTH (TONS PER SQUARE FOOT)
VERY SOFT	LESS THAN 0.25
SOFT	0.25 TO 0.50
MEDIUM STIFF	0.50 TO 1.0
STIFF	1.0 TO 2.0
VERY STIFF	2.0 TO 4.0
HARD	MORE THAN 4.0

DENSITY OF GRANULAR SOILS

DENSITY	STANDARD PENETRATION RESISTANCE[1]
VERY LOOSE	0 – 4
LOOSE	5 – 10
MEDIUM DENSE	11 – 30
DENSE	31 – 50
VERY DENSE	OVER 50

[1] STANDARD PENETRATION RESISTANCE IS THE NUMBER OF BLOWS REQUIRED TO DRIVE A 2-INCH O.D. SPLIT BARREL SAMPLER 12 INCHES USING A 140-POUND HAMMER FALLING FREELY THROUGH 30 INCHES. THE SAMPLER IS DRIVEN 18 INCHES AND THE NUMBER OF BLOWS RECORDED FOR EACH 6-INCH INTERVAL. THE SUMMATION OF THE FINAL TWO INTERVALS IS THE STANDARD PENETRATION RESISTANCE.



USCS CLASSIFICATION FOR SOILS

COARSE-GRAINED SOILS

CLEAN GRAVELS (LITTLE OR NO FINES)	GW	WELL-GRADED GRAVELS, GRAVEL-SAND MIXTURES, LITTLE OR NO FINES
	GP	POORLY-GRADED GRAVELS, GRAVEL-SAND MIXTURES, LITTLE OR NO FINES
GRAVELS WITH FINES (APPRECIABLE AMOUNT OF FINES)	GM	SILTY GRAVELS, GRAVEL-SAND-SILT MIXTURES
	GC	CLAYEY GRAVELS GRAVEL-SAND-CLAY MIXTURES
CLEAN SANDS (LITTLE OR NO FINES)	SW	WELL-GRADED SANDS, GRAVELLY SANDS, LITTLE OR NO FINES
	SP	POORLY-GRADED SANDS, GRAVELLY SANDS, LITTLE OR NO FINES
SANDS WITH FINES (APPRECIABLE AMOUNT OF FINES)	SM	SILTY SANDS, SAND-SILT MIXTURES
	SC	CLAYEY SANDS, SAND-CLAY MIXTURES

FINE-GRAINED/HIGHLY ORGANIC SOILS

SILTS AND CLAYS LIQUID LIMIT (LESS THAN 50)	ML	INORGANIC SILTS AND VERY FINE SANDS, ROCK FLOUR, SILTY OR CLAYEY FINE SANDS OR CLAYEY SILTS WITH SLIGHT PLASTICITY
	CL	INORGANIC CLAYS OF LOW TO MEDIUM PLASTICITY, GRAVELLY CLAYS, SANDY CLAYS, SILTY CLAYS, LEAN CLAYS
	OL	ORGANIC SILTS AND ORGANIC SILTY CLAYS OF LOW PLASTICITY
SILTS AND CLAYS LIQUID LIMIT (GREATER THAN 50)	MH	INORGANIC SILTS, MICACEOUS OR DIATOMACEOUS FINE SANDY OR SILTY SOILS
	CH	INORGANIC CLAYS OF HIGH PLASTICITY, FAT CLAYS
	OH	ORGANIC CLAYS OF MEDIUM TO HIGH PLASTICITY, ORGANIC SILTS
HIGHLY ORGANIC SOILS	PT	PEAT, HUMUS, SWAMP SOILS WITH HIGH ORGANIC CONTENTS

IT CORPORATION

FIGURE 7
RIVERDALE CHEMICAL COMPANY
CHICAGO HEIGHTS, ILLINOIS

LABORATORY LOG SHEET

PROJECT NAME _____

PROJECT NO. _____

PROJECT ENGINEER _____

DATE _____

BORING OR TEST PIT NO.	SAMPLE NO.						DEPTH OR LOCATION	DATE RECEIVED	DATE SAMPLE MAY BE DISCARDED	FIELD ENGINEER
	3" SHELBY	2" SHELBY	JAR	BAG	ROCK CORE					

To reduce the possibility of cross-contaminating samples, all tools, sampling equipment, and surfaces of measuring instruments will be thoroughly decontaminated between each use. The general decontamination procedures that will be observed are as follows:

- Wash with detergent (TSP) and water
- Rinse with distilled water
- Rinse with methanol, acetone or isopropyl alcohol
- Air dry
- Rinse with distilled water.

Prior to entering the site, the drill rig, drilling tools and equipment, and well pipe and casing will be steam cleaned. The drilling tools and equipment will also be steam cleaned between holes, and the split spoon sampler will be decontaminated between each sample drive.

Procedures for abandoning the boreholes upon completion of drilling are detailed in the Sampling Plan (Appendix A). Disposal procedures for potentially contaminated soils, water, and protective equipment resulting from sampling and decontamination efforts are also included in Appendix A.

5.0 SAMPLE CUSTODY

5.1 CHAIN-OF-CUSTODY PROCEDURES

Chain-of-custody procedures are intended to document sample possession from the time of collection to disposal, in accordance with federal guidelines. A copy of IT's chain-of-custody record form is included in Figure 8. For the purpose of these procedures, a sample is considered in custody if it is:

- In one's actual possession
- In view, after being in physical possession
- Locked so that no one can tamper with it, after having been in physical custody
- In a secured area, restricted to authorized personnel.

These procedures will be followed for all samples subject to chemical analysis for this project:

- Sample containers will be sealed in the field. Any samples that do not arrive at the laboratory with seals intact will not be considered to have been in valid custody.
- A chain-of-custody record will be initiated in the field for each sample. A copy of this record will accompany its sample.
- Each time responsibility for custody of the sample changes, the new custodian will sign the record and denote the date.
- Upon sample destruction or disposal, the custodian responsible for the disposal will complete the chain-of-custody record, file a copy, and send a copy to the Project Manager or to his designated representative for recordkeeping.
- The custody of individual sample containers will be documented by recording each container identification on an appropriate chain-of-custody form.
- Analyses for each sample will be recorded on an IT Analytical Services (ITAS) Request-for-Analysis form.

FIGURE 8
RIVERDALE CHEMICAL COMPANY
CHICAGO HEIGHTS, ILLINOIS

IT CORPORATION

SHEET NO. _____ OF _____

CHAIN OF CUSTODY RECORD

PROJECT NAME _____ SAMPLER(S) SIGNATURE _____

PROJECT NO. _____ _____

SAMPLE IDENTIFICATION	SAMPLING LOCATION	DATE SAMPLED	SAMPLE TYPE				COMP.	GRAB	VOLUME TO BE COLLECTED	NO. OF CONTAINERS	TIME COLLECTION BEGAN	INITIAL	TIME COLLECTION COMPLETED	INITIAL	COMMENTS
			WATER	SOLID	AIR	OIL									

RELINQUISHED BY: NAME _____ DATE/TIME _____ RECEIVED BY: NAME _____ DATE/TIME _____

RELINQUISHED BY: NAME _____ DATE/TIME _____ RECEIVED BY: NAME _____ DATE/TIME _____

RELINQUISHED BY: NAME _____ DATE/TIME _____ RECEIVED BY: NAME _____ DATE/TIME _____

AUTHORIZATION FOR DISPOSAL _____ DATE/TIME _____ DISPOSED BY: _____ DATE/TIME _____

- The following documentation will supplement the chain-of-custody records:
  - Field log book ultimately to be filed in the project files
  - Sample label on each sample
  - Sample seal on each sample
  - Field collection report
  - Photographic records (wherever practical and to the extent economically feasible)
- Prior to sampling, all personnel involved will have received copies of the chain-of-custody procedure.

5.2 FIELD COLLECTION REPORT

Field collection reports will be completed in the field for each sample acquired. The information to be provided on this report will include, but not be limited to:

- Project name and number
- Sample number (unique)
- Sampling location (e.g., boring, depth or sampling interval, and field coordinates)
- Sampling date and time
- Individual performing the sampling
- Preservation method employed
- Sample type (grab, composite, etc.)
- Sampling method description (elevation/depth of acquisition, etc.)
- Results of any field tests performed on the sample.

5.3 SAMPLE LABELING

Sample labels must contain sufficient information to uniquely identify the sample in the absence of other documentation. This will include as minimum:

- Project number
- Unique sample number
- Sample location and depth
- Sampling date and time
- Individual collecting the sample
- Preservation method employed.

The sample label will always be directly affixed to the sample container, and will always be completed using indelible ink. An example of the sample label to be used in this project is presented in Figure 9.

5.4 STANDARD OPERATING PROCEDURES

IT Analytical Services Standard Operating Procedures for internal sample coding, tracking, chain-of-custody, etc. are shown in Appendix C.

FIGURE 9
CHEMICAL ANALYSIS SAMPLE LABEL
RIVERDALE CHEMICAL COMPANY
CHICAGO HEIGHTS, ILLINOIS

IT CORPORATION

Project Name _____ Project No. _____

Sample Location _____

Boring/Well No. _____

Collector's Name _____ Date _____

Sample Type: ____ Ground Water ____ Surface Water

____ Soil ____ Sludge/Waste

Parameters _____ Preservative _____

Bottle _____ of _____ ____ Filtered ____ Nonfiltered

6.0 EQUIPMENT CALIBRATION

6.1 GENERAL CALIBRATION PROCEDURES

All laboratory and field testing equipment used for analytical determinations will be subject to periodic inspection and calibration.

Measuring and test equipment and reference standards will be calibrated at prescribed intervals and/or prior to use. Frequency will be based on the type of equipment, inherent stability, manufacturer's recommendations, values given in national standards, intended use, and experience.

Calibrated equipment shall be uniquely identified by using either the manufacturer's serial number or other means. A label with the identification number and the date when the next calibration is due will be physically attached to the equipment. If this is not possible, records traceable to the equipment will be readily available for reference.

Scheduled periodic calibration of testing equipment does not relieve field or laboratory personnel of the responsibilty of employing properly functioning equipment. If an individual suspects an equipment malfunction, he shall remove the device from service, tag it so that it is not inadvertently used, and notify the Project Manager so that recalibration can be performed or substitute equipment can be obtained.

6.2 CALIBRATION FAILURES

Equipment that fails calibration or becomes inoperable during use will be removed from service and either segregated to prevent inadvertent use, or tagged to indicate it is out of calibration. Such equipment will be repaired and recalibrated or replaced as appropriate.

Results of activities performed using equipment that has failed recalibration will be evaluated by the Project Manager. If the activity results are adversely affected, the results of the evaluation will be documented and the appropriate personnel notified.

6.3 CALIBRATION PROCEDURES FOR THE OVA AND HNu

The following equipment will be used for air monitoring and head space analysis for the soil samples:

- The HNu Model 101 photoionization analyzer equipped with the 10.2 eV lamp and calibrated for direct reading in ppm vol/vol of benzene.
- The Century Systems portable organic vapor analyzer Model OVA-128.

The calibration procedures for the OVA and HNu are shown in Appendix E, which are excerpts from the manufacturers' manuals.

7.0 ANALYTICAL PROCEDURES

7.1 OVERVIEW OF STANDARD LABORATORY OPERATING PROCEDURES

Procedures which are to be routinely followed when analyzing samples include:

- Holding times and the amount of sample available should be reviewed and the analyses prioritized
- Analyses shall be performed within holding times specified by CLP protocol
- A calibration curve consisting of five standards and a reagent blank shall be prepared as specified by CLP protocol
- Preparation and analysis of at least one procedural blank should be completed for each group of samples analyzed
- At least one sample, spiked in duplicate, shall be analyzed for every 20 samples processed to monitor the percent recovery, precision, and accuracy of the analytical procedure
- Performance evaluation samples from EMSL-LV will be provided U.S. EPA Region V for inclusion in the package of samples shipped to the laboratory. The samples will consist of one blank and one spiked sample.
- Tentative identification of the first ten volatile and first 20 semivolatile compounds greater than ten percent of the nearest internal standard will be provided.

7.2 ORGANIC COMPOUNDS

The analyses for organic HSL compounds (volatiles, acid extractables, base/neutral extractables, and pesticides/PCBs) and dioxin will be performed by IT Analytical Services Laboratories in Knoxville, Tennessee, using gas chromatography (GC) and gas chromatography/mass spectrometry (GC/MS) instrumentation. The Knoxville Laboratory is certified under the Contract Laboratory Program (CLP) for organic analyses. Procedures instituted by the CLP will be adhered to during all appropriate organic analyses pertaining to the RI/FS at the Riverdale facility. The analyses for HSL compounds will

follow methods found in EPA IFB WA-84-A267 and subsequent amendments and revisions in effect at the time of analysis. Dioxin will be analyzed following methods found in EPA IFB WA-84-A002. Sample preparation and analysis procedures specific for dioxin at the ITAS Knoxville Laboratory are shown in Appendix D.

The address for IT Analytical Services Knoxville Laboratory is as follows:

IT Analytical Services, Inc.
Stewart Laboratories Division
5815 Middlebrook Pike
Knoxville, Tennessee 37921

8.0 DATA REDUCTION, VALIDATION, AND REPORTING

All raw data pertaining to the project will be labeled as such and one copy will be submitted as a separate document with the final investigation report. When data are reduced, the method of reduction will be identified and described. The final report will also include, but not be limited to the following:

- Completed chain-of-custody record form
- Report data
- Method detection limits
- Method blank results
- Matrix spike results
- Duplicate results
- Performance evaluation sample results
- A presentation of the accuracy and precision data
- Other records as required by CLP protocol.

Procedures for assessing these aspects of the data are described in Section 12.0.

All raw data and calculations included in the final report will be checked by a person of proper technical expertise. A Xerox copy of each sheet of results or calculations will be made for checking. The checker will mark each correct calculation with a yellow felt pen. Errors will be identified with a red marker. The originator will then review the changes recommended by the checker. If the originator disagrees with the checker, the two will confer with one another until their differences are resolved.

9.0 QUALITY CONTROL PROCEDURES

9.1 FIELD QUALITY CONTROL PROCEDURES

To check the quality of data from field sampling efforts, blank and duplicate samples will be submitted to IT's Analytical Laboratory. Blank samples will be analyzed to check for container contamination or contamination induced by the sample collection procedure. Duplicate samples will be analyzed to check for sampling and analytical error causing data scatter. The confidence limits and percent level of uncertainty will be calculated and reported in the RI report. One duplicate will be prepared for every 20 samples collected and one blank will be prepared for every 20 samples (including duplicates) submitted for analysis.

Field blank samples will be prepared using diatomaceous earth for all soil samples, filling appropriate sample containers for each. Standard sampling equipment and procedures will be used for blank sampling. All blank and duplicate samples will be treated as separate samples for identification, logging, and shipping.

9.2 LABORATORY QUALITY CONTROL PROCEDURES

9.2.1 Volatile Organics

Samples for volatile organics analysis will be analyzed according to current CLP procedures. Each GC/MS will be checked and retuned (as necessary) every 12 hours to ensure that its performance on bromofluorobenzene meets the applicable U.S. EPA criteria. A calibration curve will be prepared using a mixture of volatile standards and internal standards as specified in CLP procedures. The calibration curve will be verified with a mixture of volatile standards every 12 hours.

All standards, method blanks, and samples will be spiked prior to purging (with a stream of inert gas) with surrogate standards as specified in CLP procedures. Surrogate standards are defined as non-HSL compounds used to monitor the Percent Recovery efficiencies of the analytical procedure on a sample-by-sample basis. Samples exhibiting surrogate standard responses outside the established control limits will be reanalyzed.

At least one method blank for every 20 samples will be purged and analyzed for volatile organic compounds. Volatile organics analysis requires a method blank consisting of five milliliters of organic free water spiked with the appropriate surrogate standards. Results of the method blank analysis will be maintained with the corresponding sample analyses.

One sample out of every 20 will be spiked, purged and analyzed in duplicate, following CLP procedures, to evaluate the precision of the analytical procedure. The relative percent error for each parameter will then be calculated from these duplicate analyses. Should the average relative percent error fall outside the appropriate quality control limits, the other quality control parameters will be evaluated to determine whether the duplicate sample should be re-purged and analyzed or whether the entire set of samples requires to be re-purged and analyzed.

Using the above Matrix Spike (MS) and Matrix Spike Duplicate (MSD) samples, the percent recoveries for the respective compounds will then be calculated. Thould the percent recovery values fall outside the appropriate quality control limits, the other quality control parameters will be evaluated to determine whether an error in spiking occurred or whether the entire set of samples requires re-purging and analysis.

9.2.2 Acid and Base/Neutral Extractables

Samples analyzed for acid and base/neutral extractables will be tested according to current CLP procedures. Each GC/MS will be checked and tuned (as necessary) every 12 hours to ensure that its performance on decafluorotriphenylphosphine (DFTPP) meets U.S. EPA standards. A calibration curve for quantification will be prepared using a mixture of standards composed of either organic acid or base/neutral extractable compounds and internal standards as specified by CLP procedures. The calibration curve will be verified with a mixture of HSL standards every 12 hours.

All standards, method blanks, and samples will be spiked with six surrogate compounds as specified by CLP procedures. Samples exhibiting surrogate standard responses outside the established control limits will be reanalyzed.

At least one method blank for every 20 samples will be analyzed for acid and base/neutral extractables. These compounds require a method blank consisting of one liter of distilled water spiked with the appropriate surrogate standards.

One sample out of every 20 will be spiked, extracted and analyzed in duplicate, following CLP procedures, to evaluate the precision of the analytical procedure. The relative percent error for each parameter will then be calculated from these duplicate analyses. Should the average relative percent error fall outside the appropriate quality control limits, the other quality control parameters will be evaluated to determine whether the entire set of samples requires re-extraction and analysis.

Using the above Matgrix Spike (MS) and Matrix Spike Duplicate samples, the percent recoveries for the respective compounds will then be calculated. Should the percent recovery values fall outside the appropriate quality control limits, the other quality control parameters will be evaluated to determine whether an error in spiking occurred or whether the entire set of samples requires re-extraction and analysis.

9.2.3. PCB's and Pesticides

PCB and pesticides analyses will be performed according to CLP procedures. Samples will be batched in groups of 20 or less. Blanks, spiked blanks, and spiked samples will be analyzed as specified in CLP procedures. Quality control data will be compared to CLP specifications to determine if reanalyses are necessary.

9.2.4 2,3,7,8-TCDD (Dioxin)

Isomer specificity will be demonstrated initially and verified once per 12-hour work shift. The verification will consist of injecting a mixture containing TCDD isomers which elute close to Dioxin. The Dioxin must be separated from interfering isomers, with no more than a 25% valley relative to the Dioxin peak.

Each sample will be dosed with a known quantity of $^{13}C_{12}$-2,3,7,8-TCDD as an internal standard and $^{37}Cl_4$-TCDD as a surrogate standard. The action limits

for surrogate standard results are +/- 40% of the true value. Samples showing surrogate standard results outside of these limits will be reextracted and reanalyzed unless variability is known to be due to the matrix effect.

A laboratory method blank will be run with each set of 20 or fewer samples. This method blank will be dosed with the internal and surrogate standards. Laboratory QA/AC procedures also require at least one sample in each set of 20 (or fewer) samples to be run in duplicate to determine intralaboratory precision.

Performance evaluation samples from EMSL-LV will be provided by U.S. EPA Region V. The samples will consist of one blank sample and one spiked sample. The samples will be shipped from the field in the same package as the samples collected at the Riverdale site.

The quality control procedures specific for dioxin analysis at the IT Analytical Services Laboratories in Knoxville, Tennessee are shown in Appendix F.

10.0 PERFORMANCE AND SYSTEMS AUDITS AND FREQUENCY

One audit is to be scheduled to verify compliance with IT and specific project QA/QC Program requirements. This audit will consist, as appropriate, of an evaluation of QA/QC procedures and the effectiveness of their implementation, and evaluation of work areas and activities, and a review of project documentation.

The audit will cover both field activities and report preparation. The audit will be conducted by one or more of the following IT personnel:

- Paul Mills, Quality Assurance Director of ITAS - Laboratory Audit
- Dave Troxell, Corporate Director of Quality Assurance - Project Audit
- Lee Kettren, Quality Assurance Manager - Milwaukee Office

The records of all field operations will be reviewed to verify that field-related activities were performed in accordance with appropriate project procedures. Items reviewed may include, but not be limited to: calibration records of field equipment; daily field activity logs; photographs; and all data, logs, and checkprints resulting from the field operations.

The audit will also examine, as appropriate, the documentation and verification of field and laboratory data and results; performance, documentation, and verification of analyses; preparation and verification of drawings, logs, and tables; content, consistency, and conclusions of the final report; compliance with IT and project requirements; and maintenance and filing of project records.

Audit results will be transmitted to the Project Manager and the Project Executive. Requests for corrective action will be made as described in Section 13.

11.0 PREVENTIVE MAINTENANCE

Periodic preventive maintenance is required for all sensitive equipment. Instrument manuals are kept on file for reference purposes should equipment need repair. Troubleshooting sections of manuals are often useful in assisting personnel in performing maintenance tasks.

Any equipment requiring routine maintenance will be tagged with a maintenance label indicating the date of required maintenance, the person maintaining the equipment, and the next maintenance date. Information pertaining to life histories of equipment maintenance will be kept in individual Equipment History Logs with each instrument.

12.0 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS

The following discussion describes the procedures that will be employed to evaluate the precision, accuracy, and completeness of the chemical test data generated during the investigation. Accuracy is assessed by splitting a sample into two portions, spiking (i.e., adding a known quantity of the constituents of interest) to one of the portions, and then analyzing both portions for these parameters. The difference in the concentration levels of the constituents of interest should be equal to the quantity of the spike added to one of the two portions. The actual percent recovery (%R) is calculated as follows:

$$\%R = C / C_s \times 100\%$$

where C is the measured concentration increase due to spiking and C_s is the known increase due to the spike. One hundred percent recovery is equivalent to 100% accuracy. The coefficient of variation (C_v) of the %R values is calculated as follows:

$$C_v = \frac{SD}{APR} \times 100\%$$

SD is the standard deviation of the percent recoveries for the various spiked constituents and APR is the average or mean percent recovery.

Precision is assessed by conducting separate analyses of the duplicate samples. A measure of the agreement in the reported values for the two portions is obtained by calculating the relative percent difference (RPD) in the concentration levels of each constituent, where

$$RPD_i = \frac{A_i - B_i}{\frac{(A_i + B_i)}{2}} \times 100\%$$

and A_i and B_i are the concentrations of the ith constituent.

The evaluation of the test data will be based in part on criteria adopted by the Sample Management Office of the U.S. EPA. These criteria, provide a means of categorizing a data set as being quantitative, semi-quantitative, or qualitative. They are as follows:

ORGANICS

Quantitative	APR	80% or greater
	Cv	20% or less
Semi-quantitative	APR	60% or greater
	Cv	20% to 40%
Qualitative	APR	40% or better
	Cv	70% or less

INORGANICS

Quantitative	APR	90% to 110%
	Cv	15% or less
Semi-quantitative	APR	80% or greater
	Cv	15% to 30%
Qualitative	APR	80% or less
	Cv	30% or greater

In addition to evaluating each set of data for their accuracy and precision, an assessment will also be made of the completeness of the data. This will involve computing the fraction of the reported values that remain valid after the sampling procedures have been reviewed and the results have been assessed for their precision and accuracy. The QA objectives for the investigation relative to precision, accuracy, and completeness are described in Section 3.0.

13.0 NONCONFORMANCE/CORRECTIVE ACTION PROCEDURES

Nonconforming items and activities are those which do not meet the project requirements, procurement document criteria, or approved work procedures. Nonconformances may be detected and identified by:

- Project staff - During the performance of field investigation and testing, supervision of subcontractors, and performance of audits and verification of numerical analyses
- Laboratory staff - During the preparation for and performance of laboratory testing, calibration of equipment, and quality control activities
- Quality Assurance Staff - During the performance of audits.

Each nonconformance will be documented by the person identifying or originating it. For this purpose, a Variance Log, Testing Procedure Record, Notice of Equipment Calibration Failure, results of laboratory analysis control tests, post audit report, internal memorandum, or letter will be used as appropriate. Documentation shall, when necessary, include:

- Name(s) of the individual(s) identifying or originating the nonconformance
- Description of the nonconformance
- Any required approval signatures
- Method(s) for correcting the nonconformance or description of the variance granted
- Schedule for completing corrective action.

Documentation will be made available to project, laboratory, and/or quality assurance management. Appropriate personnel will be notified by the management of any significant nonconformance detected by the project, laboratory or quality assurance staff. Implementation of corrective actions will be the

responsibility of the Project Hydrogeologist/Engineer, the Project Manager, or the Laboratory Director. In addition, the Project Manager will notify Riverdale of significant nonconformances which could impact the results of the work and indicate the corrective action taken or planned.

The Project Manager will be responsible for approving corrective actions initiated by the Project Hydrogeologist/Engineer. Completion of corrective actions for significant nonconformances will be verified by the Project Manager.

Any significant recurring nonconformance will be evaluated by project or laboratory personnel to determine its cause. Appropriate changes will then be instituted in project requirements and procedures to prevent future recurrence. When such an evaluation is performed, the results will be documented.

14.0 QUALITY ASSURANCE AUDITS AND REPORTS

To verify compliance with IT and specific project Quality Assurance/Quality Control Program requirements, the IT Quality Assurance Group shall perform planned and documented audits of project activities. These audits shall consist, as appropriate, of an evaluation of QA/QC procedures and the effectiveness of their implementation, an evaluation of work areas and activities, and a review of project documentation. Audits shall be performed in accordance with written checklists by trained members of the Quality Assurance Group and, as appropriate, technical specialists. Audit results shall be formally documented and sent to project management.

Audits may include, but not be limited to, the following areas:

- Field operations records
- Laboratory testing and records
- Equipment calibration and records
- Identification and control of samples
- Numerical analyses
- Computer program documentation and verification
- Transmittal of information
- Record control and retention.

Planned audits for this project will, as appropriate, cover the final reports. Unless significant QA problems arise, it is not anticipated that any separate reports will be issued. The final report will contain a separate QA section that summarizes the quality of the data collected during the project. Auditing will be performed in accordance with applicable requirements of Chapter 11 of the IT Engineering Services Quality Assurance Manual.

APPENDIX
A

APPENDIX A

SAMPLING PLAN

REMEDIAL INVESTIGATION/ FEASIBILITY STUDY

RIVERDALE CHEMICAL COMPANY

CHICAGO HEIGHTS, ILLINOIS

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1.0 INTRODUCTION

Remedial Investigation activities conducted by IT Corporation (IT) at the Riverdale Chemical Company's (Riverdale) plant will include the following activities noted below.

Nine soil borings will be drilled to a depth of 10-15 feet, or deeper if necessary. Approximately 100 soil samples will be collected from these borings. Initially, a total of 16 of these samples will be selected for testing of 2,3,7,8 tetrachlorodibenzo-p-dioxin (Dioxin) using rapid sample test turnaround time. Four of the 16 samples will be tested for Hazardous Substances List (HSL) organics and pesticides. Twenty surface soil samples will be collected at various on- and off-site locations. Fourteen samples will be tested for Dioxin and six composite samples will be tested for HSL organic and pesticide compounds.

Depending on the results of the initial testing, additional testing and sampling phases may need to be conducted in order to define the extent of contamination at the Riverdale site. This sampling plan details only the initial sampling and testing phase.

In the following sections of this sampling plan, information is presented on the proposed sampling locations and numbering system; drilling and sampling procedures; QA/QC sampling procedures; sample handling and analyses; decontamination procedures; field documentation procedures; organization, responsibilities, and training of the field team; and the schedule for field activities.

2.0 SAMPLING LOCATIONS, LABELING AND NUMBERING SYSTEMS

2.1 LOCATIONS

Tentative boring and surface soil sampling locations are shown in Figure 1. Exact boring and surface sampling locations will be determined in the field by either the Project Manager, the Project Hydrogeologist/Engineer or both. Selection of locations will be made in conjunction with suggestions by plant personnel.

2.2 LABELING

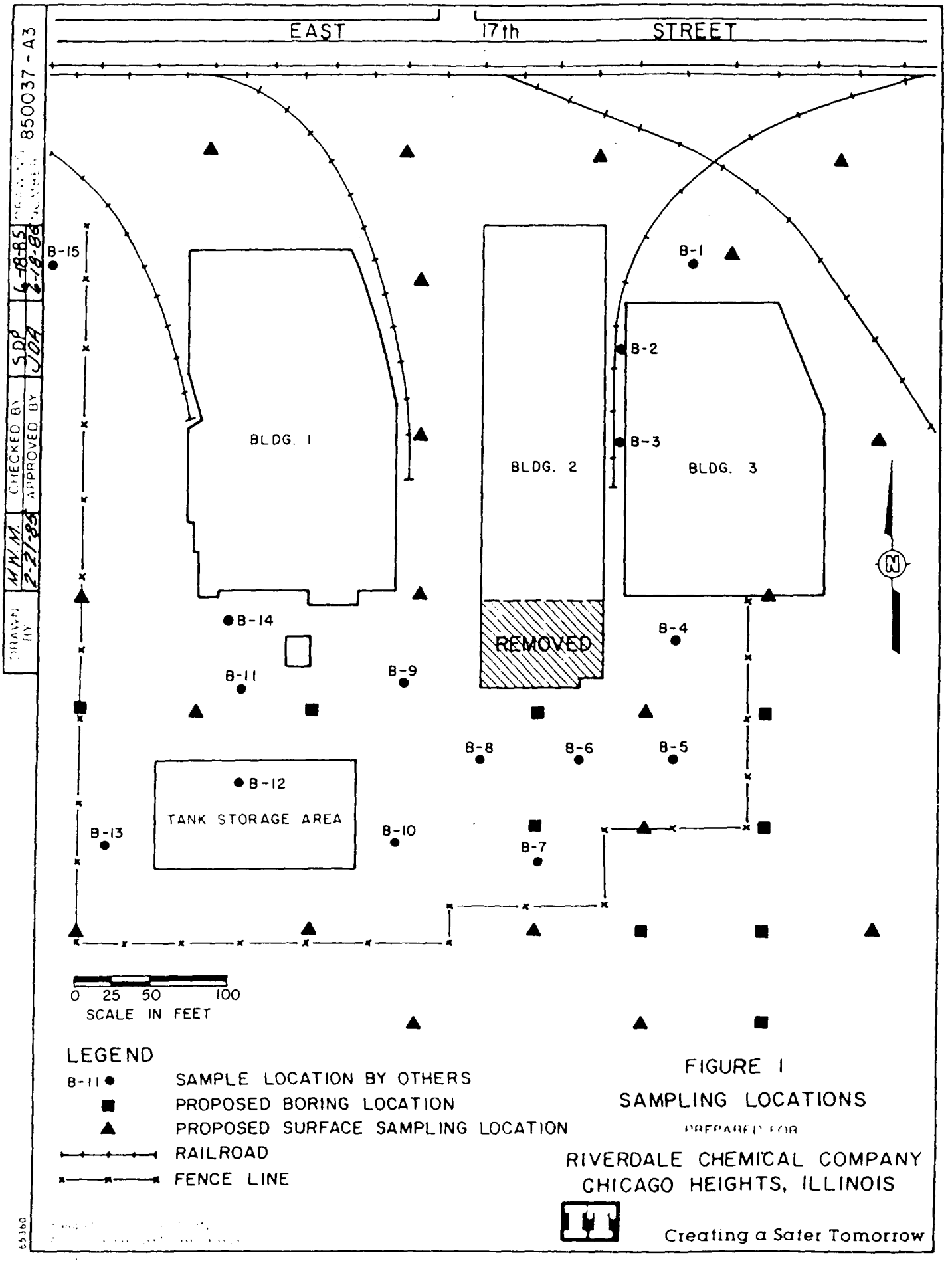
The sample containers will be labeled prior to being filled with soil at the sampling site. The sample label for surface and subsurface soil samples will show the project number, sample number, sample location and depth, date, time of sampling, and sampler's initials. The label will be filled out with waterproof ink or marker.

2.3 SAMPLE NUMBERING SYSTEM

A sample numbering system will be used to identify each sample taken during the Riverdale sampling program. This numbering system will provide a tracking procedure to allow retrieval of information about a particular sample and provide each sample with a unique number. The sample identification numbering system is described below:

- o A three letter designation will be used to identify the site where the samples are collected. For this project, it will be RIV, which stands for Riverdale
- o Each sample type collected during the sampling program will be identified by a 2-digit code:
  - SB - Soil, Boring
  - SS - Soil, Surface
- o A 2-digit number will be used to indicate boring and surface sample locations. Soil samples from borings will be identified as to the depth at which they were taken. The number will specify the deepest depth at which the sample was taken. Composite samples will be indicated by parentheses
- o A 1-digit number will be used to consecutively number sequential samples taken at a sampling site.

IT CORPORATION



Examples of sample numbers are:

- RIV-SB-01-4.5-1 - soil boring sample from boring 1, sample interval from 3 to 4.5 feet depth, sample 1
- RIV-SS-01-1 - Surface soil sample, location 01, sample 1
- RIV-SS-(01-05)-1 - Surface soil sample, composite of locations 01 through 05, sample 1

3.0 DRILLING AND SAMPLING PROCEDURES

The purpose of this task is to characterize the near surface site geology and the horizontal and vertical extent of contamination at the site. The exploration is planned to consist of nine soil borings across the site and collection of 20 surface soil samples. A qualified engineer or hydrogeologist will develop boring logs from the drilling and coordinate all field activities.

3.1 SOIL BORINGS

The nine soil borings will be drilled to a depth of 10-15 feet, or as necessary to define the near surface geology. The final depth of the borings will be determined in the field. Consideration will be given to the existence of coarse grained soils or fractured fine grained soils (which could act as migration pathways for contaminants) and the homogeneity and continuity of the site stratigraphy. Proposed boring locations are shown in Figure 1.

The boreholes will be advanced with hollow stem augers. Samples will be collected with a 3-inch diameter split-barrel sampler on 1.5 foot intervals to a depth of 15 feet. If the borings are advanced deeper than this, the sample interval will be increased to 2.5 feet. At the completion of drilling, each boring will be completely grouted with a cement/bentonite slurry to the ground surface to prevent possible migration of contaminants. (See Section 3.6).

