QUALITY ASSURANCE PROJECT PLAN

FOR

CHEMICAL WASTE MANAGEMENT, INC.
Vickery, Ohio

ADDENDUM TO THE GROUND WATER MONITORING PLAN

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VOLUME 2

PREPARED BY:

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ADDENDUM TO CWM Vickery GWMP QAPjP Appendix B-11
SOP Number: LM-RMA-4003
Title: Organochlorine Pesticides and PCBs

This addendum is specific to the Chemical Waste Management, Vickery, Ground Water Monitoring Plan QAPjP. Procedural changes or clarification statements presented herein are not applicable to Enseco SOPs for other projects, and are made here in response to an EPA letter dated March 17, 1992 to CWM containing comments (with additional attachment) regarding the CWM-Vickery GWMP QAPjP.

Section 2.2: Please refer to QAPjP Section IX for the list of project target compounds and QAPjP Appendix C-1 for the project specific quantitation limits.

Section 7.: Please refer to Section 9.1 of this SOP for additional data on instrument calibration standards and methods. QAPjP Appendix B-11A contains an example of a five point instrument calibration. The standard concentrations used for various analytes are listed as the "true" value, in ug/mL.

Section 8.2.4 and 8.2.5: Please see comment for Section 10.1, below.

Sample Cleanups: GPC and /or Acid cleanups will not be performed for this project since only aqueous samples will be analyzed.

Section 10.1: Matrix spike and matrix spike duplicate analysis are performed at a frequency of one per every group of 20 investigative samples. The compounds and concentrations used are the same as shown for duplicate control samples on pages 27 and 28 of the SOP.

Parameter retention time is variable with instrument condition and may vary slightly from calibration to calibration. Retention times recorded for an actual calibration are found in QAPjP Appendix B-11A.

SOP No.:	Revision No.:	Effective Date:
LM-RMA-4003	3.0	September 21, 1989

Supersedes:
2.0

Analysis of Organochlorine Pesticides and PCBs

1. Scope and Application

1.1 Analytes: organochlorine pesticides and polychlorinated biphenyls (see accompanying analyte lists)

1.2 Detection Limits: see analyte list in Section 2.2

1.3 Applicable matrices: water,soil, solids, oils, leachates, vegetation.

1.4 Linear Range of Instrument

1.4.1 Five point calibration curves are generated for all analytes except the multi-peak components (PCBs, chlordane, toxaphene), which have single point calibrations.

1.5 Analysis time

1.5.1 Sample prep requires at least 2 days for completion unless cleanup is required.

1.5.2 Analysis time is approximately 35 min per sample or standard.

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2. Summary of Method

2.1 Samples are extracted with methylene chloride (CH_2Cl_2). Extracts are dried, exchanged to hexane, and concentrated to a final volume of 10.0 mL. Analysis is performed on a gas chromatograph equipped with an electron capture detector. Samples are simultaneouly analyzed on two dissimilar columns. Data from one column are reported.

2.2 Reporting limits for the target parameters are presented in the following tables:

Parameter	608-PP-A Std. Prod. Reporting Limit
alpha-BHC	0.0030 ug/l
beta-BHC	0.0060
delta-BHC	0.0090
gamma-BHC (Lindane)	0.0040
Heptachlor	0.0030
Aldrin	0.0040
Heptachlor Epoxide	0.083
Endosulfan I	0.014
Dieldrin	0.0080
4,4'-DDE	0.0080
Endrin	0.0060
Endosulfan II	0.0080
4,4'-DDD	0.011
Endosulfan Sulfate	0.066
4,4'-DDT	0.012
Endrin Aldehyde	0.023
Methoxychlor	0.25
Chlordane	0.040
Toxaphene	0.60
Aroclor-1016	0.065
Aroclor-1221	0.065
Aroclor-1232	0.065
Aroclor-1242	0.065
Aroclor-1248	0.065
Aroclor-1254	0.50
Aroclor-1260	0.50

608-HSL-A & 608-AP9-A
Std. Prod.

Parameter	Reporting Limit
alpha-BHC	0.050 ug/L
beta-BHC	0.050
delta-BHC	0.050
gamma-BHC (Lindane)	0.050
Heptachlor	0.050
Aldrin	0.050
Heptachlor Epoxide	0.050
Endosulfan I	0.050
Dieldrin	0.10
4,4'-DDE	0.10
Endrin	0.10
Endosulfan II	0.10
4,4'-DDD	0.10
Endosulfan Sulfate	0.10
4,4'-DDT	0.10
Endrin Ketone	0.10
Methoxychlor	0.50
Chlordane	0.50
Toxaphene	1.0
Aroclor-1016	0.50
Aroclor-1221	0.50
Aroclor-1232	0.50
Aroclor-1242	0.50
Aroclor-1248	0.50
Aroclor-1254	1.0
Aroclor-1260	1.0
Appendix IX	
Chlorobenzilate	0.50 ug/L
Diallate	1.5
Isodrin	.050
Kepone	.10

Parameter	608-RCRA-A Reporting Limit	8080-RCRA-S Reporting Limit
Lindane	0.20 ug/L	1.0 ug/kg
Endrin	0.20	1.0
Methoxychlor	1.0	5.0
Toxaphene	2.0	10

Parameter	608-SDWAP-A Reporting Limit
Lindane	0.10 ug/L
Endrin	0.10
Methoxychlor	0.50
Toxaphene	1.0

Parameter	8080-HSL-S Reporting Limit (ug/kg)	8080-HSL-L-S Reporting Limit (ug/kg)
alpha-BHC	8.0	1.0
beta-BHC	8.0	1.0
delta-BHC	8.0	1.0
gamma-BHC (Lindane)	8.0	1.0
Heptachlor	8.0	1.0
Aldrin	8.0	1.0
Heptachlor Epoxide	8.0	1.0
Endosulfan I	8.0	1.0
Dieldrin	16	2.0
4,4'-DDE	16	2.0
Endrin	16	2.0
Endosulfan II	16	2.0
4,4'-DDD	16	2.0
Endosulfan Sulfate	16	2.0
4,4'-DDT	16	2.0
Endrin Ketone	16	2.0
Methoxychlor	80	10
Chlordane	80	10
Toxaphene	160	20
Aroclor-1016	80	10
Aroclor-1221	80	10
Aroclor-1232	80	10
Aroclor-1242	80	10
Aroclor-1248	80	10
Aroclor-1254	160	20
Aroclor-1260	160	20

8080-AP9-S & 8080-PP-S

Parameter	Reporting Limit (ug/kg)
alpha-BHC	8.0
beta-BHC	8.0
delta-BHC	8.0
gamma-BHC (Lindane)	8.0
Heptachlor	8.0
Aldrin	8.0
Heptachlor Epoxide	8.0
Endosulfan I	8.0
Dieldrin	16
4,4'-DDE	16
Endrin	16
Endosulfan II	16
4,4'-DDD	16
Endosulfan Sulfate	16
4,4'-DDT	16
Endrin Aldehyde	16
Methoxychlor	80
Chlordane	80
Toxaphene	160
Aroclor-1016	80
Aroclor-1221	80
Aroclor-1232	80
Aroclor-1242	80
Aroclor-1248	80
Aroclor-1254	160
Aroclor-1260	160

Appendix IX

Chlorobenzilate	80 ug/kg
Diallate	240
Isodrin	8.0
Kepone	16

3. Comments

3.1 Interferences are myriad. A florisil or alumina column cleanup is often required for soil samples (see Sections 8.5 and 8.6 respectively).

3.2 Helpful hints:

3.2.1 If a dirty-looking sample must be analyzed, insert a hexane blank in the following autosample position.

3.2.2 Use microvials for small sample volumes.

3.2.3 Always rinse autosampler vials with methanol (first) and hexane (last) and dry before use.

4. Safety

4.1 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. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified for the information of the analyst.

4.2 The following parameters covered by this method have been tentatively classified as known or suspected human or mammalian carcinogens: 4,4'-DDT, 4,4'-DDD, the BHCs, and the PCBs. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

5. Sample Collection, Preservation and Handling

5.1 Grab samples must be collected in glass containers. Conventional sampling practices should be followed, except that the bottle must not be pre-rinsed with sample before collection. Composite samples should be collected in refrigerated glass containers in accordance

with the requirements of the program. Automatic sampling equipment must be as free as possible of Tygon tubing and other potential sources of contamination.

5.2 All samples must be iced or refrigerated at 4°C from the time of collection until extraction.

5.3 All aqueous samples must be extracted within 7 days of collection and completely analyzed within 40 days of extraction. Soil/waste samples must be extracted within 14 days of collection and analyzed within 40 days of extraction.

6. Apparatus

6.1 Separatory funnel - 2 L, with Teflon stopcock.

6.2 Filtering Funnel - 75 mm top diameter.

6.3 Chromatographic column - 400 mm long X 22 mm ID.

6.4 Concentrator tube, Kuderna-Danish - 10 mL graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground glass stopper is used to prevent evaporation of extracts.

6.5 Evaporative flask, Kuderna-Danish - 500 mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with Springs.

6.6 Snyder column, Kuderna/Danish - 3-ball macro (Kontes K-503000-0121 or equivalent).

6.7 Vials - 10-15 mL with Teflon-lined screw cap.

6.8 Boiling chips - Approximately 10/40 mesh.

6.9 Water bath - Heated, with concentric ring over cover, capable of temperature control (± 2°C). The bath should be used in a hood.

6.10 Balance - Analytical capable of accurately weighing 0.0001g.

6.11 Sonicator - A horn-type sonicator equipped with a titanium tip should be used. The following sonicator, or an equivalent brand and model is recommended:

Ultrasonic cell disrupter: Heat Systems - Ultrasonics, Inc. Model W-385 (475 W) sonicator or equivalent (Power wattage must be a minimum of 375 W with pulsing capability and No. 200 1.2" Tapped Disrupter Horn) plus No. 207 3/4" Tapped Disrupter Horn, and No. 419 1/8" Standard Tapered microtip probe.

6.12 Sonabox - Recommended with above disrupters for decreasing cavitation sound (Heat Systems - Ultrasonics, Inc., Model 432B or equivalent).

6.13 Gas Chromatography - An analytical system complete with a gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and strip-cart recorder. A data system is recommended for measuring peak areas.

6.14 Detector - Electron capture detector.

7. Reagents and Standards

7.1 Reagent water - Reagent water is defined as a water in which an interferent is not observed at or above the method detection limit of each parameter of interest.

7.2 Sodium hydroxide solution (10 N)

7.3 Sulfuric Acid (1:1) ACS sp. gr. 1.84

7.4 Acetone, hexane, methylene chloride - pesticide quality or equivalent

7.5 Diethyl ether - nanograde, redistilled in glass if necessary. Must be free of peroxides as indicated by EM Quant test strips. After opening a bottle of diethyl ether, one must test it daily for peroxides. (Available from Scientific Products Co., Cat. NO. P1126-8, and other suppliers.) Procedures recommended for removal of peroxides are provided with the test strips. After cleanup, 20 mL ethyl alcohol preservative must be added to each liter of ether.

7.6 Sodium sulfate - (ACS) granular, anhydrous. Dry in a shallow tray at 400°C for a minimum of 4 hr to remove phthalates and other interfering organic substances. Alternatively, heat 16 hr at 450-500°C in a shallow tray or Soxhlet extract with CH_2Cl_2 for 48 hr.

7.7 Florisil - PR grade (60/100 mesh). Purchase activated at 1250°F and store in dark in glass container with ground glass stopper or foil-lined screw cap. Before use activate each batch at least 16 hr at 130°C in a foil covered glass container.

7.8 TBA-sulfite (Tetrabutylammonium sulfite)

7.9 Isopropyl alcohol

7.10 Primary standard solutions may be prepared from pure standard materials or purchased as certified solutions.

8. Extraction of Liquid Samples

8.1 Method Summary

This method is applicable to the determination of organochlorine pesticides and PCBs in aqueous samples. A 1 L water sample is extracted with CH_2Cl_2. The extract is dried, exchanged to hexane, and concentrated to a volume of 10.0 mL. Analysis is performed on a gas chromatograph equipped with an electron capture detector.

8.2 Extraction by Shakeout

8.2.1 If the sample contains particulate matter, transfer 3-4 mL of sample to a 12 mL screw top glass vial with a Teflon liner. Add approximately 1 mL of CH_2Cl_2. Shake vigorously for about 30 s and allow the layers to separate. If no emulsion forms, proceed with the shakeout technique. If an emulsion forms, use the continuous extractor method. In general, pesticide recoveries may be poor if there is an extreme amount of sediment or an extreme emulsion forms during the emulsion test.

NOTE: All glassware must be solvent rinsed with CH_2Cl_2 before use.

8.2.2 Samples that are clearly described as ground waters in the project folder are decanted from any sediment in the bottle. All others are mixed by shaking the bottle.

8.2.3 For samples in 1 L or smaller bottles, mark the meniscus on the side of the sample bottle. After emptying the

sample into a separatory funnel, fill the bottle with tap water and measure that amount in a graduated cylinder. For samples in larger bottles, measure 1.0 L into a graduated cylinder. Pour the sample into a 2 L separatory funnel. For blanks and quality control samples, pour 1.0 L of carbon filtered water into the separatory funnel. Record the volume of sample extracted on the prep sheet.

8.2.4 Add the appropriate surrogate standard to the sample using a 1 mL disposable pipet. Make sure the pipet tip is below the surface of the sample while the standard is being added. Mix the sample immediately by stirring with the disposable pipet. Record the name of the standard, verification number, amount added to sample, standard preparation date, initials of person who prepared standard, standard concentration, date added, initials of person adding standard, and initials of a witness on the prep sheet.

8.2.5 Add the spiking standard, if appropriate, using the procedure described in the preceding step.

8.2.6 Check the pH and note the initial pH on the prep sheet. Adjust the pH to 5-8, if necessary, with 10 N NaOH or 1:1 H_2SO_4. Note the adjusted pH on the prep sheet.

8.2.7 For samples that were mixed before pouring, add 60 mL CH_2Cl_2 to the sample bottle to rinse the inner walls. Do NOT cap and shake the bottle. Rinse the glass only; transfer the solvent to the separatory funnel. For samples that were decanted, add the first aliquot of CH_2Cl_2 directly to the separatory funnel. Extract the sample by shaking it for 2 min with frequent ventilation.

8.2.8 Allow the layers to separate. If there is an emulsion, break it mechanically or by centrifuging. Note any emulsions and corrective actions on the prep sheet.

8.2.9 Drain the bottom organic layer (CH_2Cl_2) into a 250 mL beaker.

8.2.10 Repeat the extraction 2 more times using a 60 mL aliquot of CH_2Cl_2 each time. Collect the solvent in the same beaker. Record the total volume of solvent recovered on the prep sheet.

8.2.11 Put a plug of glass wool in a funnel and fill about 2/3 full with Na_2SO_4. Rinse the funnel and Na_2SO_4 with 30-40 mL of CH_2Cl_2. Discard this rinsate in an appropriate waste container. Pour the extract through the Na_2SO_4 into a 500 mL Kuderna-Danish (K-D) evaporative concentrator. Rinse the beaker then the Na_2SO_4 with small amounts of CH_2Cl_2. Add these rinses to the K-D.

8.2.12 Add a boiling chip to the K-D and attach a 3 ball Snyder column to the top. Prewet the column by adding approximately 1 mL of CH_2Cl_2 through the top.

NOTE: The concentration step is critical; losses of target compounds can occur if the bath temperature is too high or too low or if extracts are allowed to boil too long.

8.2.13 Place the K-D in an 85°C hot water bath so that the receiver tube is immersed in hot water and the entire lower rounded surface is bathed in steam. At a proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume reaches 5-10 mL, remove the K-D from the bath and allow it to cool completely.

8.2.14 When the K-D has cooled, pour 50 mL of hexane through the top of the Snyder column. Add a fresh boiling chip and shake the Snyder column to make sure that the balls are not stuck. When the water bath has fully reheated to 95°C return the K-D to the water as before.

NOTE: Losses of target compounds can occur at this step. Make sure solvent loss is rapid (5-10 min). Do not allow the extract to evaporate for more than 10 min.

8.2.15 If the extract is highly colored or a precipitate forms during concentration, consult a supervisor.

8.2.16 After the K-D has cooled, rinse the Snyder column and middle flask with a small amount of hexane. Transfer the extract to a calibrated 15 mL centrifuge tube, rinsing with a small amount of hexane. Be sure to rinse all of the ground glass joints well, as compounds collect on the ground glass.

8.2.17 Carefully concentrate the extract to 10.0 mL under a gentle stream of nitrogen using the N-evap apparatus. If the extract is highly colored, forms a precipitate, or stops evaporating, consult a supervisor. Transfer the extract to a 12.0 mL vial with a teflon lined cap and mark the meniscus. Write your initials, date of final concentration, volume, parameter, solvent, project, and sample number on the vial.

8.2.18 If there is **ANY** color to the extract or precipitate, the organochlorine pesticide fraction will need a florisil column cleanup. Refer to Section 8.5.

8.2.19 Complete the prep sheet for the extraction and concentration steps.

8.3 Extraction by Continuous Extractor

8.3.1 See Section 8.2.1 for guidance on when to use the continuous extractor.

8.3.2 Put 250 mL CH_2Cl_2 in a round bottom flask and add a few boiling chips. Add 300 mL of CH_2Cl_2 to the extractor flask.

8.3.3 Carefully pour 500 mL of carbon filtered water into the extractor. Minimize the disturbance of the solvent layer and avoid getting water into either sidearm by pouring the water down the back of the extractor.

8.3.4 Open the sample container and remove a small aliquot of the sample with a pasteur pipet. Place enough of the sample on a pH paper to thoroughly soak the paper. Compare the pH paper with the chart on the container and record the initial pH on the prep sheet.

8.3.5 Aliquoting different kinds of samples

8.3.5.1 Water samples: Decant ground water samples into the extractor. Mix all other water samples by shaking the sample bottle. For samples in 1 L or smaller bottles, mark the meniscus on the side of the sample bottle. After emptying the sample into the extractor, fill the bottle with

tap water and measure that amount in a graduated cylinder. For samples in larger bottles, measure 1.0 L into a graduated cylinder. Pour the sample into the extractor. For blanks and quality control samples, pour 1.0 L of carbon filtered water into the extractor. Record the volume of sample extracted on the prep sheet.

8.3.5.2 Sludge samples: Remove an aliquot of approximately 10 g, place it in a pre-weighed aluminum boat and air-dry it overnight to determine the percent moisture. Weigh the bottle containing the sample and carefully transfer the sample to the extractor flask. Re-weigh the bottle, calculate the difference, and record it as the amount extracted on the prep sheet.

8.3.6 Add enough carbon filtered water to the extractor to bring the level of the solvent in the removable sidearm to about 4 cm from its highest point. Make sure that the extractor stays in a strictly vertical position.

8.3.7 Add the appropriate surrogate standard to the extractor using a disposable pipet. Make sure that the tip of the pipet is below the surface of the liquid in the extractor as the standard is being added. Mix the sample immediately using the disposable pipet. Record the name of the standard, verification number, amount added to sample, standard preparation date, initials of person who prepared standard, standard concentration, date added, initials of person adding standard, and initials of a witness on the prep sheet.

8.3.8 Add the spike mix, if appropriate, using the procedure described in the preceding step.

8.3.9 Adjust the pH to 5-8 with 10 N NaOH or 1:1 H_2SO_4 as appropriate.

8.3.10 Add enough carbon filtered water to the extractor flask to allow the solvent in the removable sidearm to just begin to drip into the round bottom flask. Record the total volume of carbon filtered water that was added on the prep sheet.

8.3.11 Remove the condenser from the rack and rinse the lower joint and lip with a squirt bottle (CH_2Cl_2). Place the condenser on top of the extractor. Turn on the water chiller (set at 10°C) and check the flow indicators.

8.3.12 Turn on the heating mantle so that the indicator light is barely lit. Record the starting time on the prep sheet. Check after 15 min to be sure that the solvent in the round bottom flask is boiling, that solvent is dripping from the lip of the condenser, and that the volume of the solvent in the round bottom flask is still approximately 250 mL. Adjust the rheostat so that the light is just bright after refluxing has started. Check all extractor joints for leaks with the corner of a Kimwipe.

8.3.13 Allow the extraction to proceed for a minimum of 6 hr.

8.3.14 Turn off the heating mantle and allow the apparatus to cool (30-60 min) with water flowing through the condenser.

8.3.15 The solvent (CH_2Cl_2) contained in the round bottom flask is the extract. Remove the flask from the extractor and transfer the extract to a rinsed 400 mL beaker. Rinse the flask with a small aliquot of CH_2Cl_2 and add this rinsate to the 400 mL beaker. Record the volume of extract recovered on the prep sheet.

8.3.16 Go to Section 8.2.11 and proceed with the prep.

8.4 Extraction of Soil Samples by Sonication

NOTE: All glassware must be solvent rinsed with CH_2Cl_2 before use.

8.4.1 Weigh 4 g of the sample into a 250 mL centrifuge bottle. Add 10 g of dried Na_2SO_4 and stir the mixture well with a steel spatula. The sample should have a grainy texture. If a clump forms add more Na_2SO_4 and note it on the prep sheet.

8.4.2 Add 50 mL of CH_2Cl_2 to all samples.

8.4.3 Add the appropriate surrogate standard using a disposable pipet.

NOTE: For samples being analyzed for PCBs only, the surrogate is added only to the method blank (SCS). Drizzle the standard over the surface of the sample and immediately swirl the flask to mix the standard with the sample. On the prep sheet, record all of the required information regarding the standard.

8.4.4 Add the spike, if appropriate, using the procedure described in the previous step.

8.4.5 Sonicate the samples for 1.5 min at an output setting of 10 with the 3/4 inch sonicator horn 1/2 inch below the surface of the solvent. The sonicator should be in the 1 s pulse mode, with the duty cycle set at 50%. Centrifuge the samples for 3-5 min at 35 K RPM.

8.4.6 Decant the solvent layer into a rinsed 250 mL beaker.

8.4.7 Repeat the extraction 2 more times using 50 mL aliquots of CH_2Cl_2 each time. Collect these extracts in the same beaker.