Each boring will be logged by the Project Hydrogeologist/Engineer. This individual will also provide continuous inspection of all drilling activities. The boring log will include:

- Heading information. Included will be the project number, boring number, personnel responsible for logging the hole, ground elevation and coordinates, and date started and completed
- Depths recorded in feet and tenths of feet
- Detailed soil descriptions including:
  - major soil component
  - secondary components and estimated percentages
  - classification
  - unified soil classification symbol

- color
- consistency or density
- moisture content, listed as an adjective (e.g., dry, moist, wet)
- texture
- depositional origin

o Depth/elevation interval

o Depth/elevation of strata changes

o Water-table information and method of determination, if applicable

o Sample drive and recovery

o Blow counts, hammerweight, and length of fall

o Equipment details

o Drilling sequence and comments

o Problems encountered.

After logging a sample, the hydrogeologist or sampling engineer will transfer it to a pre-labeled jar.

Jarred samples will be kept in a cooler at $4^{o}C$ until they can be transferred to a more permanent storage location.

A total of approximately 100 investigative samples will be collected for possible physical and chemical analysis during the drilling program.

Decontamination procedures for the equipment utilized in the subsurface investigation are outlined in Section 3.3.

3.2 SURFACE SOIL SAMPLING

The purpose of the surface sampling is to define the horizontal extent of contamination on the site and define the boundaries of contamination. It is anticipated that 20 surface samples will be collected. Proposed sampling locations are shown on Figure 1. The off-site samples will be collected if access to the property can be obtained. Copies of letters to property owners, requesting permission to sample, will be submitted to the U.S. EPA as verification of Riverdale's effort to obtain such access.

Samples will be collected from the upper 6-inches at each location using a shovel and stainless steel spoon or a stainless steel scoop. After collecting each sample, the sampling team will record the location, sample number, date, time, sampling personnel, and weather conditions in the field log. Decontamination of sampling equipment will be required between sampling locations and consist of the procedures detailed in Section 3.3.

3.3 DECONTAMINATION PROCEDURES

The drilling rig and associated tools will be decontaminated prior to entering the site and will be cleaned between borings. All sampling equipment will be decontaminated between samples to prevent cross contamination between borings and samples. Specific decontamination procedures are presented below:

The drilling rig should be recently painted with no surface areas showing rust. In addition, the rig will be thoroughly cleaned prior to its use. This will be accomplished by a detergent wash and scrub of the drilling rig, the augers, the tools, the bit, and anything else that will either come in contact with the soil or with the tools and augers themselves. Following the detergent wash, the equipment will be rinsed with clean water and then washed with a methanol, acetone or isopropyl alcohol solution followed by steam cleaning with potable water. A sample of the final potable water rinse may be collected for analysis as it drains from the drilling augers. The cleaned augers, tools and other drilling equipment will be placed on a clean truck bed for transportation to the drilling site.

The drill rig will also be inspected for any leakage of hydraulic fluid, oil, transmission fluid or other organic compound which could possibly contaminate the soils. The rig will be filled with gasoline or diesel fuel prior to being brought to the drilling site. Once the drill rig is brought to the site, it will be assumed that the surface soils are contaminated and no equipment will be set down on the ground where it could be contaminated. Clean plastic sheeting, aluminum foil, or cardboard will be placed on the ground to provide a work surface for each hole. The drilling rig will be steam cleaned each time it is moved to a new boring location.

The materials that will enter the borehole (augers, rods, etc.) will be carefully cleaned by an initial detergent wash with trisodium phosphate detergent (TSP), followed with a clean water rinse, a methanol, acetone or isopropyl alcohol rinse, and finally a steam cleaning using potable water.

The sample split spoons will be decontaminated after each sampling drive using the following procedure:

- Scrub with long-handled-brush in TSP and water
- Rinse in water
- Rinse in acetone, methanol or isopropyl alcohol
- Air dry
- Rinse in distilled water.

Several spoons will be used so that the solvents on the spoon can evaporate before the spoon is used again.

All surface sampling equipment will be decontaminated following the above described procedure after each sample is collected.

Drilling personnel will wear appropriate protective clothing as required by the Health and Safety Plan (see Appendix B). These measures will not only protect the driller, but will also protect the hole from cross-contamination. All protective equipment (gloves, boots, etc.) will be decontaminated prior to reuse or disposal, utilizing the procedure outlined earlier.

The drill rig, tools and other drilling equipment will be cleaned before leaving the site. A sample of the final rinse water may be collected to ascertain that the rig will not transport contaminants off the site.

3.4 LOCATING AND SAMPLING UTILITY LINES

This section outlines the provisions IT will use for identifying and locating utility lines, buried pipe and miscellaneous equipment which may be contaminated, and for determining the extent of contamination.

To locate the placement of utilities, sewers and other various buried objects on plant grounds, the plant foreman or superintendent will be contacted to

review the plant's as-built drawings. The foreman will also assist IT personnel to stake, mark or otherwise identify the underground objects near the proposed soil boring locations. This will be done to minimize accidental uncovering or damage to the utilities during drilling operations. In addition, the local public works department and utility companies will be contacted to ascertain the location of existing municipal utilities, electric, gas, and telephone lines that may be buried in the area. This information would be applicable to both on-site and off-site plant grounds. If it is necessary to expose portions of these utilities during drilling, a representative of the particular utility company will be requested to be present. The representative will witness the location and condition of the uncovered utility, as well as providing for a positive identification. The locations of buried utilities and other objects will be presented in the remedial investigation report.

To determine the extent of contamination near any buried objects, soils that are in direct contact with the objects will be sampled. The scope of work for the initial sampling phase, though, is limited to surface and near surface soil sampling, and not near buried objects. If results of the initial sample analysis indicate a need for sampling near buried objects, additional sampling will be conducted during a later phase.

3.5 DISPOSAL OF CONTAMINATED SOIL AND WATER

It is not anticipated that water will be used in the drilling process. Therefore, disposal of contaminated recirculation water is not of concern during this phase of the project.

During the drilling and sampling operations, contaminated soil, disposable health and safety gear, and water from decontamination efforts will be generated. The total amount of contaminated material produced is expected to be relatively small. The cuttings will be moved to and covered by the tarped area on the southern portion of the site. Water from the decontamination processes will be discarded near the point where the boreholes are drilled. Since no impermeable cap exists on-site, this method of disposal will not increase any potential contamination in the soils already present. Disposable

safety equipment (i.e., booties, gloves, outer coverings) will be decontaminated and disposed with other solid wastes generated by the plant.

3.6 HOLE ABANDONMENT PROCEDURES

Upon reaching the maximum depth of the boring and completion of the soil sampling, the borehole shall be abandoned using the following procedure:

- Remove the plug from the hollow stem auger
- Prepare a bentonite/cement slurry that contains at least 25 percent bentonite
- Assemble tremie rods and place in the borehole so that the discharge valve is approximately one foot above the bottom of the boring
- Back the hollow stem augers out of the hole while simultaneously pumping the bentonite/cement slurry into the hole
- After removing the augers and tremieing rods from the hole, finish off the bentonite/cement slurry at the ground surface.

4.0 QA/QC SAMPLING PROCEDURES

Duplicate and blank samples will be collected during the sampling program. In general, one duplicate will be collected for every 20 samples collected and one blank will be obtained for every 20 samples that are taken.

Duplicate soil samples will be obtained by simultaneously filling two sets of sample bottles using standard sampling equipment and procedures. These will then be treated as separate samples for labeling and shipping. Duplicate samples will be logged in the field log book.

Blank samples will be prepared using diatomaceous earth for all soil samples by filling the appropriate sample containers. Standard sampling equipment and procedures will be used for blank sampling. Soil blanks will be placed in a decontaminated stainless steel scoop prior to being placed in sample containers. Blank samples will be treated as separate samples during identification, logging, and shipping procedures.

Performance Evaluation samples from EMSL-LV will be provide by U.S. EPA Region V. The samples will consist of one spiked sample and one blank sample.

5.0 SAMPLE PROCESSING

The soil samples will be processed according to the procedures summarized in Table 1. All samples are anticipated to be low or medium concentration samples.

All soil samples collected from borings will be initially screened prior to shipping using the guidelines summarized below:

- Headspace analysis - the headspace in each container collected for organic analysis will be sampled using an HNu/OVA. The cap of each sample will be loosened and lifted only enough to allow the insertion of the intake probe of the instrument. The sample numbers and instrument readings will be recorded. Samples with relatively high or otherwise anomalous readings may be selected for analysis.
- Suspicious appearance - a visual appraisal will be made when possible and samples which exhibit unusual or anomalous appearances may be selected for analysis.
- Lithology - in some cases it may be pertinent to trace contamination along a specific horizon, and a sample may be selected based upon lithology or stratigraphic position.
- Spatial distribution - all other criteria being equal, samples will be selected to provide spatial distribution.

The initial screening may affect the selection of samples for analyses outlined in Section 6.0.

Initially, it is anticipated that a total of 30 soil samples will be collected (16 from soil borings and 14 from surface locations) for Dioxin analyses; 10 samples will be submitted (4 from soil borings and 6 from surface locations) for HSL organics and pesticides analysis. Two duplicates and two blanks will be collected for Dioxin testing and one duplicate and one blank will be collected for the HSL compound analyses.

While awaiting shipping, all low concentration samples will be stored on ice in coolers. All samples will be preserved on the same day that they are

TABLE 1
BOTTLES, PRESERVATION, SHIPPING AND PACKING REQUIREMENTS FOR CLP SAMPLES
RIVERDALE CHEMICAL COMPANY
CHICAGO HEIGHTS, ILLINOIS

ANALYSIS	BOTTLES AND JARS	PRESERVATION	HOLDING TIME	VOLUME OF SAMPLE	SHIPPING	NORMAL PACKAGING
SOILS						
Low Concentration (Organics)						
Acid extractables, base/neutral extractables, pesticides/PCB's, Dioxin	One 8-oz. wide-mouth glass jar	Iced to 4°C	Not established	Fill 3/4 full	Federal Express Priority 1	Foam Liner No. 3
Volatiles	Two 120-ml wide-mouth glass jar	Iced to 4°C	Not established	Fill completely no headspace	Federal Express Priority 1	Foam Liner No. 3
Medium Concentration (Organics)						
Acid extractables, base/neutral extractables, pesticide/PCB's, Dioxin	One 8-oz. wide-mouth glass jar	None	Not established	Fill 3/4 full	Federal Express Priority 1 w/attached shipper's certification for restricted articles	In metal paint cans vermiculite
Volatiles	Two 120-ml wide-mouth glass jars	None	Not established	Fill completely no headspace	See Above	In metal paint cans vermiculite

collected. If samples cannot be shipped on a particular day, packaging will be delayed until the following morning so that the samples can be shipped with a full load of ice. These samples will be stored on ice in coolers, and kept in a secure area. Medium concentration samples do not require ice preservation.

Coolers will be shipped via Federal Express to the IT Environmental Analytical Laboratory in Knoxville, Tennessee. Notification of shipment, including airbill number, will be phoned to the Laboratory either by close of business of the day that the samples are shipped, or if a late shipment is made, by 9:00 a.m. the following day.

A chain-of-custody record will accompany the samples from time of collection to receipt in the laboratory. A copy of IT's chain-of-custody record form is included with the QAPP.

6.0 SAMPLE ANALYSES

6.1 SOIL BORING ANALYSES

The analytical testing program will define the vertical extent of contamination on the site and define the boundaries of no contamination. This will be accomplished by a tiered effective analysis of samples. Initially, a total of 16 samples will be tested using rapid sample turnaround. If the results show significant contamination, additional testing may be accomplished within the sample hold times. The proposed initial testing will be conducted in the order listed below:

- Three samples will be tested from each of two borings to define the vertical migration of contamination. It is anticipated that the samples to be tested will be collected from depths of six inches, two feet and five feet. The depth interval may be changed in the field depending on the sample processing procedures outlined in Section 5.0 or in the laboratory depending on the results of early lab tests. The two borings will be located in the areas of highest previously recorded contamination.

- Two samples will be tested from each of three borings at depths of six inches and two feet. The two feet depth may be changed depending on the results of the initial testing.

- One sample from each of four borings will be tested at a depth of two feet to define the boundary of no contamination. The two foot depth may be changed as described above.

All of the samples will be tested for Dioxin. Additionally, four samples will be composited from the 16 samples and will be tested for HSL organics and pesticides. A list of the parameters to be tested is presented in Table 2. If the results of this testing show significant contamination, the testing program can be expanded.

6.2 SURFACE SOIL ANALYSES

All of the surface samples collected south of Buildings 1 and 3 will be tested for Dioxin. The eight samples collected from the northern area will be composited into two samples which will be tested for Dioxin. The 20 surface

samples will be composited into six samples to be tested for the HSL pollutants. Table 2 contains the list of parameters to be analyzed.

TABLE 2
PARAMETERS TO BE ANALYZED
RIVERDALE CHEMICAL COMPANY
CHICAGO HEIGHTS, ILLINOIS

VOLATILES
Chloromethane
Bromoethane
Vinyl Chloride
Chloroethane
Methylene Chloride
Acetone
Carbon Disulfide
1,1-Dichloroethene
1,1-Dichloroethane
trans-1,2-Dichloroethene
Chloroform
1,2-Dichloroethane
2-Butanone
1,1,1-Trichloroethane
Carbon Tetrachloride
Vinyl Acetate
Bromodichloromethane
1,1,2,2-Tetrachloroethane
1,2-Dichloropropane
trans-1,3-Dichloropropene
Trichloroethene
Dibromochloromethane
1,1,2-Trichloroethane
Benzene
cis-1,3-Dichloropropene
2-Chloroethyl Vinyl Ether
Bromoform
2-Hexanone
4-Methyl-2-pentanone
Tetrachloroethene
Toluene
Chlorobenzene
Ethyl Benzene
Styrene
Total Xylenes

SEMI-VOLATILES
N-Nitrosodimethylamine
Phenol
Aniline
bis(2-Chloroethyl) ether
2-Chlorophenol
1,3-Dichlorobenzene
1,4-Dichlorobenzene
Benzyl Alcohol
1,2-Dichlorobenzene
2-Methylphenol
bis(2-Chloroisopropyl) ether
4-Methylphenol
N-Nitroso-Dipropylamine
Hexachloroethane
Nitrobenzene
Isophorone
2-Nitrophenol
2,4-Dimethylphenol
Benzoic Acid
bis(2-Chloroethoxy) methane
2,4-Dichlorophenol
1,2,4-Trichlorobenzene
Naphthalene
4-Chloroaniline
Hexachlorobutadiene
4-Chloro-3-methylphenol
(para-chloro-meta-cresol)
2-Methylnaphthalene
Hexachlorocyclopentadiene
2,4,6-Trichlorophenol
2,4,5-Trichlorophenol
2-Chloronaphthalene

SEMI-VOLATILES
2-Nitroaniline
Dimethyl Phthalate
Acenaphthylene
3-Nitroaniline
Acenaphthene
2,4-Dinitrophenol
4-Nitrophenol
Dibenzofuran
2,4-Dinitrotoluene
2,6-Dinitrotoluene
Diethylphthalate
4-Chlorophenyl Phenyl ether
Fluorene
4-Nitroaniline
4,6-Dinitro-2-methylphenol
N-nitrosodiphenylamine
4-Bromophenyl Phenyl ether
Hexachlorobenzene
Pentachlorophenol
Phenanthrene
Anthracene
Di-n-butylphthalate
Fluoranthene
Benzidine
Pyrene
Butyl Benzyl Phthalate
3,3'-Dichlorobenzidine
Benzo(a)anthracene
bis(2-ethylhexyl)phthalate
Chrysene
Di-n-octyl Phthalate
Benzo(b)fluoranthene
Benzo(k) fluoranthene
Benzo(a)pyrene
Indeno(1,2,3-cd)pyrene
Dibenz(a,h)anthracene
Benzo(g,h,i)perylene

PESTICIDES
alpha-BHC
beta-BHC
delta-BHC
gamma-BHC (Lindane)
Heptachlor
Aldrin
Heptachlor Epoxide
Endosulfan I
Dieldrin
4,4'-DDE
Endrin
Endosulfan II
4,4'-DDD
Endrin Aldehyde
Endosulfan Sulfate
4,4'-DDT
Endrin Ketone
Methoxychlor
Chlrodane
Toxaphene
AROCLOR-1016
AROCLOR-1221
AROCLOR-1232
AROCLOR-1242
AROCLOR-1248
AROCLOR-1254
AROCLOR-1260

DIOXIN
2,2,7,8-TCDD

7.0 FIELD DOCUMENTATION PROCEDURES

7.1 SITE LOCATION PROCEDURE

Following identification of boring and surface soil sampling sites, a wooden stake (approximately 2" x 2" x 24") will be driven into the ground, allowing approximately 8 to 10 inches of the stake to remain visible above ground. The top portion of the stake will be painted orange and labeled for identification. The label will contain the sample location number and type. The location of each stake may be recorded by use of a transit and stadia rod.

7.2 PHOTOGRAPHS

Photographs will be taken of each sampling site with respect to the surrounding area and relative to objects used to locate the site. The picture number and roll number (if more than one roll of film is used) will be logged in the field notebook to identify which sampling site is depicted in the photograph. The film roll will be identified by taking a photograph of an informational sign on the first frame of the roll. This sign will have the job and film roll numbers written on it so as to identify the pictures contained on the roll.

For example: Riverdale
Roll Number 1
Frame Number 1 of 36
July 1, 1985 - (Photographer's name)

7.3 FIELD NOTEBOOKS

Field notebooks will provide the means of recording data collection activities performed at the site. As such, entries will be described in as much detail as possible so that the situation can be reconstructed without reliance upon memory.

Notebooks will be assigned to field personnel, but will be stored in IT's Milwaukee office when not in use. Each notebook will be identified by the project-specific document number.

The cover of each notebook will contain:

Person and Organization to whom the book is assigned
Book Number
Project Name
Start Date
End Date

Entries into the notebook will contain a variety of information. At the beginning of each entry, the date, start time, weather, all field personnel present, level of personal protection being used on-site, and the signature of the person making the entry will be entered. The names of visitors to the site and the purpose of their visit will be recorded in the field notebook.

All measurements made and samples collected will be recorded. All entries will be made in ink and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark. Wherever a sample is collected or a measurement is made, a detailed description of the location of the station will be recorded. The film roll number and number of photographs taken of the station will also be noted. All equipment used to make measurements will be identified, along with the date of calibration.

Samples will be collected following the procedures documented in this plan. The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample is collected, and the volume and number of containers into which the sample is placed in the field. Sample numbers will be assigned prior to going on-site. Significant field notebook entries shall be countersigned by another member of the project team.

A log of personnel and visitors on site will be maintained, including entry and exit times. This log may be a separate "sign-in" field notebook. Major activities being performed or other items pertinent to the history of the investigation will also be noted.

8.0 FIELD TEAM ORGANIZATION, RESPONSIBILITIES, AND TRAINING

8.1 ORGANIZATION

The field sampling team will be organized according to the sampling activity. For on-site sampling work, the actual team makeup will consist of a combination of the following:

- Project Manager
- Sampling Team Leader
- Site Safety Officer
- Hydrogeologist/Engineer
- Sampling Engineer
- Preparation Area Technician.

One person may assume more than one of the roles listed above.

Specific responsibilities and assignments of sampling team members are described below.

8.2 PROJECT MANAGER

The Project Manager (PM) will conduct the initial site briefing and be responsible for task assignments and supplying all safety equipment.

8.3 SAMPLING TEAM LEADER

The Sampling Team Leader (STL) will be responsible for the coordination of all sampling efforts, will provide for the availability and maintenance of all sampling equipment and materials, and will provide the necessary shipping and packing materials. The STL will supervise the completion of all chain-of-custody records; supervise the proper handling and shipping of the samples collected; be responsible for the accurate completion of the field notebook; and provide close coordination with the Project Manager.

8.4 SITE SAFETY OFFICE

The Site Safety Officer (SSO) will be responsible for the adherence to all site safety requirements by team members. The SSO will assist the PM in conducting the site briefing meeting. The SSO will also assist in the various sampling activities and will perform the final safety check.

Additional responsibilities will include:

- Updating equipment or procedures based upon new information gathered during the site inspection
- Upgrading or degrading the levels of protection based upon site observations
- Enforcing the "buddy system"
- Determining and posting locations and routes to medical facilities, including poison control centers; arranging for emergency transportation to medical facilities
- Notifying local public emergency officers, i.e., police and fire departments, of the nature of the team's operations and posting emergency telephone numbers
- Entering the exclusion area in emergencies when at least one other member of the field team is available to stay behind and notify emergency services; or after he/she has notified emergency services
- Examining work party members for symptoms of exposure of stress
- Providing emergency medical care and first aid as necessary on-site.

The SSO has the ultimate responsibility to stop any operation that threatens the health or safety of the team or surrounding populace.

8.5 HYDROGEOLOGIST/ENGINEER

The Hydrogeologist/Engineer will supervise drilling operations and will be responsible for ensuring that the logging requirements are met. He will also be part of the sample collection team.

8.6 SAMPLING ENGINEER

The Sampling Engineer (SE) will assist the STL in collecting surface soil and boring samples and in decontaminating all field equipment.

8.7 PREPARATION AREA TECHNICIAN

The Preparation Area Technician (PAT) will remain in the decontaminated area and will assume custody of samples from the sampling team. The PAT will be responsible for the completion of all chain-of-custody forms. The PAT will also be responsible for maintaining communications with on-site personnel.

8.8 AGENCY ROLE

It is assumed that personnel from the U.S. EPA and IEPA, if present, will be acting as observers only and will not participate directly in field sampling and related activities.

9.0 SAMPLING ACTIVITY SCHEDULE

The sampling program described in this sampling plan is expected to take two to three weeks to complete. The subsequent analyses will require a turnaround time of approximately three to four weeks. Based on the results, either the Site Assessment may be undertaken or the scope of work will be expanded and additional sampling will have to be conducted. If an additional sampling phase is deemed necessary, details pertaining to the scope of work for that phase will then be provided. The following project activities will be implemented by the milestone dates shown below:

Activity	Number of Days After Approval of QAPP
Initiate Field Activities	25
Submit Results of Phase I Analyis to U.S. EPA	70
Submit Phase II Scope of Work (if necessary) to U.S. EPA	80

APPENDIX
B

APPENDIX B

SITE SAFETY PLAN
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
RIVERDALE CHEMICAL COMPANY
CHICAGO HEIGHTS, ILLINOIS

Approved By

Harry Pullum
IT Regional Health and Safety Manager
ABIH Certification Number 2438

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1.0 INTRODUCTION

This Health and Safety Plan has been developed by IT Corporation for conducting the remedial investigation activities at the Riverdale Chemical Company's (Riverdale) plant in Chicago Heights, Illinois. The contents of this plan have been based on the manufacturing processes performed at the Riverdale facility, as well as the results of previous investigative work conducted at the site. This Health and Safety Plan will describe possible physical hazards that the remedial investigation team may encounter at the site, decontamination procedures, an emergency response plan and the employee medical monitoring program. In addition, the plan will indicate the type of protective equipment site personnel will wear in order to minimize their exposure, through inhalation or direct contact, to hazardous materials on the site. The plan will also detail the on-site monitoring program that will be conducted during the remedial investigation. Modifications of this plan may be made as additional data are gathered during subsequent site visits. At a minimum, the plan will be consistent with all applicable state and federal laws and regulations, including:

- Section 111(c) of CERCLA;
- U.S. EPA Order 1440.3 -- Respiratory Protection;
- U.S. EPA Order 1440.2 -- Health and Safety Requirements for Employees Engaged in Field Activities;
- U.S. EPA Occupational Health and Safety Manual;
- U.S. EPA Interim Standard Operating Safety Procedures and other U.S. EPA guidance as developed;
- Illinois Occupational Safety and Health Act; and
- IT Corporation Health and Safety Manual.

IT's Health and Safety Division promotes the requirements set forth in OSHA's Standard for Workplace Hazard Communication (29CFR 1910.1200, effective 1986). Through extensive internal and external training and site-specific/hazard-specific instruction, IT provides information to its employees and subcontractors concerning hazardous chemicals. This includes labels,

corporate safety standard operating procedures, Material Safety Data Sheets (MSDS), posting of site safety plans and access to other pertinent information.

2.0 SITE BACKGROUND

2.1 FACILITY DESCRIPTION

The Riverdale site encompasses approximately four to five acres and is located in the southeast section of the city of Chicago Heights, Illinois (Figures 1 and 2). On plant grounds, there exists three main buildings and a tank storage area (Figure 3). The facility has been involved since approximately 1956 primarily in the formulation of pesticides. Dioxin (2,3,7,8-tetrachlorodibenzo-p-dioxin) is a by-product of the manufacture of pesticides and other chlorinated hydrocarbons.

2.2 PREVIOUS INVESTIGATIONS AND HAZARD EVALUATION

Ecology and the Environment (E&E) conducted a Field Investigation Team (FIT) study on April 24, 1984, of the plant area as part of the National Dioxin Test Strategy and in response to concerns that contamination may be present on the site grounds with the potential for off-site migration. The FIT investigation consisted of taking 15 surface soil samples (numbered B-1 through B-15 in Figure 3). The samples were analyzed for a variety of organic compounds. The results of the analyses indicated that potentially hazardous materials were present in the surface soils at the facility and include various pesticides, volatile organic compounds (VOCs) polynuclear aromatic hydrocarbons (PAHs), phenolic compounds (phenolics) and Dioxin. The Dioxin concentrations were greatest in the soils at the far south end of the plant area.

The presence of Dioxin and the occurrence of concentrations of several pesticides found in the soils throughout the site are of concern. Many of the pesticides are listed as carcinogens, priority pollutants, and hazardous substances including aldrin, dieldrin, chlordane, heptachlor and toxaphene. Dioxin, a carcinogen, was detected at most sampling locations usually in quantities of less than 15 parts per billion (ppb) except in the soils at the south end of the plant grounds (up to 364 ppb). Several PAH's, including the listed carcinogen benzo(a) pyrene, were found primarily between buildings 2 and 3 and south of the building area. Phenolic compounds were detected in measurable concentrations only at sampling locations B-7 and B-10.

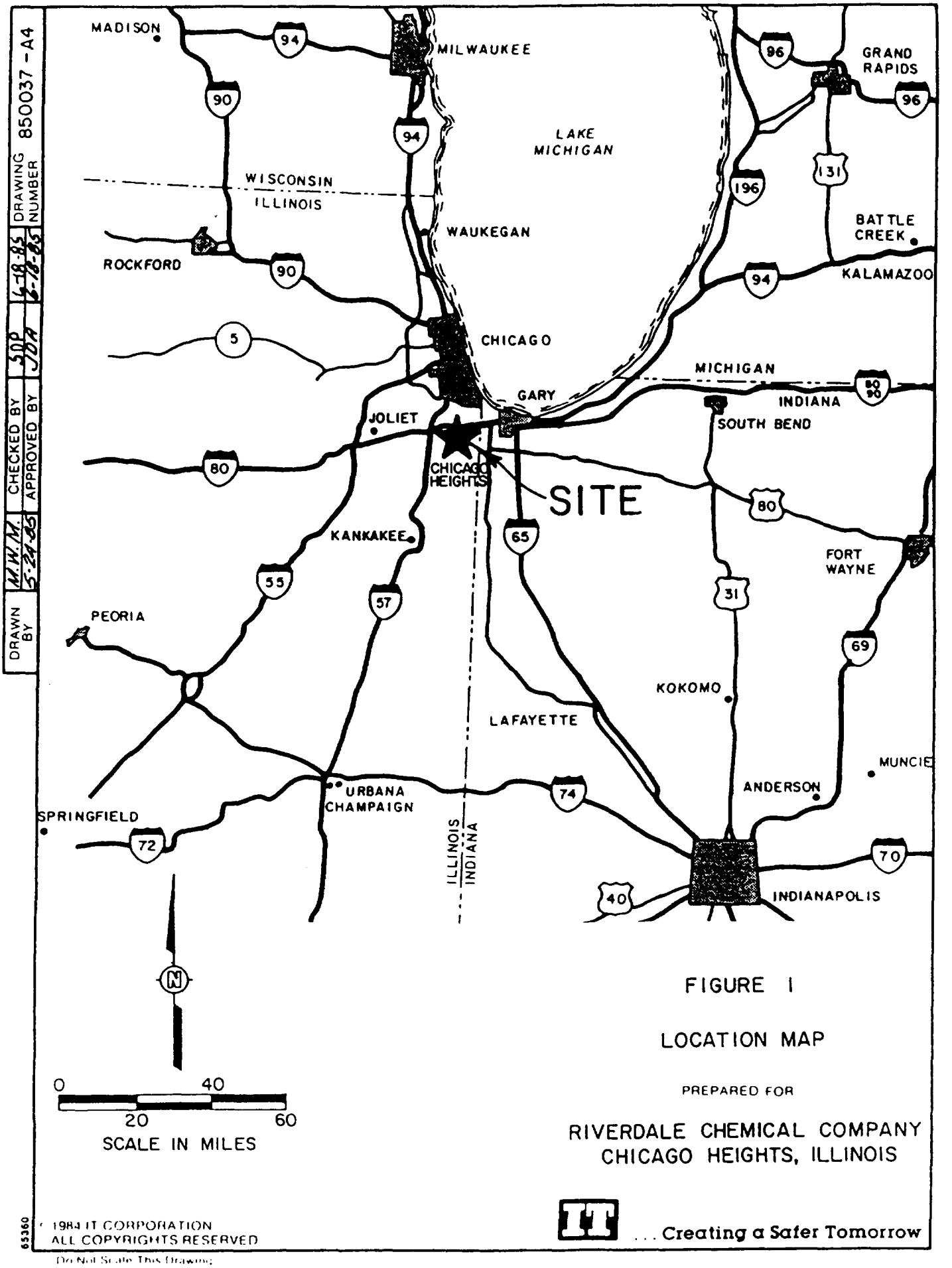

FIGURE 1

LOCATION MAP

PREPARED FOR

RIVERDALE CHEMICAL COMPANY
CHICAGO HEIGHTS, ILLINOIS

DRAWN BY M.W.M. 5-24-85
CHECKED BY SDP 6-18-85
APPROVED BY JDA 6-18-85
DRAWING NUMBER 850037-A4

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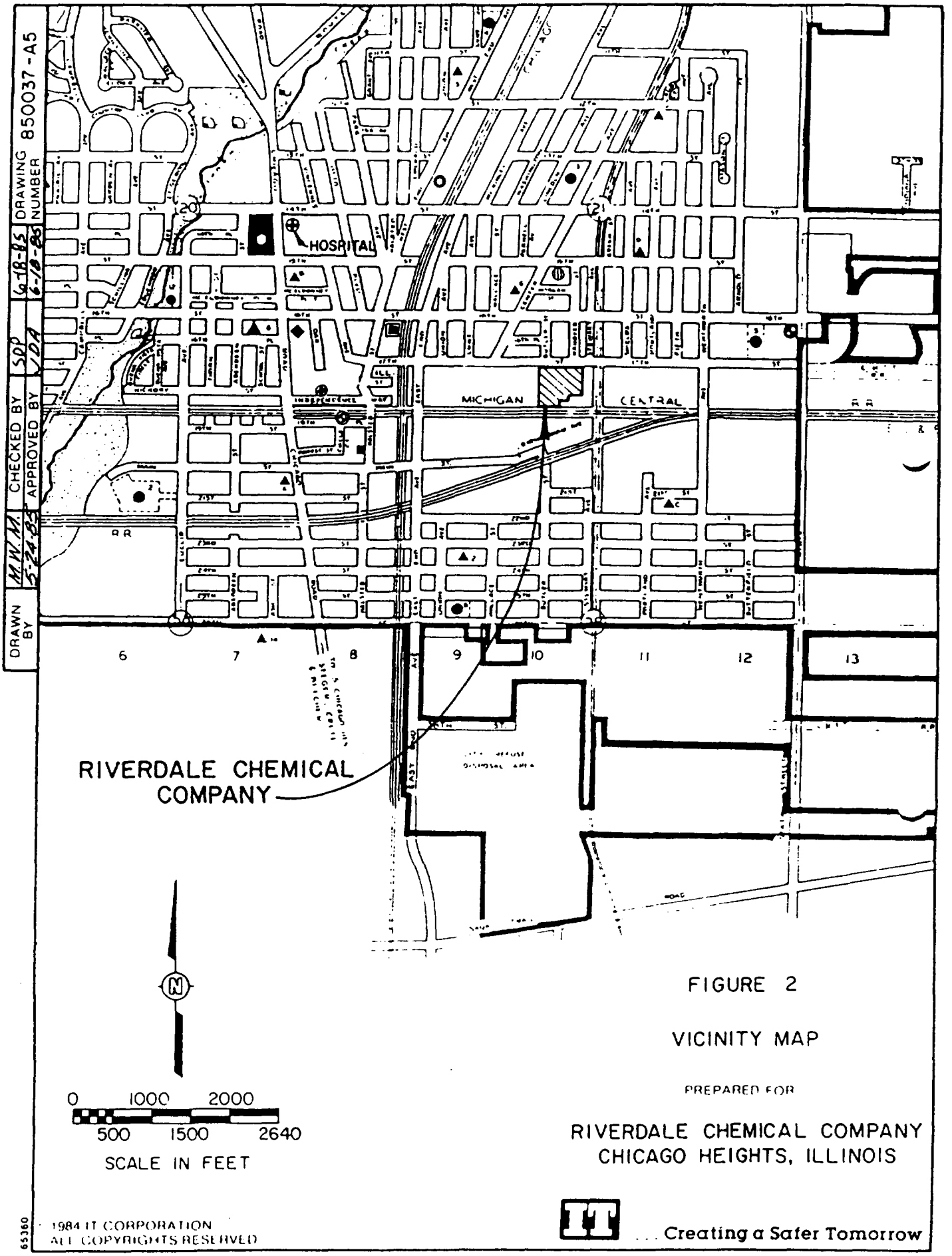
Do Not Scale This Drawing



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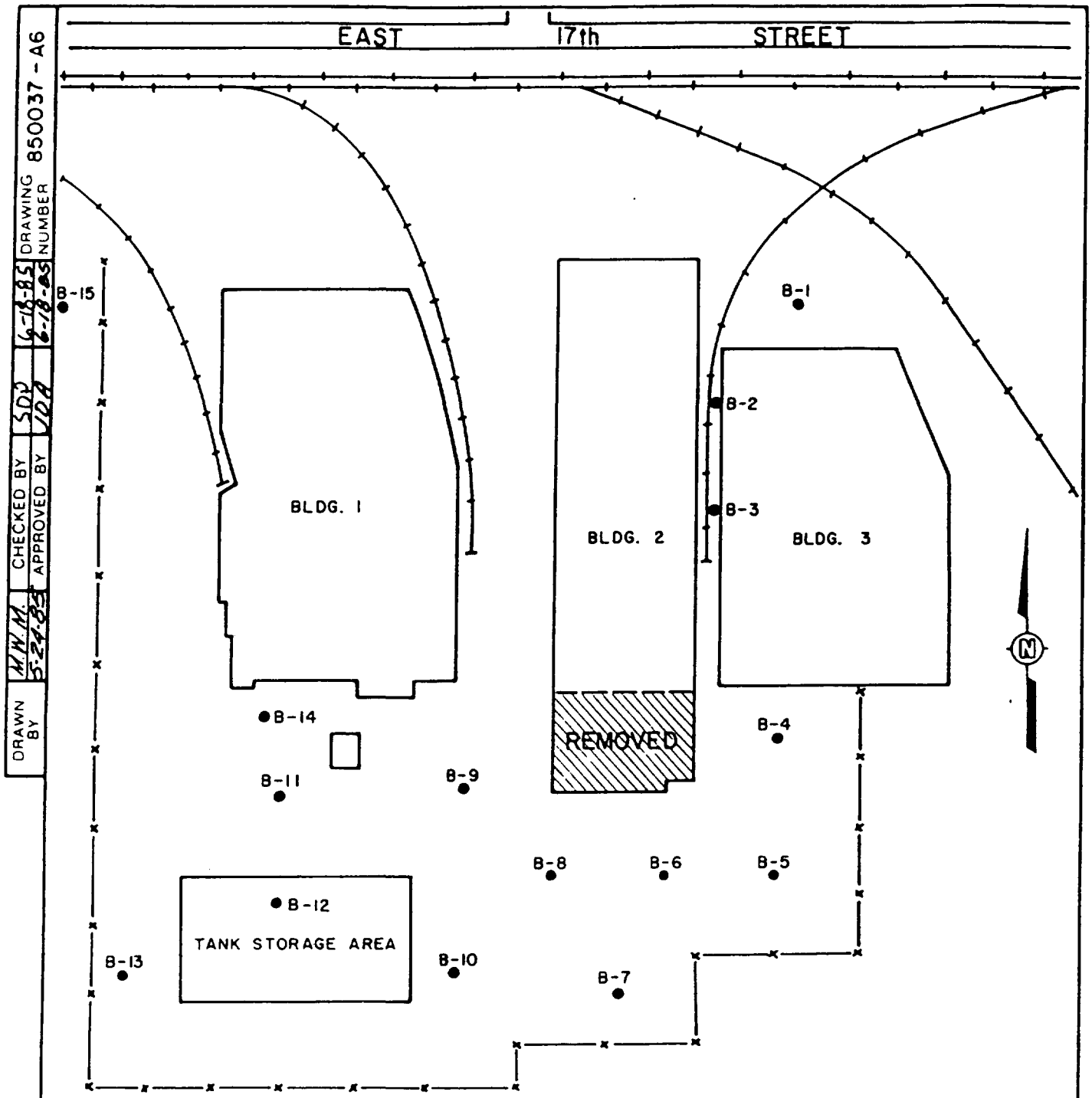

DRAWN BY	CHECKED BY	APPROVED BY	DRAWING NUMBER
M.W.M. 5-24-85	SDV 6-18-85	VDA 6-18-85	850037 - A6

FIGURE 3

SITE MAP
ECOLOGY & ENVIRONMENT
SAMPLING LOCATIONS

PREPARED FOR

RIVERDALE CHEMICAL COMPANY
CHICAGO HEIGHTS, ILLINOIS

LEGEND

B-11● SAMPLE LOCATIONS

RAILROAD

FENCE LINE

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Concentrations of VOCs were generally less than 100 parts per billion. These compounds are not expected to exist in high concentrations in the surface soils due to their volatile nature.

3.0 SITE SAFETY WORK PLAN

3.1 PURPOSE

This Health and Safety Plan prescribes workplace procedures which will be followed in order to protect the employees who are working with Dioxin, pesticides, and the other hazardous materials which may be present at the facility. The requirements listed may change as work progresses due to changing conditions, but no changes will be made without prior approval by the IT Regional Manager, Health and Safety Division. The program outlined is for both IT employees and subcontractor personnel.

Few chemicals have received the widespread notoriety that Dioxin has. "Dioxin" is a name used for compounds known chemically as dibenzo-p-dioxin. Although there are 75 different dioxin isomers, we are usually concerned with 2,3,7,8 TCDD. Dioxin is seldom mentioned or written about without the additional notation that it is one of the most toxic chemicals known to man, although the long-term health effects are not presently known. Dioxins can be breakdown products resulting from the exposure of chlorinated hydrocarbons, such as PCBs, to intense heat. (In this instance, the Dioxin was most likely generated during the production of the trichlorophenol used to manufacture 2,4,5,T and silvex acids.) Like other organochlorine compounds, such as DDT, these materials are persistent in the environment and accumulate in living tissues.

3.2 PROGRAM STRUCTURE

The Regional Manager, Health and Safety Division, will be responsible for the coordination of this plan. He, or one or more of his representatives, will be on-site for the project start-up and as necessary thereafter to supervise the worker protection program. Liaison with officers or representatives of Riverdale on matters relating to safety and health will be handled by the Health and Safety representative.

The Project Manager is responsible for field implementations of the health and safety plan, but only the Regional Manager of Health and Safety can change its provisions. This includes communicating the specific requirements to all personnel, conducting audits, and consulting with the Health and Safety

representative regarding appropriate changes in safety and health requirements.

The designated Field Health and Safety Officer will:

- Supervise the day-to-day implementation of the site-specific health and safety program.
- Train new site personnel on site specific health and safety items, interact with project personnel on health and safety matters, investigate and report accidents/incidents.
- Maintain liaison between field activities and the Project Manager.
- Perform air quality and personal monitoring as required.
- Assist the Project Manager in enforcing the requirements of this manual and the site-specific program.

All on-site personnel are responsible for understanding and complying with the requirements of this plan, and must sign a statement that they have read, understood, and will abide by the plan (Figure 4). Failure to comply with this plan will result in disciplinary action, which could lead to termination.

3.3 PERMISSIBLE EXPOSURE LIMIT GUIDELINES

Review of Dioxin risk assessments indicates that a permissible exposure limit of 18 picograms per cubic meter of air (pg/m^3) is appropriate. Therefore, until further research is done, the limit will be 18 pg/m^3 for Dioxin. Eye and skin contact as well as ingestion (improper hygiene) with Dioxin shall be prevented.

There are several other chemicals at the site to which employees may be exposed. For employees who may be exposed to these materials, the threshold limit values (TLVs) established by the ACGIH will be followed. If no TLV has been established by the ACGIH for a material in question, other sources of permissible levels will be used. Other sources would include the National Institute of Occupational Safety and Health's (NIOSH) criteria documents, the Occupational Safety and Health Administration's (OSHA) 1910.1000 standards,

FIGURE 4
EMPLOYEE HEALTH AND SAFETY PLAN CERTIFICATION
RIVERDALE CHEMICAL COMPANY
CHICAGO HEIGHTS, ILLINOIS

EMPLOYEE HEALTH AND SAFETY PLAN CERTIFICATION

Employee:

I have read and do understand the Health and Safety Plan for the _____Project. I will abide by this plan.

_____
Signature

_____
Date

toxicology textbooks, etc. The IT Regional Manager, Health and Safety Division, or his representative will evaluate appropriate data and establish personnel protection requirements. Permissible exposure limits for materials which will likely be monitored are:

- Chlordane - 0.5 milligrams per cubic meter (mg/m^3)
- Heptachlor - 0.5 mg/m^3

Where feasible, engineering controls shall be used to maintain Dioxin and other chemical exposures within the limits set forth above. This may be accomplished by the use of dust suppression techniques (i.e. misting the work area with water), coupled with protective clothing for the appropriate level for exposures.

3.4 EMPLOYEE TRAINING AND INFORMATION

All IT employees who work on site shall have completed a formal training program which shall include, as a minimum, the following:

- Basic Safety Training - This course stresses fundamentals such as the cause and prevention of slip, trip, and fall hazards, safe lifting techniques, heat stress illnesses and their prevention, etc.

- Hazards and Protection - This course deals with the identification, recognition, and safe work procedures of toxic materials. The use and limitations of applicable protective clothing, respirators, and decontamination procedures are an important part of this course. Respirator fit testing is given to each attendee.

- First Aid and Cardiopulmonary Resuscitation - A large portion of IT employees will have completed these standard Red Cross First Aid and CPR courses.

- Dioxin Hazard Awareness - This course discusses the specific nature of the operations which could result in exposure to Dioxins, a description of the medical surveillance program, the specific protective clothing and respirators to be worn on the job, the adverse health effects associated with exposure to Dioxin, and routes of exposure (skin penetration, inhalation, ingestion), and the safe work procedures associated with the employee's job assignment.

- Other Hazard Awareness - Information will be given concerning other materials to which the employee may be exposed. Information will include routes of exposure, toxic effects, appropriate protective equipment, medical surveillance, and the specific nature of the job which could result in exposure.

The following training sessions and informational materials will be provided on site for all IT and non-IT personnel.

- Tailgate Safety Meeting - A tailgate safety meeting will be conducted at the beginning of each shift or whenever new employees arrive at the job site once the job commences. This meeting discusses the health and safety considerations for that day's activities, and outlines protective equipment necessary. Figure 5 shows a copy of a Tailgate Safety Meeting form.

- Material Safety Data Sheets (MSDS) - Completed MSDS for the toxic materials present at the site shall be posted at the job site. An example of a MSDS form is attached (Figure 6).

3.5 OCCUPATIONAL HEALTH PROGRAM

All employees on this job shall have completed an IT preemployment medical examination and/or a periodic/update physical examination before assignment. The periodic/update examination must be repeated annually. The medical examination must be reviewed by a physician specializing in occupational medicine and a report must be submitted to IT which medically approves the employee for work with hazardous materials. Examples of a "medical examination report" and a "physical activity restriction" report are attached (Figures 7 and 8, respectively). The physical examination will be performed at a clinic approved by IT and results of the physical will be reviewed by IT's corporate physician.

The preemployment examinations must include at least the information included on the IT Preemployment Medical Examination form (Figure 9). The periodic/update physical examination must include at least the information included on the Update/Periodic Physical Examination form (Figure 10).

FIGURE 5
RIVERDALE CHEMICAL COMPANY
CHICAGO HEIGHTS, ILLINOIS

TAILGATE SAFETY MEETING

Division/Subsidiary _____ Facility _____

Date: _____ Time: _____ Job Number: _____

Customer _____ Address: _____

Specific Location: _____

Type of Work: _____

Chemicals Used: _____

SAFETY TOPICS PRESENTED

Protective Clothing/Equipment: _____

Chemical Hazards: _____

Physical Hazards: _____

Emergency Procedures: _____

Hospital / Clinic: _____ Phone:( ) _____ Paramedic Phone:( ) _____

Hospital Address _____

Special Equipment _____

Other _____

ATTENDEES

NAME PRINTED	SIGNATURE

Meeting conducted by: _____ _____
NAME PRINTED SIGNATURE

SUPERVISOR _____ MANAGER _____

ITC Form 9540- 1C(revised 7/81)

FIGURE 6
MATERIAL SAFETY DATA SHEET
RIVERDALE CHEMICAL COMPANY
CHICAGO HEIGHTS, ILLINOIS

U.S. DEPARTMENT OF LABOR
Occupational Safety and Health Administration

Form Approved
OMB No. 44-R1387

MATERIAL SAFETY DATA SHEET

Required under USDL Safety and Health Regulations for Ship Repairing, Shipbuilding, and Shipbreaking (29 CFR 1915, 1916, 1917)

SECTION I

MANUFACTURER'S NAME	EMERGENCY TELEPHONE NO.
ADDRESS (Number, Street, City, State, and ZIP Code)	
CHEMICAL NAME AND SYNONYMS	TRADE NAME AND SYNONYMS
CHEMICAL FAMILY	FORMULA

SECTION II - HAZARDOUS INGREDIENTS

PAINTS, PRESERVATIVES, & SOLVENTS	%	TLV (Units)	ALLOYS AND METALLIC COATINGS	%	TLV (Units)
PIGMENTS			BASE METAL		
CATALYST			ALLOYS		
VEHICLE			METALLIC COATINGS		
SOLVENTS			FILLER METAL PLUS COATING OR CORE FLUX		
ADDITIVES			OTHERS		
OTHERS					

HAZARDOUS MIXTURES OF OTHER LIQUIDS, SOLIDS, OR GASES	%	TLV (Units)

SECTION III - PHYSICAL DATA

BOILING POINT (°F.)		SPECIFIC GRAVITY (H_2O=1)	
VAPOR PRESSURE (mm Hg.)		PERCENT, VOLATILE BY VOLUME (%)	
VAPOR DENSITY (AIR=1)		EVAPORATION RATE (_____ =1)	
SOLUBILITY IN WATER			
APPEARANCE AND ODOR			

SECTION IV - FIRE AND EXPLOSION HAZARD DATA

FLASH POINT (Method used)	FLAMMABLE LIMITS	Lel	Uel
EXTINGUISHING MEDIA			
SPECIAL FIRE FIGHTING PROCEDURES			
UNUSUAL FIRE AND EXPLOSION HAZARDS			

PAGE (1)

(Continued on reverse side)

Form OSHA-20
Rev. May 73

SECTION V - HEALTH HAZARD DATA

THRESHOLD LIMIT VALUE

EFFECTS OF OVEREXPOSURE

EMERGENCY AND FIRST AID PROCEDURES

SECTION VI - REACTIVITY DATA

STABILITY	UNSTABLE		CONDITIONS TO AVOID
	STABLE		

INCOMPATABILITY (Materials to avoid)

HAZARDOUS DECOMPOSITION PRODUCTS

HAZARDOUS POLYMERIZATION	MAY OCCUR		CONDITIONS TO AVOID
	WILL NOT OCCUR		

SECTION VII - SPILL OR LEAK PROCEDURES

STEPS TO BE TAKEN IN CASE MATERIAL IS RELEASED OR SPILLED

WASTE DISPOSAL METHOD

SECTION VIII - SPECIAL PROTECTION INFORMATION

RESPIRATORY PROTECTION (Specify type)

VENTILATION	LOCAL EXHAUST	SPECIAL
	MECHANICAL (General)	OTHER

PROTECTIVE GLOVES	EYE PROTECTION

OTHER PROTECTIVE EQUIPMENT

SECTION IX - SPECIAL PRECAUTIONS

PRECAUTIONS TO BE TAKEN IN HANDLING AND STORING

OTHER PRECAUTIONS

PAGE (2)

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Rev. May 73

FIGURE 7
MEDICAL EXAMINATION REPORT
RIVERDALE CHEMICAL COMPANY
CHICAGO HEIGHTS, ILLINOIS

IT

Medical Examination Report

_____
Date

To: _____

IT Division: _____

Location: _____

_____ has received the appropriate:

- ☐ Preemployment examination
- ☐ Periodic examination
- ☐ DOT/DMV examination
- ☐ Other _____

and has been found to be:

- ☐ Acceptable: no restriction.
- ☐ Acceptable, subject to the attached "Physical Activity Restriction"*
- ☐ Not acceptable.

_____
Occupational Safety & Health Department

* For Physical Activity Restriction
1. Have employee read and sign restriction
2. Provide manager signature
3. Return form intact to the Occupational Safety & Health Department

ITC FORM 9410-1A (11/84)

FIGURE 8
PHYSICAL ACTIVITY RESTRICTION
RIVERDALE CHEMICAL COMPANY
CHICAGO HEIGHTS, ILLINOIS

IT CORPORATION

Physical Activity Restriction

EMPLOYEE'S NAME _____ DATE _____

DIVISION _____ LOCATION _____

DATE OF INJURY/EXAM _____ JOB TITLE _____

Employee is subject to the following physical activity restriction(s):

_____

_____

_____

_____

_____

_____

_____

☐ Acceptable for work under restriction stated above

☐ Not acceptable for work under restriction stated above

By _____

Date _____

Duration: ☐ Permanent

☐ Temporary

From: _____

To: _____

Employee _____ Date

Manager _____ Date

Health & Safety _____ Date

Distribution: Occupational Safety & Health
Regional Administration
Manager

ITC Form 94108
(rev. 8/83)

FIGURE 9 (PAGE 1 OF 2)
IT PREEMPLOYMENT MEDICAL EXAMINATION
RIVERDALE CHEMICAL COMPANY
CHICAGO HEIGHTS, ILLINOIS

IT CORPORATION

☐ ANALYTICAL SERVICES
☐ CORPORATION
☐ ENVIROSCIENCE
☐ ENVIRONMENTAL

☐ OIL
☐ SERVICES
☐ TRANSPORTATION
☐ NUCLEAR SERVICES

☐ BATON ROUGE
☐ BENSON RIDGE
☐ CINCINNATI
☐ EDISON

☐ HOUSTON
☐ IMPERIAL VALLEY
☐ KNOXVILLE
☐ LOS ANGELES
☐ MARTINEZ

☐ [illegible]
☐ SAN [illegible]
☐ SAN JOSE
☐ TAFT
☐ MIDLAND

PERSONAL HISTORY RECORD

NAME _____ AGE ____ SEX ____ BIRTH DATE _____ SOC. SEC. NO. _____

ADDRESS _____ CITY _____ STATE _____ ZIP _____ TELEPHONE NO. ( ) _____

APPLICANT NOTE - READ CAREFULLY

THIS INFORMATION IS TO HELP PLACE YOU IN A JOB SAFE TO YOURSELF AND TO OTHERS ACCORDING TO YOUR PHYSICAL ABILITY. ANSWER EVERY QUESTION (✓).

DO YOU OR HAVE YOU EVER HAD: (CHECK EVERY ITEM)

1 GLASSES FOR READING			28 HEMORRHOIDS			33 HIGH BLOOD PRESSURE			61 PAINFUL OR DIFFICULT JOINTS		
2 GLASSES FOR DISTANCE			29 A "BIRDER"			34 GAINED OR LOST WEIGHT			62 HEADACHES		
3 EYE TROUBLE, Other than			30 PARALYSIS			PAST YEAR			63 ASTHMATIC		
Glasses			31 HEARTBURN ATTACKS			35 BLEEDING OR BRUISING			64 A BAD BACKBONE		
4 EYE IRRITATION			32 MENTAL TROUBLE			TENDENCY			65 DIFFICULT ANGLES		
5 NOSE TROUBLE			33 BONCHITIS			36 FREQUENT DIARRHEA			66 BODY TROUBLE OR PAINFUL		
6 SINUS PAIN			34			37 FREQUENT VOMITING			FEET		
7 SINUS INFECTION			35 ALLERGIES			38 BLOOD			67 BACK TROUBLE		
8 EAR TROUBLE			36			39 STOMACH TROUBLE			68 SPINE TROUBLE		
9 EARS TROUBLE			37 SKIN TROUBLE OR RASHES			40 CHRONIC CONSTIPATION			69 RHEUMATISM (GOUT		
10 CONSIDERABLE TEETH TROUBLE			38 CHANGE IN SKIN COLOR			41 ABDOMINAL PAIN			INFECTION)		
11			39 A GOITER			42 ARTHRITIS			70 ALCOHOLISM		
12 EAR DEFECTIVE			40 CHEST PAIN			43 GALL BLADDER TROUBLE			71 DIABETES		
13 EAR TROUBLE			41 HEART TROUBLE			44 JAUNDICE			72 MALARIA		
14 SPUTUM EAR			42 CHRONIC CHEST CONDITION			45 KIDNEY TROUBLE			73 ANEMIA OR OTHER BLOOD		
15 SINUSITIS IN THE EAR			43 LUNG DISEASE			46 ULCERS			DISORDERS		
16 CONSTANTLY EAR BUZZES			44 SHORTNESS OF BREATH			47 PILES			74 CANCER		
17 NOSE TROUBLE			45 A FEELING OF TIGHTNESS IN			48 DARK URINE			75 TRACHOMA OR TROUBLE		
18 TATTOOS			CHEST			49 BED TOO OFTEN			76 ANY CHRONIC ALLERGY		
19 FREQUENT HEADACHES			46 ASTHMA			50 HAVE YOU EVER BURNED			77 BLOOD DISORDERS IN FAMILY		
20 BLOOD TROUBLE			47 RIGHT BURSAL			51 HOSPITAL CHECK			78 VACATION DURING LAST 18 YEARS		
21 SPINAL FUNCTIONS			48 BRONCHITIS			52 KIDNEY TROUBLE			FOR EMPLOYMENT OR DURING (B)		
22 BALLS CONDITION			49 ILLEGITIMATE			53 PRIVATE MEANING TROUBLE			79 NUMBER OF PREGNANCIES		
23 DEEP SPEECH			50 CHRONIC COUGH			54 VENEREAL DISEASE			100 NUMBER OF HOSPITALIZATIONS		
24 TEMPTING SPEECH			51 CHRONIC OR OF BLOOD			55 DIFFICULT FACE OR WORLDS			101 FEMALE ONLY		
25 CONVULSIONS			52 HEART TROUBLE OR A HEART			56 DIFFICULT OR PAINFUL			102 DATE LAST PREGNANCY		
26 DEFICITS			ATTACK			57			FOR FEMALE APPLICANTS		
27 LIGHT HEADED			53 FRAGMENT CHLOR			58 [illegible]			103 ARE YOUR PERIODS REGULAR		
						DESIRE			104 DATE LAST PERIOD		

EXPLAIN FULLY ITEMS CHECKED YES: _____

WHAT ILLNESS HAVE YOU HAD IN THE PAST 3 YEARS: _____

WHEN DID YOU LAST SEE A DOCTOR? _____ FOR WHAT? _____

NAME AND ADDRESS OF PERSONAL PHYSICIAN _____ DATE OF LAST VISIT _____ REASON _____

WHAT MEDICINES ARE YOU NOW TAKING? _____

HAVE YOU EVER BEEN REJECTED BECAUSE OF MEDICAL REASONS FOR MILITARY SERVICE? ☐ YES ☐ NO DATE _____ REASON _____

FOR INSURANCE? ☐ YES ☐ NO DATE _____ REASON _____

FOR EMPLOYMENT? ☐ YES ☐ NO DATE _____ REASON _____

OCCUPATIONAL HISTORY

LIST KINDS OF WORK DONE	WHAT MACHINES AND MATERIALS HAVE YOU HANDLED	NO. OF YEARS

ALL OCCUPATIONAL ILLNESS & INJURIES

DATE	NATURE OF ILLNESS OR INJURY	NAME OF COMPANY & CITY

MILITARY SERVICE - CONNECTED DISABILITIES

NATURE OF DISABILITY	%	HOT. SOC. SEC. NO.	BRANCH OF SERVICE	SERIAL NO.	CLAIM NO.	V.A. REGIONAL OFFICE

HOSPITALIZATION _____ WHY _____ WHEN _____ WHERE _____

ALL HOSPITALIZATIONS - ACCIDENTS - SICKNESS - (EXCEPT OCCUPATIONAL)

DATE	NATURE	DATE	NATURE

ALL PHYSICAL DEFECTS OR ABNORMALITIES AT THIS TIME

PARTS OF BODY	NATURE OF DEFECT

I HEREBY CERTIFY THAT TO THE BEST OF MY KNOWLEDGE THE FOREGOING ANSWERS ARE COMPLETE AND CORRECT AND I UNDERSTAND THAT ANY FALSIFICATION OF THIS RECORD IS CAUSE FOR TERMINATION. I FURTHER UNDERSTAND THAT THIS PHYSICAL EXAMINATION IS CONDUCTED FOR THE EMPLOYER AS A PREREQUISITE TO EMPLOYMENT. I AGREE THAT THIS PERSONAL AND ANY INFORMATION ACQUIRED AS A RESULT OF THIS EXAMINATION WILL BECOME THE PROPERTY OF THE EMPLOYER. I HEREBY GRANT PERMISSION TO OBTAIN RECORDS FROM MY PREVIOUS PHYSICIAN(S) OR HOSPITAL(S) WHERE I HAVE BEEN TREATED. UPON MY TERMINATION OF EMPLOYMENT I AGREE TO PARTICIPATE IN A CONCURRENT MEDICAL EXAMINATION.

JOB POSITION _____ DATE _____ SIGNATURE _____

PHYSICIAN'S REVIEW OF THE ABOVE HISTORY _____

IT Form 0410 1 (1/78)

FIGURE 9 (PAGE 2 OF 2)
IT PREEMPLOYMENT MEDICAL EXAMINATION
RIVERDALE CHEMICAL COMPANY
CHICAGO HEIGHTS, ILLINOIS

PHYSICAL EXAMINATION BY MEDICAL EXAMINER

HEIGHT	WEIGHT	BLOOD PRESS	PULSE AT REST	PULSE AFTER EXERCISE	PULSE 2 MIN. AFTER EXER	TEMP	RESP	AMBULANT ☐	RIGHT HANDED ☐	LEFT HANDED ☐

NORMAL YES	NORMAL NO	CLINICAL EXAMINATION	NOTE: DESCRIBE EVERY ABNORMALITY IN DETAIL. ENTER PERTINENT ITEM ON BEFORE EACH COMMENT
		1. GENERAL APPEARANCE	
		2. HEAD, FACE AND SCALP	
		3. EYES, PUPILS, SCLERA	
		4. EYES, FUNDOSCOPIC & MOTILITY	
		5. EYES, OCULAR TENSION	
		6. EARS, CANALS AND DRUMS	
		7. NOSE, SINUSES	
		8. MOUTH, TEETH AND PHARYNX	
		9. NECK	
		10. THORAX	
		11. LUNGS	
		12. HEART AND BLOOD VESSELS	
		13. BREASTS	
		14. ABDOMEN AND VISCERA	
		15. HERNIA	
		16. GENITALIA	
		17. ANO RECTAL	
		18. SPINE, LUMBAR AREA	
		19. UPPER EXTREMITIES	
		20. LOWER EXTREMITIES	
		21. SKIN, LYMPHATICS	
		22. NEUROLOGIC	
		23. PSYCHIATRIC	

ADDITIONAL TESTS INDICATED (LAB., X-RAY, ECG, ETC.):

URINALYSIS

SPEC. GR.	ALBUMIN	SUGAR
MICROSCOPIC		pH

X-RAY

CHEST	PSORA		14 X 17		NEGATIVE FINDING	

SUMMARY OF POSITIVE FINDS: _____

VISION

	LEFT EYE		RIGHT EYE	
DISTANCE	UNCORRECTED	CORRECTED	UNCORRECTED	CORRECTED
	20/	20/	20/	20/
NEAR DESIGNATE JAGER # USED AT 12"	UNCORRECTED	CORRECTED	UNCORRECTED	CORRECTED

DEPTH PERCEPTION GOOD ☐ POOR ☐ NONE ☐ COLOR VISION NORMAL ☐ ABNORMAL ☐

	NORMAL	ABNORMAL
CBC	☐	☐
CHEMISTRY	☐	☐
SPIROMETRY	☐	☐
ECG	☐	☐
AUDIO	☐	☐
CHEST X-RAY	☐	☐
URINALYSIS	☐	☐
SUITABLE FOR WORK WITH CONDITIONS	☐	☐
SUITABLE FOR WORK WITH RESTRICTIONS	☐	☐
OTHER	☐	☐
	☐	☐

RESTRICTIONS

AS NOTED BELOW ☐ NONE ☐

LIMITATIONS: Not to be assigned to any job requiring:

☐ 1. ANYTHING OVERHEAD THAN SEATED WORK.
☐ 2. ANY LIFTING OVER _____ POUNDS.
☐ 3. REPEATED BENDING OR CRAMPED POSITIONS.
☐ 4. PROLONGED KNEELING OR SQUATTING.
☐ 5. ANY KNEELING OR SQUATTING.
☐ 6. CONTINUOUS WALKING.
☐ 7. WALKING UP ANY STEPS.
☐ 8. WALKING UP STEPS OTHER THAN TO WORK LEVEL.
☐ 9. CONTINUOUS STANDING.
☐ 10. WORK ON LADDERS OR OVERHEAD.
☐ 11. ARMS ABOVE SHOULDER LEVEL.
☐ 12. EXPOSURE TO DUST INCLUDING DUSTING OF METALS OR PLASTICS.
☐ 13. EXPOSURE TO AIRBORNE CHEMICALS.
☐ 14. ACCURATE FAR VISION.
☐ 15. ACCURATE NEAR VISION.
☐ 16. TRUE COLOR PERCEPTION.
☐ 17. TRUE DEPTH PERCEPTION.
☐ 18. SQUEEZING OF HAND TOOLS.
☐ 19. GRAB HEARING.
☐ 20. EXPOSURE TO EXCESSIVE NOISE
☐ 21. WORK AROUND MOVING MACHINERY.
☐ 22. OPERATION OF MOVING MACHINERY
☐ 23. OPERATION OF MOTOR VEHICLE
☐ 24. USE OF RESPIRATORY TOOLS
☐ 25. EXPOSURE TO SOLVENTS, GREASES, THINNERS OR SEALERS.
☐ 26. EXPOSURE TO OILS, ASBESTOS, OR FIBERGLASS.
☐ 27. BICYCLE RIDING.
☐ 28. MENTAL TENSION OR RESPONSIBILITY.
☐ 29. ANY INDUSTRIAL TYPE OF PHYSICAL ACTIVITY.
☐ 30. ALTITUDE OVER _____ FT.
☐ 31. LIMIT USE OF _____.

THIS PERSON MUST WEAR:

☐ PROPERLY FITTED GLASSES.
☐ PROPERLY FITTED GOGGLES.
☐ PROPERLY FITTED TOOLS.

REMARKS _____

EXAMINING PHYSICIAN

NAME (Please Print) _____ PROFESSIONAL DEGREE

SIGNATURE _____

ADDRESS _____ CITY _____ STATE _____

PHONE NUMBER _____

FIG. 10
UPDATE/PERIODIC PHYSICAL EXAMINATION
RIVERDALE CHEMICAL COMPANY
CHICAGO HEIGHTS, ILLINOIS

IT CORPORATION

☐ Analytical ☐ Nuclear Services ☐ Detroit Range ☐ Knoxville ☐ Phoenix
☐ Corporation ☐ Oil ☐ Denver Range ☐ Los Angeles ☐ San Diego
☐ Environmental ☐ Services ☐ Chemical ☐ Houston ☐ San Jose
☐ Environmental ☐ Transportation ☐ Albany ☐ Tulsa
☐ Washington, DC

UPDATE/PERIODIC PHYSICAL EXAMINATION

NAME _____ WORK POSITION _____ SEX ____ AGE ____

ADDRESS _____ CITY _____ STATE ____ ZIP ____

BIRTH DATE _____ SS# _____ PHONE NO. ( )

IT IS IMPORTANT TO BRING TO THE ATTENTION OF THE DOCTOR ANY CHANGES IN YOUR HEALTH STATUS OCCURRING SINCE YOUR LAST HEALTH EXAMINATION. THEREFORE, PLEASE CAREFULLY ANSWER THE FOLLOWING QUESTIONS. EXPLAIN ALL ITEMS CHECKED YES BELOW.

	YES	NO
1. Have you had any injury or illness other than colds?		
2. Have you been hospitalized for any reason?		
3. Have you had any surgery (in or out of the hospital)?		
4. Have you had any abnormal x-rays or electrocardiograms; any abnormal blood, urine, or laboratory tests?		
5. Have you been nervous, depressed, or tense or had any emotional trouble?		
6. Have you had headaches, dizzy spells or black-outs?		
7. Have you had trouble with your eyes — change in vision, blurring, seeing double, pain or glaucoma?		
8. Have you had trouble with nose and throat — persistent hoarseness, voice change?		
9. Have you had trouble with ears — pain, change in hearing, noise exposure?		
10. Have you had chronic cough; any sputum that was bloody, excessive or colored any pain on breathing?		
11. Have you had shortness of breath, difficulty breathing, any swelling of the ankles?		
12. Have you had any chest pain, or history of heart trouble or high blood pressure?		
13. Have you had abdominal pain (or distress) or persistent vomiting?		
14. Have you had any change in bowel habits; any bloody or tarry black stools?		
15. Have you had trouble starting, stopping, or holding urine, any bloody or very dark urine, any discharge?		
16. Are you now taking any medication?		
17. Have you had skin rashes, sores, new or growing lumps, or changing moles?		
18. Have you gained or lost 10 pounds or more?		
19. Have you had any pain, swelling or stiffness of back or joints?		
20. Have you been bleeding or bruising more than at time of last examination?		
21. Has there been any change in the family medical history, such as appearance of diabetes, heart disease, strokes, blood problems or conditions that you think may be inherited?		
22. Are you a Diabetic?		
23. Are you pregnant?		
24. Have you had a Tetanus immunization within the last 10 years?		

25. If you still smoke, how much? _____ How many years have you smoked? _____
26. Present use of alcohol: social ____ moderate ____ heavy ____
27. For employee or spouse(s), number of pregnancies ____ number of miscarriages ____
28. If you have any medical problems you wish to discuss, please write them in.

EXPLAIN ITEMS CHECKED YES

_____

I hereby certify that to the best of my knowledge the foregoing answers are complete and correct and I understand that any falsification of this record is cause for termination. I agree that this form and any information acquired as a result of this examination will become the property of the employer. I hereby grant permission to obtain records from my previous physician(s) or hospital(s) where I have been treated.