8.4.8 Record the total volume of solvent recovered on the prep sheet.

8.4.9 Put a plug of glass wool in a funnel and fill 2/3 full with Na_2SO_4. Rinse the funnel and Na_2SO_4 with 30-40 mL of CH_2Cl_2, discard. Pour the extract through the Na_2SO_4 into a 500 mL Kuderna-Danish (K-D) evaporative concentrator. Rinse the beaker then the Na_2SO_4 with small amounts of CH_2Cl_2 into the K-D flask.

8.4.10 Add a boiling chip to the K-D and attach a 3 ball Snyder to the top. Pre-wet the column by adding about 1 mL of CH_2Cl_2 through the top.

NOTE: The concentration step is critical; losses of target compounds can occur if the temperature of the bath is too high or low or the extract is exposed to heat too long.

8.4.11 Place the K-D in a heated water bath set at 85°C so that the receiver tube is immersed in hot water and the entire lower rounded surface is bathed in steam. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume reaches 2-5 mL, remove the K-D from the bath and allow it to completely cool.

8.4.12 When the K-D has cooled, pour 50 mL of hexane through the top of the Snyder column. Add a fresh boiling chip and shake the Snyder column to make sure that the balls have not stuck. When the water bath has re-heated to 95°C, return the K-D to the water as before. NOTE: losses of target compounds can occur at this step. Make sure solvent loss is rapid (10-15 min). Do not allow the extract to evaporate for more than 15 min.

8.4.13 After the K-D has cooled, rinse the Snyder column and middle flask with a small amount of hexane. Transfer the extract to a calibrated 15 mL centrifuge tube, rinsing with a small amount of hexane. Be sure to rinse all of the ground glass joints well, as compounds collect on the ground glass.

8.4.14 Carefully concentrate the extract to 10.0 mL under a gentle stream of nitrogen using the N-evap apparatus. If the extract is highly colored, forms a precipitate, or stops evaporating, consult a supervisor. Transfer the extract to a labeled 12 mL screw-top vial with a Teflon lined cap, mark the meniscus, and place on a rack with other samples with the same project number, if any. Include the following information on the label: project and sample number, matrix, parameter, solvent, final volume, your initials, and date of final concentration.

8.4.15 If the extract is colored or a precipitate forms, organochlorine pesticide extracts will need a florisil column cleanup; see Section 8.5. If the extract is to be analyzed for PCBs only, the appropriate cleanup procedure may be found in Section 8.6. If there is any question, consult a supervisor.

8.4.16 Complete the prep sheet for the extraction and concentration steps.

8.5 Extraction of Oil Samples by Acid Digestion

8.5.1 Place a rinsed 50 mL ground glass stopper graduated cylinder on the top loading balance and zero the balance.

8.5.2 Mix samples well to ensure a representative aliquot.

8.5.3 Weigh 1.0g of sample oil into the graduated cylinder and note the weight on the prep sheet.

8.5.4 Dilute the sample to 10.0 mL with hexane.

8.5.5 The total volume for all samples should be 10.0 mL. Note this volume on the prep sheet.

8.5.6 Bring the total volume to 50.0 mL with concentrated H_2SO_4. Wear appropriate protective clothing.

8.5.7 Stopper the cylinder and shake it vigorously for at least one minute.

8.5.8 Allow the layers to separate and wait at least 5 minutes after the apparent separation. Some separations may require a wait of 2 hours or more. Record the volume of hexane recovered as measured by the difference in the graduated cylinder.

8.5.9 Pipet the hexane layer into a 12 mL screw top vial with a Teflon lined cap.

8.5.10 The acid goes into "PCB Contaminated Acid Waste;" all of the glassware is to be treated as PCB contaminated.

8.5.11 Label the vial as "Acid Washed," mark the meniscus, and store it in the refrigerator to be column cleaned.

8.6 Florisil Cleanup for Pesticide Samples

8.6.1 Method Summary: A sample extract in hexane is subjected to a florisil adsorption column clean-up to remove interferences and separate pesticides into discrete

fractions. The extract is introduced onto the florisil and eluted into 2 discrete fractions with solvents of varying polarities.

8.6.2 Procedure

Prepare the eluting solvents in 250 mL stoppered graduated cylinders.

Fraction 1: 200 mL 6% diethyl ether in petroleum ether
Fraction 2: 200 mL 15% diethyl ether in petroleum ether
Fraction 3: 200 mL 50% diethyl ether in petroleum ether

8.6.3 A column performance standard, OCP CPS, accompanies every set of pesticide florisil columns.

8.6.4 Place a glass wool plug in the bottom of a 20 mm I.D. chromatographic column. Add 20 g of florisil, mesh size 60-100.

8.6.5 Tap the top of the column with a rubber stopper to settle the florisil. Add 1 cm of dried Na_2SO_4 to the top of the column and tap again.

8.6.6 Pre-elute the column with 30-50 mL of petroleum ether. Collect the eluate in a waste beaker. Just prior to the exposure of the Na_2SO_4 to the air, quantitatively transfer 10.0 mL (for industrial samples) of sample extract onto the top of the column. Continue collecting in a waste beaker.

8.6.7 Just prior to the exposure of the Na_2SO_4 to the air, add Fraction 1. Collect this eluate in a rinsed 400 mL beaker labeled "Column Cleaned-A".

8.6.8 Just prior to the exposure of the Na_2SO_4 layer to the air, add Fraction 2. Collect this eluate in the same beaker as above until the solvent volume reaches about 300 mL. Replace beaker with another rinsed beaker labeled "Column Cleaned-B".

8.6.9 Again, just prior to the exposure of the Na_2SO_4 to the air, add Fraction 3. Continue collecting the eluate in Beaker B.

8.6.10 Pour the eluate from Beaker A into a Kuderna-Danish (K-D) flask. It is not necessary to pass the eluate through Na_2SO_4.

8.6.11 Add a boiling chip to the K-D. Attach a 3-ball Snyder column and prewet by adding about 1 mL of petroleum ether through the top.

8.6.12 Place the K-D on a heated water bath set at 85°C so that the receiver tube is partially immersed in hot water and the entire lower rounded surface is bathed in steam. At a proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume reaches 5-10 mL, remove the K-D from the water and allow it to cool.

8.6.13 After the flask has cooled, add the eluate from Beaker B. Add a fresh boiling chip and repeat the procedure.

8.6.14 Add 50 mL of hexane through the top of the Snyder column. Add a fresh boiling chip to the flask and shake the Snyder column to make sure the balls are not stuck.

8.6.15 When the bath has re-heated to 95°C, return the K-D to the water and concentrate as before, but this time to an apparent volume of 2-5 mL.

8.6.16 After the K-D has completely cooled, rinse the column and the middle flask with a small amount of hexane. Adjust the final volume to equal the volume of extract that was cleaned. Mark the meniscus, label the vial, and place the extract with the others in the same project, if appropriate.

8.7 Alumina/Silica Cleanup for PCB Samples

8.7.1 Method Summary: Interferences present in Polychlorinated Biphenyl (PCB) sample extracts in hexane are removed using a basic alumina and silica gel column clean-up. The chlorinated hydrocarbons are selectively eluted with a moderately polar solvent.

8.7.2 Materials:

8.7.2.1 Brockman Basic Alumina, Activity I, 80-200 mesh, or equivalent, 20g/sample. Deactivated alumina with carbon-filtered water at 0.5% by weight (w/w) (i.e., 100g alumina, 0.5g CF water). Store in a tightly capped glass bottle. Label bottle, including alumina type with mesh size and deactivation date. Re-activate and prepare at least every 10 days.

8.7.2.2 Acidified silica gel, 100-200 mesh, 6g/sample. Add H_2SO_4 (conc.) at 40% by weight (w/w) to the silica gel (i.e., 100g silica gel, 40g H_2SO_4) and shake well to distribute evenly. Silica gel should be activated prior to acidification at 200°C overnight and stored in the dessicating cabinet.

8.7.2.3 Anhydrous Na_2SO_4, 4g/sample. Dried at 400°C for 4 hr in furnace or 300°C in oven overnight.

8.7.2.4 Eluant, 20% methylene chloride in hexane, 50 mL/sample.

8.7.3 Procedure for Oil Samples:

8.7.3.1 The sample should first be acid-washed. One wash is generally enough; the sample does not need to be clear after the acid wash procedure.

NOTE: Record all cleanups, volumes, and weights in the record book and on the benchsheets.

8.7.3.2 Column preparation: Add 20g 0.5% deactivated basic alumina to a 20mm I.D. chromatographic column plugged with glass wool. Tap column to settle. Add 4g dried Na_2SO_4 (do not tap column), followed by 6g of acidified silica gel. Tap to settle.

8.7.3.3 Pre-elute the column with 40 mL of hexane. Collect the rinsate in a waste beaker.

8.7.3.4 Add 3.0 mL of the sample extract to the head of the column.

8.7.3.5 After the silica gel becomes exposed to the air, pre-elute the sample with 40 mL of hexane. Collect the rinsate in a waste beaker and discard as oil-contaminated.

8.7.3.6 Just as the silica gel becomes exposed to the air, exchange the waste beaker with a 50 mL graduated centrifuge tube (calibrated at 5.0 mL), which is the final sample collector.

8.7.3.7 Carefully add the 50 mL of 20% methylene chloride in hexane eluant, rinsing the column wall well.

8.7.3.8 After elution, columns, packing materials, glassware and waste must all be considered PCB contaminated.

NOTE: If time permits, continue with the concentration step; otherwise, double-wrap each sample with aluminum, seal with a rubber band, and store in the concentration refrigerator. Indicate at what place in the procedure you stopped on the benchsheet and in the record book.

8.7.3.9 Equilibrate the N-evap to 40°C. Immediately after the samples are placed on the N-evap, increase the bath to 63°C to ensure total removal of CH_2Cl_2. To prevent oils and PCB's (PCB's have a high affinity for glass) from sticking to the glass, continually rinse down the graduated centrifuge tube with hexane. Concentrate to slightly less than 5 mL. Before adjusting to a final volume of 5.0 mL, allow the samples to cool to room temperature.

NOTE: Label all sample vials as "column cleaned" before storing in the Pest/PCB extract refrigerator.

8.7.4 Procedure for Soils, Sludges and Water Samples

8.7.4.1 If the sample is extremely dark, it must be acid washed prior to column clean-up. If the sample extract is clear or only slightly discolored, an acid wash is not necessary. For acid washes a 10.0 mL aliquot of the extract should be used.

NOTE: Record all clean-ups, volumes and weights in the record book and on the benchsheet.

8.7.4.2 Column preparation: Add 20g of 0.5% deactivated basic alumina to a 20mm I.D. chromatographic column plugged with glass wool. Tap column to settle. (If alumina leaks from the tip of the column, repack the column or add more glass wool to the tip.) Add 4g dried Na_2SO_4 (do not tap column), followed by 6g of acidified silica gel. Tap to settle.

8.7.4.3 Pre-elute the column with 40 mL of hexane. Collect the rinsate in a waste beaker.

8.7.4.4 Add 5.0 mL of the sample extract to the head of the column.

8.7.4.5 After the silica gel becomes exposed to the air, pre-elute the sample with 40 mL of hexane. Collect the rinsate in a waste beaker and discard as oil-contaminated.

8.7.4.6 Just as the silica gel becomes exposed to the air, exchange the waste beaker with a 50 mL graduated centrifuge tube (calibrated at 5.0 mL), which is the final sample collector.

8.7.4.7 Carefully add the 50 mL of 20% methylene chloride in hexane eluant, rinsing the column wall well.

8.7.4.8 After elution, columns, packing materials, glassware and waste must all be considered PCB contaminated.

NOTE: If time permits, continue with the concentration step; otherwise double-wrap each sample with aluminum, seal with a rubber band, and store in the concentration refrigerator. Indicate at what place in the precedure you stopped on the benchsheet and in the record book.

8.7.4.9 Equilibrate the N-evap to 40°C. Immediately after the samples are placed on the N-evap, increase the bath to 63°C to ensure total removal of CH_2Cl_2. To prevent oils and PCB's (PCB's have a high affinity for glass) from sticking to the glass, continually rinse down the graduated centrifuge tube with hexane. Concentrate to slightly less than 5 mL. Allow the samples to cool to room temperature. Adjust to a final volume of 5.0 mL.

NOTE: Label all sample vials as "column cleaned" before storing in the Pest/PCB extract refrigerator.

9. Analysis

9.1 Calibration of Instrument

9.1.1 Five point calibration curves are generated for all analytes except the multi-peak components (PCBs, chlordane, toxaphene), which have single point calibrations. Most compounds are linear in the range 0.005-0.25 ug/mL. Correlation coefficients $\geq$0.995 are expected for each analyte. External standard tables are used to quantify samples.

9.1.2 Run a mid-level check standard after every 10th injection and at the end of the sequence. Precede each check standard with a solvent blank. The change in response factors (RF), as defined by an RPD calculation, should be <20%. The calculation is performed as follows for each analyte.

$$\left|\frac{X_1 - X_2}{X_1}\right| (100\%) = RPD,$$

Where X_1 = area of first standard
X_2 = area of later standard

9.1.3 EVALB is the name given to the check standard run at the beginning of every sequence. It contains aldrin, endrin, p,p'-DDT, and dibutylchlorendate at 0.10 ug/mL. This standard is used to inspect the general performance of the chromatographic system by checking peak shape and on-column degradation of endrin and p,p'-DDT, and to set the scale for the Chromatography System.

9.1.4 The analyst should examine the shape of all peaks to ascertain that they are generally symmetrical. Tailing peaks indicate that the system requires routine maintenance.

9.1.5 Degradation of endrin or p,p'-DDT on the column is limited by the EPA to <20% of either compound and this criterion is applied to all work. By the time column degradation is approaching these values, maintenance as described above is mandatory.

9.1.6 Degradation in the EVALB mixture is calculated as follows:

Using area counts, calculate endrin degradation as a percentage of total endrin:

$$\frac{\text{endrin aldehyde + endrin ketone}}{\text{endrin + endrin aldehyde + endrin ketone}} \times 100\%$$

Similarly, calculate the degradation of p,p'-DDT:

$$\frac{\text{p,p'-DDE + p,p'-DDD}}{\text{p,p'-DDT + p,p'-DDE + p,p'-DDD}} \times 100\%$$

9.1.7 There are no requirements for allowable changes in sensitivity for individual pesticide analyses. However, we impose CLP requirements. That is, changes in RFs on the quantitation column may vary by no more than 15% and on the confirmation column by no more than 20% throughout the course of the run. If these limits are exceeded, the run is stopped. Affected data is analyzed using the closest standard. The instrument must be recalibrated.

9.2 GC Conditions

9.2.1 Second column confirmation is required for all samples that contain any target compounds. Dissimilar columns may be operated simultaneously.

9.2.2 Analytical Conditions for Hewlett-Packard 5880 or 5890 equipped with 2 electron capture detectors and fitted with 2 megabore capillary columns as follows:

Column A: 007-608 (Methyl phenyl cyanopropyl silicone) 30m, 0.53mm I.D., 1.0 u film thickness.

Column B: 007-DB5 (Methyl phenyl 5% silicone) 30m, 0.53mm I.D., 1.0 u film thickness.

The following conditions are suggested for analysis with an H-P 5890 GC with dual megabore split capillary system.

Injection temperature	:	200°C
Initial temperature	:	60°C
Initial time	:	2 min
Program rate	:	40°C/min
Final temperature	:	200°C
Final time	:	2 min
Prog rate 2	:	3°C/min
Final temperature 2	:	230°C
Final time 2	:	2 min
Prog rate 3	:	2°C/min
Final temperature 3	:	240°C
Final time 3	:	10 min
Detector temperature	:	325°C
Carrier flow	:	7-10 mL/min H_2
Makeup gas	:	N_2
Total flow	:	40-50 mL/min
Run time	:	32 min.

10. QC/QA Requirements

10.1 For a standard products test duplicate lab control samples (blank spikes) are generated for every 20 samples and a single control sample (method blank) is generated with every prep and analysis set. Project-specific Q.C., usually matrix spikes or duplicates, may be requested.

10.2 Acceptance limits for Q.C. follows Enseco's Internal QC program; 80% of the accuracy values and 80% of the precision values must be acceptable for analysis to proceed.

METHOD 608
ORGANOCHLORINE PESTICIDES by GC
AQUEOUS MATRIX

Analytes	Spike Conc.	Units: ug/L Measured Conc. LCS 1	LCS 2	|-----Accuracy-----| % Recovery LCS 1	LCS 2	Control Limits	|-Precision-| RPD	Control Limits
Lindane	0.2	____	____	____	____	56-123	____	15%
Heptachlor	0.2	____	____	____	____	40-131	____	20%
Aldrin	0.2	____	____	____	____	40-120	____	22%
Dieldrin	0.5	____	____	____	____	52-126	____	18%
Endrin	0.5	____	____	____	____	56-121	____	21%
4,4'-DDT	0.5	____	____	____	____	38-127	____	27%

METHOD 8080
ORGANOCHLORINE PESTICIDES by GC
SOLID MATRIX

Analytes	Spike Conc.	Units: ug/L Measured Conc. LCS 1	LCS 2	|-----Accuracy-----| % Recovery LCS 1	LCS 2	Control Limits	|-Precision-| RPD	Control Limits
Lindane	27	____	____	____	____	46-127	____	50%
Heptachlor	27	____	____	____	____	35-130	____	31%
Aldrin	27	____	____	____	____	34-132	____	43%
Dieldrin	67	____	____	____	____	31-134	____	38%
Endrin	67	____	____	____	____	42-139	____	45%
4,4'-DDT	67	____	____	____	____	23-134	____	50%

METHOD 608
POLYCHLORINATED BIPHENYLS (PCBs) by GC
AQUEOUS MATRIX

Analytes	Spike Conc.	Units: ug/L Measured LCS 1	Conc. LCS 2	|-----Accuracy-----| % Recovery LCS 1	LCS 2	Control Limits	|-Precision-| RPD	Control Limits
Aroclor 1254	5.0	____	____	____	____	20-160	____	20%
		____	____	____	____		____	
		____	____	____	____		____	
		____	____	____	____		____	
		____	____	____	____		____	
		____	____	____	____		____	

METHOD 8080
POLYCHLORINATED BIPHENYLS (PCBs) by GC
SOLID MATRIX

Analytes	Spike Conc.	Units: ug/L Measured LCS 1	Conc. LCS 2	|-----Accuracy-----| % Recovery LCS 1	LCS 2	Control Limits	|-Precision-| RPD	Control Limits
Aroclor 1254	167	____	____	____	____	20-160	____	20%

Corrective Action: See Current Q.C. manual

11. Calculations

For Waters: $\frac{\text{sample conc ug/mL X final volume (mL) X dilution factor}}{\text{L extracted}}$

For Soils: $\frac{\text{sample conc ug/mL X final volume (mL) X dilution factor}}{\text{Kg extracted}}$

12. Reporting

12.1 Reporting units. Aqueous samples are reported in ug/L. Solid samples are reported in mg/kg.

12.2 Reporting limits are test specific. Very often the reporting limit is equivalent to the detection limit. For tests such as A010CP RCR and A160CPTCLP the reporting limit is based on the regulatory limit.

12.3 Results are reported to 2 significant figures unless a client specifies otherwise. Values below the detection limit are reported as ND-not detected.

12.4 LIMS data entry is usually done by the analyst, and reviewed by data review section.

13. Method Source

13.1 Federal Register Vol. 49, no. 209, Friday October 26, 1984

13.1.1 Deviations

The 608 method presumes a 3 point calibration curve is run and that subsequent calibration checks are within 10% of the original curve. We run a 5 level curve of all analytes of interest. We are satisfied with the results if the subsequent standards are within 20% of the curve. The rational is:

13.1.1.1 It is unrealistic to run 30 standards before analyzing any samples.

13.1.1.2 The 10% window is too tight. EPA only requires 15% and 20%.

13.2 USEPA, Office of Solid Waste and Emergency Response, Washington, DC 20460, November, 1986, SW-846, Third Edition.

APPENDIX A

Appendix A. Analysis is run with the Nelson system:

1. Generate a sequence file (sample table) on the Compaq:

   From DOS type MENU (return). From the main menu enter "3".

   Sequence file: file names are up to 10 characters, ending in "A" for column A and "B" for column B e.g. INDPESTA, THMB. Use no punctuation except for periods.

   Sample names: the field is sufficiently large to provide complete description. Use no punctuation except for periods. e.g. TOXAPHENE STD 0.25 UG PER mL

Method name: AEPI for Column A
BEPI for Column B

Method names are up to 10 characters. Our naming convention is that the first character specifies the column (A or B), the second specifies the instrument, (in this case E), and the remainder should be relevant to the analysis. PI in AEPI stands for "pesticides, individual stds".

Data file name: up to 10 characters. The first letter followed by a colon designates the data storage disk currently in use. Our naming convention again specifies column and instrument as the first 2 characters after the colon followed by 3 numbers for the Julian date (001 to 366) e.g. D:AE080. To save the sequence file, enter F9 then enter the sequence file name. When the next screen comes up enter F10,YES.

Where the system is set up so that both GC channels are fed into 1 Nelson interface, it is only necessary to generate a sequence file for Column A. The system will automatically apply this list to Column B.

2. On the 5880 integrator, use CHANNEL B with SIGNAL B ENTER keyed into the integrator.

   GET PROGRAM "NELSON B" DEVICE 16.

3. Download a Nelson interface box:

   On the Compaq from the main menu enter option 0, acquire data. Then choose F2 DOWNLOAD. If using a sequence file, enter YES and name of file including drive specification, e.g. D:INDPESTA. As the method files are all stored on drive M:, enter M:AEPI (e.g.) Enter desired parameters to the questions which follow.

   Two channel acquisition is specified in method M:AEPI. Once the interface is downloaded for channel A, the system automatically seeks method M:BEPI for channel B and automatically labels the samples on column B and stores runs from column B in _:BE_ _ _ . Stored runs are sequentially numbered and should match run numbers e.g. D:AE0801=run 1, D:AE0802=run 2, etc.

4. Always run a PS005C first. When it has run, return to main menu (F10), select 1 to modify M:AEPI. When the METGEN MENU is displayed, choose option 2 and change the millivolt settings to the desired values. The value for "plot" is that for column A and the value for "Ch 1" is that for column B.

   Exit using F9, YES to overwrite and F10 followed by YES to return to main menu.

APPENDIX B

Appendix B: Collecting and reducing all data with the Nelson system

1. Data collection:

Once the Nelson interface box is collecting data, the Compaq should be left in the acquire mode (0) in order that data is sent to disk and spooled to the printer as it is generated.

A. Setting up a library:

To reduce Nelson data, it is necessary to have a library against which to compare. Libraries are all on drive L:, access to libraries from DOS is gained by typing EDIT. Usually, each analyst has "private" libraries for individual work. Such libraries are prefixed by the analyst's initials and the privacy of these libraries should be respected. This is not to say they cannot be copied. For HP5880 instrument E, the base library is PEST101 for column A and PESTMIX for column B. To set up a new library from DOS, type:

COPY L;PEST101.LIB L:? ? ? 101.LIB and
COPY L:PESTMIX.LIB L:? ? ? MIX.LIB

The libraries typically have complete organochlorine pesticide lists, including irregularly used compounds such as the o,p'-series of DDT and its metabolites and the additional compounds required by Appendix IX.

It is important that each compound in the library have a correct retention time and reasonable retention time window.

To enter the program, type GCCOMP from DOS. The first screen gives 12 questions to be answered. Data drive, instrument, date, start cycle, end cycle are all obvious. For print standards answer yes. The sequence file does not require a drive to be specified. Libraries are usually set up for each instrument. Currently this program does all calculations on a single point, the mid-level standard which is always level 3 in the libraries. And, no, you do not wish to abort the program.

The program automatically goes through the cycles specified, first identifying standards and the components expected in them and updating the library with the new areas and the retention times. The program repeats the process with the sample runs, identifying hits and quantitating hits on both columns. Finally, it compares hits from both columns and procedures report of compounds found on both columns with calculated amounts and percent deviation from the expected retention time.

2. Nelson Libraries

All libraries are stored on drive L:, and access is gained from DOS through the EDIT program. With this program, it is possible to add (or delete) compounds to a library, change retention times and windows and method of quantitation.

Type EDIT from DOS and hit return when the screen shows Default drive = L: The option screen will show three options.

EDIT OLD LIBRARY
CREATE NEW LIBRARY
PRINT LIBRARY CONTENTS

Use the arrow keys to move the highlighted line and enter the desired option.

A. Edit Old Library

The screen will show available libraries. The basic libraries for instrument E are:

PEST101.LIB containing pesticide data for SP2100 column

PESTMIX.LIB containing pesticide data for the SP2250/SP2401 column

Use F2 to indicate which library is to be edited. The space at the end of the line is highlighted (LIBRARY:). Enter the library name, then F3, proceed and library entry 001 will come up on the screen.

Each entry consists of 2 pages and F1 allows you to go between pages. Page 1 contains RT information. Entries may be changed by moving the highlighted box using arrow keys and typing over the current entry. It is necessary to change absolute retention times if chromatographic conditions change significantly. Absolute RT are used for pesticide analyses and these entries rarely must be changed. Again, 'SINGLE' is the method primarily used for pesticide quantitation although there are 4 choices:

Average: The response factors of all active levels will be averaged and the average response factor will be used to calculate concentration.

Line: A least squares regression line ("best fit") will be drawn to encompass all active levels and the slope of this line will be the response factor used to calculate concentration.

Interpolation: Lines will connect all active levels (point-to-point) and the concentration will be calculated using the slope of the appropriate line segment as the response factor.

Single: The response factor from a single calibration level will be used to calculate concentration. The level to be used is specified in the next line "Calculation level."

Page 2 contains quantitative information. There are 7 levels of calibration possible for each entry with the first being 0,0.00. Amounts must be entered on this page. Entries for level 3 are quantities for mid-level standards (e.g. CM019C) and entries for level 4 are quantities for B-level standards (e.g. CM019B). The absolute response factor and reference data file are updated automatically by using the REPORT program.

When all information for an entry is correct exit that entry by F2. This exits but remains in the library so that other entries can be edited.

List entries - lists library entries on the screen
Print entries - makes a hard copy of the list of library entries
Change entries - allows you to change info on another entry
Add entry - appends an entry to the library
Exit to Edit

To change another entry, move highlight to "Change Entry" and enter. Answering YES to save last entry, saves any changes, NO does not. Enter the number of the library entry you wish to go to next (1-100). Exit using F3.

B. Create New Library

The screen will display a directory of libraries currently stored on Drive L: Use F2, create library and enter the name you wish to give the new library in the highlighted box. Then push F3-proceed.

Each entry has 2 pages of information associated with it. See section 1 for detailed information. Fill in both pages for all entries desired.

Use F3 to exit and save the new library.

C. Print Library Contents

This prints both pages for each library entry and can run to quite a few pages. The screen displays the libraries stored on drive L: Use F2 and enter the name of the desired library in the highlighted box. Enter F3, proceed. You have a choice of printing all entries or a range of sequential entries.

It is important to be aware that any library is accessible to all comers. If two people try to use the same library, collisions will inevitably occur. To avoid this, establish your own library by copying the base library.
B> COPY L:PEST101.LIB L:MJW101.LIB. Libraries prefixed by such initials are for the owner's use only.

3. Reprocessing Nelson Data

If chromatograms are missing but the data is available on disk (only the .PTS and .HDR file are needed); they can be reconstructed and .ATB generated using option 10 of the Nelson menu - batch reprocessing.

The answers to the questions on the screen are fairly self-evident. The base file name must include drive specification e.g. F:AE083. The default method file name is the method file to be used for integration.

When all the data has been spooled to the printer, the screen will display the status of all the Nelson interfaces. Activate the printer by simultaneously pressing F1.

Occasionally, both .ATB and .HDR files may be missing, but so long as a .PTS file is in memory, all is not lost. A .HDR file can be copied and, while some information may have to be corrected by hand, this copy and the .PTS file can be used in batch reprocessing to generate a chromatogram and .ATB file. To copy a header file:

```
B:/>COPY F:AE08013.HDR F:BE08013.HDR
```

If it is necessary or desirable to reintegrate peaks or to plot them at a different scale, this may be done using option 14 of the Nelson menu - Process Disk Data.

Once the raw data file has been entered, default values for threshold, peak width may be accepted. The timed events may or may not be edited-generally not.

After some working of the computer, a new screen will ask for a new area reject and whether or not to print a new area percent table. Internal and external standard tables are not printed for current use.

Usually the default values are accepted for amount injected and dilution factor are accepted.

The number of pages for replot permits compressing or expanding the horizontal axis of a plot. This is sometimes useful for making it easier to see complex chromatograms and to examine how baselines are drawn. Accepting the default value will leave the horizontal axis unchanged.

Answer "yes" to get a hard copy only if a copy of the original chromatogram is desired. At this point, peaks are not identified and named.

The first screen plots the first portion of the chromatogram. If this segment does not contain the peak of interest use F10 to proceed to the next segment or until the segment of interest is reached.

The first screen plots the first portion of the chromatogram. If this segment does not contain the peak of interest use F10 to proceed to the next segment or until the segment of interest is reached.

To change the way a peak is integrated (to redraw a baseline) move the cursor using the arrow keys on the right side of the keyboard. Move the cursor to the desired baseline point at the beginning of the peak and press F7 (point 1). Then move the cursor to the desired baseline at the trailing edge of the peak and press F8 (area). The new area will be printed alongside the reintegrated peak. To insert this number into the area table replacing the old value, press F9 (replace). The peak number to be entered is the appropriate peak number from the Nelson chromatogram.

A hard copy of the desired screen is obtained by pressing F6, then F1. The next screen provides the opportunity to replot the chromatogram larger or smaller than the original, stretched or compressed, or a combination. A range of times or the entire chromatogram may be replotted on as many pages (screens) as desired. Names are not put on the plot, offset is usually 0, screens are not hard copied until desired. Absolute mV full scale permits "electronic dilution." Replotting a chromatogram at 200mV which was originally plotted at 20mV will result in a 10-times down-scaling. Chromatograms may be re-scaled, reintegrated and hard copied as many times as desired before exiting the program. Screens that are hard copied by F6 are not printed until F1 is pressed and may be accumulated.

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Reduced by: MM Date: 5-20-92 Initial Calibration Report
Reviewed by: _____ Date: _____

Project: OCP05_3, Channel: 57, Analysis: C18MAY92, Node: DENCR3
Analyst: MGOETZ, Calibration File: C0518, MCRPT Method: OCPA

Calibration curve type: Linear NOT forced through zero

Sample	Level ID	Description
4	L5	Individual Mix AB 0.10ug/mL T=ST 6266
5	L4	Individual Mix AB 0.05ug/mL T=ST 6266
6	L3	Individual Mix AB 0.025ug/mL T=ST 6266
7	L2	Individual Mix AB 0.01ug/mL T=ST 6266
8	L1	Individual Mix AB 0.005ug/mL T=ST 6266

%RSD Measured	%RSD Maximum	Compound
9.1	20.0	Tetrachloro-m-xylene (TCX)
4.6	20.0	alpha-BHC
5.2	20.0	gamma-BHC
8.2	20.0	beta-BHC
8.1	20.0	Heptachlor
2.0	20.0	delta-BHC
4.1	20.0	Aldrin
6.8	20.0	Heptachlor Epoxide
6.2	20.0	Endosulfan I
3.9	20.0	4,4'-DDE
4.0	20.0	Dieldrin
4.8	20.0	Endrin
2.5	20.0	4,4'-DDD
6.2	20.0	Endosulfan II
2.8	20.0	4,4'-DDT
5.9	20.0	Endrin Aldehyde
6.5	20.0	Endosulfan Sulfate
5.6	20.0	Dibutylchlorendate (DBC)
6.5	20.0	Methoxychlor
5.7	20.0	Endrin Ketone
0.0	20.0	Octachloronapthalene
10.4	20.0	Decachlorobiphenyl (DCB)

Wed May 20 10:16:08 1992

Reduced by: MM
riewed by: _____

Initial Calibration Report

Project: OCP05_3, Cl : C18MAY92, Node: DENCR3
Analyst: MGOETZ, Cal 3, MCRPT Method: OCPA

B-11A

Calibration curve ty rced through zero

Sample	Level	ID	Description
4	L5		Individual Mix AB 0.10ug/mL T=ST 6266
5	L4		Individual Mix AB 0.05ug/mL T=ST 6266
6	L3		Individual Mix AB 0.025ug/mL T=ST 6266
7	L2		Individual Mix AB 0.01ug/mL T=ST 6266
8	L1		Individual Mix AB 0.005ug/mL T=ST 6266

%RSD Measured	%RSD Maximum	Compound
9.1	20.0	Tetrachloro-m-xylene (TCX)
4.6	20.0	alpha-BHC
5.2	20.0	gamma-BHC
8.2	20.0	beta-BHC
8.1	20.0	Heptachlor
2.0	20.0	delta-BHC
4.1	20.0	Aldrin
6.8	20.0	Heptachlor Epoxide
6.2	20.0	Endosulfan I
3.9	20.0	4,4'-DDE
4.0	20.0	Dieldrin
4.8	20.0	Endrin
2.5	20.0	4,4'-DDD
6.2	20.0	Endosulfan II
2.8	20.0	4,4'-DDT
5.9	20.0	Endrin Aldehyde
6.5	20.0	Endosulfan Sulfate
5.6	20.0	Dibutylchlorendate (DBC)
6.5	20.0	Methoxychlor
5.7	20.0	Endrin Ketone
0.0	20.0	Octachloronapthalene
10.4	20.0	Decachlorobiphenyl (DCB)

	Samp 4 Meas True %Diff	Samp 5 Meas True %Diff	Samp 6 Meas True %Diff	Samp 7 Meas True %Diff	Samp 8 Meas True %Diff	Meas True %Diff	Meas True %Diff	Meas True %Diff
Tetrachloro-m-xylene (TCX)								
	0.09047	0.05001	0.02626	0.01100	0.004724			
	0.1000	0.05000	0.02500	0.01000	0.005000			
	-9.5	0.0	5.0	10.0	-5.5			
alpha-BHC								
	0.09401	0.05078	0.02592	0.01027	0.004903			
	0.1000	0.05000	0.02500	0.01000	0.005000			
	-6.0	1.6	3.7	2.7	-1.9			
gamma-BHC								
	0.09326	0.05087	0.02603	0.01030	0.004891			
	0.1000	0.05000	0.02500	0.01000	0.005000			
	-6.7	1.7	4.1	3.0	-2.2			
beta-BHC								
	0.09037	0.05055	0.02637	0.01076	0.004775			
	0.1000	0.05000	0.02500	0.01000	0.005000			
	-9.6	1.1	5.5	7.6	-4.5			
Heptachlor								
	0.09050	0.05039	0.02650	0.01070	0.004784			
	1000	0.05000	0.02500	0.01000	0.005000			
	-9.5	0.8	6.0	7.0	-4.3			
delta-BHC								
	0.09727	0.05074	0.02535	0.01003	0.004978			
	0.1000	0.05000	0.02500	0.01000	0.005000			
	-2.7	1.5	1.4	0.3	-0.4			
Aldrin								
	0.09451	0.05089	0.02574	0.01025	0.004913			
	0.1000	0.05000	0.02500	0.01000	0.005000			
	-5.5	1.8	3.0	2.5	-1.7			
Heptachlor Epoxide								
	0.09171	0.05060	0.02623	0.01057	0.004822			
	0.1000	0.05000	0.02500	0.01000	0.005000			
	-8.3	1.2	4.9	5.7	-3.6			
Endosulfan I								
	0.09228	0.05079	0.02603	0.01052	0.004839			
	0.1000	0.05000	0.02500	0.01000	0.005000			
	-7.7	1.6	4.1	5.2	-3.2			
4,4'-DDE								
	0.09489	0.05080	0.02564	0.01027	0.004911			
	1000	0.05000	0.02500	0.01000	0.005000			
	-5.1	1.6	2.6	2.7	-1.8			

	Samp 4 Meas True %Diff	Samp 5 Meas True %Diff	Samp 6 Meas True %Diff	Samp 7 Meas True %Diff	Samp 8 Meas True %Diff	Meas True %Diff	Meas True %Diff	Meas True %Diff
Dieldrin								
	0.09466	0.05091	0.02564	0.01027	0.004910			
	0.1000	0.05000	0.02500	0.01000	0.005000			
	-5.3	1.8	2.6	2.7	-1.8			
Endrin								
	0.09390	0.05076	0.02584	0.01035	0.004886			
	0.1000	0.05000	0.02500	0.01000	0.005000			
	-6.1	1.5	3.3	3.5	-2.3			
4,4'-DDD								
	0.09670	0.05099	0.02521	0.01013	0.004957			
	0.1000	0.05000	0.02500	0.01000	0.005000			
	-3.3	2.0	0.9	1.3	-0.9			
Endosulfan II								
	0.09246	0.05064	0.02601	0.01055	0.004834			
	0.1000	0.05000	0.02500	0.01000	0.005000			
	-7.5	1.3	4.1	5.5	-3.3			
4,4'-DDT								
	^ 09626	0.05108	0.02525	0.01016	0.004948			
	1000	0.05000	0.02500	0.01000	0.005000			
	-3.7	2.2	1.0	1.6	-1.0			
Endrin Aldehyde								
	0.09336	0.05023	0.02594	0.01058	0.004831			
	0.1000	0.05000	0.02500	0.01000	0.005000			
	-6.6	0.5	3.7	5.8	-3.4			
Endosulfan Sulfate								
	0.09238	0.05051	0.02603	0.01060	0.004822			
	0.1000	0.05000	0.02500	0.01000	0.005000			
	-7.6	1.0	4.1	6.0	-3.6			
Dibutylchlorendate (DBC)								
	0.09322	0.05049	0.02597	0.01049	0.004851			
	0.1000	0.05000	0.02500	0.01000	0.005000			
	-6.8	1.0	3.9	4.9	-3.0			
Methoxychlor								
	0.09230	0.05050	0.02608	0.01059	0.004823			
	0.1000	0.05000	0.02500	0.01000	0.005000			
	-7.7	1.0	4.3	5.9	-3.5			
Endrin Ketone								
	0.09288	0.05077	0.02594	0.01048	0.004853			
	1000	0.05000	0.02500	0.01000	0.005000			
	-7.1	1.5	3.7	4.8	-2.9			

	Samp 4 Meas True %Diff	Samp 5 Meas True %Diff	Samp 6 Meas True %Diff	Samp 7 Meas True %Diff	Samp 8 Meas True %Diff	Meas True %Diff	Meas True %Diff	Meas True %Diff
Dieldrin								
	0.09233	0.05069	0.02604	0.01055	0.004834			
	0.1000	0.05000	0.02500	0.01000	0.005000			
	-7.7	1.4	4.1	5.5	-3.3			
Endrin								
	0.09411	0.05170	0.02593	0.009914	0.004982			
	0.1000	0.05000	0.02500	0.01000	0.005000			
	-5.9	3.4	3.7	-0.9	-0.4			
Endosulfan II								
	0.09120	0.05099	0.02600	0.01067	0.004804			
	0.1000	0.05000	0.02500	0.01000	0.005000			
	-8.8	2.0	4.0	6.7	-3.9			
4,4'-DDD								
	0.09381	0.05159	0.02544	0.01032	0.004902			
	0.1000	0.05000	0.02500	0.01000	0.005000			
	-6.2	3.2	1.8	3.2	-2.0			
Endrin Aldehyde								
	^ 09163	0.05071	0.02607	0.01065	0.004808			
	1000	0.05000	0.02500	0.01000	0.005000			
	-8.4	1.4	4.3	6.5	-3.8			
4,4'-DDT								
	0.1868	0.1011	0.05169	0.02102	0.009700			
	0.2000	0.1000	0.05000	0.02000	0.01000			
	-6.6	1.1	3.4	5.1	-3.0			
Endosulfan Sulfate								
	0.1868	0.1011	0.05169	0.02102	0.009700			
	0.2000	0.1000	0.05000	0.02000	0.01000			
	-6.6	1.1	3.4	5.1	-3.0			
Endrin Ketone								
	0.09200	0.05036	0.02616	0.01065	0.004808			
	0.1000	0.05000	0.02500	0.01000	0.005000			
	-8.0	0.7	4.6	6.5	-3.8			
Methoxychlor								
	0.09105	0.05028	0.02633	0.01075	0.004779			
	0.1000	0.05000	0.02500	0.01000	0.005000			
	-8.9	0.6	5.3	7.5	-4.4			
Dibutylchlorendate (DBC)								
	0.09254	0.05029	0.02606	0.01063	0.004815			
	1000	0.05000	0.02500	0.01000	0.005000			
	-7.5	0.6	4.2	6.3	-3.7			

Samp 4 Meas True %Diff	Samp 5 Meas True %Diff	Samp 6 Meas True %Diff	Samp 7 Meas True %Diff	Samp 8 Meas True %Diff	Meas True %Diff	Meas True %Diff	Meas True %Diff
Octachloronapthalene							
	0.02500						
	0.02500						
Decachlorobiphenyl (DCB)							
0.08824	0.05018	0.02684	0.01099	0.004707			
0.1000	0.05000	0.02500	0.01000	0.005000			
-11.8	0.4	7.4	9.9	-5.9			

Wed May 20 10:16:41 1992 Page 1

Reduced by: Mr Date: 5-20-92 Initial Calibration Report
Reviewed by: _____ Date: _____

Project: OCP05_3, Channel: 58, Analysis: C18MAY92, Node: DENCR3
Analyst: MGOETZ, Calibration File: C0518, MCRPT Method: OCPA

Calibration curve type: Linear NOT forced through zero

Sample	Level ID	Description
4	L5	Individual Mix AB 0.10ug/mL T=ST 6266
5	L4	Individual Mix AB 0.05ug/mL T=ST 6266
6	L3	Individual Mix AB 0.025ug/mL T=ST 6266
7	L2	Individual Mix AB 0.01ug/mL T=ST 6266
8	L1	Individual Mix AB 0.005ug/mL T=ST 6266

%RSD Measured	%RSD Maximum	Compound
8.7	20.0	Tetrachloro-m-xylene (TCX)
5.8	20.0	alpha-BHC
8.4	20.0	beta-BHC
6.6	20.0	gamma-BHC
4.8	20.0	delta-BHC
6.4	20.0	Heptachlor
5.6	20.0	Aldrin
7.3	20.0	Heptachlor Epoxide
[illegible].7	20.0	Endosulfan I
5.1	20.0	4,4'-DDE
6.3	20.0	Dieldrin
4.5	20.0	Endrin
7.3	20.0	Endosulfan II
4.7	20.0	4,4'-DDD
7.0	20.0	Endrin Aldehyde
5.5	20.0	4,4'-DDT
5.5	20.0	Endosulfan Sulfate
6.9	20.0	Endrin Ketone
7.8	20.0	Methoxychlor
6.5	20.0	Dibutylchlorendate (DBC)
2.0	20.0	Octachloronapthalene
9.6	20.0	Decachlorobiphenyl (DCB)