SIGNATURE _____ DATE _____

MEDICAL EXAMINATION

HEIGHT ____ WEIGHT ____ RESPIRATORY ____ B.P. ____ PULSE ____ TEMPERATURE ____

	WITHOUT GLASSES RIGHT	WITHOUT GLASSES LEFT	GLASSES/CONTACTS RIGHT	GLASSES/CONTACTS LEFT
1. DISTANT VISION SNELLEN	20/	20/	20/	20/
2. NEAR VISION SLOAN	20/	20/	20/	20/

	NORMAL YES	NORMAL NO	TEST NOT PERFORMED	MEDICAL EXAMINER'S FINDINGS
1. Psychiatric — Neurologic				
2. General Appearance — Physique				
3. Head & Neck				
4. Chest				
5. Cardiovascular				
6. Abdomen				
7. Back				
8. Hernia — Genitalia				
9. Arms — Legs				
10. Skin				
11. Rectal				
LABORATORY				
1. Urinalysis				
2. Complete Blood Count				
3. Chemistry Panel				
4. Audiometry				
5. Spirometry				
6. EKG				
7. Chest X-ray				
8. Back X-ray				
9. DOT/DMV				
10. May wear respirator				
11. May work with carcinogens				

SUMMARY OF EXAMINATION — WORK RESTRICTIONS — RECOMMENDATIONS

_____

EXAMINING PHYSICIAN

NAME (Please Print) _____ PROFESSIONAL DEGREE

SIGNATURE _____ DATE _____ PHONE NO. _____

ADDRESS _____ CITY _____ STATE ____ ZIP ____

In the event of any injury or accident, a "Supervisor Employee Injury Report" (Figure 11) shall be completed as soon as practical by a supervisor after the event. This shall be reviewed by the Project Manager and the Health and Safety Officer on-site. If an employee has been absent due to a work related injury or illness, a "Return to Work Authorization Following Medical Absence" form (Figure 12) must be completed before returning to their job assignment. This form shall have a medical release from a physician attached. If appropriate, a "physical activity restriction" report shall be included.

The doctor and clinic selected by IT to conduct employee medical examinations for the Milwaukee Office are:

Dr. J. Steven Moore, M.D.
Chief of Occupational Medicine
The Medical-Surgical Clinic, S.C.
2400 Lisbon Avenue
Milwaukee, Wisconsin 53215
414-671-7000

3.6 REGULATED AREAS

The site, where possible, shall be divided into three well-delineated zones, as follows:

- Contaminated Zone - This zone includes the actual areas of contamination. This zone has the highest inhalation exposure potential and/or presents a high probability of skin contact with cutaneous or percutaneous effecting chemicals. The zone will usually exist in the immediate area around the drill rig.

- Contamination Reduction Zone - This zone includes the areas immediately surrounding the contamination zone. This zone has the next highest inhalation hazard, but does not have a high probability of skin contact with cutaneous or percutaneous affecting chemicals.

FIGURE 11
RIVERDALE CHEMICAL COMPANY
CHICAGO HEIGHTS, ILLINOIS

IT CORPORATION

SUPERVISORS EMPLOYEE INJURY REPORT

☐ State Compensation
☐ Federal L & H

Date Received

Date Employer's First Submitted

This is an official document to be initiated by the Supervisor. Be thorough and accurate. Answer all questions.

Injured's Name _____ Sex ____ SS # _____ Birthdate _____

Home Address _____ City _____ State _____ Phone _____

Classification _____ Hourly Wage _____ Hire Date _____

IT Division _____ Location _____

Who work was being performed for? _____ Address _____

Exact location of incident _____

Date of Injury _____ Time Shift Began _____ Time Injured _____

Time Reported _____ Did employee leave work? _____ Left When? _____

Date & Time Employee Returned To Work _____

Supervisor/foreman _____ Leadman _____

Nature of Injury _____ Exact Body Part Affected _____

☐ NEAR MISS ☐ FIRST AID ☐ DOCTOR CASE ☐ HOSPITALIZED

Doctor's Name _____ Address _____ City _____ State _____

Hospital _____ Address _____ City _____ State _____

What was employee doing at time of incident? _____

How did incident occur? _____

Incident witnesses _____ Statements Attached ☐ Yes ☐ No Did you witness incident? ☐ Yes ☐ No

Why did incident occur? _____

Has corrective action been initiated to prevent recurrence? ☐ Yes ☐ No Explain _____

Supervisor's Signature | Date Report Prepared | Injured's Signature | Date

MANAGER

What unsafe condition or act caused incident? _____

Your recommendation? _____

Signature | Date

OCCUPATIONAL SAFETY & HEALTH

Concur with action taken? ☐ Yes ☐ No Remarks _____

Signature | Date

Distribution Insurance
Safety
Manager

IC FORM 9300 1 1 (January 1983)

FIGURE 12
RETURN TO WORK AUTHORIZATION
RIVERDALE CHEMICAL COMPANY
CHICAGO HEIGHTS, ILLINOIS

IT CORPORATION

_____

RETURN TO WORK AUTHORIZATION
FOLLOWING MEDICAL ABSENCE

_____
Date

To: _____

Company: _____

Location: _____

_____ has been absent from work due to a:

- ☐ work related illness or injury _____ (Date of injury)
- ☐ nonindustrial illness or injury
- ☐ other

and has provided a satisfactory medical release certificate for:

- ☐ return to work, without restriction
- ☐ return to work subject to the attached "Physical Activity Restriction"*

_____
Occupational Safety and Health

*For "Physical Activity Restriction":
1. Have employee read and sign restriction
2. Provide manager signature
3. Retain a copy
4. Return to the Occupational Safety & Health Department

Distribution: Occupational Safety & Health
Regional Administration
Corporate Personnel
Manager

ITC 9001-T (replaces 10-144)

- o Clean Zone - This zone covers all areas outside of the contamination reduction zone. Adverse exposure to chemicals is unlikely.

Access to Dioxin contamination work areas (contaminated and contamination reduction zones) shall be regulated and limited to authorized persons in proper protective gear. A daily roster shall be kept of all persons entering and leaving such areas.

Posting - Warning signs shall be affixed in readily visible locations in or near Dioxin work areas and at the entrances. The information contained thereon shall be arranged as in the following example:

CAUTION
DIOXIN CONTAMINATED AREA
AUTHORIZED PERSONNEL ONLY

3.7 EMPLOYEE PROTECTION

The following two sections detail the level and components of protection for site personnel during the initial phase of sampling. Due to the types of contaminants present at the site, Level C protection is prescribed for all drilling and sampling at the site.

3.7.1 Respiratory Protection

The following respiratory protection program will be followed in the initial phase of the remedial investigation:

- o The respiratory protection to be used during drilling and sampling will be full-face air purifying respirators with organic vapor and highly toxic particulate (HEPA) cartridges.
- o The respiratory protection will be upgraded if the Health and Safety Officer on-site observes an operation which could potentially result in exposures above that level which air purifying respirators would protect the employee, or if industrial hygiene monitoring results

reveal concentrations above 1.0 mg/m^3 in air for chlordane and/or heptachlor, or above the detection level (approximately 400 pg/m^3) for Dioxin, then air supplied respirators will be necessary.

- Air purifying cartridges shall be replaced at the end of each shift.
- Only employees who have had preissue qualitative fit tests, and annual fit tests thereafter, shall be allowed to work in atmospheres where respirators are required.
- If an employee has demonstrated difficulty in breathing during the fit test or during use, he or she shall have a physical examination to determine whether the employee can wear a respirator while performing the required duty.
- No employee shall be assigned to tasks requiring the use of respirators if, based on the most recent examination, a physician determines that the employee will be unable to function normally wearing a respirator or that the health or safety of the employee or other employees will be impaired by use of a respirator.
- The employee shall be permitted to change cartridges whenever an increase in breathing resistance is detected.

3.7.2 Protective Clothing

The protective apparel to be worn during activities will be:

- Polyethylene coated Tyvek disposal coveralls with hoods,
- White uncoated Tyvek disposable coverall as an undergarment,
- Nitrile or neoprene gloves with latex surgical undergloves,
- Polyvinyl chloride (PVC) or neoprene steel-toed boots with latex boot covers, and
- Hardhat (with chin strap-optional).

The protective clothing sleeves will be taped to the gloves, the legs taped to the boots, and the hood to the respirator; all openings shall be sealed. The protective coveralls shall be removed in the inside out fashion.

3.8 EMPLOYEE DECONTAMINATION

The following employee decontamination will be followed:

- A decontamination area shall be positioned at the entrance of the contamination reduction zone with a step-off area just inside the contamination reduction zone. All persons entering the contamination zones shall pass through the decontamination area to don protective clothing. All persons leaving the contamination zones shall pass through the area to remove the protective clothing before donning their street clothing.
- All employees shall be required to shower at the end of the work shift.
- An area shall be designated as the break area. Employees shall wash their face and hands before eating, drinking, or smoking.
- Eyewashes and showers shall be provided at strategic locations in the immediate work area for employees who may come into contact with contaminated materials.
- If there is a rip or tear in an employee's protective clothing and if there was contact with potentially contaminated material, the employee shall return to the decontamination area immediately, wash the affected skin area, and report the incident to the on-site Health and Safety Officer. The officer will then determine and authorize, if appropriate, the employee to don new protective clothing and return to the work area.

3.9 MONITORING

Industrial hygiene monitoring will be conducted at the beginning of the job and periodically thereafter to determine the employees' exposures to Dioxin and other materials. The results of the monitoring will dictate the selection and subsequent changes of the personal protective equipment to be used in initial site sampling activities.

There are no established or published procedures for industrial hygiene monitoring of Dioxins. The method outlined below is an internal IT Corporation procedure which has been found to be acceptable by clients and government agencies, including U.S. EPA (Regions II and VII) and the New Jersey Department of Environmental Protection. The procedure was developed by certified industrial hygienists and has been successfully used in the field for more than one year.

The industrial hygiene Dioxin samples will be collected by using a personal pump sampling air at a rate of 3.0 liters per minute or greater. The Dioxin will be collected on a glass fiber filter in a three-piece cassette, operated in the open face mode. A minimum sampling time of 420 minutes (1,260 liters) will be necessary. Appropriate analytical methods can detect a level of 0.5 nanograms per sample; therefore, the lower detection limit will be 0.4 nanograms per cubic meter for 1,260 liters of air collected.

Since Dioxin has low vapor pressures, very little gaseous phase Dioxin is expected to be present. Therefore, sorbent tube samples will only be taken during the early phases of the job (baseline and during solvent use). If no Dioxin is detected or if correlations can be made concerning percent breakthrough, this sampling method will be discontinued. To obtain these samples, XAD-2 sorbent tubes will be used as a backup to the filters described previously. Therefore, a similar volume of air will be sampled and detection limits will be the same.

The Dioxin samples taken will be prepared for analysis by extraction of the filters (or sorbent tubes) with subsequent cleanup by liquid chromatography. Analysis will be high resolution gas chromatography/low resolution mass spectrometry operating in a selected ion mode.

Chlordane and heptachlor will also be sampled due to their high concentration in the soil. The results of these samples will be used as an indicator for

all contaminants on the site. Therefore, if levels for Dioxin, chlordane, and heptachlor are at concentrations at which protective equipment is adequate, it will be assumed that the employee will be protected for all contaminants on the site.

The quality assurance/quality control procedures will be as follows:

- The personal sampling pumps will be calibrated before and after each use with the standard bubble meter. All volumes will be adjusted to standard temperature and pressure, if necessary.
- A chain-of-custody form will be kept for each sample. The form will be signed by the person taking the sample, the person(s) transferring the sample to the laboratory, and by the laboratory receiving the sample.
- There will be one blank filter and one blank spiked with 0.5 to 2.5 nanograms of labeled Dioxin included with each set of Dioxin samples sent to the laboratory. These blanks will have been prepared by the laboratory. The blanks and spikes will be labeled in such a way as to be indistinguishable from the field sample. The blanks and spikes will be utilized to determine precision and accuracy of the laboratory.

Low concentrations of VOCs were detected in the soils at the site. Therefore, intermittent air monitoring using an HNu (11.7 lamp) or an organic vapor analyzer (OVA) will be conducted during drilling operations. Head space analysis using the HNu/OVA will also be performed on the soil samples which are collected. The maximum values for each reading will be recorded in the field notebook and on the appropriate sampling forms.

3.10 GENERAL WORK PRACTICES

The following work practices will be adhered to during the remedial investigation:

- Contaminated protective equipment, such as respirators, hoses, boots, etc., shall not be removed from the regulated area until it has been properly cleaned, packaged and labeled.

- Legible and understandable precautionary labels shall be affixed prominently to containers of contaminated scrap, waste, debris, and clothing.

- Contaminated materials shall be stored in tightly-closed containers in well-ventilated areas.

- Containers shall be moved only with the proper equipment and shall be secured to prevent dropping or loss of control during transport.

- Removal of Dioxin contaminated particulates from protective clothing or equipment by blowing, shaking, or any other means which disperses contaminants into the air is prohibited.

- No food or beverages shall be present or consumed in the regulated area.

- No tobacco products shall be present or used, and cosmetics shall not be applied in the regulated area.

- Emergency equipment shall be located outside storage areas in readily accessible locations which will remain minimally contaminated in an emergency.

- All areas that have been determined as uncontaminated inside the regulated area will be clearly marked as such. No personnel, equipment, etc., shall be in these areas until they have been decontaminated.

3.11 HEAT STRESS

The heat stress of employees on site may be monitored by the Wet Bulb Globe Temperature Index (WBGT) technique. This method will require the use of a heat stress monitoring device, such as the Wibget Heat Stress Monitor (Reuter Stokes).

The WBGT shall be compared to the Threshold Limit Values (TLV) outlined in the ACGIH TLV's Manual, and a work-rest regimen established, as necessary, according to the WBGT obtained. Note that 3°C must be subtracted from the TLVs for

heat stress listed to compensate for the wearing of impermeable protective clothing.

The Health and Safety Officer shall continuously visually monitor all personnel to note for signs of heat stress. In addition, all field personnel will be instructed to observe for symptoms of heat stress and methods on how to control it. One or more of the following control measures can be used to help control heat stress:

- Provision of adequate liquids to replace lost body fluids. Employees must replace water and salt lost from sweating. Employees must be encouraged to drink more than the amount required to satisfy thirst. Thirst satisfaction is not an accurate indicator of adequate salt and fluid replacement.
- Replacement fluids can be a 0.1% salt water solution, commercial mixes such as Gatorade or Quick Kick, or a combination of these with fresh water. Employees should be encouraged to salt their foods more heavily.
- Establishment of a work regimen that will provide adequate rest periods for cooling down. This may require additional shifts of workers.
- Cooling devices such as vortex tubes or cooling vests can be worn beneath protective garments.
- All breaks are to be taken in a cool rest area (77°F is best).
- Employees shall remove impermeable protective garments during rest periods.
- Employees shall not be assigned other tasks during rest periods.
- All employees shall be informed of the importance of adequate rest, acclimation, and proper diet in the prevention of heat stress.

3.12 EMERGENCY PROCEDURES

In the event of an emergency such as an injury/illness or fire, the following procedures will be followed:

- Emergency procedures must be initiated by the first person recognizing the emergency situation.

- Air horns or similar warning devices must be located in the area of each work crew.

- Two blasts of the warning system indicates an injury or illness at the location of the blasts. A designated two-man crew will proceed to the area to render assistance to the injured person. The members of this crew will have had first aid and CPR training (see Training Section of the Health and Safety Plan) and will be wearing all appropriate protective equipment. First aid kits will be strategically located to assist the crew with the injury/illness. At the same time personnel will contact the ambulance service and hospital listed below.

- A continuous blast of the warning system indicates that the area or building needs to be evacuated. All employees will assemble in the regulated area in the vicinity of the decontamination unit. The senior management employee on site will act as assembly coordinator and direct further actions.

- The Riverdale Chemical Company emergency procedures will be followed. All employees on-site will be instructed on these procedures.

- Once the nature of the emergency is known, Riverdale will be notified. If it is determined that outside services are warranted, and Riverdale authorizes IT, the appropriate agencies listed below will be notified by the clerk:

- - Hospital
  St. James Hospital
  Chicago Road and Lincoln Highway
  Chicago Heights, Illinois
  312-756-1000

  - Ambulance Service
  312-754-2121

- Fire Department
  312-754-2121

- Police Department
  312-754-2121

- U.S. EPA
  Region V Emergency Remedial Response
  312-353-9773

o The IT Health and Safety Officer will be notified as soon as possible.

o Facilities will be located in the work area for employees to be able to immediately wash any affected skin area or the eyes if this comes into contact with contaminated materials.

o If an injury/illness is the result of a chemical exposure, steps shall be made to identify the chemical, and this information will be given to the attending physician.

In addition, the health concerns for the community are of utmost priority. In the unlikely event that there is an emergency which could affect the health of the community, the following agencies will be notified, as appropriate:

- Fire Department
  Chicago Heights
  312-754-2121

- Police Department
  Chicago Heights
  312-754-2121

- U.S. EPA Region V
  312-353-9773

- U.S. Coast Guard
  800-424-8802

APPENDIX
C

STANDARD OPERATING PROCEDURE

IT CORPORATION

IT ANALYTICAL SERVICES

STEWART LABORATORIES DIVISION

TITLE:	SOP NO: CD-841010R0 DATE INITIATED: 10/10/84 REVISION NO: 0 DATE REVISED: PAGE 1 of 4
Receipt and Logging in of EPA Contract Laboratory Program Samples	

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
JES	Jack R Mall	12-19-84	Gary H Woody	12/19/84

1.0 Sample Receipt

1.1 Samples Received on Weekends

1.1.1 After notification from the Sample Management Office (SMO) that samples are to be received on a weekend, the ITAS CLP Project Manager assigns weekend sample receipt to a specific lab employee.

1.1.2 Upon delivery of samples, the designated lab employee must

1.1.2.1 Sign for shipment after verifying that the number of packages received agrees with airbill/waybill.

1.1.2.2 Fill in date/time samples received on Sample Receipt Log (Figure 1).

1.1.2.3 Check ice chests for the presence and condition of Custody Seals. A Custody Seal should be positioned so that it would have to be cut, broken, or somehow disrupted for the ice chest to be opened. The condition of the Custody Seal, intact or disrupted, is noted on the Sample Receipt Log.

1.1.2.4 Move ice chest(s) to the refrigerated EPA-CLP sample storage area. Sign and date the Sample Receipt Log-relinquishing chain of custody to storage.

1.1.2.5 Place shipping papers and the sample Receipt Log in Coding Room.

1.1.3 The following Monday (or next regular work day), coding personnel must .

1.1.3.1 Remove ice chests from refrigerated storage and move to the Coding Room, documenting chain of custody on Sample Receipt Log.

1.1.3.2 Review existing sample receipt information and contact the SMO if Custody Seals have been disrupted.

1.2 Samples Received on Weekdays

1.2.1 Coding personnel are notified as soon as sample shipments arrive and must

1.2.1.1 Sign for shipment after verifying that the number of packages received agrees with airbill/waybill.

1.2.1.2 Fill in date/time samples received on Sample Receipt Log (Figure 1).

1.2.1.3 Check ice chests for the presence, position, and condition of Custody Seals. A Custody Seal should be positioned so that it would have to be cut, broken, or somehow disrupted for the ice chest to be opened. The condition of the Custody Seal, intact or disrupted, is noted on the Sample Receipt Log. If Seal has been disrupted, notify the SMO before proceeding with coding.

1.2.1.4 Move ice chest(s) and shipping papers to the Coding Room.

2.0 Project/Sample Log In

2.1 Samples received on a given day are grouped together by EPA Case No., under an ITAS Project Code consisting of the 4-letter client code EPAL and a 5-digit number. The 4-letter client code is client specific and the 5-digit number is sequentially assigned as Projects are coded in. Both the Project Code and the EPA Case No. are entered on the Sample Receipt Log.

2.2 Ice chests are opened under a vented hood immediately and samples inspected as to sample condition and presence/condition of Custody Seals. This information is then entered on the Sample Receipt Log and the accompanying EPA Organics Traffic Reports. The coding personnel signs each Organics Traffic Report and enters the date sample was received.

2.3 Each sample in a Project is given an ITAS sample number consisting of a one-letter prefix and a four-digit number. Pre-numbered label tape is affixed to each sample container. These numbers are sequentially assigned as they are coded in.. Samples are processed through the laboratory by sample number. The following sample information is entered on the Sample Receipt Log:

- -EPA Sample No.
- -Sample Type (from Traffic Report)
- -Sample Concentration (from Traffic Report)
- -Extract Tag No.
- -ITAS Extract Sample No.
- -Where Extract Sample Stored
- -VOA Tag No.
- -ITAS VOA Sample No.
- -Where VOA Sample Stored
- -VOA Condition (no. of vials received/no. of vials without air bubbles)
- -If sample information does not agree between Traffic Reports, Sample Tags, and Chain of Custody forms, the "NA" column is checked and the SMO office contacted. Details on discrepancies are entered in the "Notes" column.

2.4 Samples are then stored under refrigeration in secured refrigerator space designated for CLP samples only. The Sample Receipt Log is signed and dated--relinquishing chain of custody to storage.

2.5 Project/Sample information is then entered into the ITAS customized Perkin-Elmer LIMS 2000 computer data base. After the Project is entered into the computer, a Project/Sample information output is printed (Figure 2).

2.5.1 The following information is entered for the PROJECT CODE items:

Item 2-DUE DATE: (30 days from receipt of samples)

Item 4-CHAIN OF CUSTODY?: (Y)

Item 7-SPECIAL QC?: (Y, ?

Item 8-CLIENT TYPE (C,G,I)?: (G)

Item 15-PURCHASE ORDER NO.: (Enter EPA Case No.)

Item 16-CONTRACT NO.: ?

Item 18-JOB CODE: (EPAL001)

Items 31,32,33-NOTES: (Use the following test assignments for CLP samples:

?

2.5.2 Answer "Y" to PRL prompt, "Activate Data Entry Into Sort Fields (Y/N)?".

2.5.3 The following information is entered for SAMPLE items:

Item 1-SAMPLE TYPE: (Determine appropriate ITAS code from Organics Traffic Report items 3, Sample Matrix, and 8, Sample Description)

Item 3-SPEC SAMPLE DISPOSAL: (Enter "Do not dispose of sample without authorization from ITAS CLP Project Manager")

Item 4-NOTES: (Enter "low concentration" or "medium concentration", as checked in Item 2 of the Organics Traffic Report)

Item 8-SAMPLE DESCRIPTION: (Enter the description found in the first column of Item 6 on the Organics Traffic Report, i.e. "Water (Ext.)" or "Water (VOA)", etc.

Item 12-CLIENT SAMPLE NO.: (Enter "EPA Case No.-EPA Sample No.", i.e. "7740-C4401")

Item 13-SORT CODE 1: (Enter EPA Case No.)

Item 14-SORT CODE 2: (Enter EPA Sample No.)

Item 22-FIELD SAMPLE NO.: (Enter EPA Tag No.)

2.6 Each Project is assigned a file folder labeled with the Project Code and EPA Case No. All coding information printouts, Chain of Custody forms, Traffic Reports, Sample Receipt Logs, EPA Sample Tags, and shipping papers are filed in the Project Folder. These Project Folders are then filed in the in-coming Project box in the room set aside for EPA-CLP document storage and administrative activities.

-K-CDU[illegible])

SAMPLE RECEIPT LOG
EPA CONTRACTUAL SAMPLES
ITAS-Knoxville

ect Code _____
ract # _____
ase # _____

Date/Time Received _____
Required Turnaround _____

Sample Condition
Cold Yes__ No__
Intact Yes__ No__

dy Seals	On Ice Chests	On Samples	Forms	Received	Agree*
nt	Yes___ No___	Yes___ No___	Federal Express Airbill	Yes___ No___	
rly Placed	Yes___ No___	Yes___ No___	Traffic Report	Yes___ No___	Yes___ No___
t	Yes___ No___	Yes___ No___	Sample Tags	Yes___ No___	Yes___ No___
			Chain of Custody	Yes___ No___	Yes___ No___

No, then "x" in "NA" column below will indicate which samples are affected by Non-Agreement.

le #	Sample Type	Sample Conc.	Extract Tag #	ITAS Extract Sample #	Where Stored	NA	VOA Tag #	ITAS VOA Sample #	Where Stored	VOA Cond.	NA	Notes

leekend Receipt:
: chests put in refrigerated storage _____ (Date) _____ (Initials) Samples put in refrigerated storage _____ (Date)

chests removed from refrigerated storage _____ (Date) _____ (Initials) _____ (Signature)

Figure 2

```
PROJECT=ITCM17943                DATE=06/27/84                                    TIME= 16:51:3

     PROJECT CODE                ITCM17943
     DATE-TIME ENTERED           05/08/84 11:10:50
     DATE-TIME MODIFIED          05/30/84 17:23:21
     USAGE COUNT(PROJ)           001
     DATE SAMPLES REC'VD         05/07/84
     DUE DATE                    05/11/84
     PROJ.STATUS                 R
     CHAIN OF CUSTODY?           N
     RUSH STATUS?                Y
     CALL REPORT?                Y
     SPECIAL QC?                 Y
     CLIENT TYPE (C,G,I)?        Y
     CALL REPORT TO              Robert Galbraith or Beitz Heindrick at (415) 228-8400
     BILLING CODE                I/C
     PROFIT CENTER               2320
     CLIENT PC#                  0221
     PROJECT STATUS HIST.        RPOMLR
     QUOTATION                   B050484-1
     PURCHASE ORDER NO.
     CONTRACT NO.
     PROPOSAL NO.
     JOB CODE
     INITIALS-QC APPROVAL        ARM
     TASK CODE
     INITIALS-PROJ.RELEAS        ARM
     DATE REPORTED               05/15/84
     REPORT FORMAT
     DATE INVOICED               05/29/84
     DATE $ REC'VD               05/30/84
     OTHER SUBMISSION INF        See quote sheet
     PROJ.DESCRIP1               Sixty two (62) water samples & seven (7) splits
     PROJ.DESCRIP2
     SAMPLE #S ASSIGNED          M2241\M2289,M2292\M2311
     SAMPLE #S ASSIGN
     NOTES #1                          M2241\M2289 (Arsenic,Lead,Zinc,Copper, & Cadmium)
     NOTES #2                          M2292\M2311 (pH & Specific Conductance)
     NOTES #3                    SEE WORK FOLDER FOR SPECIAL INSTRUCTIONS & DETECTION LIMITS
     PROJECT NUMBER              17943
     SUBPROJECT ?                N
     TRACKING MET                PR
     DEN NO.
     LIMS FILE

     SAMPLES M2241 \M2289
     TEST    AG01 003            Arsenic, Total, in Water & Effl. by Graphite Furna  EPA Total Metals Prep
     TEST    AG01 039            Lead, Total in Water & Effl. by Graphite Furnace    EPA Total Metals Prep
     TEST    AA01 045            Zinc, Total, in Water and Effluent                  EPA Total Metals Prep
     TEST    AA01 014            Copper, Total, in Water & Effluent                  EPA Total Metals Prep
     TEST    AA01 010            Cadmium, Total, in Water & Effluent                 EPA Total Metals Prep
     SAMPLES M2292 \M2311
     TEST    WW   032            pH in Water & Effluent
     TEST    WW   046            Specific Conductance in Water & Effluent

*****************************************
*****************************************

     M2241     WSS-145,Sea Water    STAT=01 TYPE=01 TMET=PR PROJ=ITCM17943 TASK=          JOB=
       SAMPLE DESCRIPT. : WSS-145,Sea Water,1:30 P


PROJECT=ITCM17943                DATE=06/27/84                                    TIME= 16:51:3

                 NOTES :
     M2242     WSS-129,U61,11:00    STAT=01 TYPE=01 TMET=PR PROJ=ITCM17943 TASK=          JOB=
       SAMPLE DESCRIPT. : WSS-129,U61,11:00
                 NOTES :
     M2243     WSS-131,U61,11:00    STAT=01 TYPE=01 TMET=PR PROJ=ITCM17943 TASK=          JOB=
       SAMPLE DESCRIPT. : WSS-131,U61,11:00
                 NOTES :
     M2244     WSS-101,1A,11:09 A   STAT=01 TYPE=01 TMET=PR PROJ=ITCM17943 TASK=          JOB=
       SAMPLE DESCRIPT. : WSS-101,1A,11:09 A
                 NOTES :
     M2245     WSS-102,1A,filtered  STAT=01 TYPE=01 TMET=PR PROJ=ITCM17943 TASK=          JOB=
       SAMPLE DESCRIPT. : WSS-102,1A,11:50 A,filtered
                 NOTES :
     M2246     WSS-113,2A,1:40P     STAT=01 TYPE=01 TMET=PR PROJ=ITCM17943 TASK=          JOB=
       SAMPLE DESCRIPT. : WSS-113,2A,1:40 P
                 NOTES :
     M2247     WSS-114,2A,filtered  STAT=01 TYPE=01 TMET=PR PROJ=ITCM17943 TASK=          JOB=
       SAMPLE DESCRIPT. : WSS-114,2A,1:40 P,filtered
                 NOTES :
     M2248     WSS-132,3A,8:40      STAT=01 TYPE=01 TMET=PR PROJ=ITCM17943 TASK=          JOB=
```

STANDARD OPERATING PROCEDURE

IT CORPORATION

IT ANALYTICAL SERVICES

STEWART LABORATORIES DIVISION

TITLE:	SOP NO: MA841214R0 DATE INITIATED: 12/14/84 REVISION NO: 0 DATE REVISED: PAGE 1 of 1
Temperature Monitoring of Refrigerated Storage Areas	

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
JES	Jack R Hall	12-19-84	Gary M Wood	12/19/84

1.0 Purpose and Applications

1.1 The purpose of this procedure is to ensure compliance with the CLP contract requirements regarding refrigerated sample storage.

1.2 This procedure applies to all refrigerated areas designated for storage of CLP samples and extracts.

2.0 Procedure

2.1 Mechanically driven temperature recorders shall be installed in all such refrigerated areas.

2.2 Such recorders shall be capable of continuously recording the temperature for not less than seven (7) days.

2.3 Each Wednesday, a designated person shall change the charts, dating and initialling them when they are installed and removed.

2.4 Completed charts shall be delivered to the Document Control Coordinator for filing.

2.5 Deviations from the contract stipulated temperature (4°C) shall be immediately reported to the QC Coordinator and appropriate corrective action taken.

STANDARD OPERATING PROCEDURE

IT CORPORATION

IT ANALYTICAL SERVICES

STEWART LABORATORIES DIVISION

TITLE:	SOP NO: QA841113RO DATE INITIATED: 11/13/84 REVISION NO: 0 DATE REVISED: PAGE 1 of 2
Sample Storage for EPA Contract Laboratory Program	

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
JES	Jack R Hall	12-19-84	Gary M Woody	12/19/84

1.0 Samples and extracts will be stored in secured, refrigerated areas designated for EPA-CLP samples. Separate areas will be defined for raw extract samples, VOA samples, GC/MS extracts, GC extracts, and archival of sample extracts and raw sample after completion.

1.1 Refrigerators will be kept locked at all times. The following ITAS employees will be issued keys to the refrigerated areas: Sample Custodians, EPA-CLP Project Manager, EPA-CLP Document Coordinator, GC/MS Group Leader, GC Group Leader, and Organic Prep Group Leader. The EPA-CLP Project Manager may also sign out spare keys to designated employees in order to cover weekend and shift operations.

1.2 Refrigerated areas will be designated, operated, and monitored according to contract specifications or other EPA directives. ITAS Support Services personnel are responsible for the monitoring and operation of refrigerators and refrigerated areas in accordance with SOP MA841214RO.

2.0 Whenever samples or extracts are transferred into or out of "in-process" storage, the transfer will be documented using EPA-CLP Sample Receipt Logs or the Project Work Sheets used for analysis assignments to the laboratory groups. Documentation will be in accordance with SOP CD841010RO, "Receipt and Logging in of EPA Contract Laboratory Program Samples" and SOP PM841011RO, "Work Assignments, Analysis Tracking and Sample Chain of Custody for the EPA Contract Laboratory Program".

3.0 Samples and extracts will be stored/archived after completion of analyses. Sample Archive Logs (Figure 1) will be used for documenting transfer into or out of archive storage, including disposal. These Logs will be attached to the sample archive refrigerators or refrigerated rooms. Disposal of samples will occur after one of the following:

(1) 180 days after data submission, the ITAS Project Manager will submit to the EPA Project Officer, a written request to dispose of samples. Upon written authorization back from the Project Officer, samples will be disposed.

(2) Seven days after receipt of written disposal request from the EPA Project Officer or the Sample Management Office.

3.1 Samples will be disposed of in an appropriate manner as set forth in the IT Corporation Laboratory Safety Manual.

SAMPLE ARCHIVE LOG
EPA-CLP Program

Refrigerator ID: _____ Page No. _____

Laboratory Sample No.	Date/Time Archived	Date/Time Removed	Reason (Re-analysis, Disposal, Return to EPA, etc.)	Date/Time Returned

STANDARD OPERATING PROCEDURE

IT CORPORATION

IT ANALYTICAL SERVICES

STEWART LABORATORIES DIVISION

TITLE: Water Purification System Monitoring	SOP NO: QA841214RO-6 DATE INITIATED: 12/14/84 REVISION NO: 0 DATE REVISED: PAGE 1 of 1

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
JES	John R. Hall	12-17-84	Gary M Woody	12/17/84

1.0 Purpose and Applications

1.1 The purpose of this SOP is to ensure compliance with the EPA-CLP contract requirements regarding the purity of water to be used in extractions, analyses, etc., associated with work done under the contract.

1.2 This procedure applies to all water purification systems which produce reagent grade water which may be used with samples associated with the CLP contract.

2.0 Procedure

2.1 Each working day, a designated person shall measure the resistivity of the purified water from each such system.

2.2 Measurements may be made either by a built-in meter or by an external meter, as appropriate.

2.3 Prior to taking such measurements, the meter calibration shall be checked using a built-in test switch or a suitable standard solution of known conductivity, as appropriate.

2.4 Should the resistivity of the water be less than 10 megohm-centimeters, cartridges and filters, etc., shall be charged, as necessary.