	Samp 4 Meas True %Diff	Samp 5 Meas True %Diff	Samp 6 Meas True %Diff	Samp 7 Meas True %Diff	Samp 8 Meas True %Diff	Meas True %Diff	Meas True %Diff	Meas True %Diff
Tetrachloro-m-xylene (TCX)								
	0.09039	0.05005	0.02648	0.01086	0.004749			
	0.1000	0.05000	0.02500	0.01000	0.005000			
	-9.6	0.1	5.9	8.6	-5.0			
alpha-BHC								
	0.09295	0.05049	0.02604	0.01050	0.004847			
	0.1000	0.05000	0.02500	0.01000	0.005000			
	-7.0	1.0	4.2	5.0	-3.1			
beta-BHC								
	0.09043	0.05016	0.02651	0.01079	0.004764			
	0.1000	0.05000	0.02500	0.01000	0.005000			
	-9.6	0.3	6.0	7.9	-4.7			
gamma-BHC								
	0.09234	0.05033	0.02615	0.01060	0.004820			
	0.1000	0.05000	0.02500	0.01000	0.005000			
	-7.7	0.7	4.6	6.0	-3.6			
delta-BHC								
	0.09384	0.05064	0.02594	0.01034	0.004886			
	1000	0.05000	0.02500	0.01000	0.005000			
	-6.2	1.3	3.7	3.4	-2.3			
Heptachlor								
	0.09226	0.05033	0.02632	0.01051	0.004836			
	0.1000	0.05000	0.02500	0.01000	0.005000			
	-7.7	0.7	5.3	5.1	-3.3			
Aldrin								
	0.09310	0.05058	0.02601	0.01046	0.004857			
	0.1000	0.05000	0.02500	0.01000	0.005000			
	-6.9	1.2	4.0	4.6	-2.9			
Heptachlor Epoxide								
	0.09143	0.05032	0.02634	0.01066	0.004800			
	0.1000	0.05000	0.02500	0.01000	0.005000			
	-8.6	0.6	5.3	6.6	-4.0			
Endosulfan I								
	0.09099	0.05022	0.02656	0.01064	0.004799			
	0.1000	0.05000	0.02500	0.01000	0.005000			
	-9.0	0.4	6.2	6.4	-4.0			
4,4'-DDE								
	0.09382	0.05054	0.02582	0.01045	0.004864			
	1000	0.05000	0.02500	0.01000	0.005000			
	-6.2	1.1	3.3	4.5	-2.7			

Samp 4 Meas True %Diff	Samp 5 Meas True %Diff	Samp 6 Meas True %Diff	Samp 7 Meas True %Diff	Samp 8 Meas True %Diff	Meas True %Diff	Meas True %Diff	Meas True %Diff
Octachloronapthalene							
0.02557	0.02454	0.02483	0.02473	0.02533			
0.02500	0.02500	0.02500	0.02500	0.02500			
2.3	-1.8	-0.7	-1.1	1.3			
Decachlorobiphenyl (DCB)							
0.08937	0.05009	0.02664	0.01094	0.004725			
0.1000	0.05000	0.02500	0.01000	0.005000			
-10.6	0.2	6.6	9.4	-5.5			

Injection Report

Acquired on 18-MAY-1992 at 22:47

RMAL Chromatography System on DENCR3

Analyst Name : MGOETZ
Lims Id : 2uL injected
Comment : C-5890,DUAL ECD Rtx-35,0.53,1.00um, Rtx-5, 0.53,1.5um
Method Title :
Sample Name : Individual Mix AB 0.10ug/mL T=ST 6266
Sample Id : Ver #6266
Sample Type : Standard Amount=1.00000
Bottle No : 4

MG 5-20-92

PEAK INFORMATION

Peak	GRP	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
8		8.684	8.684	613240	1905705	0.09047	Tetrachloro-m-xylene (TCX)	3.2
16		10.382	10.382	830491	3089166	0.09401	alpha-BHC	3.7
18		11.564	11.564	708293	2934263	0.09326	gamma-BHC	4.3
19		11.778	11.778	342842	1418176	0.09037	beta-BHC	4.0
23		12.951	12.951	540109	2575855	0.09050	Heptachlor	5.3A
24		13.040	13.040	623140	2911044	0.09727	delta-BHC	4.3A
27		14.196	14.196	512100	2745175	0.09451	Aldrin	5.3
30		16.400	16.400	441598	2548017	0.09171	Heptachlor Epoxide	5.9
35		17.982	17.982	402846	2447469	0.09228	Endosulfan I	6.1
36		18.969	18.969	440065	2451121	0.09489	4,4'-DDE	5.3
37		19.329	19.329	406076	2456270	0.09466	Dieldrin	6.1
40		20.902	20.902	358639	2243752	0.09390	Endrin	6.4
42		21.560	21.560	330017	1916298	0.09670	4,4'-DDD	5.9
43		21.782	21.787	348365	2227929	0.09246	Endosulfan II	6.1
46		23.080	23.080	319925	1920985	0.09626	4,4'-DDT	6.1
47		23.249	23.249	256239	1661321	0.09336	Endrin Aldehyde	6.4
48		24.067	24.067	306793	2056558	0.09238	Endosulfan Sulfate	6.4
51		26.196	26.196	251219	1688102	0.09322	Dibutylchlorendate (DBC)	6.7
52		27.120	27.124	166876	1083984	0.09230	Methoxychlor	6.4
53		27.507	27.511	310666	2069253	0.09288	Endrin Ketone	6.7
60		33.467	33.467	113109	831877	0.02551	Octachloronapthalene	7.2
61		33.724	33.729	236585	1852081	0.08824	Decachlorobiphenyl (DCB)	7.7

Totals			
Unknowns	426479	1945643	N/A
Quantified	8859233	47034396	1.98115
Grand Total	9285712	48980040	1.98115

MISSING PEAKS

No missing peaks.

PEAK GROUP INFORMATION

No peak group information.

ANALYSIS SUMMARY

Method..... COCP
Run sequence..... C0518
Calibration..... C0518
External standard calibration using area
Calibration last modified on 20-MAY-1992 at 14:02

Uncalibrated peaks use user factor (0.0000)

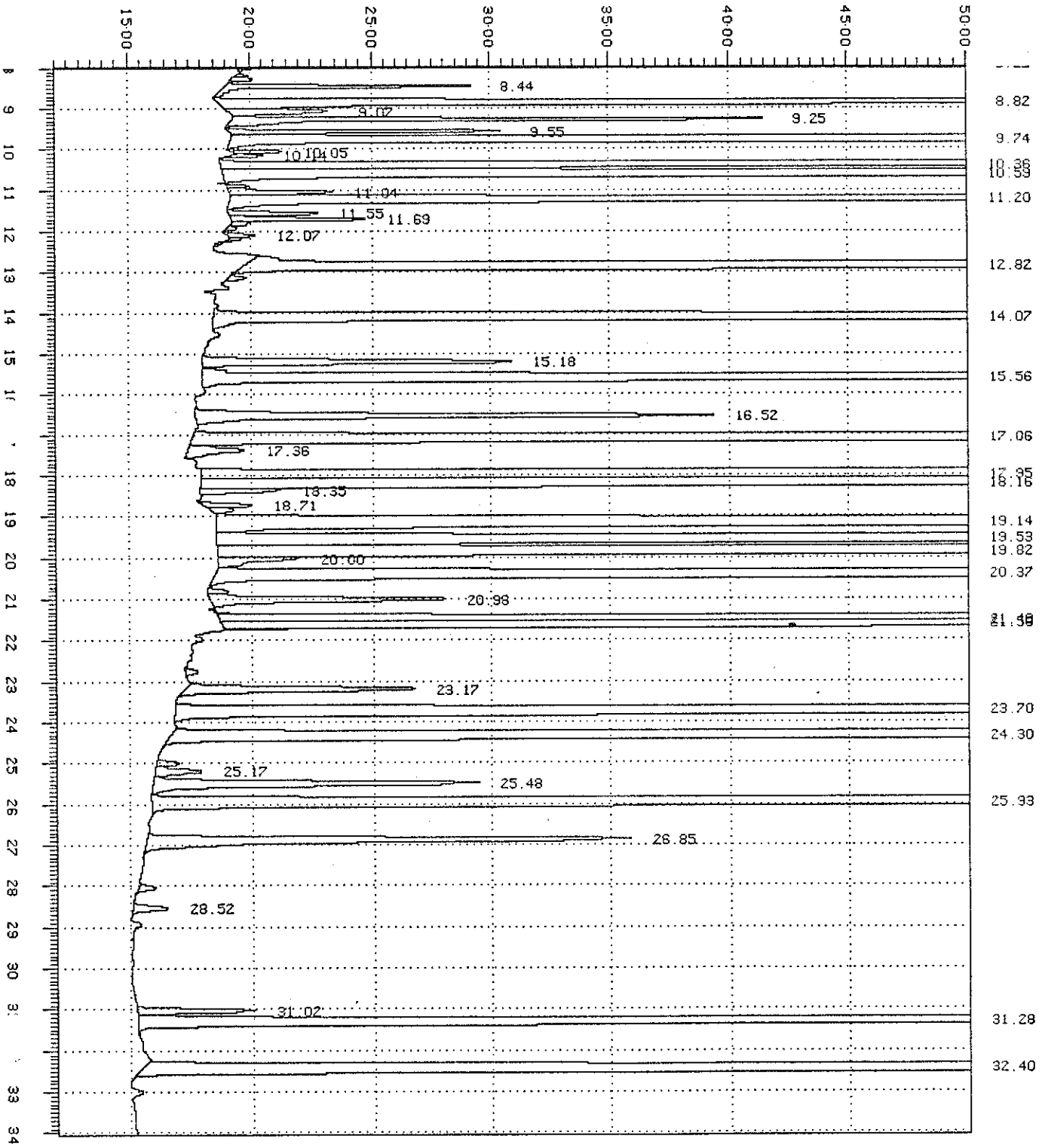
RMAL Chromatography System on DENCR3

[OCP05_3] 58 C18MAY92,4,1

Individual Mix AB 0.10ug/mL T=ST 6266. Amount : 1.000.

Acquired on 18-MAY-1992 at 22:47
Reported on 20-MAY-1992 at 12:12
Box 1 (of 1)

MB 5-20-92

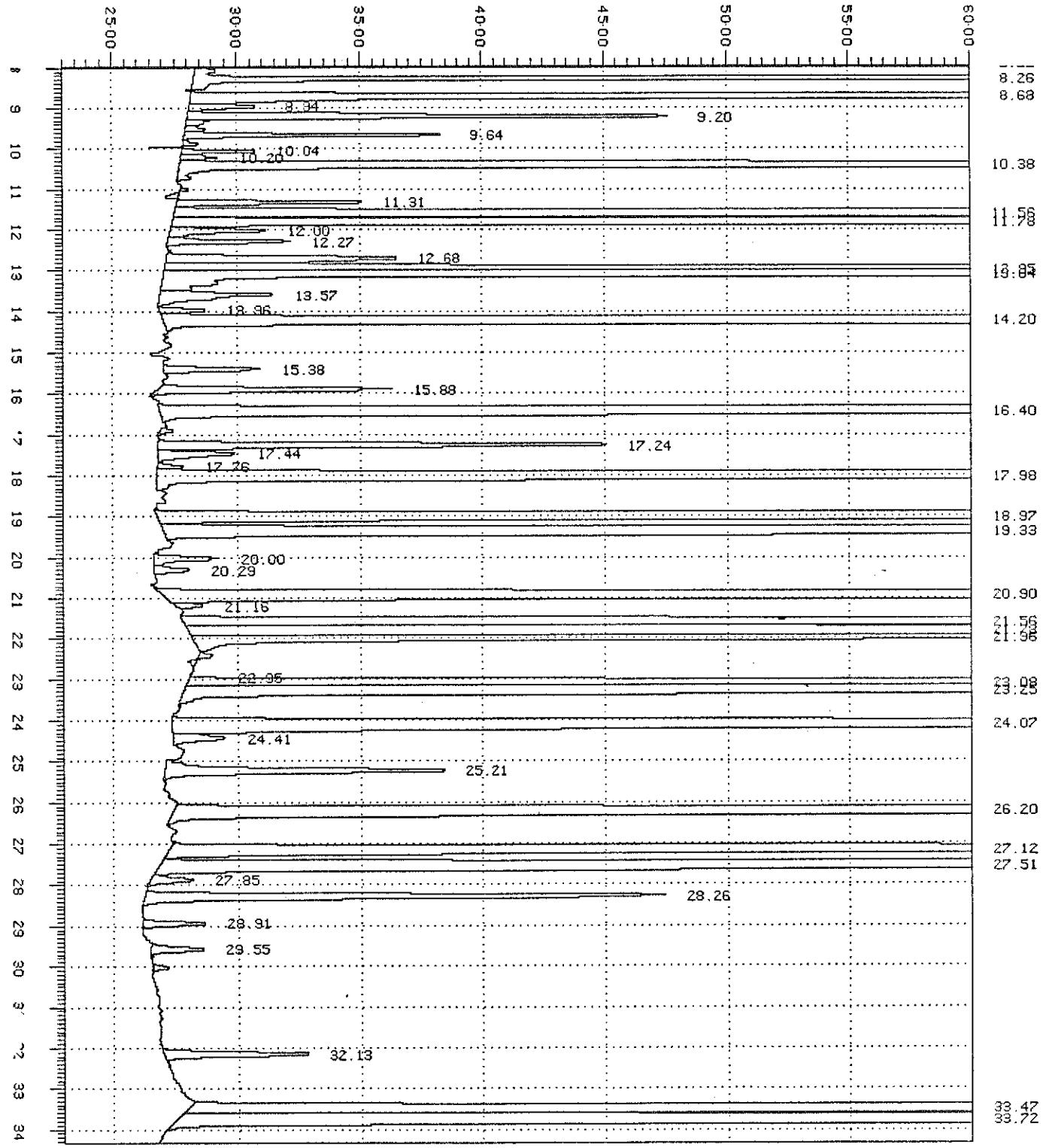


RMAL Chromatography System on DENCR3

[OCP05_3] 57 C18MAY92,4,1

Individual Mix AB 0.10ug/mL T=ST 6266. Amount : 1.000.
C_5890,Dual ECD,Column: RTx-35,0.53mm,0.53um,30m
Acquired on 18-MAY-1992 at 22:47
Reported on 20-MAY-1992 at 12:06
Box 1 (of 1)

MW 5-20-92



Injection Report

Acquired on 18-MAY-1992 at 22:47

RMAL Chromatography System on DENCR3

Analyst Name : MGOETZ
Lims Id : 2uL injected
Comment : C-5890,DUAL ECD Rtx-35,0.53,1.00um, Rtx-5, 0.53,1.5um
Method Title :
Sample Name : Individual Mix AB 0.10ug/mL T=ST 6266
Sample Id : Ver #6266
Sample Type : Standard Amount=1.00000
Bottle No : 4

MG 5-20-92

PEAK INFORMATION

Peak	GRP	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
7		8.822	8.827	598861	1775461	0.09039	Tetrachloro-m-xylene (TCX)	2.9
11		9.738	9.738	910876	2986549	0.09295	alpha-BHC	3.2
14		10.360	10.364	369852	1331282	0.09043	beta-BHC	3.5
15		10.587	10.587	780192	2831592	0.09234	gamma-BHC	3.7
18		11.200	11.204	710933	2688614	0.09384	delta-BHC	3.7
24		12.819	12.822	598222	2788530	0.09226	Heptachlor	4.5
26		14.071	14.071	563610	2740091	0.09310	Aldrin	4.8
28		15.560	15.564	476683	2527183	0.09143	Heptachlor Epoxide	5.3
30		17.062	17.067	438823	2472665	0.09099	Endosulfan I	5.6
32		17.951	17.951	458165	2489581	0.09382	4,4'-DDE	5.3
33		18.164	18.169	448097	2534477	0.09233	Dieldrin	5.6
37		19.138	19.138	413085	2643046	0.09411	Endrin	5.9
38		19.529	19.533	369553	2207144	0.09120	Endosulfan II	5.9
39		19.818	19.827	350379	1995986	0.09381	4,4'-DDD	5.6
41		20.373	20.373	284753	1750003	0.09163	Endrin Aldehyde	6.1
C1			21.476	722538	4086879	0.18685	4,4'-DDT/Endosulfan Sulfate	
44		21.476	21.476	349264	1882361	0.08430	- C1	6.4A
45		21.556	21.476	373274	2204518	0.09929	- C1	6.4A
48		23.698	23.702	337058	2169725	0.09200	Endrin Ketone	6.4
49		24.302	24.307	187418	1192824	0.09105	Methoxychlor	6.4
53		25.929	25.933	251191	1654225	0.09254	Dibutylchlorendate (DBC)	6.4
58		31.276	31.280	122178	751596	0.02557	Octachloronapthalene	5.9
59		32.404	32.409	196842	1353746	0.08937	Decachlorobiphenyl (DCB)	6.7

Totals			
Unknowns	330432	1533625	N/A
Quantified	9589310	46971200	1.96203
Grand Total	9919742	48504824	1.96203

MISSING PEAKS

No missing peaks.

PEAK GROUP INFORMATION

No peak group information.

ANALYSIS SUMMARY

Method..... COCP
Run sequence..... C0518
Calibration..... C0518
External standard calibration using area
Calibration last modified on 19-MAY-1992 at 13:25

Uncalibrated peaks use user factor (0.0000)

Injection Report

Acquired on 18-MAY-1992 at 23:34

RMAL Chromatography System on DENCR3

Analyst Name : MGOETZ
Lims Id : 2uL injected
Comment : C-5890,DUAL ECD Rtx-35,0.53,1.00um, Rtx-5, 0.53,1.5um
Method Title :
Sample Name : Individual Mix AB 0.05ug/mL T=ST 6266
Sample Id : Ver #6266
Sample Type : Standard Amount=1.00000
Bottle No : 5

MG 5-20-92

PEAK INFORMATION

Peak	GRP	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
9		8.689	8.684	345031	1074835	0.05001	Tetrachloro-m-xylene (TCX)	3.2
15		10.387	10.382	460285	1674345	0.05078	alpha-BHC	3.5
17		11.573	11.564	393050	1611298	0.05087	gamma-BHC	4.0
18		11.787	11.778	195252	806096	0.05055	beta-BHC	4.0
22		12.956	12.951	306239	1467364	0.05039	Heptachlor	4.8A
23		13.044	13.040	340177	1520565	0.05074	delta-BHC	4.8A
26		14.204	14.196	282365	1491674	0.05089	Aldrin	5.3
29		16.404	16.400	251175	1428215	0.05060	Heptachlor Epoxide	5.6
32		17.991	17.982	226408	1366939	0.05079	Endosulfan I	5.9
33		18.973	18.969	238099	1323696	0.05080	4,4'-DDE	5.6
34		19.338	19.329	222183	1334502	0.05091	Dieldrin	5.9
37		20.911	20.902	198011	1227352	0.05076	Endrin	6.1
39		21.569	21.560	176732	1015597	0.05099	4,4'-DDD	5.6
40		21.791	21.787	194928	1239298	0.05064	Endosulfan II	6.1
42		23.089	23.080	170882	1026270	0.05108	4,4'-DDT	5.9
43		23.253	23.249	142836	907091	0.05023	Endrin Aldehyde	6.4
44		24.076	24.067	169515	1139762	0.05051	Endosulfan Sulfate	6.4
48		26.200	26.196	137162	929746	0.05049	Dibutylchlorendate (DBC)	6.7
49		27.129	27.124	93266	602084	0.05050	Methoxychlor	6.4
50		27.516	27.511	171577	1145513	0.05077	Endrin Ketone	6.4
56		33.471	33.467	110624	815166	0.02500	Octachloronapthalene	7.2
57		33.733	33.729	138854	1079615	0.05018	Decachlorobiphenyl (DCB)	7.5

Totals			
Unknowns	313473	1482178	N/A
Quantified	4964651	26227020	1.08848
Grand Total	5278124	27709198	1.08848

MISSING PEAKS

No missing peaks.

PEAK GROUP INFORMATION

No peak group information.

ANALYSIS SUMMARY

Method..... COCP
Run sequence..... C0518
Calibration..... C0518
External standard calibration using area
Calibration last modified on 20-MAY-1992 at 14:02

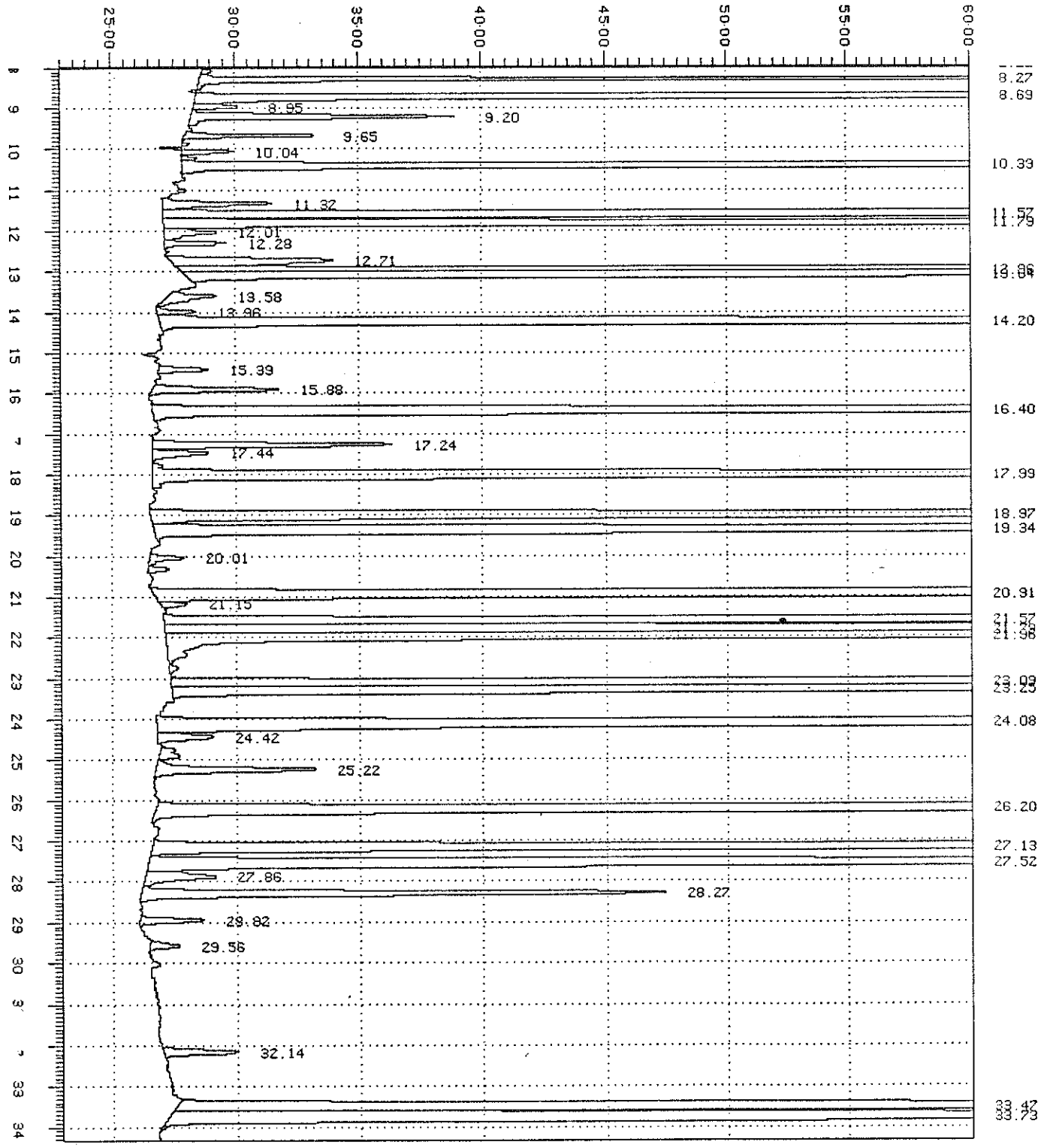
Uncalibrated peaks use user factor (0.0000)

RMAL Chromatography System on DENCR3

[OCP05_3] 57 C18MAY92,5,1

Individual Mix AB 0.05ug/mL T=ST 6266. Amount : 1.000.
C_5890,Dual ECD,Column: RTx-35,0.53mm,0.53um,30m
Acquired on 18-MAY-1992 at 23:34
Reported on 20-MAY-1992 at 12:06
Box 1 (of 1)

MB 5-20-92



Injection Report

Acquired on 18-MAY-1992 at 23:34

RMAL Chromatography System on DENCR3

Analyst Name : MGOETZ
Lims Id : 2uL injected
Comment : C-5890,DUAL ECD Rtx-35,0.53,1.00um, Rtx-5, 0.53,1.5um
Method Title :
Sample Name : Individual Mix AB 0.05ug/mL T=ST 6266
Sample Id : Ver #6266
Sample Type : Standard Amount=1.00000
Bottle No : 5

MG 5-20-92

PEAK INFORMATION

Peak	GRP	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
7		8.831	8.827	332616	1000672	0.05005	Tetrachloro-m-xylene (TCX)	3.2
11		9.742	9.738	498622	1639665	0.05049	alpha-BHC	3.5
14		10.369	10.364	206968	754707	0.05016	beta-BHC	3.5
15		10.591	10.587	429830	1564254	0.05033	gamma-BHC	3.5
18		11.209	11.204	386394	1462418	0.05064	delta-BHC	3.7
22		12.826	12.822	339542	1551819	0.05033	Heptachlor	4.5
23		14.076	14.071	311634	1508106	0.05058	Aldrin	4.8
25		15.564	15.564	271284	1418329	0.05032	Heptachlor Epoxide	5.1
27		17.067	17.067	248211	1390889	0.05022	Endosulfan I	5.6
29		17.956	17.951	250452	1355454	0.05054	4,4'-DDE	5.3
30		18.169	18.169	248593	1409590	0.05069	Dieldrin	5.6
33		19.142	19.138	231743	1619376	0.05170	Endrin	6.1
34		19.533	19.533	208185	1255993	0.05099	Endosulfan II	5.9
35		19.827	19.827	190266	1106419	0.05159	4,4'-DDD	5.6
36		20.378	20.373	159895	985219	0.05071	Endrin Aldehyde	5.9
C1			21.476	394940	2243606	0.10111	4,4'-DDT/Endosulfan Sulfate	
39		21.484	21.476	192952	1078220	0.04690	- C1	6.9A
40		21.560	21.476	201988	1165387	0.05095	- C1	6.4A
42		23.707	23.702	190829	1209187	0.05036	Endrin Ketone	6.4
43		24.307	24.307	106189	670909	0.05028	Methoxychlor	6.1
46		25.938	25.933	138056	916655	0.05029	Dibutylchlorendate (DBC)	6.4
51		31.280	31.280	118792	734268	0.02454	Octachloronapthalene	6.1
52		32.413	32.409	112684	778358	0.05009	Decachlorobiphenyl (DCB)	6.7

Totals			
Unknowns	233462	1106295	N/A
Quantified	5375726	26575892	1.08601
Grand Total	5609188	27682188	1.08601

MISSING PEAKS

No missing peaks.

PEAK GROUP INFORMATION

No peak group information.

ANALYSIS SUMMARY

Method..... COCP
Run sequence..... C0518
Calibration..... C0518
External standard calibration using area
Calibration last modified on 19-MAY-1992 at 13:25

Uncalibrated peaks use user factor (0.0000)

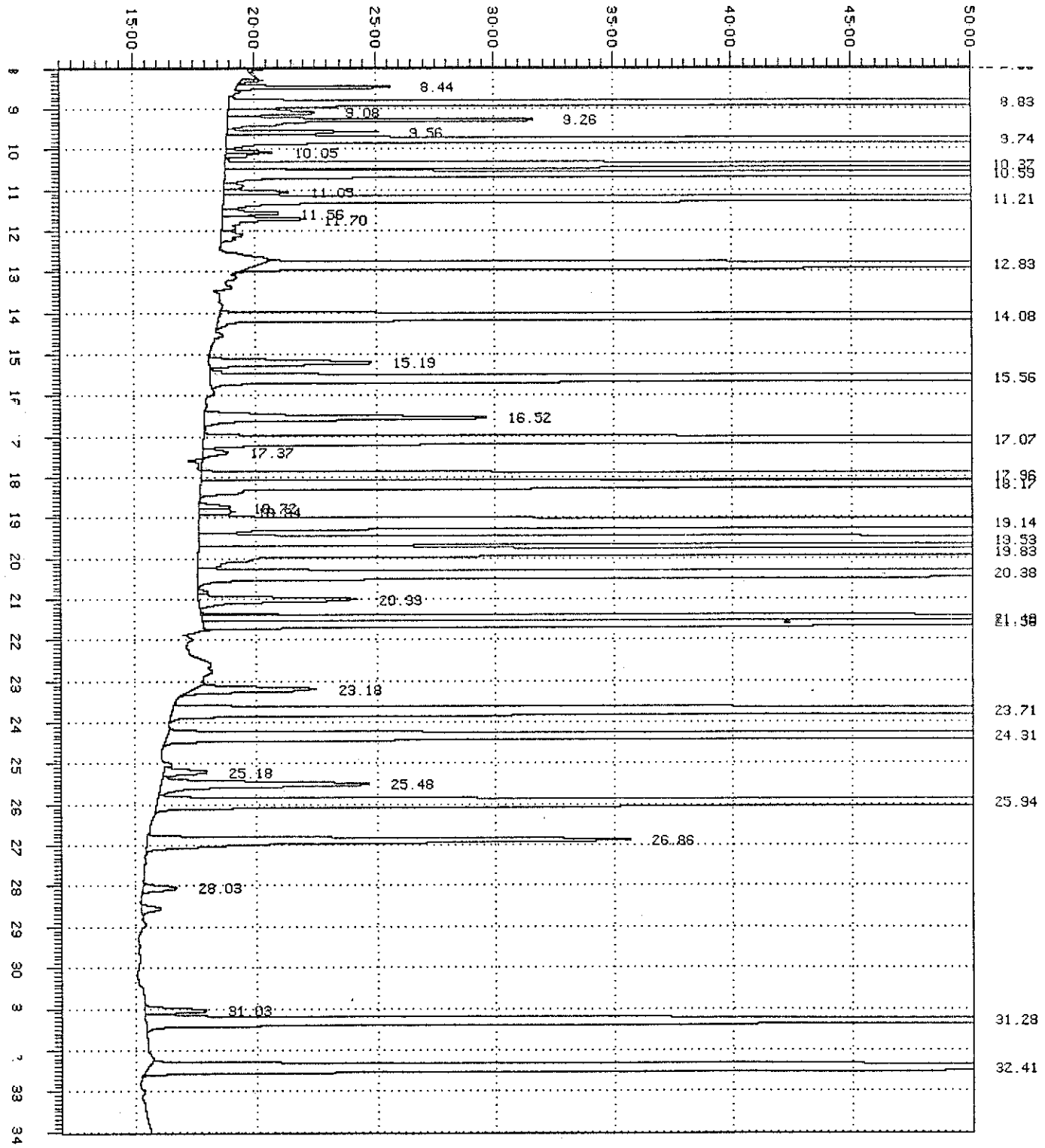
RMAL Chromatography System on DENCR3

[OCP05_3] 58 C18MAY92,5,1

Individual Mix AB 0.05ug/mL T=ST 6266. Amount : 1.000.

Acquired on 18-MAY-1992 at 23:34
Reported on 20-MAY-1992 at 12:12
Box 1 (of 1)

mr 5-20-92



Injection Report

Acquired on 19-MAY-1992 at 00:22

RMAL Chromatography System on DENCR3

Analyst Name : MGOETZ
Lims Id : 2uL injected
Comment : C-5890,DUAL ECD Rtx-35,0.53,1.00um, Rtx-5, 0.53,1.5um
Method Title :
Sample Name : Individual Mix AB 0.025ug/mL T=ST 6266
Sample Id : Ver #6266
Sample Type : Standard Amount=1.00000
Bottle No : 6

MG 5-20-92

PEAK INFORMATION

Peak	GRP	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
8		8.684	8.684	187370	587152	0.02626	Tetrachloro-m-xylene (TCX)	3.2
14		10.382	10.382	239254	860975	0.02592	alpha-BHC	3.7
17		11.564	11.564	208959	835945	0.02603	gamma-BHC	4.0
18		11.778	11.778	105509	434515	0.02637	beta-BHC	4.0
22		12.951	12.951	165485	807314	0.02650	Heptachlor	5.3A
23		13.040	13.040	173205	761696	0.02535	delta-BHC	4.3A
26		14.196	14.196	147879	769001	0.02574	Aldrin	5.1
29		16.400	16.400	134288	764474	0.02623	Heptachlor Epoxide	5.6
32		17.982	17.982	121981	721927	0.02603	Endosulfan I	5.6
33		18.969	18.969	124328	680386	0.02564	4,4'-DDE	5.3
34		19.329	19.329	115723	686848	0.02564	Dieldrin	5.9
36		20.902	20.902	103582	640329	0.02584	Endrin	5.9
38		21.560	21.560	88312	507576	0.02521	4,4'-DDD	5.6
39		21.787	21.787	104101	657062	0.02601	Endosulfan II	6.1
41		23.080	23.080	86611	514669	0.02525	4,4'-DDT	5.9
42		23.249	23.249	74580	482256	0.02594	Endrin Aldehyde	6.4
43		24.067	24.067	90316	603919	0.02603	Endosulfan Sulfate	6.4
48		26.196	26.196	73184	494448	0.02597	Dibutylchlorendate (DBC)	6.4
49		27.124	27.124	49849	320414	0.02608	Methoxychlor	6.4
50		27.511	27.511	92328	600858	0.02594	Endrin Ketone	6.4
55		33.467	33.467	112393	821399	0.02519	Octachloronapthalene	7.2
56		33.729	33.729	78518	606004	0.02684	Decachlorobiphenyl (DCB)	7.5

Totals			
Unknowns	271861	1327149	N/A
Quantified	2677755	14159166	0.57002
Grand Total	2949616	15486315	0.57002

MISSING PEAKS

No missing peaks.

PEAK GROUP INFORMATION

No peak group information.

ANALYSIS SUMMARY

Method..... COCP
Run sequence..... C0518
Calibration..... C0518
External standard calibration using area
Calibration last modified on 20-MAY-1992 at 14:02

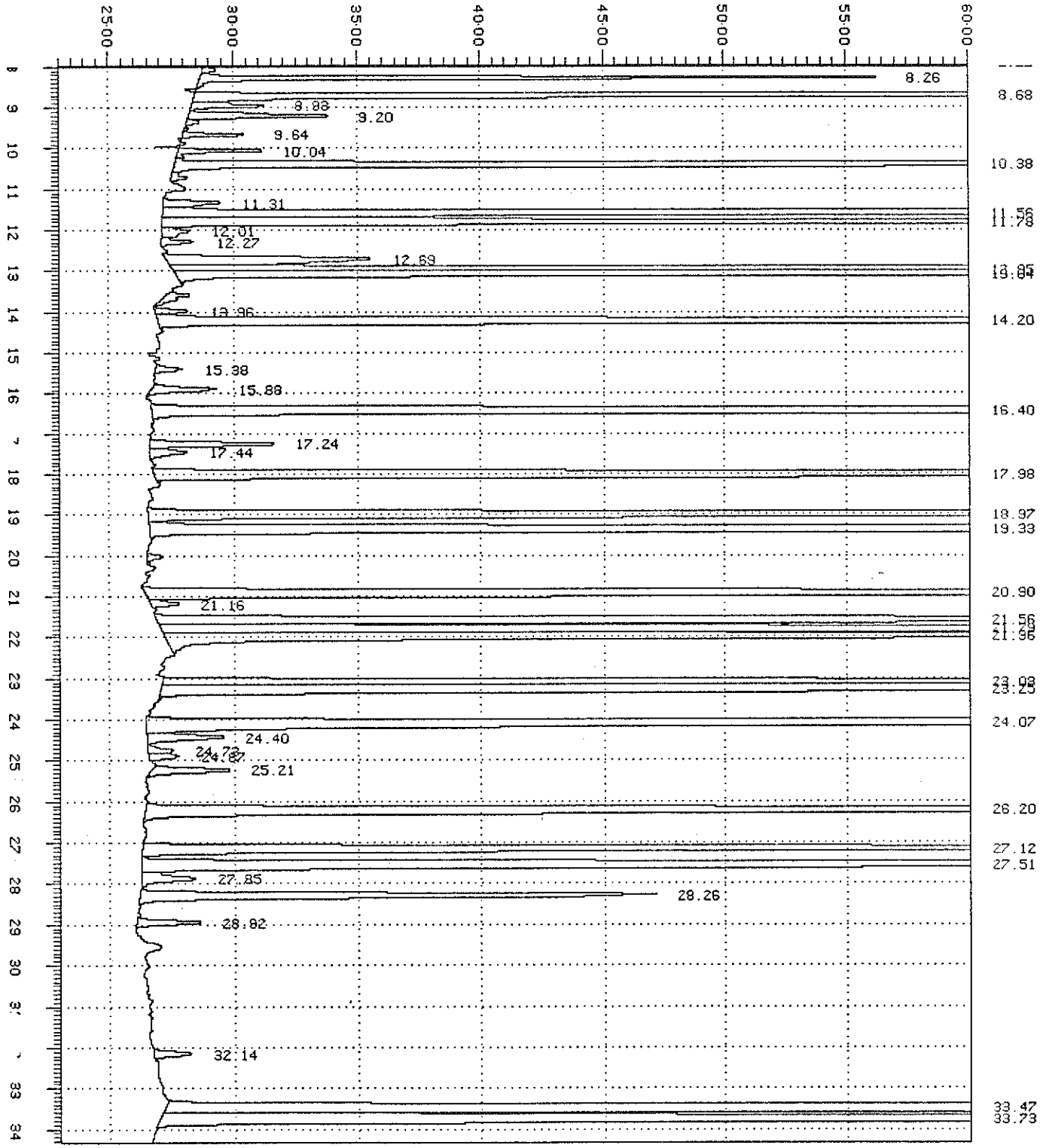
Uncalibrated peaks use user factor (0.0000)

RMAL Chromatography System on DENCR3

[OCP05_3] 57 C18MAY92,6,1

Individual Mix AB 0.025ug/mL T=ST 6266. Amount : 1.000.
C_5890,Dual ECD,Column: RTx-35,0.53mm,0.53um,30m
Acquired on 19-MAY-1992 at 00:22
Reported on 20-MAY-1992 at 12:07
Box 1 (of 1)

MB 5-20-92



Injection Report

Acquired on 19-MAY-1992 at 00:22

RMAL Chromatography System on DENCR3

Analyst Name : MGOETZ
Lims Id : 2uL injected
Comment : C-5890,DUAL ECD Rtx-35,0.53,1.00um, Rtx-5, 0.53,1.5um
Method Title :
Sample Name : Individual Mix AB 0.025ug/mL T=ST 6266
Sample Id : Ver #6266
Sample Type : Standard Amount=1.00000
Bottle No : 6

M 5-20-92

PEAK INFORMATION

Peak	GRP	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
7		8.827	8.827	179687	547931	0.02648	Tetrachloro-m-xylene (TCX)	2.9
12		9.738	9.738	262708	864227	0.02604	alpha-BHC	3.5
14		10.364	10.364	112410	416097	0.02651	beta-BHC	3.7
15		10.587	10.587	227881	834601	0.02615	gamma-BHC	3.5
17		11.204	11.204	200514	761188	0.02594	delta-BHC	3.7
20		12.822	12.822	185972	843479	0.02632	Heptachlor	4.5
21		14.071	14.071	167284	796468	0.02601	Aldrin	4.8
23		15.564	15.564	147017	771453	0.02634	Heptachlor Epoxide	5.3
25		17.067	17.067	134955	763194	0.02656	Endosulfan I	5.6
26		17.951	17.951	131328	707636	0.02582	4,4'-DDE	5.3
27		18.169	18.169	131568	743640	0.02604	Dieldrin	5.3
29		19.138	19.138	126948	997320	0.02593	Endrin	7.7
30		19.533	19.533	113122	664829	0.02600	Endosulfan II	5.9
31		19.827	19.827	98100	555445	0.02544	4,4'-DDD	5.6
32		20.373	20.373	86407	524652	0.02607	Endrin Aldehyde	5.9
C1			21.476	209624	1181304	0.05169	4,4'-DDT/Endosulfan Sulfate	
34		21.484	21.476	104807	587629	0.02408	- C1	6.9A
35		21.556	21.476	104817	593675	0.02436	- C1	6.9A
37		23.702	23.702	102896	650942	0.02616	Endrin Ketone	6.1
38		24.307	24.307	57987	364238	0.02633	Methoxychlor	6.1
41		25.933	25.933	74848	493732	0.02606	Dibutylchlorendate (DBC)	6.7
45		31.280	31.280	118814	739054	0.02483	Octachloronapthalene	5.9
46		32.409	32.409	62911	434943	0.02664	Decachlorobiphenyl (DCB)	6.7

Totals			
Unknowns	205477	880144	N/A
Quantified	2932981	14656372	0.57334
Grand Total	3138459	15536516	0.57334

MISSING PEAKS

No missing peaks.

PEAK GROUP INFORMATION

No peak group information.

ANALYSIS SUMMARY

Method..... COCP
Run sequence..... C0518
Calibration..... C0518
External standard calibration using area
Calibration last modified on 19-MAY-1992 at 13:25

Uncalibrated peaks use user factor (0.0000)

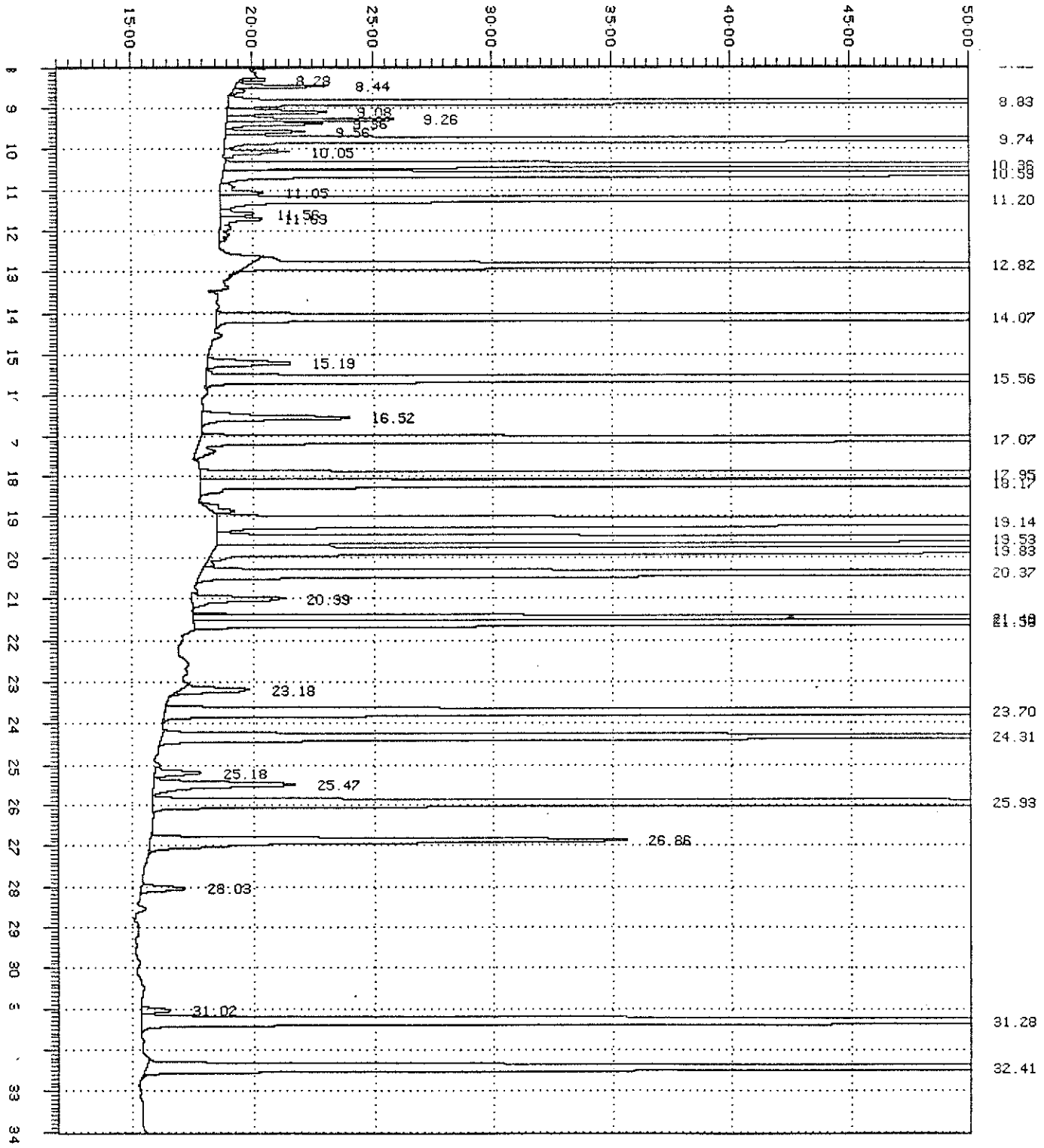
RMAL Chromatography System on DENCR3

[OCP05_3] 58 C18MAY92,6,1

Individual Mix AB 0.025ug/mL T=ST 6266. Amount : 1.000.