2.5 The resistivity and any changes made to the system(s) shall be recorded, with date and initials, in a bound logbook kept at the water system.

2.6 Any problems regarding the purity of the water, whether observed by the above designated person or by any of the analytical groups, shall be immediately brought to the attention of the QC Coordinator and appropriate corrective action will be taken.

STANDARD OPERATING PROCEDURE

IT CORPORATION

IT ANALYTICAL SERVICES

STEWART LABORATORIES DIVISION

TITLE:	SOP NO: QA841114R0 DATE INITIATED: 11/14/84 REVISION NO: 0 DATE REVISED: PAGE 1 of 1
Facility Security	

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
JES	Jack R Hall	12-18-84	Gary M Woody	12/19/84

1.0 During normal working hours (8:00 am - 5:00 pm, Monday through Friday) when a receptionist is on duty, access to the laboratory will be through the monitored reception area at the front of the building.

1.1 Visitors will be required to sign-in and be accompanied by an ITAS employee while in the laboratory.

2.0 During hours when a receptionist is not on duty, the front doors will be kept locked.

3.0 During hours when no employees are in the building, the facility is secured by the use of an electronic detection system monitored by a local security service.

4.0 Outside doors at the back and sides of the building will be kept locked at all times.

5.0 When deliveries are made at the back of the building, an ITAS employee will be required to remain with the delivery service personnel at all times.

STANDARD OPERATING PROCEDURE

IT CORPORATION

IT ANALYTICAL SERVICES

STEWART LABORATORIES DIVISION

TITLE: GC Sample Analysis and Tracking	SOP NO: GC841213R0 DATE INITIATED: 12/13/84 REVISION NO: 0 DATE REVISED: PAGE 1 of 2

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
NDS	Jack R Hall	12-19-84	Gary M Woody	12/19/84

1.0 Scope

1.1 The purpose of outlining the sample flow through the GC laboratory is to know or determine the progress of any sample. Tracking this flow also documents every sample analysis and treatment.

2.0 Summary

2.1 The flow of every sample analysis, dilution or treatment is documented and retained for sample control and historical retrievability.

3.0 Receiving Sample Extracts

3.1 When preparation of the sample extract(s) is completed, the sample extract(s) are delivered to the GC analyst or technician along with all chain of custody documentation - prep worksheets, chain of custody sheets, project identification index cards.

3.2 The GC analyst or technician signs the chain of custody sheet, filling out all requested information. Prep worksheets and chain of custody sheets are placed in GC incoming work file.

3.3 Sample extracts are placed in the sample extract refrigerator in the GC lab. Sample extract(s) are stored at 4°C at all times, except when they are removed for analysis.

4.0 Sample Custody

4.1 Each time a sample extract is removed from the refrigerator for analysis, the person removing the sample will sign the chain of custody form and fill out requested information. When samples are returned to the refrigerator, chain of custody information is again required to be completed.

5.0 Sample Analysis

5.1 Samples and standards will be analyzed following the specifications of the contract.

5.2 Analysis Procedure - fill autosampler vial to proper level with sample.

5.3 The autosampler vial is placed in the autosampler tray preceded by a solvent vial. The tray number and position are recorded in the sample log under the respective instrument and date of analysis. Samples should be kept in numerical order when possible.

5.4 When the samples for that date have finished, the chromatograms are removed from the data station and labeled according to the sample log.

5.5 The GC technician will determine if a dilution or specific clean-up treatment is required for each sample; whichever is needed will be annotated in the sample log to the left of the particular sample number.

5.6 Samples that were diluted or cleaned up will be reanalyzed by the gas chromatograph. After sample has been rerun, cross out annotation previously made on the log sheet. Record new dilution or treatment beside sample number on log sheet.

6.0 Data Flow

6.1 When the GC technician completes labeling and review of chromatograms, he will submit them to the GC analyst.

6.2 The project identification index cards will be transferred to a holding box until all sample extracts of that project are disposed of or sent back to EPA, as specified by the contract.

7.0 Sample Archive

7.1 The remaining sample extract will be placed in a sealed vial, labeled and stored by project number in the GC sample archive storage area.

7.2 Sample extracts will be held for the time period specified by EPA in the contract.

CASE NO._____

Instrument_____
Column_____
Method_____

Date_____
Operator_____

Tray_____

1._____
2._____
3._____
4._____
5._____
6._____
7._____
8._____
9._____
10._____
11._____
12._____
13._____
14._____
15._____

Tray_____

1._____
3._____
5._____
7._____
9._____
11._____
13._____
15._____

Tray_____

1._____
3._____
5._____
7._____
9._____
11._____
13._____
15._____

Tray_____

1._____
3._____
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11._____
13._____
15._____

Tray_____

1._____
3._____
5._____
7._____
9_____
11._____
13._____
15._____

Tray_____

1._____
3._____
5._____
7._____
9_____
11._____
13._____
15._____

NOTE: EVEN NUMBERED TRAY POSITIONS ARE SOLVENT RINSES.

GC Sample Log Format

(General Format)	(Example of Format)
Date of Analysis	5/27/84
Instrument Number	T-560-1
Tray # _____	Tray # 8

1.	Tray position for samples	1.	--
3.	Sample number, dilution, &/or cleanup*	3.	M2001 (F)
5.		5.	M2002 (F)
7.		7.	M2010 1/10, Hg
9.		1/5 9.	M2015
11.		Hg, 1/5 11.	M2016
13.		1/5 13.	M2017
15.		Acid, 1/5 15.	M2018

* = Annotate dilution &/or cleanup needed.

NOTE: Even numbered tray positions are solvent rinses.

STANDARD OPERATING PROCEDURE

IT CORPORATION

IT ANALYTICAL SERVICES

STEWART LABORATORIES DIVISION

TITLE: GC/MS Sample Analysis and Tracking	SOP NO: M841218-R0 DATE INITIATED: 12/18/84 REVISION NO: 0 DATE REVISED: PAGE 1 of 2

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
SAM	Jack R. Hall	12/19/84	Gary M Woody	12/19/84

1.0 Scope

1.1 The purpose of outlining the sample flow through the GC/MS laboratory is to know or determine the progress of any sample. Tracking this flow also documents every sample analysis and treatment.

2.0 Summary

2.1 The flow of every sample analysis, dilution or treatment is documented and retained for sample control and historical retrievability.

3.0 Receiving Sample Extracts

3.1 When preparation of the sample extract(s) is completed, the sample extract(s) are delivered to the GC/MS analyst or technician along with all chain of custody documentation - prep worksheets, chain of custody sheets, project identification index cards.

3.2 The GC/MS analyst or technician signs the chain of custody sheet, filling out all requested information. Prep worksheets and chain of custody sheets are placed in GC/MS incoming work file.

3.3 Sample extracts are placed in the sample extract refrigerator in the GC/MS lab. Sample extract(s) are stored at 4°C at all times, except when they are removed for analysis.

4.0 Sample Custody

4.1 Each time a sample extract is removed from the refrigerator for analysis, the person removing the sample will sign the chain of custody form and fill out requested information. When samples are returned to the refrigerator, chain of custody information is again required to be completed.

5.0 Sample Analysis

5.1 Samples and standards will be analyzed following the specifications of the contract.

5.2 All sample analysis data is entered into the instrument log at the time of analysis. Internal standard areas and surrogate recoveries are entered after data reduction.

5.3 All dilutions and sample reruns are recorded in the instrument log as if they were discrete samples.

6.0 Data Flow

6.1 All RIC's, Quan Lists, and Spectra are grouped by sample and project and submitted with the completed report sheet to the GC/MS Group Leader for approval.

6.2 After approval of report, all data is submitted to the document custodian for numbering, inventory and filing.

7.0 Sample Archive

7.1 The remaining sample extract will be placed in a sealed vial, labeled and stored by project number in the GC/MS sample archive storage area.

7.2 Sample extracts will be held for the time period specified by EPA in the contract.

STANDARD OPERATING PROCEDURE

IT CORPORATION

IT ANALYTICAL SERVICES

STEWART LABORATORIES DIVISION

TITLE:	
Document Numbering and Inventory Procedure for Use in EPA Contract Laboratory Program	SOP NO: QA841213R0 DATE INITIATED: 12/13/84 REVISION NO: 0 DATE REVISED: PAGE 1 of 2

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
JES	Jack R. Hall	12-19-84	Gary M Woody	12/19/84

1.0 Document Numbering

1.1 The document custodian will assign a document inventory number to each document of a case. The document number will consist of:

1.1.1 SMO case number
1.1.2 EPA region number
1.1.3 Serialized document number

1.2 All documents pertaining to each case will be given a document number and inventoried. The documents will be numbered in the following order:

1.2.1 Document file inventory
1.2.2 Chain of Custody form(s)
1.2.3 Sample tag(s)
1.2.4 Airbill(s)
1.2.5 Organics Traffic Report(s)
1.2.6 Organics laboratory chronicles (extraction)
1.2.7 Organics laboratory chronicles (analysis)
1.2.8 Sample Receipt Log
1.2.9 Prep worksheets & sample tracking forms
1.2.10 GC/MS data, analysis sheets, & logs
1.2.11 GC chromatograms, analysis sheets, & logs
1.2.12 Related correspondence and/or memos
1.2.13 All other related documents

2.0 Document Inventory

2.1 A document inventory list which includes all documents and their document number will be prepared and filed with each case.

2.2 In the event that all case documents are sent to an EPA Region for enforcement action, or other action, the laboratory will retain a copy of the document inventory list for that case.

3.0 Case File Assembly

3.1 All documents will be compiled in case file folders for submission to EPA.

3.2 Using appropriate file folders, assign one folder to each case according to SMO case number.

3.3 Place all documents, sample tags, SMO forms, and laboratory generated data pertaining to one case in the folder.

3.4 Documents should be arranged by document type within the case folders, i.e., all sample tags together, all traffic reports together, all hard-copy mass spectra together, etc.

3.5 The document case files will be stored in secured file cabinets arranged by SMO case number.

STANDARD OPERATING PROCEDURE

IT CORPORATION

IT ANALYTICAL SERVICES

STEWART LABORATORIES DIVISION

TITLE: Laboratory Data Recording	SOP NO: QA841214R0-5 DATE INITIATED: 12/14/84 REVISION NO: 0 DATE REVISED: PAGE 1 of 1

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
JES	Jack R Hall	12-19-84	Gary M Woody	12/19/84

1.0 Purpose and Applications

1.1 The purpose of this SOP is to ensure compliance with generally recognized good laboratory practices and, in particular, the requirements of the EPA-CLP contract requirements regarding recordkeeping.

1.2 This procedure applies to all laboratory data sheets, assignment sheets, work sheets, tabulations of results, logbooks, etc.

2.0 Procedure

2.1 All such records are to be kept in ink, preferably blue or black to enable legible copying when necessary.

2.2 Corrections shall be made by drawing a single line through the incorrect data and writing in the corrected value beside or above. The use of "white-out" is expressly prohibited.

2.3 All corrections shall be dated and initialled.

2.4 All data sheets shall be dated and signed by the chemist/technician performing the work. In the event that sheets are used for multiple tests by different people, each individual shall date and initial those parts for which he or she is responsible, and the completed sheet reviewed, signed and dated by the Group Leader or Coordinator.

2.5 Unused space on data sheets should be "Z-ed out".

2.6 Insofar as is possible, only preprinted forms as appropriate should be used.

2.7 Any problems arising from cramped or confusing forms should be brought to the attention of the QC Coordinator and appropriate corrective action be taken.

STANDARD OPERATING PROCEDURE

IT CORPORATION

IT ANALYTICAL SERVICES

STEWART LABORATORIES DIVISION

TITLE: Balance Calibration	SOP NO: QA841214RO-3 DATE INITIATED: 12/14/84 REVISION NO: 0 DATE REVISED: PAGE 1 of 1

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
JES	Jack R. Hall	12-19-84	Gary M Woody	12/19/84

1.0 Purpose and Application

1.1 The purpose of this SOP is to ensure compliance with the EPA-CLP contract requirements regarding calibration of balances.

1.2 This procedure applies to all balances which may be used to weigh samples, standards, reagents, etc., associated with analyses done under the CLP contract.

2.0 Procedure

2.1 Each working day, a designated person shall check and record the calibration of all such balances.

2.2 Indicated weights shall be recorded in bound logbooks.

2.3 Only weights meeting Class 5 specifications of NBS Circular 547 shall be used.

2.4 Any deviations from the nominal values of the weights shall be immediately brought to the attention of the QC Coordinator and appropriate corrective action will be taken.

2.5 At least quarterly, all balances shall be calibrated and adjusted by a certified laboratory balance service engineer.

STANDARD OPERATING PROCEDURE

IT CORPORATION

IT ANALYTICAL SERVICES

STEWART LABORATORIES DIVISION

TITLE:	SOP NO: QA841214RO-2 DATE INITIATED: 12/14/84 REVISION NO: 0 DATE REVISED: PAGE 1 of 1
Glassware Clean-Up for Organic Extractions and Analyses	

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
PJK	[signature]	12-14-84	[signature]	12/17/84

1.0 Purpose and Applications

1.1 The purpose of this SOP is to reduce the possibility of cross-contamination of samples.

1.2 This procedure shall be followed for all glassware used in the extraction, concentration, and analysis of samples for organic compounds by GC or GC/MS.

2.0 Procedure

2.1 Glassware must be cleaned as soon as possible after being used. All glassware is to be cleaned and put away before you leave for the day.

2.2 Rinse glassware with solvent being used for the extraction immediately after use.

2.3 Wash with hot soapy water. Be sure to rub all surfaces of the glass well with the brush.

2.4 Rinse at least five (5) times with tap water.

2.5 Rinse at least three (3) times with DI water.

2.6 Dry outside with paper towel.

2.7 Rinse twice with acetone.

2.8 Rinse twice with hexane.

2.9 Store glassware in its proper place.

STANDARD OPERATING PROCEDURE

IT CORPORATION

IT ANALYTICAL SERVICES

STEWART LABORATORIES DIVISION

TITLE:	SOP NO: QA841214RO-1 DATE INITIATED: 12/14/84 REVISION NO: 0 DATE REVISED: PAGE 1 of 2
Work Assignments, Analysis Tracking, and Sample Chain of Custody for the EPA Contract Laboratory Program	

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
JES	Jack A Hall	12-18-84	Gary M Woody	12/19/84

1.0 After projects have been coded in and samples stored under refrigeration according to SOP ITAS-K-CD841010RO, coding personnel are responsible for notifying the EPA-CLP Document Coordinator that samples have arrived.

1.1 The Document Coordinator reviews coding information from the Project Folder and verifies the test assignments. If needed, the Document Coordinator modifies the computer test assignments and updates Project Status to "Test Assigned/Modified by GRL's". After the Project is verified, the Document Coordinator updates the Project Status to "Test Assignments Verified by Coordinators".

1.2 After verification, the Document Coordinator is responsible for filling out Project Work Sheets for analysis assignments to the laboratory Groups. Copies of the Work Sheets for GC, GC/MS, and Organic Prep are shown in Figures 1-6. The Work Sheets are then given to the Group Leaders involved.

1.3 Group Leaders check their Work Sheets and update their Master Project Logs shown in Figures 7-9. Work assignments are distributed to Group personnel by use of the Work Sheets.

1.4 The Project Work Sheets are held in the various laboratory Groups until completion of the analysis or prep for all samples. They are signed and dated by analysts or prep technicians after analysis or prep has been completed and then turned in to the Group Leader in charge. After review, the Group Leader approves the work and either sends the Work Sheet to the analysis Group involved (in the case of Organic Prep) or attaches the Work Sheet to the analysis forms and turns it in to the Document Coordinator (in the case of analysis Groups).

1.4.1 Chain of Custody of samples moving through the laboratory for analysis will be documented by the use of Project Work Sheets. Spaces are provided for entering employee name, date, and location where samples were taken from storage, where extracts were stored, and where left over sample and completed extracts were stored.

1.5 Computer analysis status updates are entered by analysts after completion of assignments and by Group Leaders after reviewing and approving data. Figure 10 is a list of Project and Sample/Analysis status codes.

1.5.1 Using the computer system, the status of samples can be monitored at the Project, Sample, Analysis, or Case level. Printouts can be created at any time or information viewed on CRT's.

1.6 The Document Coordinator organizes and assembles the data packages using the EPA-CLP Data Package Checklist shown in Figure 11. The data package is reviewed by both the ITAS CLP Project Manager and the QC Coordinator before data is released.

1.7 After approval of the data packages associated with a Project Code, the Document Coordinator updates the computer Project Status to "Report Released (Mgt)", sends out the data packages, and updates the Project Status to "Report Has Been Sent Out".

1.8 Completed Projects are filed in the EPA-CLP room by Case No. in locked filing cabinets. All records associated with the CLP samples are stored together--shipping papers, SMO documents, coding information, Work Sheets, raw data, and the copy of the final data packages.

PROJECT & TEST ASSIGNMENT VERIFICATION FORM

___ VW Group: Additional tests assigned? ___
___ Metals Group: Additional tests assigned? ___
___ GC & Misc. Org. Groups: Additional tests assigned? ___
___ GC/MS Group: Additional tests assigned? ___

**** Last Group Leader should route to Analysis Coordinator, GMW ****

___ GMW (Misc. Inorg., SC, SP, LM & check to see that ALL tests have been assigned): Additional tests assigned? ___

**** GMW is responsible for updating Project Status to "C" and/or "D" using VIP--ARM in GMW's absence ****

Figure 1

PROJECT WORKSHEET

GC/MS ANALYSIS

PROJECT CODE: _____ DATE ISSUED: _____

SAMPLE NO: _____

_____ DATE DUE: _____

SAMPLE TYPE: _____ SAMPLE LOCATION: _____

	TEST #	ANALYSIS DESCRIPTION	METHOD
____	MV01 804	PUBLIC WATER SUPPLY TRIHALOMETHANES	
____	MV01 806	PRIORITY POLLUTANT P&T VOLATILE ORGANICS	EPA 624
____	MV04 806	PRIORITY POLLUTANT VOAS IN SOIL/SLUDGE/WASTE	SW 846
____	ME02 806	PRIORITY POLLUTANT BN TESTS IN WATER & EFFLUENT	EPA 625
____	ME03 806	PRIORITY POLLUTANT AE TESTS IN WATER & EFFLUENT	EPA 625
____	ME05 806	PRIORITY POLLUTANT BN/AE TESTS IN SOIL/SED.	SW 846
____	ME02 811	PRIORITY POLLUTANT PEST/PCBS IN WATER & EFFLUENT	EPA 625

OTHER: _____

ADDITIONAL COMPOUNDS: _____

QA/QC: _____

NOTES/SPECIAL INSTRUCTIONS: _____

ANALYST/INSTRUMENT: _____ DATE OF ANALYSIS: _____

Figure 2

WORKSHEET

Organic Prep for GC/MS

PROJECT CODE: _____ DATE ISSUED: _____

SAMPLE NO: _____ DATE DUE: _____

_____ SAMPLE LOCATION: _____

SAMPLE TYPE: _____ _____

==========

Analyte(s): _____

Prep Method	Method #	Description
_____	ME 02	Base Neutral Extraction for Water & Effluent EPA 625
_____	ME 03	Acid Extraction for Water & Effluent EPA 625
_____	ME 05	Sonication Extraction (BN/AE for Soil/Sediment EPA 3550)
_____	ME 06	ACYB Prep (P.P. in sludge)

_____ Other _____

_____

_____

Special Instructions _____

_____

_____

_____

Special QA/QC _____

_____

_____

Prepped by _____ Date _____

Sample Extraction Verified _____ Date _____

Figure 3

PROJECT WORKSHEET
SAMPLE PREP
Organic Extraction

Prepped By _____

PROJECT CODE: _____ Date Prepped _____

ample No.	Sample Vol (ml) or Wgt (g)	Initials	Final Extract Vol(ml)	Initials	Florisil & Initials	Additional Information (Comments)

ITAS-K-QA003R0

INTERNAL CHAIN OF CUSTODY FORM
IT Analytical Services-Knoxville

Sample No.	DATE	Extract* Type or Original Sample?	Moved From	Moved To	Reason	Signature

* O - Original Sample
AE - Acid Extract
P - Pesticide/PCB Extract
BNA - BN/AE Extract
BN - Base Neutral Extract
VOA - VOA Sample

Figure 4A

WORKSHEET

Organic Analysis

PROJECT CODE: _____ DATE ISSUED: _____

SAMPLE NO: _____ DATE DUE: _____

SAMPLE TYPE: _____ SAMPLE LOCATION: _____

==========

Analyte(s): _____

Method #			Description
_____	GC 09	802	City Water Pesticides
_____	GC 10	803	City Water Herbicides
_____	GC 12	804	Trihalomethanes
_____	GC 01	807	PCB's in Water and Effluent
_____	GC 03	808	PCB's in Oil (ASTM)
_____	GC	811	Priority Pollutant Pesticides & PCB's in Water and Effluent
_____	GC	812	PCB's in Soil/Sediment/Sludge (ITAS SOP)
_____	GC 04	813	PCB's in Oil (EPA)
_____	GC		PCB's Calculated by Webb & McCall Method
_____	OM 003		Total Organic Halogen
_____	OM 005		IR Scan
_____	GC		Industrial Hygiene
_____	Other		_____

_____

_____

Special Instructions _____

_____

_____

Analysis Summary _____

_____

_____

Special QA/QC _____

_____

_____

Analyst _____ DATE _____

_____ DATE _____

Figure 5

PROJECT WORKSHEET

Organic Prep- GC1

PROJECT CODE: _____ DATE ISSUED: _____

SAMPLE NO: _____ DATE DUE: _____

_____ SAMPLE LOCATION: _____

SAMPLE TYPE: _____ _____

==========

Analyte(s): _____

Prep Method	Method #	Description
_____	GC 01	EPA 617 PCB's
_____	GC 02	ITAS SOP for PCB's in SOIL/SEDIMENT
_____	GC 03	ASTM Prep for PCB's in Oil
_____	GC 05	Sonication Extraction for Soil/Sediment
_____	GC 06	Mechanical Dispersion Ext. for Sediment/Fish
_____	GC 09	Pesticide for Public Water Supplies
_____	GC 10	Herbicides for Public Water Supplies
_____	GC 01	EPA 617 Priority Pollutant Pesticides and PCB's
_____	Other	_____

Special Instructions _____

Special QA/QC _____

Prepped by _____ Date _____

Sample Extraction Verified _____ Date _____

Figure 6

WORKSHEET

Organic Prep - GC2

PROJECT CODE: _____ DATE ISSUED: _____

SAMPLE NO: _____ DATE DUE: _____

_____ SAMPLE LOCATION: _____

SAMPLE TYPE: _____ _____

==========

Analyte(s): _____

Method #		Description
_____	GC 04	EPA Prep for PCB's in Oil
_____	GC 14	Priority Pollutants in Sludge
_____	GC 11	NIOSH P&CAM, Organic Solvents (Charcoal Tubes)
_____	GC 13	Carbon Tetrachloride Extraction
_____	GC 15	FDA Prep for Fish
_____	GC 16	Soxhlet Extraction for PP Pest & PCB's in Soil/Sediment
_____	Other	_____

Special Instructions _____

Special QA/QC _____

Prepped by _____ Date _____

Sample Extraction Verified _____ Date _____

Figure 7

GC/MS Project Work Sheet

Project Code	Sample	Numbers		VO	BN	AE	OTHER		Due Date	Pre Due
UB 18287	M3977	-3980		THMs					7-24-84	
LI 18289	M3988			THMPE					7-20-84	
LI 18290	M3990	-3991			X	X		P.E.	7-20-84	
CU 18295	M4007	-4010		THMs					7-25-84	
CU 18298	M4014	-4017		THMs					7-25-84	
SWSM 18277	M4038	-4039		X					7-30-84	
MI 18309	M4048	-4049		X					7-25-84	
TEK 18310	M4054	-4065					PCBs	RUSH	7-20-84	
UB 18312	M4078	-4080		X	X	X			8-10-84	
PAL 18314	M4098	-4120		X	X	X	Performance	Eval.	7-31-84	
IKU 18322	M4162	-4165		THMs					8-1-84	
OM 18323	M4081	-4087		X	X	X			8-1-84	
TEK 18324	M4166	-4170					PCBs	RUSH	7-24-84	
AW 18325	M4171	-4172		X					8-2-84	
YE 18336	M4274	-4279		X	X	X	Performance	Evaluation	8-10-84	
AW 18348	M4332	-4334		X					8-7-84	
E 18354	M4375			THMs					8-8-84	

GC and MISCELLANEOUS ORGANIC ANALY SES

Project Code	Sample Type	Samples Nos.	Type of Analysis		Du Dat

PROJECT STATUS

A	Samples Logged-In
B	Samples Logged-In & All Tests Assigned @ A
C	Test Assignments Completed by GRLs or Coordinators
D	Test Assignments Verified by Coordinators
E	On Hold
F	Help Requested
G	In-Process
H	Re-Work
I	Lab Work Completed
J	Lab Work Release In-Process
K	All Lab Work Released
L	QC Approved
M	Report Released (Mgt.)
N	Report Called to Client
O	Report Was Been Sent Out
P	Billed
Q	Partially Paid
R	Full Payment Received
S	Uncollectable
T	Cancelled
U	No Charge Project
Y	Special Long-Term, On-Going Projects--Will Have Sub-Projects
Z	Special Projects Which Will Never Have Samples
1	Samples Added before Status C
2	All Tests Assigned by Coding after Project Log-In (PRL)
3	Partial Test Assignment at Coding
4	Tests Assigned/Modified by GRLs
5	Samples Added after Status of D
6	Tests Added after Status of D
7	Samples Deleted after Status of D
8	Tests Deleted after Status of D
9	Not Released from Coding(Problem)

SAMPLE STATUS

1	Logged In
2	On Hold
3	Rejected
4	Help Requested
5	In Process
6	Re-Work
7	Complete
8	Released
9	Cancelled
10	Active (1-7 above)

Figure 10

TABLE I

ORGANICS

Each file must contain the following documents or a memo explaining their absence (one memo may cover several documents):

- File inventory
- * Chain-of-custody form
- * Sample tag(s)
- * Airbill(s)
- * Organics Traffic Report(s)
- * Organics Laboratory Chronicles (Extraction)
- * Organics Laboratory Chronicles (Analysis)
- Organics Analysis Data Summaries
- Copies of analyst's notebook pages
- Benchsheets and worksheets
- Copies of instrument logbook pages
- Sample tracking documents
  - Sample receipt logbook pages
  - ** Internal custody records
- Hard copies of mass spectra and chromatograms
- QA/QC package
  - DFTPP/BFB calibration spectra and worksheets
  - Quality Control Reports
  - Standards Analysis Report forms, worksheets, and Spectra/Chromatograms
  - Duplicate, Matrix, Surrogate Spike Results
- GC/MS computer library search worksheets and accompanying spectra
- Related correspondence and/or memos
- All other related documents

* If received with sample shipment

** If used to supplement sample tracking system

Figure 11

APPENDIX
D

SAMPLE PREPARATION PROCEDURES FOR DIOXIN IN WATER

1.0 SUMMARY OF METHOD

1.1 A one-liter water sample is extracted with methylene chloride using a separatory funnel technique after spiking with $^{37}Cl_4$-2,3,7,8-TCDD and $^{13}C_{12}$-2,3,7,8-TCDD. After the sample is exchanged to hexane and concentrated, column cleanup steps are performed, followed by GC/MS analysis.

2.0 REAGENTS

2.1 Spiking standard solution (contains both internal and surrogate standards). $^{13}C_{12}$-2,3,7,8-TCDD internal standard at a concentration of 500 ng/ml and $^{37}Cl_4$-2,3,7,8-TCDD surrogate standard at a concentration of 100 ng/ml, both in the same acetone solution. The standard ID number is 374:68-1. The GC/MS IS mixture is prepared at a concentration of 200 ng/ml in isooctane.

2.2 Sulfuric acid (concentrated); ACS grade; specific gravity 1.84.

2.3 Methylene chloride; pesticide quality or equivalent.

2.4 Hexane; pesticide quality or equivalent.

2.5 Acetone; pesticide quality or equivalent.

2.6 Sodium sulfate; ACS, granular, anhydrous. Prepare by a 24-hour methylene chloride extraction. After preparation, store in an oven maintained at 110°C.

2.7 Silica gel; type 60, EM reagent, 70-230 mesh, or equivalent. Prepare by soxhlet extraction with methylene chloride overnight, drying, and then activating in an aluminum foil covered glass container for 24 hours at 130°C.

2.8 Alumina-neutral; Fisher brand, 80-200 mesh. Prepare by soxhlet extraction with methylene chloride, followed by drying and then activating in an aluminum foil-covered glass container for 24 hours at 190°C.

2.9 Sulfuric acid; impregnated silica gel, 40% w/w. To prepare, add two parts (200 g) concentrated sulfuric acid to three parts silica gel (300 g) contained in a glass one-liter bottle equipped with a Teflon-lined screw cap. Mix thoroughly with a glass rod until no lumps are visible. Label the bottle with "Sulfuric Acid - impregnated silica gel" and "caution - contains concentrated sulfuric acid;" date prepared and person preparing the reagent. All information will be recorded in the laboratory standard notebook.

2.10 Sodium hydroxide modified silica gel, 33% w/w. To prepare, add one part 1M NaOH to two parts activated silica gel contained in a glass jar

equipped with a Teflon-lined screw cap. Mix thoroughly with a glass rod until no lumps are visible. Label bottle with "NaOH - impregnated silica gel," date prepared and the person preparing the reagent. All information will be recorded in the laboratory standard notebook.

2.11 Carbopak C (activated carbon) on Celite: Prepare by thoroughly mixing 3.6 g of Carbopak C (80/100 mesh) and 16.4 g of Celite 545 in a 40-ml vial. Activate at 130°C for six hours. Store in a desiccator. CAUTION: Check each new batch of mixed Carbopak/Celite to ensure TCDD recovery of > 50%. Subject the low level concentration calibration solution to the procedure in section 6.3.0-6.3.12 and measure the quantity of labeled and unlabeled TCDD. Label bottle with "Activated Carbopack/Celite," date prepared and the person preparing the reagent. All information will be recorded in the laboratory standard notebook.

3.0 CAUTIONS

3.1 Samples received for this preparation procedure are of unknown composition but may be potentially carcinogenic, mutagenic, toxic or in other ways hazardous.

3.2 It is mandatory that the initial weighing of the samples, addition of isotopically labeled 2,3,7,8-TCDD, and mixing of the samples be performed inside the designated dioxin hood in the high hazard laboratory.

3.3 Personnel involved in this sample preparation procedure should be thoroughly familiar with laboratory SOPs on the processing of high hazard samples.

4.0 EQUIPMENT AND MATERIALS

4.1 Separatory funnels, 2000 ml and 125 ml, with Teflon stopcocks.

4.2 Steam baths, with concentric ring covers.

4.3 Kuderna-Danish concentration apparatuses, consisting of a three-ball macro Snyder column, a 500-ml evaporative flask, and a 10-ml graduated concentrator tube.

4.4 Mini vials (Reacti-vials); 1.0 ml capacity with conical interiors and graduated at 0.1 ml; equipped with Teflon-faced rubber septa and screw caps.

4.5 Concentration vials; 20-ml screw top, septum sealed, scintillation vials.

4.6 Concentration device; nitrogen blowdown apparatus, Organomation and Pierce concentration devices, or equivalent.

4.7 Glass filtering funnels, short stem.

4.8 Filter paper, Whatman No. 4 or equivalent.

4.9 Chromatography columns, 200 mm X 10 mm, with removable Teflon stopcock.

5.0 SAMPLE EXTRACTION

5.1 Prepare designated dioxin hood as instructed in laboratory SOP for processing high hazard samples.

5.2 Mark sample meniscus on side of bottle, for volume determination later. Pour into a two-liter separatory funnel.

5.3 Add 100 ml of spiking solution containing both internal and surrogate standards to the separatory funnel.

5.4 Rinse sample bottle with 60 ml methylene chloride and add to separatory funnel. Extract sample by shaking funnel for two minutes; allow phases to separate for at least 10 minutes. If an emulsion forms, phase separation should be attempted using stirring, filtration through glass wool or centrifugation. Collect the methylene chloride extract in a Kuderna-Danish (K-D) concentrator.

5.5 Rinse the sample bottle with another 60 mL volume of methylene chloride and repeat the extraction. Combine this methylene chloride extract with the first extract in the K-D concentrator. Perform a third extract in the same manner.

5.6 Concentrate the extract volume to approximately 3 ml with the K-D apparatus. Remove the K-D apparatus from the steam bath and allow it to cool for 10 minutes.

5.7 Remove the Synder column, add 50 ml hexane to the K-D flask and replace the column. Concentrate the extract volume to 3 ml.

5.8 Transfer the concentrated extract to a 20-ml scintillation vial. Rinse evaporator flask with three 5-ml portions of hexane; transfer each rinse into the scintillation vial. During these transfers, evaporate the solvent using a gentle stream of dry nitrogen.

5.9 After the final rinse has been added, reduce the extract volume to approximately 1 ml.

6.0 DUAL COLUMN CLEAN-UP

6.1 Either the necessary columns have been prepared and are in a drying oven, or the silica gel and alumina columns need preparation. If columns need to be prepared, obtain enough 1 X 20 cm columns (for the silica gel columns) and 1 X 30 cm columns for the alumina columns.

6.2 Place a small wad of glass wool in the bottom of the 1 X 20 cm columns and add 1.0 g of silica gel, 2.0 g of the NaOH impregnated silica gel, 1 g silica gel, 4 g of the 40% w/w sulfuric acid treated silica gel and

2.0 g of silica gel. Gently tap the columns to allow the contents to settle after each addition.

6.3 Place a small wad of glass wool in the bottom of the 1 X 30 cm columns and add 6 g of alumina and a 1 cm layer of sodium sulfate. Gently tap the columns to allow the contents to settle.

6.4 Attach the silica gel and alumina columns to lab supports so that the silica gel column is above the alumina column and the lower tip of the silica gel column is inserted into the top of the alumina column.