Acquired on 19-MAY-1992 at 00:22
Reported on 20-MAY-1992 at 12:12
Box 1 (of 1)

m 5-20-92



Injection Report

Acquired on 19-MAY-1992 at 01:09

RMAL Chromatography System on DENCR3

Analyst Name : MGOETZ
Lims Id : 2uL injected
Comment : C-5890,DUAL ECD Rtx-35,0.53,1.00um, Rtx-5, 0.53,1.5um
Method Title :
Sample Name : Individual Mix AB 0.01ug/mL T=ST 6266
Sample Id : Ver #6266
Sample Type : Standard Amount=1.00000
Bottle No : 7

MG 5-20-92

PEAK INFORMATION

Peak	GRP	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
6		8.693	8.684	83999	273738	0.01100	Tetrachloro-m-xylene (TCX)	2.9
11		10.391	10.382	98324	348735	0.01027	alpha-BHC	3.5
13		11.573	11.564	87696	345061	0.01030	gamma-BHC	4.0
14		11.791	11.778	47364	194581	0.01076	beta-BHC	4.0
17		12.960	12.951	73447	370858	0.01070	Heptachlor	4.8A
18		13.049	13.040	69635	303812	0.01003	delta-BHC	4.3A
20		14.209	14.196	62688	323890	0.01025	Aldrin	5.1
22		16.409	16.400	59877	338045	0.01057	Heptachlor Epoxide	5.3
25		17.991	17.982	53743	318124	0.01052	Endosulfan I	5.6
26		18.978	18.969	51901	287427	0.01027	4,4'-DDE	5.3
27		19.338	19.329	49247	292815	0.01027	Dieldrin	5.6
28		20.911	20.902	45319	275507	0.01035	Endrin	5.9
30		21.569	21.560	36484	210354	0.01013	4,4'-DDD	5.6
31		21.796	21.787	46268	291568	0.01055	Endosulfan II	6.4
33		23.093	23.080	36229	215928	0.01016	4,4'-DDT	5.9
34		23.258	23.249	33322	213755	0.01058	Endrin Aldehyde	6.4
35		24.076	24.067	40043	266058	0.01060	Endosulfan Sulfate	6.1
39		26.204	26.196	32478	219574	0.01049	Dibutylchlorendate (DBC)	6.7
40		27.133	27.124	22273	141913	0.01059	Methoxychlor	6.4
41		27.520	27.511	39831	261779	0.01048	Endrin Ketone	6.4
46		33.480	33.467	110856	819377	0.02513	Octachloronapthalene	7.2
47		33.738	33.729	38246	284233	0.01099	Decachlorobiphenyl (DCB)	7.5

Totals			
Unknowns	215595	1053543	N/A
Quantified	1219271	6597132	0.24502
Grand Total	1434867	7650675	0.24502

MISSING PEAKS

No missing peaks.

PEAK GROUP INFORMATION

No peak group information.

ANALYSIS SUMMARY

Method..... COCP
Run sequence..... C0518
Calibration..... C0518
External standard calibration using area
Calibration last modified on 20-MAY-1992 at 14:02

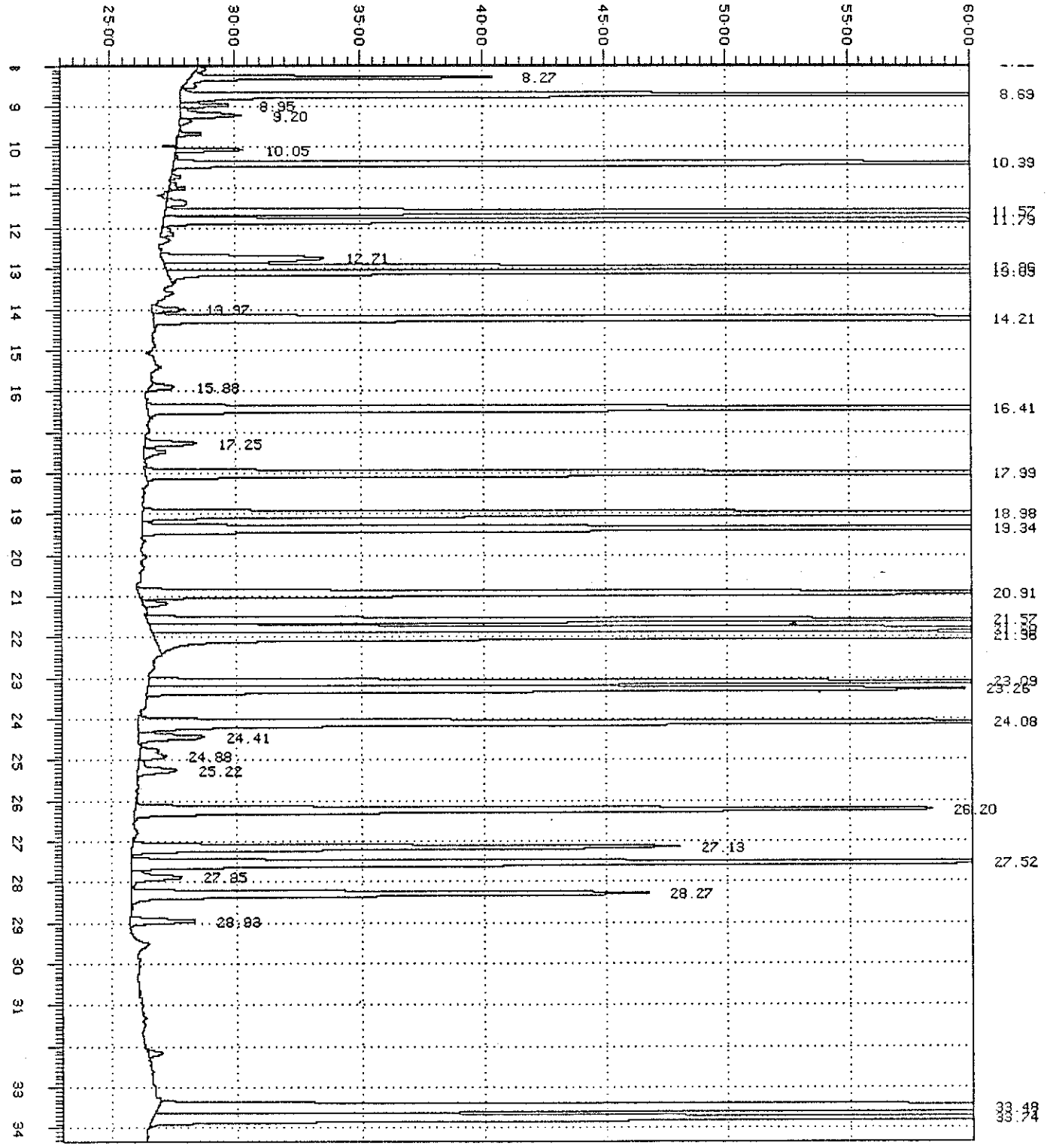
Uncalibrated peaks use user factor (0.0000)

RMAL Chromatography System on DENCR3

[OCP05_3] 57 C18MAY92,7,1

Individual Mix AB 0.01ug/mL T=ST 6266. Amount : 1.000.
C_5890,Dual ECD,Column: RTx-35,0.53mm,0.53um,30m
Acquired on 19-MAY-1992 at 01:09
Reported on 20-MAY-1992 at 12:07
Box 1 (of 1)

MA 5-20-92



Injection Report

Acquired on 19-MAY-1992 at 01:09

RMAL Chromatography System on DENCR3

Analyst Name : MGOETZ
Lims Id : 2uL injected
Comment : C-5890,DUAL ECD Rtx-35,0.53,1.00um, Rtx-5, 0.53,1.5um
Method Title :
Sample Name : Individual Mix AB 0.01ug/mL T=ST 6266
Sample Id : Ver #6266
Sample Type : Standard Amount=1.00000
Bottle No : 7

MG 5-20-92

PEAK INFORMATION

Peak	GRP	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
6		8.836	8.827	81040	248023	0.01086	Tetrachloro-m-xylene (TCX)	2.9
11		9.747	9.738	114159	371157	0.01050	alpha-BHC	3.2
13		10.373	10.364	51946	191019	0.01079	beta-BHC	3.5
14		10.596	10.587	100215	365577	0.01060	gamma-BHC	3.7
16		11.213	11.204	84843	318540	0.01034	delta-BHC	3.7
18		12.824	12.822	84218	377256	0.01051	Heptachlor	4.5
19		14.080	14.071	73156	345909	0.01046	Aldrin	4.8
21		15.569	15.564	67153	348624	0.01066	Heptachlor Epoxide	5.1
23		17.071	17.067	61613	340770	0.01064	Endosulfan I	5.3
24		17.960	17.951	56882	304823	0.01045	4,4'-DDE	5.3
25		18.173	18.169	57191	325154	0.01055	Dieldrin	5.6
27		19.133	19.138	62541	610827	0.00991	Endrin	9.9
28		19.542	19.533	51169	302074	0.01067	Endosulfan II	5.9
29		19.831	19.827	41905	236749	0.01032	4,4'-DDD	5.6
30		20.382	20.373	39281	236502	0.01065	Endrin Aldehyde	5.9
C1			21.476	93424	521900	0.02102	4,4'-DDT/Endosulfan Sulfate	
32		21.489	21.476	47252	268431	0.00923	- C1	6.4A
33		21.564	21.476	46172	253469	0.00853	- C1	6.4A
35		23.711	23.702	46414	293263	0.01065	Endrin Ketone	6.4
36		24.311	24.307	26603	164794	0.01075	Methoxychlor	6.1
39		25.942	25.933	34041	224441	0.01063	Dibutylchlorendate (DBC)	6.7
43		31.284	31.280	118247	737453	0.02473	Octachloronapthalene	6.1
44		32.413	32.409	30282	204977	0.01094	Decachlorobiphenyl (DCB)	6.7

Totals			
Unknowns	165703	661939	N/A
Quantified	1376323	7069832	0.24662
Grand Total	1542026	7731771	0.24662

MISSING PEAKS

No missing peaks.

PEAK GROUP INFORMATION

No peak group information.

ANALYSIS SUMMARY

Method..... COCP
Run sequence..... C0518
Calibration..... C0518
External standard calibration using area
Calibration last modified on 19-MAY-1992 at 13:25

Uncalibrated peaks use user factor (0.0000)

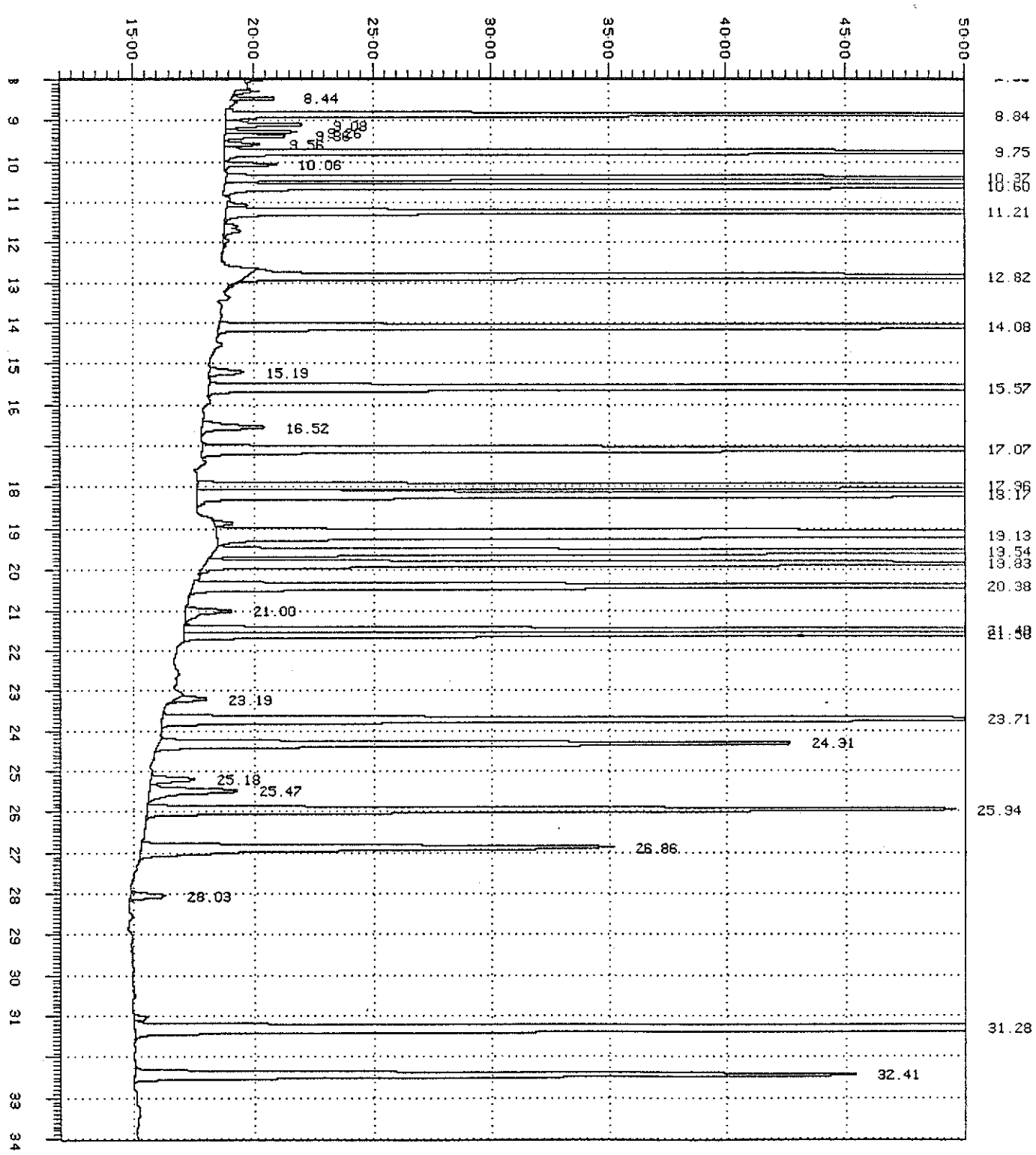
RMAL Chromatography System on DENCR3

[OCP05_3] 58 C18MAY92,7,1

Individual Mix AB 0.01ug/mL T=ST 6266. Amount : 1.000.

Acquired on 19-MAY-1992 at 01:09
Reported on 20-MAY-1992 at 12:12
Box 1 (of 1)

M 5-20-92



Injection Report

Acquired on 19-MAY-1992 at 01:57

RMAL Chromatography System on DENCR3

Analyst Name : MGOETZ
Lims Id : 2uL injected
Comment : C-5890,DUAL ECD Rtx-35,0.53,1.00um, Rtx-5, 0.53,1.5um
Method Title :
Sample Name : Individual Mix AB 0.005ug/mL T=ST 6266
Sample Id : Ver #6266
Sample Type : Standard Amount=1.00000
Bottle No : 8

MG 5-20-92

PEAK INFORMATION

Peak	GRP	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
6		8.711	8.684	45229	144872	0.00472	Tetrachloro-m-xylene (TCX)	3.2
11		10.409	10.382	48922	173204	0.00490	alpha-BHC	3.5
14		11.591	11.564	44026	176083	0.00489	gamma-BHC	4.0
15		11.809	11.778	25501	102645	0.00477	beta-BHC	4.0
17		12.973	12.951	40017	207259	0.00478	Heptachlor	4.8A
18		13.067	13.040	34494	152913	0.00498	delta-BHC	4.3A
20		14.222	14.196	32002	170478	0.00491	Aldrin	5.1
21		16.427	16.400	32554	181441	0.00482	Heptachlor Epoxide	5.3
23		18.009	17.982	29301	170043	0.00484	Endosulfan I	5.6
24		18.996	18.969	26711	150283	0.00491	4,4'-DDE	5.3
25		19.356	19.329	25672	155286	0.00491	Dieldrin	5.9
26		20.929	20.902	23852	146768	0.00489	Endrin	5.9
28		21.587	21.560	18580	108352	0.00496	4,4'-DDD	5.6
29		21.813	21.787	24951	156395	0.00483	Endosulfan II	6.4
31		23.111	23.080	18926	112690	0.00495	4,4'-DDT	5.9
32		23.276	23.249	17886	113187	0.00483	Endrin Aldehyde	6.4
33		24.093	24.067	20892	139436	0.00482	Endosulfan Sulfate	6.1
37		26.222	26.196	17445	119510	0.00485	Dibutylchlorendate (DBC)	6.7
38		27.151	27.124	11840	75359	0.00482	Methoxychlor	6.1
39		27.538	27.511	21014	138438	0.00485	Endrin Ketone	6.4
43		33.493	33.467	113143	833827	0.02557	Octachloronapthalene	7.2
44		33.756	33.729	21009	156753	0.00471	Decachlorobiphenyl (DCB)	7.5

Totals			
Unknowns	194433	976965	N/A
Quantified	693966	3885223	0.12753
Grand Total	888399	4862188	0.12753

MISSING PEAKS

No missing peaks.

PEAK GROUP INFORMATION

No peak group information.

ANALYSIS SUMMARY

Method..... COCP
Run sequence..... C0518
Calibration..... C0518
External standard calibration using area
Calibration last modified on 20-MAY-1992 at 14:02

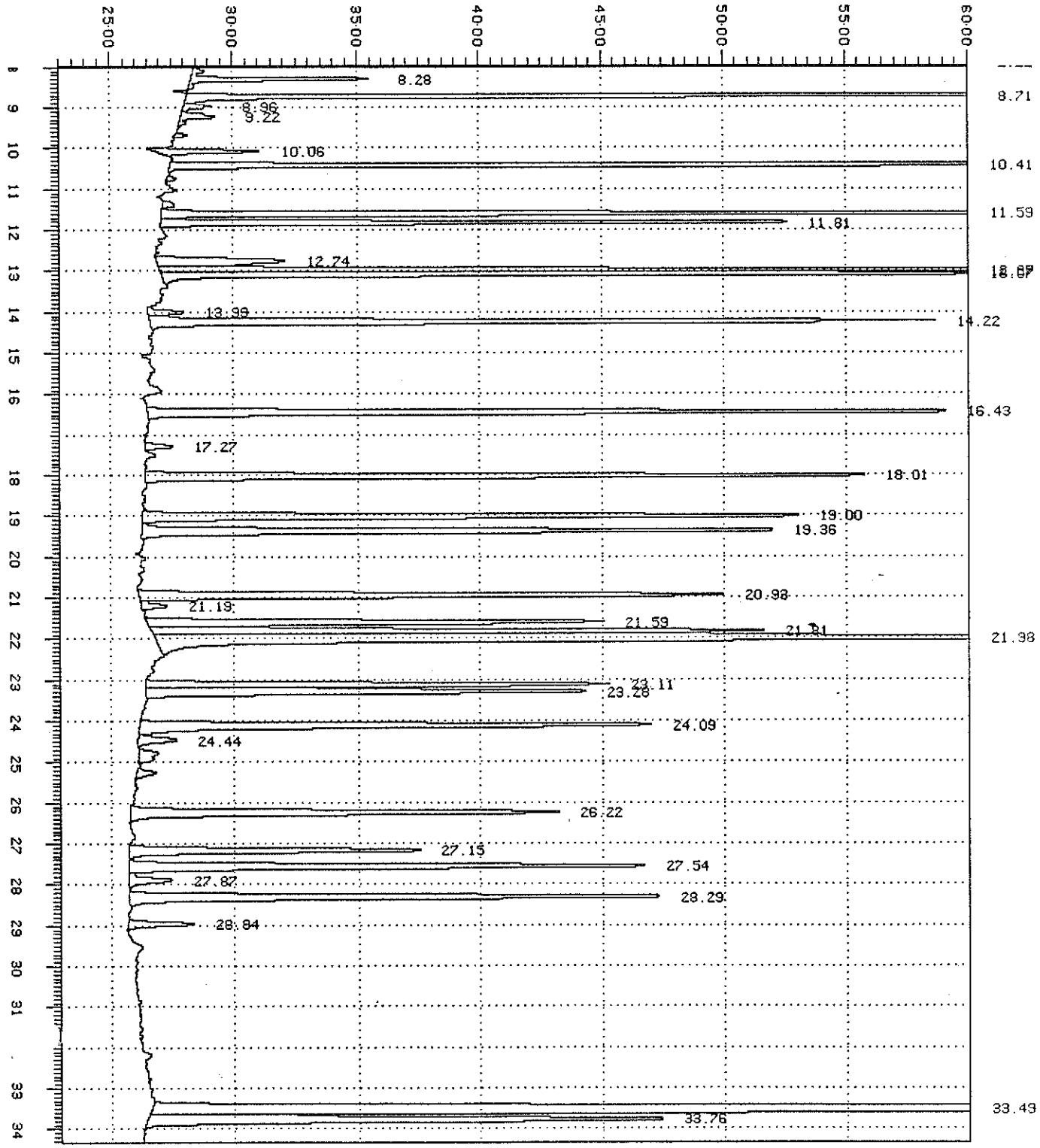
Uncalibrated peaks use user factor (0.0000)

RMAL Chromatography System on DENCR3

[OCP05_3] 57 C18MAY92,8,1

Individual Mix AB 0.005ug/mL T=ST 6266. Amount : 1.000.
C_5890,Dual ECD,Column: RTx-35,0.53mm,0.53um,30m
Acquired on 19-MAY-1992 at 01:57
Reported on 20-MAY-1992 at 12:07
Box 1 (of 1)

M 5-20-92



Injection Report

Acquired on 19-MAY-1992 at 01:57

RMAL Chromatography System on DENCR3

Analyst Name : MGOETZ
Lims Id : 2uL injected
Comment : C-5890,DUAL ECD Rtx-35,0.53,1.00um, Rtx-5, 0.53,1.5um
Method Title :
Sample Name : Individual Mix AB 0.005ug/mL T=ST 6266
Sample Id : Ver #6266
Sample Type : Standard Amount=1.00000
Bottle No : 8

MG 5-20-92

PEAK INFORMATION

Peak	GRP	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
6		8.831	8.827	44123	130647	0.00475	Tetrachloro-m-xylene (TCX)	3.2
11		9.742	9.738	58998	191944	0.00485	alpha-BHC	3.2
13		10.369	10.364	28492	104702	0.00476	beta-BHC	3.7
14		10.591	10.587	52753	191179	0.00482	gamma-BHC	3.7
16		11.209	11.204	43015	163726	0.00489	delta-BHC	3.7
17		12.823	12.822	45496	209989	0.00484	Heptachlor	4.5
18		14.076	14.071	38703	183671	0.00486	Aldrin	4.5
20		15.569	15.564	36955	190597	0.00480	Heptachlor Epoxide	5.1
22		17.067	17.067	33753	185834	0.00480	Endosulfan I	5.6
23		17.956	17.951	29846	158383	0.00486	4,4'-DDE	5.3
24		18.173	18.169	30682	170860	0.00483	Dieldrin	5.3
26		19.071	19.138	56559	491801	0.00498	Endrin	9.1
27		19.542	19.533	27918	163277	0.00480	Endosulfan II	5.9
28		19.831	19.827	21918	122643	0.00490	4,4'-DDD	5.3
29		20.382	20.373	21495	127301	0.00481	Endrin Aldehyde	5.9
C1			21.476	49596	278577	0.00970	4,4'-DDT/Endosulfan Sulfate	
31		21.484	21.476	25228	145778	0.00352	- C1	6.4A
32		21.564	21.476	24368	132799	0.00292	- C1	6.4A
34		23.711	23.702	25737	158522	0.00481	Endrin Ketone	6.1
35		24.311	24.307	14516	88383	0.00478	Methoxychlor	6.1
38		25.938	25.933	18824	122861	0.00481	Dibutylchlorendate (DBC)	6.4
41		31.280	31.280	121073	747563	0.02533	Octachloronapthalene	6.1
42		32.413	32.409	17184	113943	0.00473	Decachlorobiphenyl (DCB)	6.7

Totals			
Unknowns	139229	540976	N/A
Quantified	817638	4296404	0.12671
Grand Total	956867	4837380	0.12671

MISSING PEAKS

No missing peaks.

PEAK GROUP INFORMATION

No peak group information.

ANALYSIS SUMMARY

Method..... COCP
Run sequence..... C0518
Calibration..... C0518
External standard calibration using area
Calibration last modified on 19-MAY-1992 at 13:25

Uncalibrated peaks use user factor (0.0000)

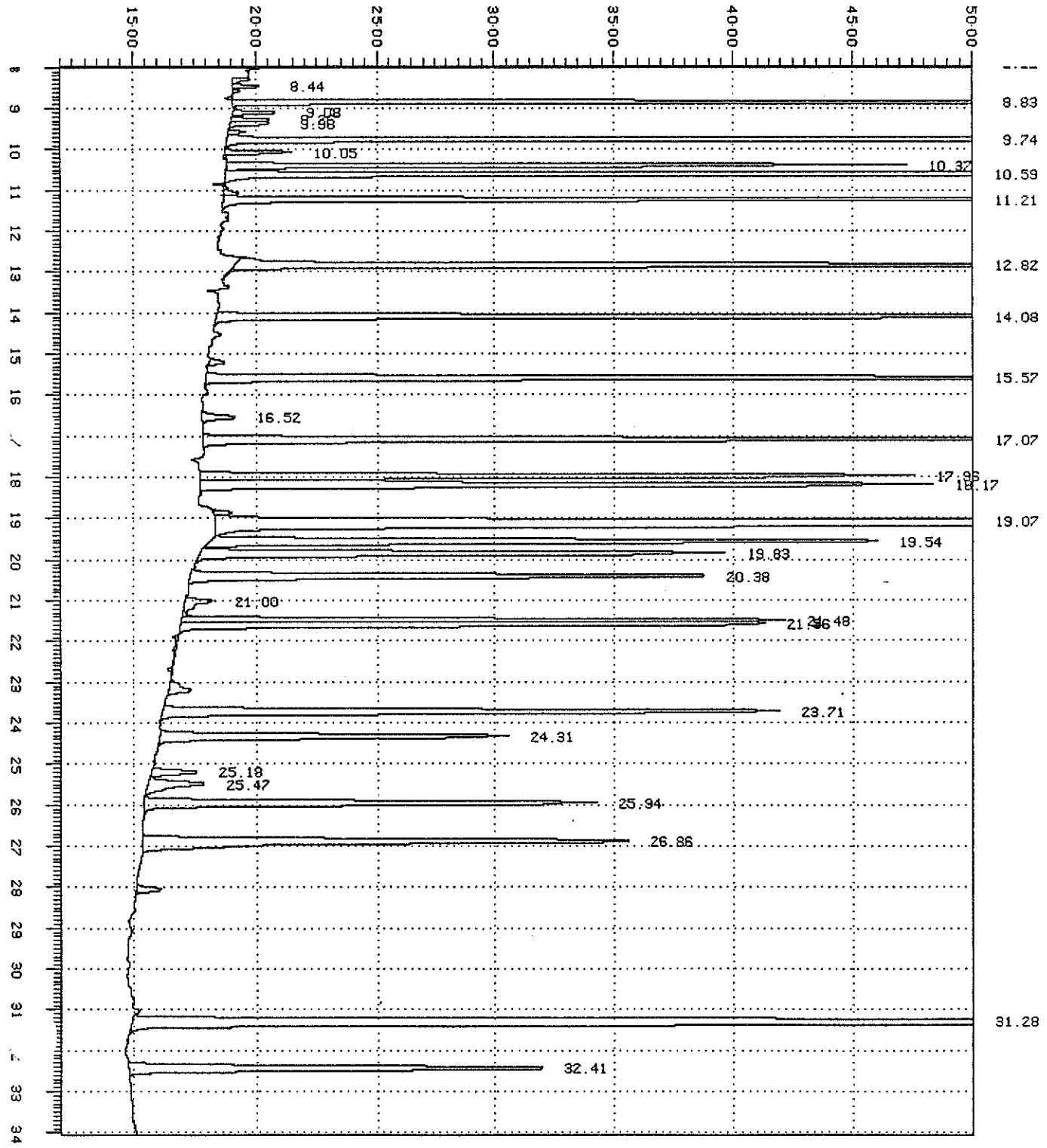
RMAL Chromatography System on DENCR3

[OCP05_3] 58 C18MAY92,8,1

Individual Mix AB 0.005ug/mL T=ST 6266. Amount : 1.000.

Acquired on 19-MAY-1992 at 01:57
Reported on 20-MAY-1992 at 12:13
Box 1 (of 1)

M 5-20-92



Enseco
A Corning Company

STANDARD
OPERATING
PROCEDURE

Subject or Title: OCP SCS STOCK		Page 1 of 2
SOP No.: LS-RMA-2250	Revision No.: 3.0	Effective Date: 03/14/91
Supersedes:	Revision 2.0	

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1. TYPE OF STANDARD: Sample control spike stock.

2. SOLVENT/MATRIX: Methanol.

3. PREPARATION NOTES:

   3.1 Standards made for the extraction laboratory (DCSs, SCSs, MS/MSDs) and the instrumentation laboratory (calibration standards, check standards) must each be prepared from different sources. The other source may be another chemical supplier, the EPA, or the National Institute of Standards and Technologies (NIST), refer to SOP: LP-RMA-0019.

Prepared by: Anne Yardy	Date: 03/14/91
Management Approval: Debra Henderson	Date: 03-18-91
QA Officer Approval: Jan Ridenbarger	Date: 3/18/91

3.2 To a 50.0 mL volumetric flask add approximately 40 mL of methanol. Using a 5.0 mL Hamilton gas tight syringe add 2.0 mL of dibutylchlorendate (DBC) at 5000 ug/mL to the flask. Bring to volume with methanol. Transfer solution to two 35 mL screw top bottles.

4. SHELF LIFE: 6 months.

5. SAFETY ISSUES: Refer to the MSDS's.

6. CONSTITUENTS/CONCENTRATION:

CHEMICAL/STOCK NAME (Initial Conc.)	FINAL CONC.
Dibutylchlorendate (5000 ug/mL, methanol)	200.0 ug/mL

7. NOTES:

Subject or Title: OCP DCS STOCK		Page 1 of 2
SOP No.: LS-RMA-2253	Revision No.: 3.0	Effective Date: 03/14/91
Supersedes:	Revision 2.0	

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1. TYPE OF STANDARD: Laboratory control spike stock.

2. SOLVENT/MATRIX: Methanol.

3. PREPARATION NOTES:

   3.1 Standards made for the extraction laboratory (DCSs, SCSs, MS/MSDs) and the instrumentation laboratory (calibration standards, check standards) must each be prepared from different sources. The other source may be another chemical supplier, the EPA, or the National Institute of Standards and Technologies (NIST), refer to SOP: LP-RMA-0019.

Prepared by: Anne Yardy	Date: 03/14/91
Management Approval: Alisia Henderson	Date: 03-18-91
QA Officer Approval: Jan Redenbarger	Date: 3/18/91

3.2 To a 25.0 mL volumetric flask add approximately 20 mL of methanol. Using a 1.0 mL Hamilton gas tight syringe add 250 uL of Ultra Scientific Pesticides Matrix Spiking Solution at 2000/5000 ug/mL to the flask. Bring to volume with methanol. Transfer solution to a 35 mL screw top bottle.

4. SHELF LIFE: 6 months.

5. SAFETY ISSUES: Refer to the MSDS's.

6. CONSTITUENTS/CONCENTRATION:

CHEMICAL/STOCK NAME (Initial Conc.)		FINAL CONC.
Ultra Scientific Pesticides Matrix Spiking Solution	(2000/5000 ug/mL, methanol)	
Lindane	(2000 ug/mL, methanol)	20.0 ug/mL
Heptachlor	(2000 ug/mL, methanol)	20.0 ug/mL
Aldrin	(2000 ug/mL, methanol)	20.0 ug/mL
Endrin	(5000 ug/mL, methanol)	50.0 ug/mL
Dieldrin	(5000 ug/mL, methanol)	50.0 ug/mL
p,p'-DDT	(5000 ug/mL, methanol)	50.0 ug/mL

7. NOTES:

Enseco
A Corning Company

STANDARD
OPERATING
PROCEDURE

Subject or Title: GC-INDAB-CSA (Individual Mix AB)		Page 1 of 2
SOP No.: LS-RMA-2256	Revision No.: 3.0	Effective Date: 03/14/91
Supersedes:	Revision 2.0	

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1. TYPE OF STANDARD: Calibration standard.

2. SOLVENT/MATRIX: Hexane.

3. PREPARATION NOTES:

   3.1 Standards made for the extraction laboratory (DCSs, SCSs, MS/MSDs) and the instrumentation laboratory (calibration standards, check standards) must each be prepared from different sources. The other source may be another chemical supplier, the EPA, or the National Institute of Standards and Technologies (NIST), refer to SOP: LP-RMA-0019.

Prepared by: Anne Yardy	Date: 03/14/91
Management Approval: Debra Henderson	Date: 03-18-91
QA Officer Approval: Jan Redenburg	Date: 3/18/91

3.2 To a 10.0 mL volumetric flask add approximately 5 mL of hexane. Using a 1.0 mL Hamilton gas tight syringe add 500 uL of Supelpreme-HC Pesticide Mix at 2000 ug/mL, add 500 uL of endrin ketone at 2000 ug/mL, add 200 uL of methopychlor at 5000 ug/mL, add 200 uL of dibutylchlorendate at 5000 ug/mL to the flask. Bring to volume with hexane. Transfer solution to a 12 mL screw top amber vial.

4. SHELF LIFE: 6 months.

5. SAFETY ISSUES: Refer to the MSDS's.

6. CONSTITUENTS/CONCENTRATION:

CHEMICAL/STOCK NAME (Initial Conc.)		FINAL CONC.
Supelpreme-HC Pesticides Mix (2000 ug/mL, Tol/Hex)		100.0 ug/mL
Aldrin	Supelco	100.0 ug/mL
beta-BHC	Supelco	100.0 ug/mL
alpha-BHC	Supelco	100.0 ug/mL
delta-BHC	Supelco	100.0 ug/mL
4,4'-DDE	Supelco	100.0 ug/mL
Dieldrin	Supelco	100.0 ug/mL
Endosulfan A	Supelco	100.0 ug/mL
Endrin	Supelco	100.0 ug/mL
Heptachlor	Supelco	100.0 ug/mL
gamma-BHC	Supelco	100.0 ug/mL
4,4'-DDD	Supelco	100.0 ug/mL
4,4'-DDT	Supelco	100.0 ug/mL
Endosulfan B	Supelco	100.0 ug/mL
Endosulfan Sulfate	Supelco	100.0 ug/mL
Endrin Aldehyde	Supelco	100.0 ug/mL
Heptachlor Epoxide	Supelco	100.0 ug/mL
Endrin Ketone (2000 ug/mL, ethyl acetate)		100.0 ug/mL
Methoxychlor (5000 ug/mL, hexane)		100.0 ug/mL
Dibutylchlorendate (5000 ug/mL, methanol)		100.0 ug/mL

7. NOTES:

Subject or Title: GC-EVAL-CSA		Page 1 of 2
SOP No.: LS-RMA-2258	Revision No.: 3.0	Effective Date: 03/14/91
Supersedes:	Revision 2.0	

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1. TYPE OF STANDARD: Calibration standard stock.

2. SOLVENT/MATRIX: Methanol.

3. PREPARATION NOTES:

   3.1 Standards made for the extraction laboratory (DCSs, SCSs, MS/MSDs) and the instrumentation laboratory (calibration standards, check standards) must each be prepared from different sources. The other source may be another chemical supplier, the EPA, or the National Institute of Standards and Technologies (NIST), refer to SOP: LP-RMA-0019.

Prepared by: Anne Yardy	Date: 03/14/91
Management Approval: Debra Henderer	Date: 03-18-91
QA Officer Approval: Jan Redenbarger	Date: 3/18/91

3.2 To a 25.0 mL volumetric flask add approximately 20 mL of methanol. Using a 1.0 mL Hamilton gas tight syringe add 500 uL of aldrin at 5000 ug/mL, add 500 uL of endrin at 5000 ug/mL, and 500 uL of p,p'-DDT at 5000 ug/mL, and 500 uL of dibutylchlorendate at 5000 ug/mL to the flask. Bring to volume with methanol. Transfer solution to a 35 mL screw top bottle.

4. SHELF LIFE: 6 months.

5. SAFETY ISSUES: Refer to the MSDS's.

6. CONSTITUENTS/CONCENTRATION:

CHEMICAL/STOCK NAME (Initial Conc.)	FINAL CONC.
Aldrin (5000 ug/mL, methanol)	100.0 ug/mL
Endrin (5000 ug/mL, methanol)	100.0 ug/mL
p,p'-DDT (5000 ug/mL, methanol)	100.0 ug/mL
Dibutylchlorendate (5000 ug/mL, methanol)	100.0 ug/mL

7. NOTES:

STANDARD
OPERATING
PROCEDURE

Subject or Title: GC-AP90CP-CSA		Page _1_ of _2_
SOP No.: LS-RMA-2261	Revision No.: 4.0	Effective Date 06/25/92
Supersedes: 3.0		

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1. TYPE OF STANDARD: Calibration standard stock.

2. SOLVENT/MATRIX: methanol

3. PREPARATION NOTES:

   3.1 Standards made for the extraction laboratory (DCSs, SCSs, MS/MSDs) and the instrumentation laboratory (calibration standards, check standards) must each be prepared from different sources. The other source may be another chemical supplier, the EPA, or the National Institute of Standards and Technologies (NIST). Refer to SOP: LP-RMA-0019.

Prepared by: Heather Esbenson	Date: 06/25/92
Management Approval: Paul Williams	Date: 6/25/92
QA Officer Approval: Cindy Pattison	Date: 7/2/92

3.2 To a 10.0mL volumetric flask add approximately 6 mL of methanol. Using a 1.0 mL Hamiltion gas tight syringe add 500 uL of each of the following stocks at 500 ug/ml, 2000 ug/ml, and 5000 ug/ml to the flask. Bring to volume with methanol. Transfer to a 12 mL amber vial.

4. SHELF LIFE: 6 months

5. SAFETY ISSUES: Refer to the MSDS's.

6. CONSTITUENTS/CONCENTRATION:

CHEMICAL/STOCK NAME (Conc., open date, etc.)	FINAL CONC.
Chlorobenzilate (2000 ug/ml)	100.0 ug/ml
DiAllate (2000 ug/ml)	100.0 ug/ml
Kepone (5000 ug/ml)	250.0 ug/ml
Isodrin (500 ug/ml)	25.0 ug/ml

7. NOTES:

Subject or Title: GC-a,g-CHLORDANE-CSA		Page 1 of 2
SOP No.: LS-RMA-2262	Revision No.: 3.0	Effective Date: 03/14/91
Supersedes:	Revision 2.0	

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1. TYPE OF STANDARD: Calibration standard stock.

2. SOLVENT/MATRIX: Hexane.

3. PREPARATION NOTES:

   3.1 Standards made for the extraction laboratory (DCSs, SCSs, MS/MSDs) and the instrumentation laboratory (calibration standards, check standards) must each be prepared from different sources. The other source may be another chemical supplier, the EPA, or the National Institute of Standards and Technologies (NIST), refer to SOP: LP-RMA-0019.

Prepared by: Anne Yardy	Date: 03/14/91
Management Approval: Debra Henderer	Date: 03-18-91
QA Officer Approval: Jan Redenbaugh	Date: 3/18/91

3.2 To a 10.0 mL volumetric flask add approximately 8 mL of hexane. Using a 1.0 mL Hamilton gas tight syringe add 200 uL of alpha-chlordane at 5000 ug/mL, add 200 uL of gamma-chlordane at 5000 ug/mL, add 200 uL of dibutylchlorendate at 5000 ug/mL to the flask. Bring to volume with hexane. Transfer solution to a 12 mL screw top amber vial.

4. SHELF LIFE: 6 months.

5. SAFETY ISSUES: Refer to the MSDS's.

6. CONSTITUENTS/CONCENTRATION:

CHEMICAL/STOCK NAME (Initial Conc.)	FINAL CONC.
alpha-Chlordane (5000 ug/mL, hexane)	100.0 ug/mL
gamma-Chlordane (5000 ug/mL, hexane)	100.0 ug/mL
Dibutylchlorendate (5000 ug/mL, hexane)	100.0 ug/mL

7. NOTES:

Subject or Title: GC-TOXAPHENE-CSA		Page 1 of 2
SOP No.: LS-RMA-2264	Revision No.: 3.0	Effective Date: 03/14/91
Supersedes:	Revision 2.0	

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1. TYPE OF STANDARD: Calibration standard.

2. SOLVENT/MATRIX: Methanol.

3. PREPARATION NOTES:

   3.1 Standards made for the extraction laboratory (DCSs, SCSs, MS/MSDs) and the instrumentation laboratory (calibration standards, check standards) must each be prepared from different sources. The other source may be another chemical supplier, the EPA, or the National Institute of Standards and Technologies (NIST), refer to SOP: LP-RMA-0019.

Prepared by: Anne Yardy	Date: 03/14/91
Management Approval: Debra Henderer	Date: 03-18-91
QA Officer Approval: Jan Redenbarger	Date: 3/18/91

3.2 To a 10.0 mL volumetric flask add approximately 8 mL of methanol. Using a 1.0 mL Hamilton gas tight syringe add 500 uL of toxaphene at 5000 ug/mL, add 250 uL dibutylchlorendate at 2000 ug/mL to the flask. Bring to volume with methanol. Transfer solution to a 12 mL screw top amber vial.

4. SHELF LIFE: 6 months.

5. SAFETY ISSUES: Refer to the MSDS's.

6. CONSTITUENTS/CONCENTRATION:

CHEMICAL/STOCK NAME (Initial Conc.)	FINAL CONC.
Toxaphene (5000 ug/mL, methanol)	250.0 ug/mL
Dibutylchlorendate (2000 ug/mL, methanol)	50.0 ug/mL

7. NOTES:

Subject or Title: OCP CPS STOCK		Page 1 of 2
SOP No.: LS-RMA-2269	Revision No.: 3.0	Effective Date: 03/14/91
Supersedes:	Revision 2.0	

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1. TYPE OF STANDARD: Column performance standard.

2. SOLVENT/MATRIX: Methanol.

3. PREPARATION NOTES:

   3.1 Standards made for the extraction laboratory (DCSs, SCSs, MS/MSDs) and the instrumentation laboratory (calibration standards, check standards) must each be prepared from different sources. The other source may be another chemical supplier, the EPA, or the National Institute of Standards and Technologies (NIST), refer to SOP: LP-RMA-0019.

Prepared by: Anne Yardy	Date: 03/14/91
Management Approval: Debra Hendrues	Date: 03-18-91
QA Officer Approval: Jan Redenbarger	Date: 3/18/91

3.2 To a 10.0 mL volumetric flask add approximately 8 mL of methanol. Using a 1.0 mL Hamilton gas tight syringe add 500 uL of GC-EVAL-CSA at 100 ug/mL to the flask. Bring to volume with methanol. Transfer solution to a 12 mL screw top amber vial.