6.5 Rinse both columns with hexane to remove any air bubbles and discard the hexane.

6.6 When the silica gel and alumina columns have stopped dripping hexane, place a clean Erlenmeyer flask under the alumina column and transfer the hexane concentrate obtained from step 5.15 to the top of the silica gel column. Rinse the scintillation vial with two 0.5 ml portions of hexane and add the washings to the top of the silica gel column.

6.7 Wash the silica gel column with 120 ml of hexane, added in aliquots by means of a transfer pipet. Remove the silica gel column.

6.8 Wash the alumina column by placing 20 ml of hexane on the column and eluting until the hexane has dropped below the sodium sulfate layer. Remove the Erlenmeyer flask and retain (with aluminum foil cap) until directions are given to discard.

6.9 Place a clean, labeled 125-ml Erlenmeyer flask under the alumina column and place 20 ml of 20% v/v methylene chloride in hexane on the alumina column, catching the eluent in the Erlenmeyer flask.

6.10 Place the 125-ml Erlenmeyer flask on the Organomation or Pierce concentration apparatus and reduce the volume of the solvent until less than 10 ml of solvent remains, but do not evaporate to dryness.

6.11 Quantitatively transfer the hexane extract to labeled 10-ml K-D concentrator tubes. Rinse the Erlenmeyer flask with several small (1-2 ml) portions of hexane, adding the washes to the K-D concentrator tube.

6.12 Transfer the K-D concentrator tubes to the concentration device and concentrate the hexane extract to approximately 1.0 ml, using a gentle stream of nitrogen and heat if necessary.

6.13 After the 1.0 ml volume has been obtained, remove the K-D concentrator tube and quantitatively transfer the contents of the K-D concentrator tube to designated conical mini-vials. Rinse the concentrator tube with two 0.5-ml portions of hexane and transfer the washes to the concentrate vial. Complete necessary paperwork (see QA section).

6.14 Store the hexane concentrate in a freezer until just prior to GC/MS analysis.

6.15 Concentrate the hexane to near dryness and add 50 µl of a 200 ppb standard of an appropriate Is. Return sample to the reach-in refrigerator.

7.0 ACTIVATED CARBON CLEAN-UP

7.1 After GC/MS analysis of samples processed by the dual column method in Section 6.0, the possibility exists that certain matrices may produce indeterminable results. In such cases an activated carbon column clean-up technique will be employed.

7.2 Obtain from the desiccator the activated Celite/Carbopak (from Section 2.13).

7.3 Insert a small wad of glass wool into a small (7 mm O.D. X 15 cm) disposable pipet and, using vacuum aspiration at the pointed tip of the pipet, add the Celite/Carbopak mixture until a 2-cm column is obtained.

7.4 Prepare the column by adding the following solvents/solvent mixtures in the designated aliquot sizes: (place a clean 125-ml Erlenmeyer flask under the column)

- 2 ml toluene,
- 1 ml 75/20/5 (v/v/v) methylene chloride/methanol/benzene,
- 1 ml 50/50 (v/v) cyclohexane/methylene chloride,
- 2 ml hexane.

7.5 When the bottom of the hexane meniscus just touches the top of the Celite/Carbopak material, quantitatively transfer the 50 µl of sample onto the column.

7.6 Rinse the sample container with two 1-ml portions of hexane, adding the rinses onto the column.

7.7 Sequentially wash the column with the following solvents/solvent mixtures in the designated aliquot sizes:

- 1 ml 50/50 (v/v) cyclohexane/methylene chloride
- 1 ml 75/20/5 (v/v/v) methylene chloride/methanol/benzene.

7.8 Remove the 125-ml Erlenmeyer flask and replace with a concentrator tube or Reacti-vial.

7.9 Elute the TCDD from the column with 2 ml of toluene into the concentrator tube or Reacti-vial.

7.10 Store the toluene eluent in a freezer until the GC/MS analysis is to be performed.

7.11 Shortly before the analysis, concentrate the extract to near dryness and add 50 µl of isooctane for GC/MS analysis.

7.12 Complete any paperwork requirements and store the concentrate in the reach-in refrigerator (See QA section for examples of paperwork).

8.0 CAUSTIC AND ACID CLEANUP (OPTIONAL)

8.1 Certain samples may require additional cleanup before column chromatography in order to achieve acceptable detection limits. The following is a procedure which involves acid and caustic wash of the sample.

8.2 After step 5.9 is completed, quantitatively transfer the extract to a 125-ml separatory funnel.

8.3 Wash the extract with 30 ml of 20% aqueous potassium hydroxide by shaking for 10 minutes. Let stand for 10 minutes and discard the aqueous layer.

8.4 Wash the extract with 25 ml of distilled deionized water by shaking for 2 minutes. Let stand for 10 minutes and discard the aqueous layer.

8.5 Slowly add 50 ml of concentrated sulfuric acid to the extract and shake for 10 minutes. Let stand for 10 minutes and discard the acid layer. Repeat until acid layer remains colorless after extraction.

8.6 Wash extract with 20 ml of distilled/deionized water by shaking for 2 minutes. Let stand for 10 minutes and discard the aqueous layer.

8.7 Quantitatively transfer the organic layer to a 20-ml scintillation vial and dry over 10 g anhydrous sodium sulfate.

8.8 Reduce the extract volume to approximately 1 ml.

8.9 Proceed to dual column cleanup if required.

9.0 GLASSWARE PREPARATION PROCEDURES

9.1 Rinse glassware with the last solvent used in it. Wash with hot water containing detergent. Rinse with copious amounts of tap water and several portions of distilled water; drain dry. Rinse with high purity acetone and hexane and allow to air dry. When dry, heat in a muffle furnace to 400°C for 1 hour. (Volumetric glassware should not be heated in a muffle furnace.) Remove from the oven when cool; store inverted in a clean environment.

SAMPLE PREPARATION PROCEDURES FOR DIOXIN IN SOILS AND SEDIMENTS

1.0 SUMMARY OF METHOD

1.1 Soil and sediment samples are extracted with a methanol/hexane mixture using a jar extraction technique after spiking the sample with $^{37}Cl_4$-2,3,7,8-TCDD and $^{13}C_{12}$-2,3,7,8-TCDD. After filtering, sample cleanup procedures are followed before analysis by GC/MS is performed.

2.0 REAGENTS

2.1 Spiking standard solution (contains both internal and surrogate standards). $^{13}C_{12}$-2,3,7,8-TCDD internal standard at a concentration of 500 ng/ml and $^{37}Cl_4$-2,3,7,8-TCDD surrogate standard at a concentration of 100 ng/ml, both in the same hexane solution. The standard ID number is 358:32-IFB SOIL. GC/MS IS mixture is prepared at a concentration of 200 ng/ml in isooctane.

2.2 Sulfuric acid (concentrated); ACS grade; specific gravity 1.84.

2.3 Potassium hydroxide; 20% aqueous. Prepare by cautiously adding, with stirring, 200 g of potassium hydroxide pellets to 800 ml of distilled/deionized water contained in a beaker in a cold water bath. After the potassium hydroxide has dissolved and the solution is at room temperature, transfer to a plastic bottle.

2.4 Methylene chloride; pesticide quality or equivalent.

2.5 Hexane; pesticide quality or equivalent.

2.6 Methanol; pesticide quality or equivalent.

2.7 Toluene; pesticide quality or equivalent.

2.8 Sodium sulfate; ACS, granular, anhydrous. Prepare by a 24 hour methylene chloride extraction. After preparation, store in an oven maintained at 110°C.

2.9 Silica gel; type 60, EM reagent, 70-230 mesh, or equivalent. Prepare by soxhlet extraction with methylene chloride overnight, drying, and then activating in an aluminum foil covered glass container for 24 hours at 130°C.

2.10 Alumina-neutral; Fisher brand, 80-200 mesh. Prepare by soxhlet extraction with methylene chloride, followed by drying and then activating, in an aluminum foil-covered glass container for 24 hours at 190°C.

2.11 Sulfuric acid; impregnated silica gel, 40% w/w. To prepare, add two parts (200 g) concentrated sulfuric acid to three parts silica gel (300 g) contained in a one-glass liter bottle equipped with a Teflon-lined screw cap. Mix thoroughly with a glass rod until no lumps are visible. Label the bottle with "Sulfuric Acid - impregnated silica gel"

and "Caution - contains concentrated sulfuric acid," the date prepared and the person preparing the reagent. All information will be recorded in the laboratory standard notebook.

2.12 Sodium hydroxide modified silica gel, 33% w/w. To prepare, add one part 1M NaOH to two parts activated silica gel contained in a glass jar equipped with a Teflon-lined screw cap. Mix thoroughly with a glass rod until no lumps are visible. Label bottle with "NaOH - impregnated silica gel," date prepared and the person preparing the reagent. All information will be recorded in lab standard notebook.

2.13 Carbopak C (activated carbon) on Celite: Prepare by thoroughly mixing 3.6 g of Carbopak C (80/100 mesh) and 16.4 g of Celite 545 in a 40-ml vial. Activate at 130°C for six hours. Store in a desiccator. CAUTION: Check each new batch of mixed Carbopak/Celite to ensure TCDD recovery of > 50%. Subject the low level concentration calibration solution to the procedure in section 6.3.0-6.3.12 and measure the quantity of labeled and unlabeled TCDD. Label bottle with "Activated Carbopak/Celite," the date prepared and the person preparing the reagent. All information will be recorded in the lab standard notebook.

3.0 CAUTIONS

3.1 Samples received for this preparation procedure are of unknown composition but may be potentially carcinogenic, mutagenic, toxic or in other ways hazardous.

3.2 It is mandatory that the initial weighing of the samples, addition of isotopically labeled 2,3,7,8-TCDD, and mixing of the sample, be performed inside the designated dioxin hood in the high hazard laboratory.

3.3 Personnel involved in this sample preparation procedure should be thoroughly familiar with laboratory SOPs on the processing of high hazard samples.

4.0 EQUIPMENT AND MATERIALS

4.1 Electrical platform shakers.

4.2 500-ml glass jars (amber) with Teflon-lined screw caps to be used on the platform shakers.

4.3 Kuderna-Danish concentration apparatuses, consisting of a three-ball macro Snyder column, a 500-ml evaporative flask, and a 10-ml graduated concentrator tube.

4.4 Mini vials (Reacti-vials); 1.0 ml. capacity with conical interiors and graduated at 0.1 ml; equipped with Teflon-faced rubber septa and screw caps.

4.5 Concentration vials; 20 ml screw top, septum sealed, scintillation vials.

4.6 Concentration device; nitrogen blowdown apparatus, Organomation and Pierce concentration devices, or equivalent.

4.7 Glass filtering funnels, short stem.

4.8 Filter paper, Whatman No. 4 or equivalent.

5.0 SAMPLE EXTRACTION

Jar extraction. NOTE: Extremely wet samples may require centrifuging to remove water before addition of sodium sulfate. The liquid will be analyzed as a water sample (see water method).

5.1 Prepare designated dioxin hood as instructed in laboratory SOP for processing high hazard samples. This includes obtaining, preparing, and labeling the requisite number of 500-ml jars before placing in the hood.

5.2 Transfer 10 g of the soil or sediment (wet weight) to a tared 500 ml jar (± 0.5 g weighed to 3 significant figures).

5.3 Spike the sample with 100 µl of spiking solution, containing both internal and surrogate standards, adding the solution at several sites over the surface of the sample.

5.4 Add 20 g of anhydrous sodium sulfate. Stir the mixture thoroughly with a stainless steel spatula.

5.5 Allow the mixture to stand for two hours, mix thoroughly with the spatula and allow the mixture to stand for an additional six hours. Mix the sample again, ensuring that no lumps are present. During the period that the samples set, turn off the light in the hood and close the hood sash.

5.6 Add 20 ml of methanol, stir, and add 150 ml of hexane. Remove the spatula while rinsing it with hexane.

5.7 Place the extraction jar containing the soil, sodium sulfate, and solvents in the shaker and shake for at least three hours.

5.8 After the three hour shaking period, turn off the shaker and allow the solids to settle before proceeding.

5.9 Into the top of a 500-ml KD flask, insert a glass filter funnel containing Whatman No. 4 filter paper (or equivalent) rinsed with hexane.

5.10 Carefully decant the extract through the filter funnel, using a stainless steel spatula to facilitate the transfer process.

5.11 Rinse the inside of the jar and contents with hexane, using the spatula to mix the hexane with the solid material remaining in the beaker.

5.12 Decant the washing into the filter funnel, using the spatula to facilitate the transfer.

5.13 Concentrate the extract volume to approximately 3 ml with a Kuderna-Danish apparatus.

5.14 Transfer the concentrated extract to a 20-ml scintillation vial. Rinse the evaporator flask with three 5-ml portions of hexane; transfer each rinse into the scintillation vial. During these transfers, evaporate the solvent using a gentle stream of dry nitrogen.

5.15 After the final rinse has been added, reduce the extract volume to approximately 1 ml.

6.0 DUAL COLUMN CLEAN-UP

6.1 Either the necessary columns have been prepared and are in a drying oven, or the silica gel and alumina columns need preparation. If columns need to be prepared, obtain enough 1 X 20 cm columns (for the silica gel columns) and 1 X 30 cm columns for the alumina columns.

6.2 Place a small wad of glass wool in the bottom of the 1 X 20 cm columns and add 1.0 g of silica gel, 2.0 g of the NaOH impregnated silica gel, 1 g of silica gel, 4 g of the 40% w/w sulfuric acid treated silica gel and 2.0 g of silica gel. Gently tap the columns to allow the contents to settle after each addition.

6.3 Place a small wad of glass wool in the bottom of the 1 X 30 cm columns and add 6 g of alumina and a 1 cm layer of sodium sulfate. Gently tap the columns to allow the contents to settle.

6.4 Attach the silica gel and alumina columns to lab supports so that the silica gel column is above the alumina column and the lower tip of the silica gel column is inserted into the top of the alumina column.

6.5 Rinse both columns with hexane to remove any air bubbles and discard the hexane.

6.6 When the silica gel and alumina columns have stopped dripping hexane, place a clean Erlenmeyer flask under the alumina column and transfer the hexane concentrate obtained from step 5.15 to the top of the silica gel column. Rinse the scintillation vial with two 0.5 ml portions of hexane and add the washings to the top of the silica gel column.

6.7 Wash the silica gel column with 120 ml of hexane, added in aliquots by means of a transfer pipet. Remove the silica gel column.

6.8 Wash the alumina column by placing 20 ml of hexane on the column and eluting until the hexane has dropped below the sodium sulfate layer. Remove the Erlenmeyer flask and retain (with aluminum foil cap) until directions are given to discard.

6.9 Place a clean, labeled 125-ml Erlenmeyer flask under the alumina column and place 20 ml of 20% v/v methylene chloride in hexane on the alumina column, catching the eluent in the Erlenmeyer flask.

6.10 Place the 125-Erlenmeyer flask on the Organomation or Pierce concentration apparatus and reduce the volume of the solvent until less than 10 ml of solvent remains, but do not evaporate to dryness.

6.11 Quantitatively transfer the hexane extract to labeled 10-ml K-D concentrator tubes. Rinse the Erlenmeyer flask with several small (1 to 2 ml) portions of hexane, adding the washes to the K-D concentrator tube.

6.12 Transfer the K-D concentrator tubes to the concentration device and concentrate the hexane extract to approximately 1.0 ml, using a gentle stream of nitrogen and heat if necessary.

6.13 After the 1.0 ml volume has been obtained, remove the K-D concentrator tube and quantitatively transfer the contents of the K-D concentrator tube to designated conical mini-vials. Rinse the concentrator tube with two 0.5 ml portions of hexane and transfer the washes to the concentrate vial. Complete necessary paperwork (see QA section).

6.14 Store the hexane concentrate in a freezer until just prior to GC/MS analysis.

6.15 Concentrate the hexane to near dryness and add 50 µl of 200 ppb standard of an appropriate Is. Return sample to the reach-in refrigerator.

7.0 ACTIVATED CARBON CLEAN-UP

7.1 After GC/MS analysis of samples processed by the dual column method in Section 6.0, the possibility exists that certain matrices may produce indeterminable results. In such cases an activated carbon column clean-up technique will be employed.

7.2 Obtain from the desiccator the activated Celite/Carbopak (from Section 2.13).

7.3 Insert a small wad of glass wool into a small (7 mm O.D. X 15 cm) disposable pipet and, using vacuum aspiration at the pointed tip of the pipet, add the Celite/Carbopak mixture until a 2 cm column is obtained.

7.4 Prepare the column by adding the following solvents/solvent mixtures in the designated aliquot sizes: (place a clean 125 ml Erlenmeyer flask under the column)

- 2 ml toluene,
- 1 ml 75/20/5 (v/v/v) methylene chloride/methanol/benzene,
- 1 ml 50/50 (v/v) cyclohexane/methylene chloride,
- 2 ml hexane.

7.5 When the bottom of the hexane meniscus just touches the top of the Celite/Carbopak material, quantitatively transfer the 50 µl of sample onto the column.

7.6 Rinse the sample container with two 1-ml portions of hexane, adding the rinses onto the column.

7.7 Sequentially wash the column with the following solvents/solvent mixtures in the designated aliquot sizes:

- 1 ml 50/50 (v/v) cyclohexane/methylene chloride
- 1 ml 75/20/5 (v/v/v) methylene chloride/methanol/benzene.

7.8 Remove the 125-ml Erlenmeyer flask and replace with a concentrator tube or Reacti-vial.

7.9 Elute the TCDD from the column with 2 ml of toluene into the concentrator tube or Reacti-vial.

7.10 Store the toluene eluent in a freezer until the GC/MS analysis is to be performed.

7.11 Shortly before the analysis, concentrate the extract to near dryness and add 50 µl of isooctane for GC/MS analysis.

7.12 Complete any paperwork requirements and store the concentrate in the reach-in refrigerator (see QA section for examples of paperwork).

8.0 CAUSTIC AND ACID CLEANUP (OPTIONAL)

8.1 Certain samples may require additional cleanup before column chromatography in order to achieve acceptable detection limits. The following is a procedure which involves acid and caustic wash of the sample.

8.2 After step 5.15 is completed, quantitatively transfer the extract to a 125-ml separatory funnel.

8.3 Wash the extract with 30 ml of 20% aqueous potassium hydroxide by shaking for 10 minutes. Let stand for 10 minutes and discard the aqueous layer.

8.4 Wash the extract with 25 ml of distilled deionized water by shaking for 2 minutes. Let stand for 10 minutes and discard the aqueous layer.

8.5 Slowly add 50 ml of concentrated sulfuric acid to the extract and shake for 10 minutes. Let stand for 10 minutes and discard the acid layer. Repeat until acid layer remains colorless after extraction.

8.6 Wash extract with 20 ml distilled/deionized water by shaking for 2 minutes. Let stand for 10 minutes and discard the aqueous layer.

8.7 Quantitatively transfer the organic layer to a 20-ml scintillation vial and dry over 10 g anhydrous sodium sulfate.

8.8 Reduce the extract volume to approximately 1 ml.

8.9 Proceed to dual column cleanup if required.

9.0 GLASSWARE PREPARATION PROCEDURES

9.1 Rinse glassware with the last solvent used in it. Wash with hot water containing detergent. Rinse with copious amounts of tap water and several portions of distilled water; drain dry. Rinse with high purity acetone and hexane and allow to air dry. When dry, heat in a muffle furnace to 400°C for 1 hour. (Volumetric glassware should not be heated in a muffle furnace.) Remove from the oven when cool; store inverted in a clean environment.

D150-SS

2,3,,8-TETRACHLORO-P-DIOXIN ANALYSIS PROCEDURES BY MULTIPLE ION DETECTION (MID) HIGH RESOLUTION GAS CHROMATOGRAPHY/LOW RESOLUTION MASS SPECFROMETRY (HRGC/LRMS)

1.0 INTRODUCTION

1.1 This is a qualitative and quantitative (high resolution) GC/(low resolution) MS analysis specific for the 2,3,7,8 isomer of tetrachlorodibenzo-p-dioxin using selected ion monitoring. A sample is spiked with isotopically labeled $^{13}C_{12}$-2,3,7,8-TCDD as internal standard and with $^{37}Cl_4$-2,3,7,8-TCDD as surrogate. Quantitation is based on the response of native TCDD relative to the internal standard. Performance is based on surrogate standard results. Percent recovery of the IS/SURR mixture is based on the GC/MS internal standard.

2.0 SAFETY

2.1 Samples are sent to IT Corporation from suspected or known hazardous waste sites. Samples are to be handled from receipt to storage by qualified personnel only. Analysts must have a working knowledge of safety protocols and be adept at safety procedures. GC/MS instruments must be equipped with vapor contamination traps on the capillary split and sweep vents and on the rough pump effluent lines prior to use (see Safety SOP).

3.0 SET-UP AND INSTALLATION

3.1 Install a 60 meter, 0.25 mm ID, fused silica SP2330, 0.20 micron film thickness capillary column. Set the head pressure to approximately 20 to 25 psi and the split and sweep flows to 30 ml/min and 8 ml/min respectively.

3.2 Create a reasonable unit resolution tune, for PFTBA. Adjust the zero according to the instrument manufacturers suggested settings. Set the preamp sensitivity to 10^{-8} amps/volts. The electron multiplier must be set to achieve 800,000 area units for 2 ng of $^{13}C_{12}$-TCDD as injected for M/E 334. Calibrate the instrument.

3.3 Establish the following MID descriptors:

3.3.1 NAME "TC" for running column performance mixture

MID DESC: TC
INST: FINN CALI: CAL39

MASS DEFECT AT 100 AMU	30 MMU
MASTER RATE	1024
TOTAL ACQU TIME	0.318 SECS
TOTAL SCAN TIME	0.450 SECS
CENT SAMP INT	0.200 MS
CALI MASS RANGE	44 TO 614 AMU

INT NO.	BEGIN MASS	END MASS	TIME REQUEST	(SECS) ACTUAL	MPW	MFW	MA	TH	BL	ION
1	319.649	320.150	0.050	0.054	1	100	1	1	0	POS
2	321.649	322.150	0.050	0.052	1	100	1	1	0	POS
3	322.649	323.105	0.050	0.052	1	100	1	1	0	POS
4	327.649	328.150	0.050	0.054	1	100	1	1	0	POS
5	331.649	332.150	0.050	0.052	1	100	1	1	0	POS
6	333.649	334.150	0.050	0.052	1	100	1	1	0	POS

3.3.2 NAME "TD" for running standards and samples

MID DESC: TD
INST: FINN CALI CAL39

MASS DEFECT AT 100 AMU	30 MMU
MASTER RATE	1024
TOATL ACQU TIME	0.430 SECS
TOTAL SCAN TIME	0.450 SECS
CENT SAMP INT	0.200 MS
CALI MASS RANGE	44 TO 614 AMU

INT NO.	BEGIN MASS	END MASS	TIME REQUEST	(SECS) ACTUAL	MPW	MFW	MA	TH	BL	ION
1	256.649	257.150	0.050	0.052	1	100	1	1	0	POS
2	319.649	320.150	0.050	0.054	1	100	1	1	0	POS
3	321.649	322.150	0.050	0.052	1	100	1	1	0	POS
4	327.649	328.150	0.050	0.054	1	100	1	1	0	POS
5	331.649	332.150	0.050	0.052	1	100	1	1	0	POS
6	333.649	334.150	0.050	0.052	1	100	1	1	0	POS
7	315.649	316.150	0.050	0.052	1	100	1	1	0	POS
8	317.649	318.150	0.050	0.054	1	100	1	1	0	POS

3.4 Set the GC conditions as follows:

Injection Port Temp	260°C
Separator Temp	260°C
Initial Temp	70°C
Initial Time	4 min
Ramp Rate 1	20°C/min
Temp 2	200°C
Hold Time 2	0 min
Ramp Rate 2	4°C/min
Temp 3	260°C
Hold Time 3	5 min*
Split/Sweep	85 sec
Filament/Mult turn on time	10 min

* Hold for at least 2 min beyond the retention time of the last isomer of TCDD in the performance mixture.

3.5 Analyze the 7 isomer EPA test mixture. If no isomers are co-eluting with 2,3,7,8-TCDD, conditions stated above are acceptable to proceed. If co-elution does occur with 2,3,7,8-TCDD, the column must be changed or conditions modified in order to stop co-elution.

3.6 Create a library with the following entries:

Entry 1 - ^{13}C - 2,3,7,8-TCDD (Internal standard)
- delete all masses except 322/334
- amount = 1000
- units = pg/µl
- quant mass = 332

Entry 2 - ^{13}C - 2,3,7,8-TCDD (Secondary ion)
- delete all masses except 322/334
- amount = 1000
- units = pg/µl
- quant mass = 334

Entry 3 - 2,3,7,8-TCDD
- delete all masses except 320, 322, 332, 257
- amount = 1000
- units = pg/µl
- quant mass = 320

Entry 4 - 2,3,7,8-TCDD (Secondary ion)
- delete all masses except 320, 322, 332, 257
- amount = 1000
- units = pg/µl
- quant mass = 322

Entry 5 - 2,3,7,8-TCDD (Tertiary ion)
- delete all masses except 320, 322, 332, 328
- amount = 1000
- units = pg/µl
- quant mass = 257

Entry 6 - ^{37}Cl -2,3,7,8-TCDD (Recovery int. std.)
- delete all masses except 328
- amount = 200
- units = pg/µl
- quant mass = 328

4.0 ANALYSIS

4.1 General Description

4.1.1 A five point calibration consisting of a 200 pg/µl (1 ppb equivalent), a 1000 pg/µl (5 ppb equivalent), a 5000 pg/µl (25 ppb equivalent), a 20,000 pg/µl (100 ppb equivalent), and a 20,000 pg/µl (200 ppb equivalent) standard must be run in triplicate and a linear response curve generated before samples are

analyzed. The 200 pg/µl standard is analyzed at the beginning of each eight hour shift to verify system performance and conformity to the multipoint calibration (see QA section on standard paperwork). Samples are received in 50 µl volumes and require no further preparation by the GC/MS laboratory.

4.1.2 COMPOSITION OF CONCENTRATION CALIBRATION SOLUTIONS

Solution No.	Concentration of 2,3,7,8-TCDD			
	Isotopically Labeled			Unlabeled
	$^{13}C_{12}$	$^{37}Cl_4$	Is	
1	1 ng/µL	0.06 ng/µL	0.2 ng/µl	0.2 ng/µL
2	1 ng/µL	0.12 ng/µL	0.2 ng/µl	1 ng/µL
3	1 ng/µL	0.2 ng/µL	0.2 ng/µl	5 ng/µL
4	1 ng/µL	0	0.2 ng/µl	20 ng/µL
5	1 ng/µL	0	0.2 ng/µl	40 ng/µL

4.2 Procedures for GC/MS Analysis Initial Calibration

4.2.1 The GC conditions for all standards, samples, and the column performance mixture are as stated in Section 3.4.

4.2.2 Tune and calibrate the instrument as in step 3.2 or verify that the instrument has been tuned and calibrated within the past week and has performed satisfactorily when last used. If the method has not been performed successfully within the last seven days, check the tune and recalibrate.

4.2.3 Acquire the seven isomer EPA test mix. If no isomers are co-eluting with 2,3,7,8-TCDD, proceed with 4.2.4. If co-elution does occur, the conditions must be modified or the column must be changed. The MID descriptor TC must be used for this analysis (section 3.3).

4.2.3.1 Determine and document acceptable system performance with the following criteria:

A. Five data points for each GC peak are acquired.

B. GC column performance -- The valley between 2,3,7,8-TCDD and the peaks representing all other TCDD isomers must be resolved with a valley $\leq$25%. Valley % = x/y X 100 when y is peak height of 2,3,7,8-TCDD and x is baseline to valley height (Fig. 1).

C. Ratio of integrated ion current for m/z 320 to m/z 322 for 2,3,7,8-TCDD must be $\geq$ 0.67 and $\leq$ 0.87.

D. MS resolution -- Ratio of integrated ion current for m/z 323 relative to m/z 322 for unlabeled 2,3,7,8-TCDD should be $\geq$ 0.07 and $\leq$ 0.20.

E. Ratio of integrated ion current for m/z 332 to m/z 334 for $^{13}C_{12}$-2,3,7,8-TCDD must be $\geq$ 0.67 and $\leq$ 0.87.

F. Response factor for $^{37}Cl_4$-2,3,7,8-TCDD relative to $^{13}C_{12}$-2,3,7,8-TCDD must be within $\pm$ 10% of the mean value established by triplicate analyses of the concentration calibration solutions (section 4.1.2).

4.2.3.2 Remedial action must be taken if all of the criteria are not met.

4.2.4 Using the same GC conditions that produced acceptable results with the performance solution, analyze 2 µl of each of the five concentration calibration solutions in section 4.1.2. MID descriptor TD (3.3.2) is used for all calibration standards and samples.

4.2.4.1 Repeat section 4.2.4 two times to produce triplicate data sets for each solution.

4.2.5 Calculate the response factor for $^{37}Cl_4$-2,3,7,8-TCDD and for unlabeled 2,3,7,8-TCDD relative to $^{13}C_{12}$-2,3,7,8-TCDD:

$$RF = \frac{A_x \bullet Q_{is}}{A_{is} \bullet Q_x}$$

where A_x = integrated ion abundance (corrected for native contribution) of m/z 328 for $^{37}Cl_4$-2,3,7,8-TCDD or the sum of integrated ion abundances of m/z 320 and m/z 322 for unlabeled 2,3,7,8-TCDD,

A_{is} = the sum of integrated abundances of m/z 332 and m/z 334 for $^{13}C_{12}$-2,3,7,8-TCDD,

Q_{is} = quantity of $^{13}C_{12}$-2,3,7,8-TCDD, and

Q_x = quantity of unlabeled 2,3,7,8-TCDD or $^{37}Cl_4$-2,3,7,8-TCDD injected.

RF is a unitless number; units used to express quantities must be equivalent.

4.2.6 For both $^{37}Cl_4$-2,3,7,8-TCDD and unlabeled 2,3,7,8-TCDD, calculate the mean RF and its relative standard deviation (RSD) from triplicate analyses of each of the five concentration calibration solutions. Variation of the RF calculated for each compound at each concentration level must not exceed 10% RSD. If the five

mean RFs for each compound do not differ by more than ± 10%, the RF can be considered to be independent of analyte quantity for the calibration concentration range, and the mean of the five mean RFs shall be used for concentration calculations.

4.2.7 Fill out all necessary paperwork for the standard calibration QA/QC (see QA/QC section for paperwork).

4.2.8 Plot the response factor vs concentration for the five point calibration curve for QA/QC reporting.

4.3 Calibration before the start of each eight hour shift

4.3.1 Inject 2 µl of the performance check solution as in 4.2.3 - 4.2.3.1F.

4.3.2 Inject 2 µl of the concentration calibration solution No. 1 (200 pg/µl) determine and document acceptable performance for

4.3.2.1 MS sensitivity - signal-to-noise (S/N) ratio of ≥ 2.5 for m/z 257 and ≥ 10 for m/z 322 for unlabeled 2,3,7,8-TCDD. The ratio of integrated ion current for m/z 257 to m/z 322 must be ≥ 0.20 and ≤ 0.45.

4.3.2.2 Measured response factor for unlabeled 2,3,7,8-TCDD relative to $^{13}C_{12}$-2,3,7,8-TCDD is within ± 10% of the mean values established (Section 4.2) by triplicate analyses of the concentration calibration solutions.

4.3.2.3 If both these criteria are met, fill out the bottom portion of form 248A (QA/QC section). If the RF ratios are within 10% of the calibration average, samples may then be analyzed. DO NOT UPDATE the shift standard to the response list. Use R;S;T only. If the 10% criteria are not met, reanalyze the shift standard. If still out of bounds, a new multipoint must be run. Multipoints may continue to be used for as long as the shift standards conform to this criteria. Xerox a copy of Form 248A for inclusion with the shift standard packages and an extra copy to be placed in the instrument log book so that subsequent shift standard entries may be made on the same form.

4.3.2.4 Acquire sample analyses. Samples may be analyzed following a successful shift standard analysis. Performance standards must be rerun within every eight hours. The injection procedure must be carefully adhered to to avoid cross contamination. If the background of a sample analysis remains high towards the end of an acquisition, the column should be baked out for an extra period of time to avoid possible chromatographic carryover into the next sample injection.

Septa should be changed after approximately 40 injections. Capillary injection port liners should be cleaned or exchanged with every other septum change. Good sense and experience prevail.

4.3.3 After eight hours from the injection of the column performance check solution (4.3.1), the tune is over and the performance check solution must be analyzed again. If all criteria (4.2.3.1 a-f) are met, the samples analyzed during that eight hour period are acceptable. If the criteria are not met, the samples must be reanalyzed.

4.4 For all injections, a hot needle injection technique is used.

4.4.1 Injection Technique (Hot Needle) - The syringe must be thoroughly cleaned between injections to avoid cross-contamination. Remove the plunger between injections and wipe it thoroughly with a Kimwipe. Rinse the syringe with ten to fifteen full syringe volumes of hexane solvent wash. Replace the solvent wash with pesticide quality hexane daily. If a Hamilton syringe cleaner is available that is equipped with a vacuum source, use this also. Do not use the Hamilton syringe cleaner if there is no vacuum pump attached. Insert the needle into the septum port, wait approximately ten seconds for the needle to heat, then pump the plunger back and forth a few times. Rinse with the solvent wash hexane again. Work the plunger up and down in the syringe barrel to reduce excess hexane wash. There should be approximately 0.5 µl of solvent left in the syringe barrel following this final rinse.

Draw back the plunger so that there are about 2 µl of air in the barrel. Draw 2.1 ml of sample into the needle. Usually to get a total of 2 µl of sample, it is necessary to pull the plunger back approximately 1.2 µl. The sample should be drawn up into the barrel and the amount confirmed to be 2 µl. If it is not, the sample should be expelled and process repeated.

After getting 2.0 µl of sample into the barrel, insert the needle into the injector port and wait 6 seconds. Rapidly make the injection.

After making the injection, remove the needle as quickly as possible. As soon as the injection is made, start the GC.

4.5 Identification criteria for native 2,3,7,8-TCDD

4.5.1 Retention time (at maximum peak height) of the sample component must be within 3 seconds of the retention time of the $^{13}C_{12}$-2,3,7,8-TCDD. Retention times are required for all chromatograms, but scan numbers are optional. These parameters should be printed next to the appropriate peak.

4.5.2 The integrated ion currents detected for m/z 257, 320, and 322 must maximize simultaneously. If there are peaks that will

affect the maximization or quantitation of peaks of interest, attempts should be made to narrow the scan window to eliminate the interfering peaks. This should be reported on a separate chromatogram.

4.5.3 The integrated ion current for each analyte and surrogate compound ion (m/z 257, 320, 322 and 328) must be at least 2.5 times background noise and must not have saturated the detector; internal standard ions (m/z 332 and 334) must be at least 10 times background and must not have saturated the detector.

4.5.4 Relative abundance of m/z 257 to m/z 322 should be $\geq$ 20% and $\leq$ 45%.

4.5.5 Abundance of integrated ion counts detected for m/z 320 must be $\geq$ 67% and $\leq$ 87% of integrated ion counts detected for m/z 322.

5.0 DELIVERABLES

5.1 Each sample "package" must include the following:

a) RIC (1000 - end of run)
b) Complete quantitation report. (Input area and scan No. manually if missed)
c) (EICP of m/e 332; m/e 334; m/e 316, and m/e 318)
d) (EICP of 320; 322; 332; and 257)
e) (EICP of 320; 322; 332; and 328)
f) Quan (320; 322; 257; 5 scans) The center of the 5 scan window is the retention time of $^{13}C_{12}$-2,3,7,8-TCDD
g) A standard package including all of (a) through (e) plus an attached copy of the TCDD calibration summary (Form 248A)

5.1.1 See QA/QC section for batch report deliverables

6.0 TOTAL ION CONFORMATION (Highest positive sample in each batch (see 4.1.8 QA/QC Section 5.9).

6.1 Inject 2 µl of a DFTPP solution into the GC/MS system using the same GC conditions as stated in section 3.4 with the MS scanning from 35-450 at sec/scan. Required calibration criteria for DFTPP shall be:

m/z	Relative Intensity
51	30 - 60 percent of base peak
68	< 2 percent of m/z = 69
70	< 2 percent of m/z = 69
127	40 - 60 percent of base peak
197	< 1 percent of base peak
198	100 percent (base peak)
199	5 - 9 percent of base peak
275	10 - 30 percent of base peak
365	> 1 percent of base peak
441	less than m/z = 443
442	> 40 percent of base peak
443	17 - 23 percent of m/z = 442

6.2 Inject 4 µl of the positive TCDD sample using the same GC conditions in section 3.4. MS data acquisition requirements shall be:

6.2.1 Cycle time $\leq$ 1.5 seconds.

6.2.2 Acquisition of $\geq$ 5 spectra during elution of 2,3,7,8-TCDD from the GC.

6.2.3 MS scanning from 150-350 at 1 sec/scan.

6.3 Subtract an appropriate background spectrum, and plot a spectrum of 2,3,7,8-TCDD after background subtraction. (The person responsible for MS data interpretation is responsible for demonstrating that the background spectrum selected for subtraction was an appropriate spectrum.) Provide a hard copy of the background spectrum, the TCDD spectrum before subtraction, and the TCDD spectrum after subtraction. The quality of the plotted spectrum will be affected by other sample components that have approximately the same GC retention time and will be highly variable. Desired spectral features are:

Base peak = m/z 322
Ratio of m/z 320 to 322 = 0.77
Ratio of m/z 320 to 324 = 1.58
Ratio of m/z 257 to 322 = 0.32
Ratio of m/z 257 to 259 = 1.03
Ratio of m/z 194 to 196 = 1.54
m/z 160 and 161 = $\geq$ 10% of m/z 322

Because $^{13}C_{12}$-2,3,7,8-TCDD, the internal standard, is present in every sample and has essentially the same retention time as unlabeled 2,3,7,8-TCDD, the spectrum after background subtraction will represent a mixture. When $^{13}C_{12}$-2,3,7,8-TCDD is present at a higher concentration than unlabeled 2,3,7,8-TCDD, the resultant spectrum must be normalized to m/z 322 to demonstrate desired spectral features.

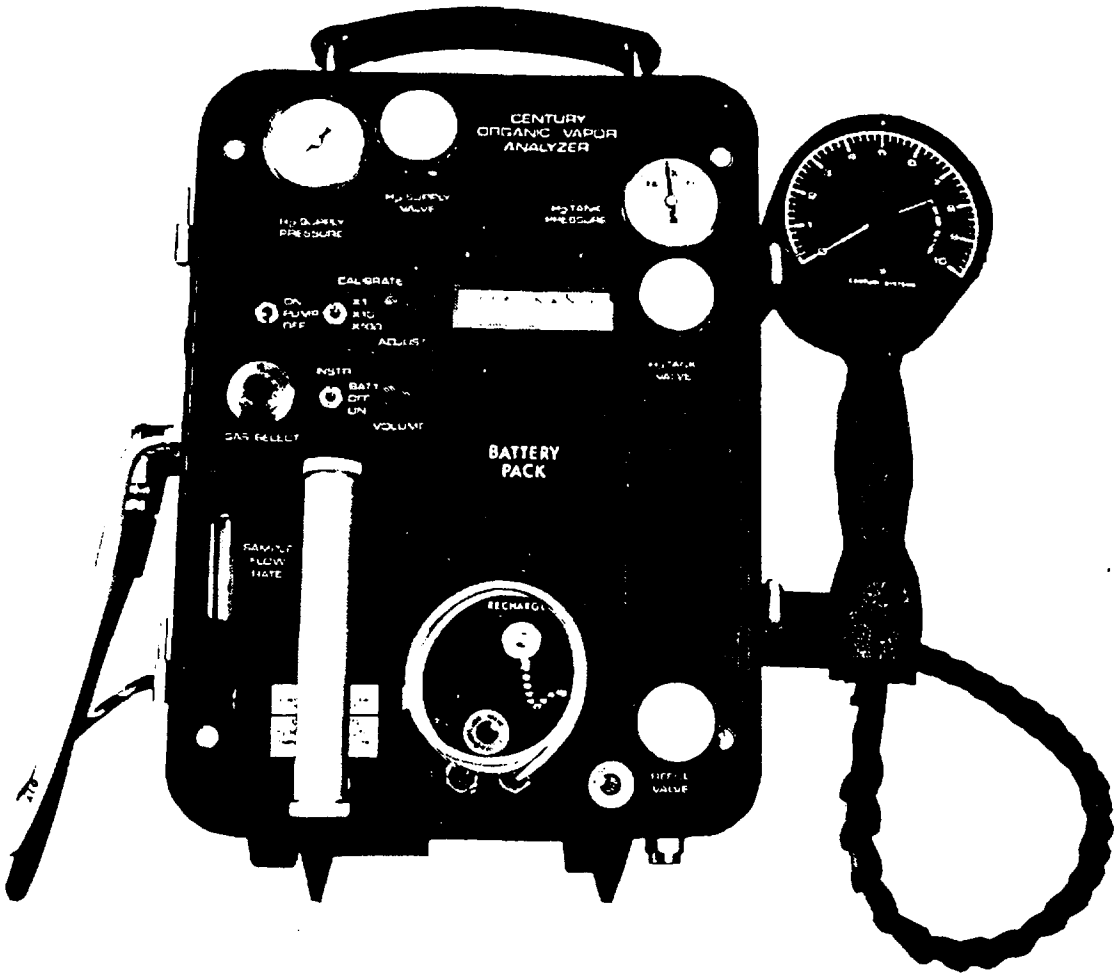
7.0 SEE QA/QC SECTION FOR EXAMPLES OF ALL PAPERWORK NECESSARY FOR COMPLETE REPORTING OF GC/MS DATA

APPENDIX
E

Instruction & Service Manual

MI

2R900AC



CENTURY SYSTEMS

Portable Organic Vapor Analyzer
Model OVA-128

Foxboro Analytical
A Division of The Foxboro Company

FOXBORO®

damaging the batteries. When finished, move the battery charger switch to OFF and disconnect from the Side Pack Assembly.

The following are special instructions relative to batteries which have been allowed to completely discharge.

It has been established that the above battery recharging procedures may not be sufficient when the operator of the instrument has inadvertently left the INSTR Switch ON for a period of time without recharging and allowed the battery to completely discharge.

When this happens and the above procedures fail to recharge the battery, the following should be accomplished:

1) Remove the battery from the instrument case.
2) Connect to any variable DC power supply.
3) Apply 40 volts at 1/2 amp maximum.
4) Observe the meter on the power supply frequently and as soon as the battery begins to draw current, reduce the voltage on the power supply at a slow rate until the meter reads approximately 15 volts. NOTE: The time required to reach the 15 volt reading will depend on degree of discharge.
5) Repeat steps a), b), c), and d) above to continue charging.

2.7.2 DC CHARGER

a) The optional DC charger is designed to both charge the battery and to provide power for operating the instrument from a 12 volt DC source, such as vehicle power.
b) Connect the DC charger cord to the connector on the battery cover of the Side Pack Assembly. Plug the line cord into the vehicle cigarette lighter or other power source connection.
c) In mobile applications, the DC charger is used to supply vehicle power to the instrument. Therefore, it may be left connected at all times.

2.8 CHARCOAL FILTERING

When it is desired to preferentially remove the heavier hydrocarbons, such as those associated with automobile exhaust, gasoline, etc., simply remove the pickup fixture from the end of the probe and install the optional charcoal filter assembly.

This same charcoal filter assembly can be installed directly into the Readout Assembly by using the adapter provided.

2.9 MOISTURE FILTERING

Filtering of moisture in the sample is not normally required. However, when moving in and out of buildings in cold weather, excessive condensation can form in the lines and detector chamber. In this case, the charcoal filter adapter can be filled with a desiccant such as "Drierite" which will filter out the moisture contained in the sample.

SECTION 3

SUMMARIZED OPERATING PROCEDURES

3.1 GENERAL

The procedures presented in this section are intended for use by personnel generally familiar with the operation of the instrument. Section 2 presents the comprehensive detailed operating procedures.

It is assumed that, prior to start up the positions of all switches and valves are in shut down configuration as described in paragraph 3.3.

3.2 START UP

a) Move PUMP Switch to ON and check battery condition by moving the INSTR Switch to the BATT position.
b) Move INSTR Switch to ON and allow five (5) minutes for warm-up.
c) Set Alarm Level Adjust Knob on back of Readout Assembly to desired level.
d) Set CALIBRATE Switch to X10 position, use CALIBRATE Knob and set meter to read 0.
e) Move PUMP Switch to ON position then place instrument panel in vertical position and check SAMPLE FLOW RATE indication.
f) Open the H2 TANK VALVE and the H2 SUPPLY VALVE.
g) Depress Igniter Button until burner lights. Do not depress Igniter Button for more than six (6) seconds. (If burner does not ignite, let instrument run for several minutes and again attempt ignition.)
h) Use CALIBRATE Knob to "zero" out ambient background. For maximum sensitivity below 1 ppm, set CALIBRATE Switch to X1 and readjust zero on meter. To avoid false flame-out alarm indication, set meter to 1 ppm with CALIBRATE Knob and make differential readings from there.

3.3 SHUT DOWN

a) Close the H2 SUPPLY VALVE and the H2 TANK VALVE.
b) Move the INSTR Switch and PUMP Switch to OFF.
c) Instrument is now in shut down configuration.

SECTION 4

CALIBRATION

4.1 GENERAL

The OVA is capable of responding to nearly all organic compounds. For precise analyses it will be necessary to calibrate the instrument with the specific compound of interest. This is especially true for materials containing elements other than carbon and hydrogen.

The instrument is factory calibrated to a methane in air standard. However, it can be easily and rapidly calibrated to a variety of organic compounds. A GAS SELECT control is incorporated on the instrument panel which is used to set the electronic gain to a particular organic compound.

Internal electronic adjustments are provided to calibrate and align the electronic circuits. There are four (4) such adjustments all located on the electronics board. One adjustment potentiometer, R-38, is used to set the power supply voltage and is a one-time factory adjustment. The remaining three adjustments, R-31, R-32 and R-33 are used for setting the electronic amplifier gain for each of the three (3) calibrate ranges. Access to the adjustments is accomplished by removing the instrument from its case. Figure 4-1 indicates the location of the adjustments.

4.2 ELECTRONIC ADJUSTMENTS

Primary calibration of this instrument is accomplished at the factory using methane in air sample gases.

4.2.1 GAIN ADJUSTMENT

a) Place instrument in normal operation with CALIBRATE Switch set to X10 and GAS SELECT control set to 300.
b) Use the CALIBRATE ADJUST (zero) Knob and adjust the meter reading to zero.
c) Introduce a methane sample of a known concentration (near 100 ppm) and adjust trimpot R-32 on circuit board (see Figure 4-1 for location) so that meter reads equivalent to the known sample.
d) This sets the instrument gain for methane with the panel mounted gain adjustment (GAS SELECT) set at a reference number of 300.
e) Turn off H2 SUPPLY VALVE to put out flame.

4.2.2 BIAS ADJUSTMENT

a) Leave CALIBRATE Switch on X10 position and use CALIBRATE ADJUST (zero) Knob to adjust meter reading to 4 ppm.
b) Place CALIBRATE Switch in X1 position and, using trimpot R-31 on circuit board, adjust meter reading to 4 ppm. (See Figure 4-1)
c) Move CALIBRATE Switch to X10 position again. Use CALIBRATE ADJUST (zero) Knob to adjust meter to a reading of 40 ppm.
d) Move CALIBRATE Switch to X100 position and use trimpot R-33 on circuit board to adjust meter reading to 40 ppm.
e) Move CALIBRATE Switch to X10 position and use CALIBRATE ADJUST (zero) Knob to adjust meter reading to zero.
f) Unit is now balanced from range to range, calibrated to methane, and ready to be placed in normal service.

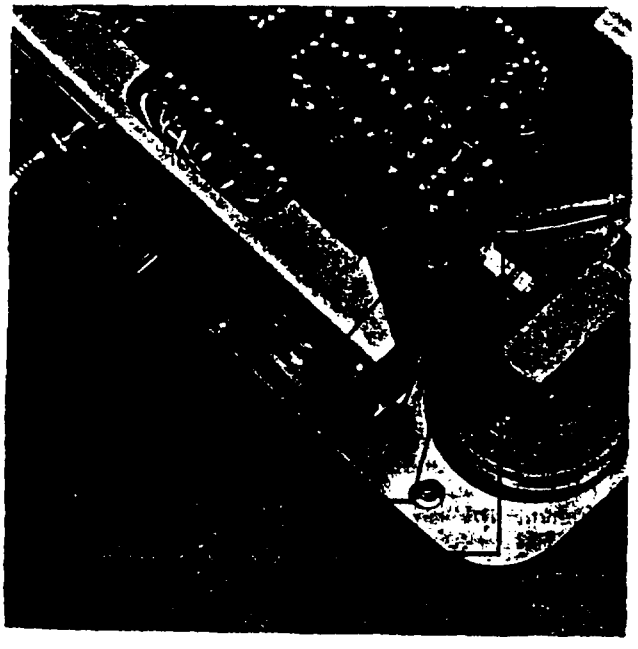


FIGURE 4-1. LOCATION OF ELECTRONIC ADJUSTMENTS
(Model OVA-118 shown; location typical to OVA-128)

4.3 CALIBRATION TO OTHER ORGANIC VAPORS

4.3.1 SETTING GAS SELECT CONTROL (Span)

Primary calibration of the instrument is accomplished using a known mixture of a specific organic vapor compound. After the instrument is in operation and the "normal background" is "zeroed out", draw a sample of the calibration gas into the instrument. The GAS SELECT Knob on the panel is then used to shift the readout meter indication to correspond to the concentration of the calibration gas mixture.

The instrument is then calibrated for the vapor mixture being used. After this adjustment, the setting on the "digidial" is read and recorded for that particular organic vapor compound. This exercise can be performed for a large variety of compounds and when desiring to read a particular compound the GAS SELECT control is turned to the predetermined setting for the compound. Calibration on any one range automatically calibrates the other two ranges.

4.3.2 USING EMPIRICAL DATA

Relative response data may be obtained, which can then be used to estimate concentrations of various vapors. With the instrument calibrated to methane, obtain the concentration reading for a calibration sample of the test vapor. The relative response, in percent, for that test vapor would then be the concentration *read/concentration of the calibrated sample* X 100.

4.3.3 PREPARATION OF CALIBRATION STANDARDS

4.3.3.1 COMMERCIAL SAMPLES

Commercially available standard samples offer the most convenient and reliable calibration standards and are recommended for the most precise analyses. Always remember to obtain the cylinder with the desired sample and the "balance as air". Sample should be drawn from the cylinder into a collapsed sample bag, then drawn from the bag by the instrument to prevent a pressure or vacuum at the sample inlet.

4.3.3.2 PURE GASEOUS SAMPLES

Obtain a large collapsible sample bag, preferably polyethylene such as a 40 gallon trash can liner. Insert a tube into the bag opening and tie shut around the tube. The tubing should have a shut-off valve or plug and be suitable for connecting the OVA input tube. Determine the volume of the bag by appropriate means (i.e., wet-test meter, dimensions of the bag, etc.). Forty gallon polyethylene bags provide a volume of approximately 140-160 liters. For gas samples, flush a 10 cc hypodermic syringe with the compound to be tested and then inject a 10 cc sample through the wall of the air-filled bag. Immediately after withdrawing the needle, cover the hole with a piece of plastic tape. Allow a few minutes for the sample to completely diffuse throughout the bag. Agitation will ensure complete diffusion. Connect the outlet tube to the OVA and take a reading. To verify repeatability of sampling technique, disconnect the bag and inject a second sample of the gas into the bag without emptying. Since only 2 or 3 liters will have been removed, the overall volume change will be small and the instrument reading should now be twice that of the

original. The concentration in ppm (V/V) will be equal to the sample size in cc divided by the volume of the bag in liters times 1000. For example, a 10 cc gas sample when placed in a 160 liter bag will provide a sample of 63 ppm, i.e., 10 X 1000/160 equals 63 ppm.

4.3.3.3 GASEOUS AND LIQUID SAMPLES (Alternate Method)

Obtain a five (5) gallon glass bottle and determine its volume by measuring the volume of water needed to fill it (use of a 1000 ml graduated cylinder, obtainable from scientific supply houses, is convenient). Another approach is to weigh the empty bottle, fill it with water and weigh again. The difference between the two values is the weight of water. By multiplying the weight of water in pounds by 0.455, you obtain the volume of the bottle in liters. Empty the water out and allow the bottle to dry. Place a one-foot piece of plastic tubing in the flask to aid in mixing the vapors uniformly with the air. The volume of such a bottle should be about 20 liters, which is 20,000 ml. If the volume were 20,000 ml, then a 2 ml sample of a gas placed in the bottle would be equivalent to 200 ml per 2 million ml or 100 ppm (V/V). Use of a gas tight syringe, readable in 0.01 ml, allows the preparation of mixtures in the 1 - 2 ppm range, which are sufficient for the quantitative estimation of concentrations. A rubber stopper is loosely fitted to the top of the bottle and the needle of the syringe placed inside the jug neck and the stopper squeezed against the needle to decrease leakage during sample introduction. Inject the sample into the bottle and withdraw the needle without removing the stopper. Put the stopper in tight and shake the bottle for a few minutes with sufficient vigor that the plastic tubing in the bottle moves around to ensure good mixture of the vapors with the air.

For liquid samples, use of the following equation will allow the calculation of the number of microliters of organic liquid needed to be placed into the bottle to make 100 ppm (V/V) of vapor.

V1 equals V2 X Mw/244D

- V1 - Volume of liquid in microliters needed to make an air mixture of 100 ppm (V/V)
- V2 - Volume of bottle in liters
- Mw - Molecular weight of substance
- D - Density of substance

This procedure has the advantage that you can see when all of the organic liquid has vaporized and the volume can be determined readily.

For liquid samples, an alternate procedure involves the use of a diffusion dilution device such as that described by Desty, Geach and Goldup in "Gas Chromatography", R.P.W. Scott, ed., Academic Press, New York, 1961.

4.4 THEORY

Theoretical background and empirical data related to the Century Organic Vapor Analyzer is presented in 4.4.1 and 4.4.2.

4.4.1 HYDROCARBONS

In general, a hydrogen flame ionization detector is more sensitive for hydrocarbons than any other class of organic compounds. The response of the OVA varies from compound to compound, but gives excellent repeatable results with all types of hydrocarbons; i.e., saturated hydrocarbons (alkanes), unsaturated hydrocarbons (alkenes and alkynes) and aromatic hydrocarbons.

The typical relative response of various hydrocarbons to methane is as follows:

Compound	Relative Response (percent)
Methane	100 (reference)
Propane	64
N-butane	61
N-pentane	100
Ethylene	85
Acetylene	200
Benzene	150
Toluene	120
Ethane	90

4.4.2 OTHER ORGANIC COMPOUNDS

Compounds containing oxygen, such as alcohc ethers, aldehydes, carbolic acid and esters give somewhat lower response than that observed for hydrocarbons. This is particularly noticeable with those compounds having a high ratio of oxygen to carbon such as found in the lower members of each series which have only one, two or three carbons. With compounds containing higher numbers of carbons, the effect of the oxygen is diminished to such an extent that the response is similar to that of the corresponding hydrocarbons.

Nitrogen-containing compounds (i.e., amines, amides and nitriles) respond in a manner similar to that observed for oxygenated materials. Halogenated compounds also show a lower relative response as compared with hydrocarbons. Materials containing no hydrogen, such as carbon tetrachloride, give the lowest response; the presence of hydrogen in the compounds results in higher relative responses. Thus, $CHCl_3$ gives a much higher response than does CCl_4. As in the other cases, when the carbon to halogen ratio is 5:1 greater, the response will be similar to that observed simple hydrocarbons.

The typical relative response of various compounds to methane is as follows:

Methane	100 (calibration sample)
Ketones	
Acetone	60
Methyl ethyl ketone	80
Methyl isobutyl ketone	100
Alcohols	
Methyl alcohol	15
Ethyl	25
Isopropyl	65

Halogen compounds

Carbon tetrachloride	10
Chloroform	65
Trichloroethylene	70
Vinyl chloride	35

The OVA has negligible response to carbon monoxide and carbon dioxide which evidently, due to their structure, do not product appreciable ions in the detector flame. Thus, other organic materials may be analyzed in the presence of CO and CO_2.

EPA #
19/829

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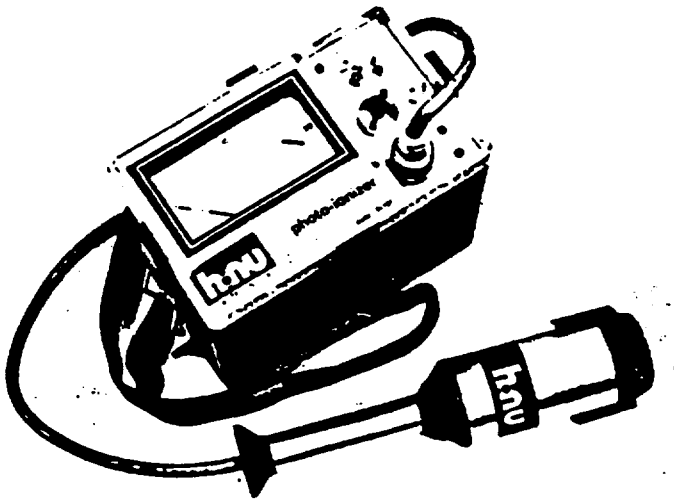
INSTRUCTION MANUAL

FOR

MODEL PI 101

PHOTOIONIZATION ANALYZER



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SECTION 3

CALIBRATION

Static or dynamic gas generation systems can be utilized for calibration of the instrument. A number of such systems for generating test atmospheres for various gases have been described by G. O. Nelson in "Controlled Test Atmospheres," Ann Arbor Science Publishers, Ann Arbor, Michigan (1971).

The most convenient packages for calibration are the non-toxic analyzed gas mixtures available from HNU Systems in pressurized containers (Catalogue #101-350).

A rapid procedure for calibration involves bringing the probe and readout in close proximity to the calibration gas, cracking the valve on the tank and checking the instrument reading. This provides a useful spot check for the instrument.

The recommended and most accurate procedure for calibration of the instrument from a pressurized container is to connect one side of a "T" to the pressurized container of calibration gas, another side of the "T" to a rotameter and the third side of the "T" directly to the 8" extension to the photoionization probe (see Figure 5). Crack the valve of the pressurized container until a slight flow is indicated on the rotameter. The instrument draws in the volume of sample required for detection, and the flow in the rotameter indicates an excess of sample. Now adjust the span pot so that the instrument is reading the exact value of the calibration gas. (If the instrument span setting is changed, the instrument should be turned back to the standby position and the electronic zero should be readjusted, if necessary.)

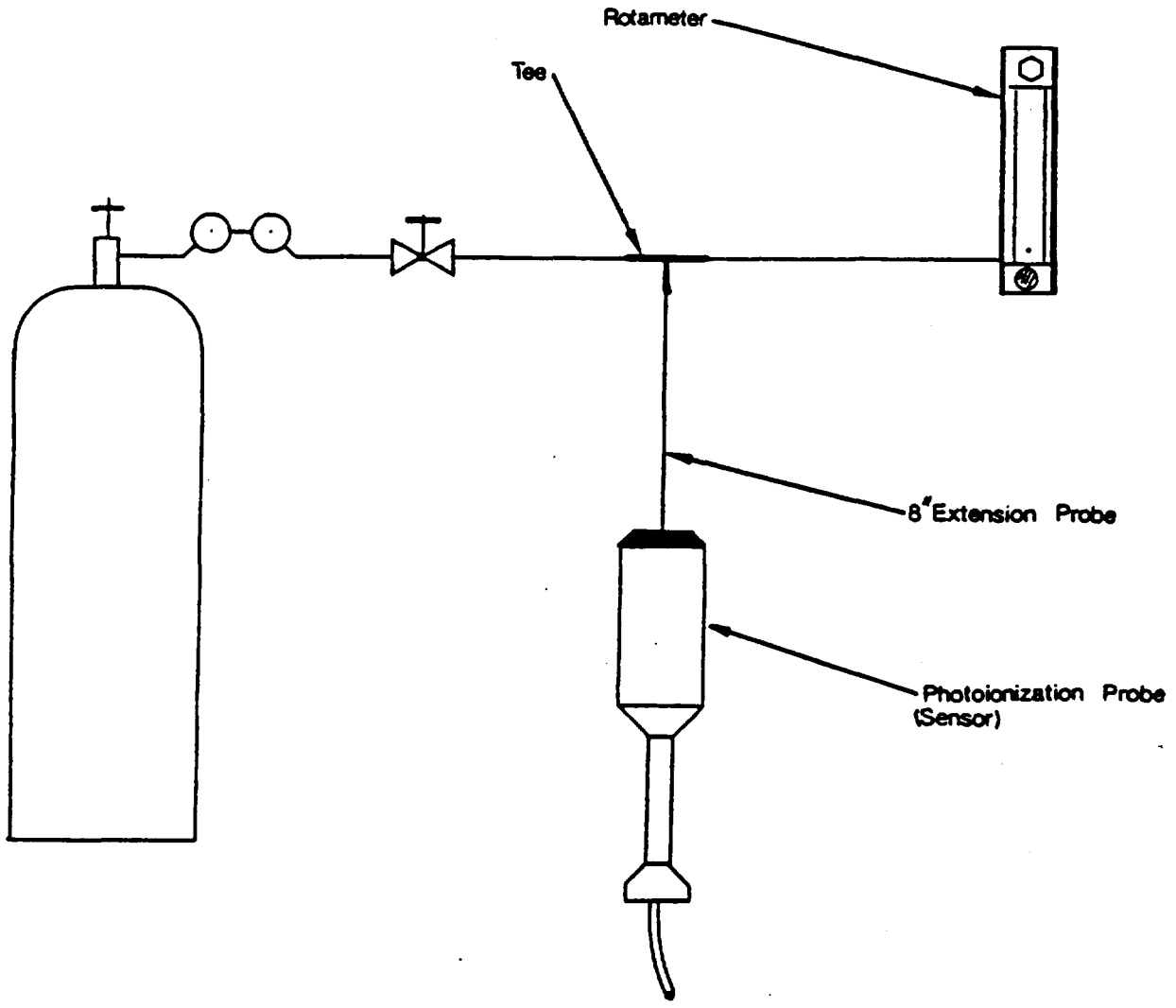

Figure 5. Recommended Calibration Procedure for Photoionization Analyzer

The calibration gas* should be prepared in the same matrix (air, nitrogen, hydrogen, etc.) in which it is to be measured, otherwise an inaccurate reading may be obtained. The increased response which is seen in oxygen free gases can be attributed to a reduction in the quenching of ions by oxygen (actually O_2^-) and is typical of any ionization detector. The quenching effect of oxygen is constant from about ten percent O_2 to very high levels.

If a gas standard prepared in nitrogen is to be used for measurements in air, fill a 0.5 or 1 liter bag with the standard then add 50 or 100 cc of pure oxygen to bring the level to 10-12%.

Any error between this value and 20% oxygen is quite small.

If the sample to be measured is in nitrogen, standards should be prepared in nitrogen. This will result in an increase in sensitivity of approximately 4.

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* Calibration with toxic gases should be performed in a hood since the 101 is a non-destructive analyzer.

APPENDIX F

QA/QC PROCEDURES
FOR DIOXIN

1.0 REQUIRED ANALYSIS FORMS

1.1 Sample Tracking: Samples arrive at the back entrance to the high hazard laboratory. The chain-of-custody forms are signed and the sample tracking book is filled out (see Appendix I for a page from that book). Each batch of samples (24 or less) will be scheduled for extraction together. Appendix II is a copy of the extraction schedule form. A laboratory reagent blank is assigned to each batch of samples along with a duplicate sample analysis. Each sample is given a worksheet (Appendix III) that will travel with the sample from receipt at the back door until final filing. In addition to filling out the above listed paperwork, the chemist involved in the extraction maintains a laboratory notebook that includes all necessary information to duplicate the information about each sample in the unlikely event of lost paperwork.

1.2 After extraction (all paperwork filled out), the samples are stored in a reach-in refrigerator until ready for GC/MS analysis. All of the paperwork generated from 1.1 is filed with the exception of the worksheet (Appendix III). Samples are scheduled for GC/MS analysis by due date. The GC/MS schedule forms (Appendix IV) are sent to the GC/MS laboratory along with all worksheets from above. After GC/MS analysis, all paperwork returns to the files, the extracts are returned to the reach-in refrigerator, and all computer generated-hardcopies from the GC/MS analysis are attached to the worksheet to proceed with data review. The GC/MS analyst also keeps a logbook of all samples analyzed in chronological order.

1.3 Data Review (calculation paperwork): Deliverables are generated as described in 5.0 of the analysis procedures section. A deliverables checklist is filled out during data review to ensure completeness of data package (Appendix V). A form (Appendix VI) is included to serve as a calculation sheet for $^{37}Cl_4$-2,3,7,8-TCDD surrogate concentration and percent accuracy and native 2,3,7,8-TCDD concentration. A form (Appendix VII) is included to serve as a calculation sheet for the detection limit estimation for native 2,3,7,8-TCDD which is to be used for those samples that have no detectable level of native 2,3,7,8-TCDD. The form in Appendix VIII will serve as a sample compound list. This compound list will be compiled on a batch basis and reported as a group with all other samples and blanks from that group (Appendix IX).

1.4 Initial and Routine Calibration Paperwork: The initial calibration procedure is described in Section 4.2 of the analysis section. Complete the top part of the TCDD calibration summary form (Appendix X) labeled "Multipoint." Use the form in Appendix XI to calculate the combined mass response factors. Section 4.2 of the analysis section explains the acceptable linearity requirements. The form in Appendix X remains by the GC/MS instrument and is updated with every shift standard analyzed. A copy of this form is attached to the shift standard while the original

stays by the instrument. To update this form, fill out the bottom of the form as described in Section 4.3 of the analysis section.

1.5 Partial Scan Results: Section 6.0 of the analysis section describes the total ion confirmation analysis for one sample from every batch. A reporting sheet is completed for that sample (Appendix XII).

2.0 BATCH DELIVERABLES

2.1 All reports and documentation required, including chromatograms and mass spectra, shall be clearly labeled with the sample number and associated sample/traffic report number.

2.2 Data Package

2.2.1 Results of initial triplicate analysis of five concentration calibration solutions, including all selected ion current profiles (SICPs) as per section 5.1 of the analysis section, calculated response factors, plotted concentration calibration curves, and computer-generated quantitation reports.

2.2.2 Completed data reporting sheets (Appendix IX, XII, XIII, XIV) with appropriate SICPs and spectra attached. Data results of levels less than 10 ppb shall be reported to two significant figures; results greater than 10 ppb shall be reported to three significant figures. Apply the rounding rules found in Section 7.2.2, "Handbook for Analytical Quality Control in Water and Wastewater Laboratories," EPA 600/4-79-019. Each SICP and spectrum shall include computer-generated header information indicating instrumental (GC and MS) operating parameters during data acquisition. When samples are analyzed more than once, all sample data shall be reported. Rejected sample runs must be separated and attached to the back of the data package and marked on the SICP as "Rejected."

2.2.3 SICPs generated during each performance check solution analysis and each concentration calibration solution analysis.

2.2.4 Documentation of acceptable MS calibration (see Section 6.1, Analysis Section) for each confirmatory analysis.

2.2.5 A chronological list of all analyses performed. If more than one GC/MS system is used, a chronological list is required for each system. The list must provide the data system file name, the sample number, and (if appropriate) the contractor laboratory sample number, for each sample, blank, concentration calibration solution, performance check solution, including labeled peaks for TCDD isomers, or other pertinent analytical matrix, including partial scan confirmation spectra. This list shall specify date and time of beginning of analysis. All sample/blank analyses performed during an 8-hour period must be accompanied by two performance check solution analyses, one preceding and one following sample/blank analyses. If multiple shifts are used, the ending

performance check sample analysis from one 8-hour period shall serve as the beginning analysis for the next 8-hour period.

2.3 GC/MS Raw Data Tapes
Magnetic tapes containing raw GC/MS data for all analyses (samples, blanks, concentration calibration solutions, performance check solutions, and performance evaluation samples) shall be maintained.

2.4 Sample Extracts and Unused Sample Portions
Unused portions of samples and sample extracts shall be retained by the laboratory for a period agreed upon by both the laboratory and the client of six months after receipt. When directed in writing by the client, the laboratory shall ship (not at the laboratory's expense but in accordance with Department of Transportation Regulations) specific samples and/or extracts to a specified location and person. After the agreed upon time, remaining samples and extracts shall be disposed of at the laboratory's expense, in accordance with applicable regulations concerning the disposal of such materials.

2.5 Document Control and Chain-of-Custody Package
The Document Control and Chain-of-Custody Package includes all laboratory records received or generated for a specific case or sample batch, that have not been previously submitted as a deliverable. These items include but are not limited to: sample tags, custody records, sample tracking records, analysts logbook pages, bench sheets, chromatographic charts, computer printouts, raw data summaries, instrument logbook pages, correspondence, and the document inventory.

2.6 Monthly Sample Status Report
The monthly sample status report shall provide the status of all samples the laboratory has received or has had in-house during the calendar month. Required status information includes: samples received, samples extracted, samples analyzed, samples rerun, and samples which required special cleanup.

3.0 SAMPLE RERUN REQUIREMENTS

3.1 Scope and Application
The laboratory shall be required to reextract and reanalyze certain samples or batches of samples in a variety of situations that may occur in the process of contract performance. (For purposes of this SOP, the term "rerun" shall mean sample reextraction and reanalysis.)

3.2 Required Sample Reruns

3.2.1 Automatic sample reruns.

3.2.1.1 If acceptable detection limits have been demonstrated using Calibration Standard No. 1 (Section 4.1.2 Analysis Section) and matrix effects or interferences on a specific sample prevent the contract required detection limit of 1.0 ppb from being achieved, the Contractor shall

subject the extract to additional cleanup, and reanalyze the sample.

3.2.1.2 If the calculated unlabeled TCDD amount was outside the upper initial calibration range, the Contractor shall reextract the sample, using a smaller sample aliquot, and reanalyze the sample.

3.2.1.3 If the percent accuracy for the surrogate standard, $^{37}Cl_4$-2,3,7,8-TCDD, was outside of the acceptance limits of $\pm$ 40%, the Contractor shall reextract and reanalyze the sample, utilizing the additional cleanup.

3.2.1.4 If the internal standard was not found to be present with at least 10:1 signal to noise ratio at mass 332 and 334, the laboratory shall reextract and reanalyze the sample.

3.2.1.5 If the internal standard 332/334 ratio was outside of the contract specified control limits of 0.67-0.87, t laboratory shall subject the extract to additional cleanup, and reanalyze the sample.

3.2.2 Sample reruns to be performed due to the laboratory's materials, equipment, or instrumentation problems.

3.2.2.1 If the laboratory reagent blank was found to contain TCDD, the laboratory shall reanalyze the entire associated batch of samples.

3.2.2.2 If the performance check (PC) solution does not meet specified criteria, the laboratory shall reanalyze all samples run during the time period between the last acceptable PC run and the unacceptable PC run.

3.2.2.3 If the accuracy of measured concentration of $^{37}Cl_4$-2,3,7,8-TCDD in the spiked (fortified) field blank is $>\pm$40%, the laboratory shall reextract and reanalyze a second aliquot of the field blank sample.

3.2.2.4 If a false positive is reported for an uncontaminated soil (blind QC) sample, upon notification by the QA/QC Coordinator the laboratory shall reextract and reanalyze the entire associated batch of samples.

3.2.2.5 If the analysis results for a performance evaluation blind QC sample fall outside established acceptance windows, upon notification of the QA/QC Coordinator the laboratory shall reextract and reanalyze the entire associated batch of samples.

3.2.2.6 If the isotope abundance ratio for m/z 320/322 is less than 0.67 or greater than 0.87, and all other criteria

contained in Section 4.5.1 through 4.5.6 of Analysis Section are met, then the sample must be rerun.

4.0 QA/QC REQUIREMENTS

SUMMARY OF QC ANALYSES

4.1 Initial and periodic calibration and instrument performance checks.

4.2 Laboratory reagent blank analyses (Section 4.4.1); minimum of one blank shall be analyzed with each sample batch; an additional blank analyzed when new reagents are used.

4.3 Analysis of a batch of samples with accompanying QC analyses:

4.3.1 Sample Batch -- 24 samples, including field blank and rinsate sample(s).

4.3.2 Additional QC Analyses Per Batch:

Laboratory reagent blank	1
Duplicate sample analysis	1
Confirmatory partial scan analysis	1
TOTAL	3

4.4 "Blind" QC samples may be submitted to laboratory as an ordinary soil or sediment sample included among the batch of samples. Blind samples include:

4.4.1 Uncontaminated soil/water

4.4.2 Split samples

4.4.3 Unlabeled duplicates

4.4.4 Performance evaluation samples (field spikes)

5.0 QUALITY CONTROL

5.1 Performance Evaluation Samples -- Included among samples in some batches will be samples containing known amounts of unlabeled 2,3,7,8-TCDD that may or may not be marked as other than ordinary samples.

5.2 Performance Check Solution

5.2.1 At the beginning of each 8-hour period during which samples are to be analyzed, an aliquot of the performance check solution and an aliquot of concentration calibration solution No. 1 shall be analyzed to demonstrate adequate GC and MS resolution and sensitivity, response factor reproducibility, and mass range calibration. These procedures are described in Section 4.2 of Analysis Section If any required criteria are not met, remedial action must be taken before any samples are analyzed.

5.2.2 To validate sample data, the performance check solution must be analyzed also at the end of each 8-hour period during which samples are analyzed.

5.2.2.1 If the laboratory operates only during one 8-hour period (shift) each day, the performance check solution must be analyzed twice (at the beginning and end of the 8-hour period) to validate data acquired during the interim period.

5.2.2.2 If the contractor laboratory operates during consecutive 8-hour periods (shifts), analysis of the performance check solution at the beginning of each 8-hour period and at the end of the final 8-hour period is sufficient.

5.2.3 Results of at least two analyses of the performance check solution must be reported with sample data collected during an 8-hour period.

5.2.4 Deviations from criteria specified for the performance check solution invalidate all sample data collected between analyses of the performance check solution, and samples shall be rerun.

5.3 Concentrations of all solutions containing unlabeled 2,3,7,8-TCDD and not obtained from EMSL-LV must be verified by comparison to the unlabeled 2,3,7,8-TCDD standard solution (concentration of 7.87 µg/mL) that is available from EMSL-LV.

5.4 Blanks

5.4.1 Laboratory reagent blank -- Perform all steps in the analytical procedure using all reagents, standards, equipment, apparatus, glassware, and solvents that would be used for a sample analysis, but omit an aliquot of soil or sediment.

5.4.1.1 A laboratory reagent blank must contain the same amount of $^{37}Cl_4$-2,3,7,8-TCDD or $^{13}C_{12}$-2,3,7,8-TCDD that is added to samples before extraction.

5.4.1.2 Analyze a laboratory reagent blank before any samples are extracted and analyzed.

5.4.1.3 Analyze two laboratory reagent blanks before a new batch of solvents or reagents is used for sample extraction or for column chromatographic procedures. Do not add any $^{37}Cl_4$-2,3,7,8-TCDD or $^{13}C_{12}$-2,3,7,8-TCDD to one blank, to demonstrate that reagents contain no impurities producing an ion current above the level of background noise for m/z 328, 332 and 334.

5.4.1.4 Analyze a laboratory reagent blank along with each batch of samples.

5.4.1.5 All resins used for air sampling will be prewashed by soxhlet extraction and packed in the sampling tubes at IT Knoxville. An aliquot of each new batch is analyzed before shipment is made (one spiked and one unspiked).

5.4.1.6 Acceptable laboratory reagent blanks contain no ion current above the level of background signal-to-noise for any of the selected characteristic ions (m/z 257, 320, 322) for unlabeled 2,3,7,8-TCDD. If the reagent blank which was extracted along with a batch of samples is contaminated, the entire batch of samples must be rerun.

5.4.1.6.1 If the above criterion is not met, check solvents, reagents, apparatus, and glassware to locate and eliminate the source of contamination before any samples are extracted and analyzed.

5.4.1.6.2 If new batches of reagents or solvents contain interfering contaminants, purify or discard them.

5.4.2 Field Blanks -- Each batch of samples contains a sample of uncontaminated sample that is to be fortified with unlabeled 2,3,7,8-TCDD at a concentration of 1 µg/kg before analysis. In addition to that field blank, a batch of samples may include a rinsate, that is a portion of solvent (usually trichloroethylene) that was used to rinse sampling equipment. The rinsate is analyzed to assure that samples have not been contaminated by sample equipment.

5.4.2.1 Unfortified field blank -- Analyze with procedures used for environmental samples. This blank may or may not be labeled as such (i.e., it may be a "blind" QC sample).

5.4.2.2 Fortified (Spiked) Field Blank

5.4.2.2.1 Take an appropriate aliquot of the specified field blank sample and add 100 µL of the solution containing 0.1 ng/µL of unlabeled 2,3,7,8TCDD, 0.5 ng/µL of $^{13}C_{12}$-2,3,7,8-TCDD, and 0.1 ng/µL of $^{37}Cl_4$-2,3,7,8-TCDD. (Analysis before fortification is not required because this field blank is known not to contain a detectable concentration of unlabeled 2,3,7,8-TCDD.)

5.4.2.2.2 Extract with the appropriate procedure and analyze a 2-µL aliquot.

5.4.2.2.3 Calculate concentration of both $^{37}Cl_4$-2,3,7,8-TCDD and unlabeled 2,3,7,8-TCDD, and accuracy of each measured concentration.

5.4.2.2.3.1 If accuracy of measured concentration of $^{37}Cl_4$-2,3,7,8-TCDD is > ± 40%, discard the results and repeat the fortified field blank extraction and analysis with a second aliquot of the specified field blank sample.

5.4.2.3 Rinsate Sample

5.4.2.3.1 To a 100-ml aliquot of equipment rinse solvent (rinsate sample), add 100 µl of the solution containing 0.5 ng/µl of $^{13}C_{12}$-2,3,7,8-TCDD and 0.1 ng/µl solution of $^{37}Cl_4$-2,3,7,8-TCDD.

5.4.2.3.2 Using a Kuderna-Danish apparatus, concentrate the volume to approximately 5 ml.

5.4.2.3.3 Transfer the total 5-ml concentrate in 1-ml portions to a 1 ml-amber mini-vial, reducing volume as necessary with a gentle stream of dry nitrogen.

5.4.2.3.4 Rinse container with two 0.5 ml portions of hexane and transfer rinses to the 1-ml amber mini-vial.

5.4.2.3.5 Just before analysis, reduce volume to near dryness; make to final volume of 50 µl with the Is solution. (Column chromatography is not required.)

5.4.2.3.6 Analyze an aliquot with the same procedures used to analyze samples.

5.5 Duplicate Analyses

5.5.1 Laboratory duplicates -- In each batch of samples, locate the sample specified for duplicate analyses and analyze a second 10-g sample aliquot.

5.5.1.1 Results of laboratory duplicates must agree within 50% relative difference (difference expressed as percentage of the mean). If relative difference is > 50%, Contractor shall immediately contact the Sample Management Office for resolution of the problem. Report all results.

5.5.1.2 Recommended actions to help locate problem:

5.5.1.2.1 Analyze an aliquot of the performance check sample to verify satisfactory instrument performance.

5.5.1.2.2 If possible, determine that no error was made while weighing sample aliquots.

5.5.1.2.3 Review analytical procedures with performing laboratory personnel.

5.6 Accuracy of Measured Concentrations of $^{37}Cl_4$-2,3,7,8-TCDD -- For each sample and blank, calculate the percent accuracy of the measured concentration of $^{37}Cl_4$-2,3,7,8-TCDD. If percent accuracy is > ± 40% for a sample, analyze a second aliquot of that sample and report both results. NOTE: Low or high accuracy for a blank does not require discarding sample data but indicates a potential problem with future sample data. Percent recovery will be monitored with typical recovery values expected between 40-120% low recoveries from some matrixes may indicate a potential problem with the method and signal the laboratory to modify the procedures.

5.7 Identification Criteria

5.7.1 If any of the four initial identification criteria (Sections 4.5.1-4.5.4 Analysis Section) are not met, the sample is reported not to contain unlabeled 2,3,7,8-TCDD at the calculated detection limit.

5.7.2 When the four initial identification criteria are met, but the fifth criteria, the isotopic abundance ratio for m/z 320 and 322 (Section 4.5.5 Analysis Section) is not met, that sample is presumed to contain interfering contaminants. Contractor shall use the second column chromatography procedure (Section 6.3 Sample Prep) to remove interferences from the extract, and shall reanalyze the sample.

5.8 Blind QC Samples -- Included among samples may be QC samples that are not specified as such to the performing laboratory. Types that may be included are:

5.8.1 Uncontaminated soil, water, wipes, air trap resins.

5.8.1.1 If a false positive is reported for this sample, the laboratory shall be required to rerun the entire associated batch of samples.

5.8.2 Split samples -- composited sample aliquots sent to more than one laboratory.

5.8.3 Unlabeled field duplicates -- two aliquots of a composited sample.

5.8.4 Performance evaluation sample -- sample containing a known amount of unlabeled 2,3,7,8-TCDD.

5.8.4.1 If the performance evaluation sample result falls outside the acceptance windows established by ITC, the

laboratory shall be required to rerun the entire associated batch of samples. NOTE: Acceptance windows are based on historical data results.

5.9 Confirmatory Partial Scan Analysis

5.9.1 From each sample batch, select the sample extract containing the highest concentration of unlabeled 2,3,7,8-TCDD and analyze an aliquot by GC/MS under the same GC conditions used previously but with the MS tuned and calibrated to acquire data for the mass range m/z 150 to m/z 350. (If no sample in a batch contains unlabeled 2,3,7,8-TCDD, no confirmatory analysis is required for that batch.) Required calibration criteria for decafluorotriphenylphosphine introduced through the GC column shall be:

m/z	Relative Intensity
51	30 - 60 percent of base peak
68	< 2 percent of m/z = 69
70	< 2 percent of m/z = 69
127	40 - 60 percent of base peak
197	< 1 percent of base peak
198	100 percent (base peak)
199	5 - 9 percent of base peak
275	10 - 30 percent of base peak
365	> 1 percent of base peak
441	less than m/z = 443
442	> 40 percent of base peak
443	17 - 23 percent of m/z = 442

5.9.2 MS data acquisition requirements shall be:

5.9.2.1 Cycle time $\leq$ 1.5 seconds.

5.9.2.2 Acquisition of $\geq$ 5 spectra during elution of 2,3,7,8-TCDD from the GC.

5.9.3 Subtract an appropriate background spectrum, and plot a spectrum of 2,3,7,8-TCDD after background subtraction. (The person responsible for MS data interpretation is responsible for demonstrating that the background spectrum selected for subtraction was an appropriate spectrum.) Provide a hard copy of the background spectrum, the TCDD spectrum before subtraction, and the TCDD spectrum after subtraction. The quality of the plotted spectrum will be affected by other sample components that have approximately the same GC retention time and will be highly variable. Desired spectral features are:

Base peak = m/z322
Ratio of m/z 320 to 322 = 0.77
Ratio of m/z 320 to 324 = 1.58
Ratio of m/z 257 to 322 = 0.32

Ratio of m/z 257 to 259 = 1.03
Ratio of m/z 194 to 196 = 1.54
m/z 160 and 161 = $\geq$ 10% of m/z 322

Because $^{13}C_{12}$-2,3,7,8-TCDD, the internal standard, is present in every sample and has essentially the same retention time as unlabeled 2,3,7,8-TCDD, the spectrum after background subtraction will represent a mixture. When $^{13}C_{12}$-2,3,7,8-TCDD is present at a higher concentration than unlabeled 2,3,7,8-TCDD, the resultant spectrum must be normalized to m/z 322 to demonstrate desired spectral features.

5.10 Records - At the laboratory, records must be maintained on site for six months after contract completion to document the quality of all data generated during contract performance. Before any records are disposed of, written concurrence of the Contracting Officer must be obtained.

5.11 Magnetic tapes containing all raw GC/MS data (including performance check solution, blanks, and concentration calibration solutions) must be kept.

5.12 Unused portions of samples and sample extracts must be preserved for six months after sample receipt.

5.13 Reuse of glassware is to be minimized to avoid the risk of using contaminated glassware.

6.0 Standard Verification

1.0 SUMMARY

1.1 All standards bought commercially must be checked for both purity and accuracy of concentration. The benchmark standard for verification of accuracy is a 7.87 µg/ml standard of native 2,3,7,8-TCDD supplied by the USEPA.

2.0 RECEIPT OF STANDARDS AND STANDARD CHECKS OF STOCK SOLUTIONS

2.1 Standards received from commercial suppliers of dioxin are checked into the building and logged into a standard notebook. The standards are transferred to volumetric flasks and brought up to volume in toluene. A known dilution of the stock is analyzed on GC/MS to check for both purity and accuracy of concentration vs. an already approved standard of the USEPA benchmark standard. If the response factor agrees $\pm$ 10% from the check standard, the new lot is approved for use. The hardcopies of the standard check will contain EICPs for all compounds of interest from both the new standard and the benchmark standard and will be filed for future reference.

3.0 STORAGE OF APPROVED STANDARDS

3.1 The approved stock will be transferred to tared numbered and labeled vials (2 ml size). The weights of the standards will be recorded (Form

1). When a new standard bottle is used, it is weighed first to ensure no volume loss during storage. The standards are kept at -4°C for no longer than six months.

4.0 WORKING STANDARD SOLUTIONS

4.1 Dilutions of the stock solutions are made for all working standards to be used in the extraction laboratory and the GC/MS laboratory.

4.2 Before use of the working level standards on sample analysis, the solutions must be approved for concentration and purity as in the stock solutions. The extraction lab standard is analyzed by GC/MS and compared to the shift calibration solution. If the calculated amounts are ± 10% of expected amount, the standard is approved for use in the extraction laboratory. When new GC/MS shift standards are made and approved as above, a new five-point calibration in triplicate must be run.

4.3 Working standard solutions are stored as per Section 3.0.

5.0 STANDARD NOTEBOOK

5.1 All standards made are logged into a standard notebook that includes volumes used, solvents, any problems encountered, date made, date expired, weights of each standard container, concentration, and approval date (see Fig. 4.1-1).

7.0 SAFETY PROCEDURES

1.0 SUMMARY

1.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level. The laboratory maintains a file of current OSHA regulations regarding the safe handling of the chemicals used in this method.

1.2 Protective Equipment: Disposable plastic gloves, disposable lab coats, safety glasses, lab hoods adequte for radioactive work segregated for dioxin work only.

1.3 Confinement: Limited access laboratory held under constant negative pressure with air lock doors, posted with signs. The limited access laboratory contains four isolated work areas under negative pressure with each work area having a lab hood, segregated glassware and tools, and plastic-backed absorbent paper on benchtops and in hoods. The limited access laboratory is a change-in, shower-out area restricted to authorized persons only.

1.4 Training: Workers are trained in the proper method of removing contaminated gloves and clothing without contacting the exterior surfaces.

Standard or Stock Solution # _____ Ref. # _____ Lot # _____

Compound(s): _____

Original Conc. _____ Solvent _____ Total Volume _____

Sol'n Prepared By _____ at Date/Time _____

Container Tare (grams)_____ Container + Solvent (grams) _____

Container Gross (grams) _____

Special Notes : _____

Date Used	User Initials	Weight Before Use (grams)	Weight After Use (grams)	Volume Removed (mls/µl)	Weight After Septum (grams) Replacement

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Fig. 4.1-1. Standard Logbook Page

1.5 Personal Hygiene: Thorough washing of hands and forearms after each manipulation with mild soap and plenty of scrubbing action. Shower out before lunch breaks and before going home.

1.6 Waste: Contaminated waste is kept to a minimum. Plastic bag-lined waste cans are used and when full they are put in a trash compactor.

1.6.1 Disposal of Waste: All paper waste is put in a trash compactor; when full the waste is transferred to a 55-gallon drum for solid waste. Liquid wastes are allowed to evaporate in a disposable container, the residue and the container are put in the above mentioned 55-gallon drum. The drum is disposed of through commerical channels that are capable of handling extremely toxic waste.

1.7 Glassware, Tools, and Surfaces: Satisfactory cleaning is accomplished by rinsing with 1,1,1-trichloroethane or the solvent last used in the glassware (solvents are treated as liquid waste as per 1.6.1), followed by washing with detergent and water. Dishwater is disposed of to the sewer.

1.8 Laundry: Disposable garments are used for all TCDD work. The laundry is disposed of as paper waste as per 1.6.1.

1.9 Wipe Tests: All work surfaces and tools are wiped with a piece of filter paper every six months, or when an accident has occurred. The filter paper is extracted and analyzed by GC/MS. Less than 0.1 μg/wipe indicates acceptable cleanliness. Anything higher warrants further cleaning. More than 10 μg/wipe indicates an acute hazard that requires prompt cleaning before further use of the work area and indicates unacceptable work practices have been employed in the past.

1.10 All work on TCDD is done in the hoods when the potential for inhalation. occurs. All hoods are fitted with hepa filters to avoid gross losses to the atmosphere.

1.11 The effluents of sample splitters for the gas chromatograph and roughing pumps on the GC/MS are vented to a fume hood. The hood is fitted with hepa filter to avoid gross losses to the atmosphere.

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Appendix I

Sample Tracking

IT No.	Client No.	Client Name	Method	Date In	Date Due	Notebook	Extraction Page	Extraction Date	GC/MS Date	Date O

EXTRACTION SCHEDULE FORM

Page # _____

Sample ID	Method	Wt Samples	Extracted By	Date	Cleanup	Final Volume
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23 QC (Dup)						
24 QC (Spike)						
25 QC TBLANK *						
26 QC TCOLUMN **						

*Insert page # on line

**Use only if new batch of alumina or silica

Spiked with _____ µl

Lot # _____

IT #_____

Method_____

EXTRACTION INFORMATION

Wt. of sample _____g

IS/Surr Std ID #_____

Vol IS/Surr added _____µl

Final volume extract _____ µl

Cleanup method(s) used _____

Associated method blank # T_____

Location of extract:

Extracted by _____

Date _____

Log ref._____

GM/MS INFORMATION

Shift Std ID _____

Std Lot #_____

Disc ID _____

GC/MS operator_____

Date Run_____

REVIEW INFORMATION

_____% accuracy

Reviewed by_____

COMMENTS:

APPENDIX III

GC/MS SCHEDULE FORM

TYPE OF ANALYSIS_____

DATE DUE_____

IT #	DATE RUN	OPERATOR	COMMENTS

APPENDIX IV

D154-T-3

TCDD REPORT FORM (EPA SOIL)
Deliverable Check List

	Yes	No
1. RIC (500 to end of run)	____	____
2. EICP of ions 332 and 334	____	____
3. EICP of ions of 257; 320; 322; 332	____	____
4. EICP of ions 320; 322; 328; 332	____	____
5. EICP of ions 320; 322; 257 for 5 scans centered on the retention time of $^{13}C_{12}$-2,3,7,8	____	____

Surrogate/Internal Standard

1. 332; 334; 328 ions present [signal to noise > 10:1]	____	____
2. 332/334 ratio of peak intensities in range 0.67-0.87	____	____

2,3,7,8 TCDD

1. 320/322 ratio in range 0.67-0.87	____	____
2. 320; 322; 257 ions all present and maximize together (± 1 scan)	____	____
3. Signal to noise (intensity) for 320; 322; and 257 >2.5:1	____	____
4. Elution time of 2,3,7,8-TCDD within 3 seconds of $^{13}C_{12}$-2,3,7,8-TCDD	____	____
5. Relative abundance of m/z 257 to m/z 322 in range 0.20-0.45	____	____

APPENDIX V

D154-T-4

PERCENT ACCURACY AND CONCENTRATION CALCULATIONS

Sample # _____

Calculated By _____

I. Percent Accuracy (All areas from sample)

$$\% \text{ Accuracy} = \frac{\text{Amount Surrogate Measured}}{\text{Amount Surrogate Added}} \times 100\%$$

$$= \frac{\text{Amount } {}^{13}C_{12}\text{-2,3,7,8}}{\text{Areas m/e 332 + m/e 334}} \times \frac{\text{Area m/e 328 - (0.009 x m/e 322)}}{\text{RF}}$$

$$\frac{\qquad\qquad}{\text{Amount Added}} \times 100$$

$$= 1000 \times \frac{\text{Area m/e 328 - (0.009 x m/e 322)}}{\text{(Areas m/e 332 + m/e 334) x RF}}$$

$$= 1000 \times \frac{\qquad - (\qquad)}{(\qquad) \times} = \frac{\qquad}{200} \text{conc} \times 100\% = \_\_\_\_\%$$

Where RF = Average RF of multipoint for surrogate ${}^{37}Cl_4$

II. 2,3,7,8-TCDD Concentration by Internal Standard =

$$\text{Native Amt} = \frac{\text{Amt } {}^{13}C \text{ in Standard}}{\text{Combined Areas 332 + 334 in Sample}} \times \frac{\text{Combined Areas 320 + 322 in Sample}}{\text{RF}}$$

Where RF = Average RF of multipoint (Form 248A)

$$= \frac{1000}{\text{Area 332 + Area 334}} \times \frac{\text{Area 320 + 322}}{\text{RF}}$$

$$= \frac{1000}{\qquad} \times \frac{\qquad}{\qquad} = \_\_\_\_\_ \text{ pg/µl}$$

To convert pg/µl to PPB =

$$\text{ppb} = \text{Native Amount (pg/µl)} \times \frac{50 \text{ µl} \times 1 \text{ ng}}{\text{\# g sample extracted} \times 1000 \text{ pg}}$$

$$= \text{Native Amount} \times \frac{1}{\text{\# g extracted} \times 20}$$

$$= \frac{\qquad}{\qquad} \times \frac{1}{\qquad \times 20}$$

(Use three sig figs for values above 10.0 ppb)

= _____ppb (Use two sig figs for values below 10 ppb)

ESTIMATED DETECTION LIMIT OF 2,3,7,8-TCDD CALCULATION

Sample # _____

Calculated By _____

I. Signal to Noise x 2.5

= 2.5 x Background intensity of m/e 320 or background intensity m/e 322

= 2.5 x _____ or _____ = _____

II. Conc. of IS. x Signal to noise (ppb)

$$\text{ppb} = \frac{\text{Amount int. std.}}{\text{Intensity m/e 332 or Intensity m/e 334}} \times \frac{I}{RF}$$

Where RF = Multipoint average native response factor obtained from multipoint

$$= \frac{1000}{\text{Peak height m/e 332 or peak height m/e 334}} \times \frac{I}{RF}$$

$$= \frac{1000}{\quad} \times \frac{\quad}{\quad}$$

$$= \frac{1000}{\quad} \times \frac{\quad}{\quad} = \frac{\quad}{\quad} \text{ pg/µl*}$$

$$\text{Native amount (pg/µl)} \times \frac{50\ \mu l \times 1\ ng}{\#\ g\ \text{sample extracted} \times 1000\ pg}$$

$$= \frac{\quad}{\quad} \times \frac{1}{\#\ g\ \text{extracted} \times 20}$$

$$= \frac{\quad}{\quad} \times \frac{1}{\times 20} = \frac{\quad}{\quad} \text{ ppb*}$$

* Use one significant figure

APPENDIX VII

LAB: _____ DATE ANALYZED: _____

CASE: _____ TIME ANALYZED: _____

BATCH SHIPMENT #: _____

ITE SAMPLE # _____

Extra Cleanup	Net Weight (Grams)	2378-TCDD Conc. (ppb)	Detection Limit (ppb)	320/322	332/334	Surrogate Measured (ppb)	Surrogate Percent Accuracy

Area 332	Area 334	Area 320	Area 322	Area 257	Area* 328

COMMENTS:

MB - Method Blank
N - Native TCDD Spike
D - Duplicate (Intralab)
P - Partial Scan

ND - Not Detected
J - Jar Extraction
FB - Field Blank

* Corrected area 328 [328 - (0.009 x 322)]

APPENDIX VIII

D154-T-7

FORM B-1. TCDD DATA REPORT FORM

Lab: _____

Case No: _____

Batch/Shipment No. _____

Report Date: _____

Column: _____

Sample Number	Extra Cleanup*	Aliquot Wet Wt.(g)	PPB TCDD Meas.	PPB TCDD DL	GC/MS Analysis Instr. ID	GC/MS Analysis Date	GC/MS Analysis Time	Rel. Ion Abund 320/322	Rel. Ion Abund 332/334	PPB Surrogate Meas.	PPB Surrogate % Acc'y	Relative Ion Abundance 320	Relative Ion Abundance 322	Relative Ion Abundance 257	Relative Ion Abundance 328*4	Relative Ion Abundance 332	Relative Ion Abundance 334	Comments

RB = Reagent Blank
P = Partial Scan/Confirmatory Analysis
N = Native TCDD Spike
D = Duplicate/Fortified Field Blank

FB = Field Blank
ND = Not Detected
DL = Detection Limit

*Corrected for contribution by native TCDD; 0.9% of m/z 322 subtracted.

2,4,8 TCDD CALIBRATION STANDARD

DATE	CONC. 2378(*)	FILENAME	NATIVE TCDD RF(**)	NATIVE TCDD Ave. RF	NATIVE TCDD % RSD	SURROGATE $^{37}Cl_4$ RF(**)	SURROGATE $^{37}Cl_4$ Ave. RF	SURROGATE $^{37}Cl_4$ % RSD
	200 pg/ul							
	1000 pg/ul							
	5000 pg/ul							
	20000 pg/µl							
	40000 pg/µl							

DATE	SHIFT	CONC 2378(*)	FILENAME	NATIVE(**) RF	NATIVE† RF RATIO	$^{37}Cl_4$(**) RF	$^{37}Cl_4$† RF RATIO	OPERATOR
		200 pg/ul						
		200 pg/ul						
		200 pg/ul						
		200 pg/ul						
		200 pg/ul						
		200 pg/ul						
		200 pg/ul						
		200 pg/ul						
		200 pg/ul						
		200 pg/ul						
		200 pg/ul						
		200 pg/ul						
		200 pg/ul						
		200 pg/ul						
		200 pg/ul						
		200 pg/ul						
		200 pg/ul						

200 pg/ul is equivalent to 1 ppb assuming 10 g sample extraction and a final extract volume of 50 ul.

) See Form 248B for RF calculations

atio of both the native and surrogate shift standard response factors to respective multipoint average RFs must be within ange of 0.90 - 1.10

Form 248A

MULTIPOINT COMBINED RESPONSE CALCULATION SHEET

A. RRF for native 2,3,7,8-TCDD

Formula used: $\frac{\text{Amt } ^{13}\text{C-TCDD}}{\text{Amt native TCDD}} \times \frac{\text{Area m/e 320 + Area m/e 322}}{\text{Area m/e 332 + Area m/e 334}}$

RRF (200 pg/µ) = 5 x _____ + _____ / _____ +

= 5 x _____ = _____

RRF (1000 pg/µ) = 1 x _____ + _____ / _____ +

= 1 x _____ = _____

RRF (5000 pg/µ) = 0.2 x _____ + _____ / _____ +

= 0.2 x _____ = _____

RRF (20000 pg/µl) = 0.5 x _____ + _____ / _____ +

= 0.5 x _____ = _____

RRF (40000 pg/µl) = 0.025 x _____ + _____ / _____ +

= 0.025 x _____ = _____

B. RRF for surrogate $^{37}Cl_4$-2,3,7,8-TCDD

Formula used: $\frac{\text{Amt } ^{13}\text{C-TCDD}}{\text{Amt } ^{37}\text{Cl-TCDD}} \times \frac{\text{Area m/e 328 - (0.009 x Area m/e 322)}}{\text{Area m/e 332 + Area m/e 334}}$

RRF (200 pg/µl) 16.67 x _____ - (_____) / _____ +

= 16.67 x _____ = _____

RRF (1000 pg/µl) 8.33 x _____ - (_____)

8.33 x _____ = _____

RRF (5000 pg/µl) 5 x _____ - (_____)

5 x _____ = _____

No ^{37}Cl added to 20 ng and 40 ng standards

INITIAL CALIBRATION SUMMARY

Date	Instr. ID	Sol. ID	Measured RF Native Surrogate	Mean RF Used Native Surrogate	TCDD Isomer Resolution Percent Valley (Applies for PC only)

Solution ID Codes:

PC = Performance check solution
CC1 = Concentration calibration solution #1 = 0.2 ng/µl
CC2 = Concentration calibration solution #2 = 1.0 ng/µl
CC3 = Concentration calibration solution #3 = 5.0 ng/µl
CC4 = Concentration calibration solution #4 = 20.0 ng/µl
CC5 = Concentration calibration solution #5 = 40.0 ng/µl

APPENDIX XIII

TCDD DATA REPORT - PARTIAL SCAN CONFIRMATION

Sample Number	Response Ratios					% Relative Abundances*								
	320/322	320/324	257/322	257/259	194/196	160	161	194	196	257	259	320	322	324

*Relative to m/e 322.

CONTINUING CALIBRATION SUMMARY

Date	Instr. ID	Sol. ID	Measured RF Native / Surrogate	Mean RF Used Native / Surrogate	TCDD Isomer Resolution Percent Valley (Applies for PC only)

Solution ID Codes:

PC = Performance check solution
CC1 = Concentration calibration solution #1 = 0.2 ng/µl
CC2 = Concentration calibration solution #2 = 1.0 ng/µl
CC3 = Concentration calibration solution #3 = 5.0 ng/µl
CC4 = Concentration calibration solution #4 = 20.0 ng/µl
CC5 = Concentration calibration solution #5 = 40.0 ng/µl

QUALITY CONTROL SUMMARY

Mean Accuracy, Surrogate Measurements: _____ # of Date Points: _____

Accuracy, Fortified/Spike Field Blank: _____ Sample # _____

Rel. Diff. (%), Duplicate Analysis: _____ Sample # _____