4. SHELF LIFE: 6 months.

5. SAFETY ISSUES: Refer to the MSDS's.

6. CONSTITUENTS/CONCENTRATION:

CHEMICAL/STOCK NAME (Initial Conc.)	FINAL CONC.
GC-EVAL-CSA:	
Aldrin (100 ug/mL, hexane)	5.0 ug/mL
Endrin (100 ug/mL, hexane)	5.0 ug/mL
p,p'-DDT (100 ug/mL, hexane)	5.0 ug/mL
Dibutylchlorendate (100 ug/mL, hexane)	5.0 ug/mL

7. NOTES:

STANDARD
OPERATING
PROCEDURE

Subject or Title: GC-AR1016/1260-CSA		Page 1 of 2
SOP No.: LS-RMA-2757	Revision No.: 2.0	Effective Date: 7/29/91
Supersedes: 1.0		

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1. TYPE OF STANDARD: Calibration standard stock.

2. SOLVENT/MATRIX: Hexane.

3. PREPARATION NOTES:

   3.1 Standards made for the extraction laboratory (DCSs, SCSs, MS/MSDs) and the instrumentation laboratory (calibration standards, check standards) must each be prepared from different sources. The other source may be another chemical supplier, the EPA, or the National Institute of Standards and Technologies (NIST). Refer to SOP: LP-RMA-0019.

Prepared by: Anne Yardy	Date: 07/29/91
Management Approval: Debra Henderson 08/01/91	Date:
QA Officer Approval: Jan Ridenbarger	Date: 8/2/91

3.2 To a 10.0 mL volumetric flask add approximately 8 mL of hexane. Using a 500 uL Hamilton gas tight syringe add 100 uL of Aroclor 1016 at 1000 ug/mL, add 100 uL of Aroclor 1260 at 1000 ug/mL, add 100 uL of Decachlorobiphenyl at 200 ug/mL and add 100 uL of Tetrachloro-m-xylene at 200 ug/mL to the flask. Bring to volume with hexane. Transfer solution to a 12 mL screw top amber vial.

4. SHELF LIFE: 6 months.

5. SAFETY ISSUES: Refer to the MSDS's.

6. CONSTITUENTS/CONCENTRATION:

CHEMICAL/STOCK NAME (Conc.)	FINAL CONC.
Aroclor 1016 (1000 ug/mL, hexane)	10.0 ug/mL
Aroclor 1260 (1000 ug/mL, hexane)	10.0 ug/mL
Decachlorobiphenyl (200 ug/mL, isooctane)	2.0 ug/mL
Tetrachloro-m-xylene (200 ug/mL, isooctane)	2.0 ug/mL

7. NOTES:

STANDARD
OPERATING
PROCEDURE

Subject or Title: GC-AR1221-CSA		Page 1 of 2
SOP No.: LS-RMA-2758	Revision No.: 2.0	Effective Date: 07/29/91
Supersedes: 1.0		

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1. TYPE OF STANDARD: Calibration standard stock.

2. SOLVENT/MATRIX: Hexane.

3. PREPARATION NOTES:

   3.1 Standards made for the extraction laboratory (DCSs, SCSs, MS/MSDs) and the instrumentation laboratory (calibration standards, check standards) must each be prepared from different sources. The other source may be another chemical supplier, the EPA, or the National Institute of Standards and Technologies (NIST). Refer to SOP: LP-RMA-0019.

Prepared by: Anne Yardy	Date: 07/29/91
Management Approval: Debra Harderer 08-01-91	Date:
QA Officer Approval: Jan Ridenbarger	Date: 8/2/91

3.2 To a 10.0 mL volumetric flask add approximately 8 mL of hexane. Using a 500 uL Hamilton gas tight syringe add 200 uL of Aroclor 1221 at 1000 ug/mL, add 100 uL of Decachlorobiphenyl at 200 ug/mL and add 100 uL of Tetrachloro-m-xylene at 200 ug/mL to the flask. Bring to volume with hexane. Transfer solution to a 12 mL screw top amber vial.

4. SHELF LIFE: 6 months.

5. SAFETY ISSUES: Refer to the MSDS's.

6. CONSTITUENTS/CONCENTRATION:

CHEMICAL/STOCK NAME (Conc.)	FINAL CONC.
Aroclor 1221 (1000 ug/mL, hexane)	20.0 ug/mL
Decachlorobiphenyl (200 ug/mL, isooctane)	2.0 ug/mL
Tetrachloro-m-xylene (200 ug/mL, isooctane)	2.0 ug/mL

7. NOTES:

STANDARD
OPERATING
PROCEDURE

Subject or Title: GC-AR1232-CSA		Page 1 of 2
SOP No.: LS-RMA-2759	Revision No.: 2.0	Effective Date: 07/29/91
Supersedes: 1.0		

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1. TYPE OF STANDARD: Calibration standard stock.

2. SOLVENT/MATRIX: Hexane.

3. PREPARATION NOTES:

   3.1 Standards made for the extraction laboratory (DCSs, SCSs, MS/MSDs) and the instrumentation laboratory (calibration standards, check standards) must each be prepared from different sources. The other source may be another chemical supplier, the EPA, or the National Institute of Standards and Technologies (NIST). Refer to SOP: LP-RMA-0019.

Prepared by: Anne Yardy	Date: 07/29/91
Management Approval: Debra Henderer 08-01-91	Date:
QA Officer Approval: Jan Pedenbaugh	Date: 8/2/91

3.2 To a 10.0 mL volumetric flask add approximately 8 mL of hexane. Using a 500 uL Hamilton gas tight syringe add 100 uL of Aroclor 1232 at 1000 ug/mL, add 100 uL of Decachlorobiphenyl at 200 ug/mL and add 100 uL of Tetrachloro-m-xylene at 200 ug/mL to the flask. Bring to volume with hexane. Transfer solution to a 12 mL screw top amber vial.

4. SHELF LIFE: 6 months.

5. SAFETY ISSUES: Refer to the MSDS's.

6. CONSTITUENTS/CONCENTRATION:

CHEMICAL/STOCK NAME (Conc.)	FINAL CONC.
Aroclor 1232 (1000 ug/mL, hexane)	10.0 ug/mL
Decachlorobiphenyl (200 ug/mL, isooctane)	2.0 ug/mL
Tetrachoro-m-xylene (200 ug/mL, isooctane)	2.0 ug/mL

7. NOTES:

STANDARD
OPERATING
PROCEDURE

Subject or Title: GC-AR1242-CSA		Page 1 of 2
SOP No.: LS-RMA-2760	Revision No.: 2.0	Effective Date: 07/29/91
Supersedes: 1.0		

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1. TYPE OF STANDARD: Calibration standard stock.

2. SOLVENT/MATRIX: Hexane.

3. PREPARATION NOTES:

   3.1 Standards made for the extraction laboratory (DCSs, SCSs, MS/MSDs) and the instrumentation laboratory (calibration standards, check standards) must each be prepared from different sources. The other source may be another chemical supplier, the EPA, or the National Institute of Standards and Technologies (NIST). Refer to SOP: LP-RMA-0019.

Prepared by: Anne Yardy	Date: 07/29/91
Management Approval: Debra Henderer 08-01-91	Date:
QA Officer Approval: Ken Redenbarger	Date: 8/2/91

3.2 To a 10.0 mL volumetric flask add approximately 8 mL of hexane. Using a 500 uL Hamilton gas tight syringe add 100 uL of Aroclor 1242 at 1000 ug/mL, add 100 uL of Decachlorobiphenyl at 200 ug/mL and add 100 uL of tetrachloro-m-xylene at 200 ug/mL to the flask. Bring to volume with hexane. Transfer solution to a 12 mL screw top amber vial.

4. SHELF LIFE: 6 months.

5. SAFETY ISSUES: Refer to the MSDS's.

6. CONSTITUENTS/CONCENTRATION:

CHEMICAL/STOCK NAME (Conc.)	FINAL CONC.
Aroclor 1242 (1000 ug/mL, hexane)	10.0 ug/mL
Decachlorobiphenyl (200 ug/mL, isooctane)	2.0 ug/mL
Tetrachloro-m-xylene (200 ug/mL, isooctane)	2.0 ug/mL

7. NOTES:

STANDARD
OPERATING
PROCEDURE

Subject or Title: GC-AR1248-CSA		Page 1 of 2
SOP No.: LS-RMA-2761	Revision No.: 2.0	Effective Date: 07/29/91
Supersedes: 1.0		

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1. TYPE OF STANDARD: Calibration standard stock.

2. SOLVENT/MATRIX: Hexane.

3. PREPARATION NOTES:

   3.1 Standards made for the extraction laboratory (DCSs, SCSs, MS/MSDs) and the instrumentation laboratory (calibration standards, check standards) must each be prepared from different sources. The other source may be another chemical supplier, the EPA, or the National Institute of Standards and Technologies (NIST). Refer to SOP: LP-RMA-0019.

Prepared by: Anne Yardy	Date: 07/29/91
Management Approval: Debra Henderson 08-01-91	Date:
QA Officer Approval: Jan Redenberger	Date: 8/2/91

3.2 To a 10.0 mL volumetric flask add approximately 8 mL of hexane. Using a 500 uL Hamilton gas tight syringe add 100 uL of Aroclor 1248 at 1000 ug/mL, add 100 uL of Decachlorobiphenyl at 200 ug/mL and add 100 uL of Tetrachloro-m-xylene at 200 ug/mL to the flask. Bring to volume with hexane. Transfer solution to a 12 mL screw top amber vial.

4. SHELF LIFE: 6 months.

5. SAFETY ISSUES: Refer to the MSDS's.

6. CONSTITUENTS/CONCENTRATION:

CHEMICAL/STOCK NAME (Conc.)	FINAL CONC.
Aroclor 1248 (1000 ug/mL, hexane)	10.0 ug/mL
Decachlorobiphenyl (200 ug/mL, isooctane)	2.0 ug/mL
Tetrachloro-m-xylene (200 ug/mL, isooctane)	2.0 ug/mL

7. NOTES:

STANDARD
OPERATING
PROCEDURE

Subject or Title: GC-AR1254-CSA		Page 1 of 2
SOP No.: LS-RMA-2762	Revision No.: 2.0	Effective Date: 07/29/91
Supersedes: 1.0		

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1. TYPE OF STANDARD: Calibration standard stock.

2. SOLVENT/MATRIX: Hexane.

3. PREPARATION NOTES:

   3.1 Standards made for the extraction laboratory (DCSs, SCSs, MS/MSDs) and the instrumentation laboratory (calibration standards, check standards) must each be prepared from different sources. The other source may be another chemical supplier, the EPA, or the National Institute of Standards and Technologies (NIST). Refer to SOP: LP-RMA-0019.

Prepared by: Anne Yardy	Date: 07/29/91
Management Approval: Debra Henderson 08-01-91	Date:
QA Officer Approval: Jan Redenbarger	Date: 8-2-91

3.2 To a 10.0 mL volumetric flask add approximately 8 mL of hexane. Using a 500 uL Hamilton gas tight syringe add 100 uL of Aroclor 1254 at 1000 ug/mL, add 100 uL of Decachlorobiphenyl at 200 ug/mL and add 100 uL of Tetrachloro-m-xylene at 200 ug/mL to the flask. Bring to volume with hexane. Transfer solution to a 12 mL screw top amber vial.

4. SHELF LIFE: 6 months.

5. SAFETY ISSUES: Refer to the MSDS's.

6. CONSTITUENTS/CONCENTRATION:

CHEMICAL/STOCK NAME (Conc.)	FINAL CONC.
Aroclor 1254 (1000 ug/mL, hexane)	10.0 ug/mL
Decachlorobiphenyl (200 ug/mL, isooctane)	2.0 ug/mL
Tetrachloro-m-xylene (200 ug/mL, isooctane)	2.0 ug/mL

7. NOTES:

This addendum is specific to the Chemical Waste Management, Vickery, Ground Water Monitoring Plan QAPjP. Procedural changes or clarification statements presented herein are not applicable to Enseco SOPs for other projects, and are made here in response to EPA letters dated March 17, 1992 and September 15, 1992 to CWM containing comments (with additional attachments) regarding the CWM-Vickery GWMP QAPjP.

Section 1.2: Dilutions of samples are performed within accepted EPA guidelines in SW-846 and the CLP methods. All samples are screened prior to analysis. The screening results are used to optimize the sample analysis to achieve the lowest possible reporting limits. Dilutions are only performed relative to compounds that would interfere with the analysis. Thus, a high concentration of inorganic ions such as sodium would not result in a dilution being performed for semivolatile organics. Only those compounds that have the property of a "semivolatile organic" would cause a dilution.

Section 2.2: "Solid" samples will not be analyzed within the scope of this project. For the purposes of this project specific QAPjP, Section 2.2 of this SOP is deleted.

Sections 7.6 to 7.10:
Appendix B, containing the referenced standard solutions is contained within the SOP. See pages 55 through 59 of 64 for the appropriate information.

Section 8.2.1 Enseco-RMA plans to use CLLE (continuous liquid-liquid extraction) for all GC/MS semi-volatile analyses.

Section 8.8.2: Initial calibration utilizing standard solutions prepared at concentrations of 20, 40, 80, 120 and 160 ug/mL while utilizing a reporting limit of 10 ug/L is based on method 8270 requirements in SW-846 (1986). In Section 5.6 of method 8270 the language states that "one of the calibration standards should be near, but above, the method detection limit". Enseco-RMAL performs MDL studies on regular basis that support the 10 ug/L reporting limits.

For the purposes of this project, the fifth paragraph under Section 8.8.2 (beginning with ....."If the samples are NOT being analyzed for these specific compounds....") is deleted.

Section 8.8.2.1: The 1-2 uL injection volume referenced is correct. Actual injection volume is documented in the raw data and accounted for in the calculations.

On the last line of this section, the reference to (Section) 8.7, should actually refer to (Section) 8.6 of the SOP, dealing with instrument conditions during calibration.

Section 8.8.2.5: For the purposes of this project, the last sentence under Section 8.8.2.5 is deleted.

Section 8.8.3.1: The 1-2 uL injection volume referenced is correct. Actual injection volume is documented in the raw data and accounted for in the calculations.

On the last line of this section, the reference to (Section) 8.7, should actually refer to (Section) 8.6 of the SOP, dealing with instrument conditions during calibration.

Section 8.10.6: Library searches of up to 20 unknown peaks will be performed.

Section 8.10.10: Mass spectra, chromatographs, tuning and calibration data, daily standard and continuing calibration check information et., will be included as a part of raw data packages. These data will be available to the data validation organization and to regulatory agencies for auduting.

Section 9.5: Matrix spike/matrix spike duplicate analysis will be performed, however, for drilling mud matrix samples taken during drilling no recovery control limit requirements will be applicable. For any ground water matrix samples, control limits of 80% - 120% will be an objective, but not a requirement. After one year of experience working with the ground water matrix, control limit reqirements will be set.

Table A-1: For a list of Appendix IX parameter reporting limits, please refer to QAPjP Appendix A-10.

Table B-5 The compounds and concentrations listed are calibration standards to be utilized.

DFTPP Ions: DFTPP key ions and ion abundance criteria are included in Table C-1, on page 60 of 64 in the SOP.

SOP Appendix A: This SOP is generic in nature, and intended to be sufficiently flexible to address the requirements of multiple clients and/or regulatory agencies. For the purposes of this particular project, Appendix A is not applicable.

Relative Retention Times:
Target parameters and and their respective retention times are indicated QAPjP Appendix B-12A.

Summary tables of target compounds are found in the GWMP Section 7, pages 9 through 11, and in Table 7-1.

SOP Number: LM-RMA-3013 BNA 625

Title: GC/MS Analysis of Semivolatile Organics

This addendum is specific to the Chemical Waste Management, Vickery, Ground Water Monitoring Plan QAPjP. Procedural changes or clarification statements presented herein are not applicable to Enseco SOPs for other projects, and are made here in response to an EPA letter dated March 17, 1992 to CWM containing comments (with additional attachment) regarding the CWM-Vickery GWMP QAPjP.

Section 1.2: Dilutions of samples are performed within accepted EPA guidelines in SW-846 and the CLP methods. All samples are screened prior to analysis. The screening results are used to optimize the sample analysis to achieve the lowest possible reporting limits. Dilutions are only performed relative to compounds that would interfere with the analysis. Thus, a high concentration of inorganic ions such as sodium would not result in a dilution being performed for semivolatile organics. Only those compounds that have the property of a "semivolatile organic" would cause a dilution.

Section 2.2: "Solid" samples will not be analyzed within the scope of this project. For the purposes of this project specific QAPjP, Section 2.2 of this SOP is deleted.

Sections 7.6 to 7.10: Appendix B, containing the referenced standard solutions is contained within the SOP. See pages 55 through 59 of 64 for the appropriate information.

Section 8.2.1 Enseco-RMA plans to use CLLE (continuous liquid-liquid extraction) for all GC/MS semi-volatile analyses.

Section 8.8.2: Initial calibration utilizing standard solutions prepared at concentrations of 20, 40 80, 120 and 160 ug/mL is based on CLP requirements, and will not be changed for this project.

For the purposes of this project, the fifth paragraph under Section 8.8.2 (beginning with ....."If the samples are NOT being analyzed for these specific compounds....") is deleted.

Section 8.8.2.1:	The 1-2 uL injection volume referenced is correct. Actual injection volume is documented in the raw data and accounted for in the calculations. On the last line of this section, the reference to (Section) 8.7, should actually refer to (Section) 8.6 of the SOP, dealing with instrument conditions during calibration.
Section 8.8.2.5:	For the purposes of this project, the last sentence under Section 8.8.2.5 is deleted.
Section 8.8.3.1:	The 1-2 uL injection volume referenced is correct. Actual injection volume is documented in the raw data and accounted for in the calculations. On the last line of this section, the reference to (Section) 8.7, should actually refer to (Section) 8.6 of the SOP, dealing with instrument conditions during calibration.
Section 8.10.6:	Library searches of up to 20 unknown peaks will be performed.
Section 8.10.10:	Mass spectra, chromatographs, tuning and calibration data, daily standard and continuing calibration check information et., are <u>not</u> planned to be part of the normal reporting package, but are included as a part of raw data packages (not being routinely supplied). These data will be made available to regulatory agencies for auditing. Such additional data will be provided to regulatory agencies upon 30 days notice.
Section 9.5:	Matrix spike/matrix spike duplicate analysis will be performed.
Table A-1:	For a list of Appendix IX parameter reporting limits, please refer to QAPjP Appendix A-10.
Table B-5	The compounds and concentrations listed are calibration standards to be utilized.
DFTPP Ions:	DFTPP key ions and ion abundance criteria are included in Table C-1, on page 60 of 64 in the SOP.

SOP Appendix A: This SOP is generic in nature, and intended to be sufficiently flexible to address the requirements of multiple clients and/or regulatory agencies. For the purposes of this particular project, Appendix A is not applicable.

Summary tables of target compounds is found in the GWMP Section 7, pages 9 through 11, and in Table 7-1.

STANDARD
OPERATING
PROCEDURE

Subject or Title:

GC/MS Analysis of Semivolatile Organics

SOP No.: LM-RMA-3013 BNA 625	Revision No.: 1.0	Effective Date: 12/01/89

Supersedes: Original and LM-RMA-3002

1. Scope and Application

Capillary GC/MS is used to analyze a wide range of extractable organic compounds in a variety of matrices. Although the method for preparation of samples is dependent on the matrix, most aspects of the instrumental analysis are not dependent on the matrix or compounds to be determined, consequently the scope of the method may be extended to include quantitative analysis of extractable, chromatographable compounds not explicitly listed in Appendix A provided they are amenable to analysis as semivolatiles and a standard is analyzed and evaluated for those compounds.

Several Analytical Test References are contained in Appendix F. The Analytical Test References document analyses currently performed at RMAL which deviate in target compound list, analytical protocol and/or data package requirements from those specified in Appendix A and Sections 8.7 through 12.3.

The applicability of this Standard Operating Procedure LM-RMA-3013 to particular matrices and/or analytical requests not specifically stated in this S.O.P. is determined by the GC/MS department manager and communicated to the client manager. The GC/MS department manager has the authority to make modifications to the S.O.P. for certain matrices and/or analyses if his technical judgment deems it necessary and appropriate.

1.1 Analytes: Quantitative analysis is performed for the compounds listed in Appendix A. Qualitative analysis by mass spectral identification can be performed on other extractable, chromatographable compounds.

Prepared by:	Date:
[signature]	12-4-89
Management Approval:	Date:
[signature]	2/15/90
QA Officer Approval:	Date:
[signature]	8-23-90

1.2 Reporting Limits: For most compounds, the instrument reporting limit is 10 ug/mL. The extraction recovery of some compounds and consequently their detection limits depend on the sample matrix (e.g. soil, water, waste). The detection limit also depends on the characteristics of individual samples. All aspects of this S.O.P. assume target compounds will be found and quantitated at levels within the specified range of calibration for each analysis. Therefore, if a sample contains a high concentration of target compounds or a large amount of interfering material, it will be diluted prior to analysis. This increases the reporting limit proportionally to the dilution. If the sample extract is diluted to 10% (i.e. 1:10 dilution) or less, the surrogates and spiked compounds will be diluted to levels below reporting limits and will not be reported. Refer to Appendix A for HSL (Hazardous Substance List) and PP (Priority Pollutant) semivolatiles and their nominal reporting limits for each matrix. Refer to Appendix F for summaries of other common analyses performed at RMAL. Included in each Analytical Test Summary is the typical range of calibration for analytes.

1.3 Applicable matrices: Water, soil, solids, sludge, waste and TCLP leachates (Toxicity characterization leaching procedure.)

1.4 Dynamic range: Analytes can typically be quantitated between 5ug/mL and 160 ug/mL. Samples which exceed this concentration range for any analyte in Table A-1 should be diluted to be within the dynamic range of the method. Sample extracts should also be diluted when any other semivolatile component of the sample exceeds this range. Reporting limits will be adjusted to reflect the dilution performed.

1.5 Analysis time: Approximate analytical time is 50 minutes per GC/MS run. The time required for data reduction will be dependent upon the complexity of the sample.

2. Method Summary

2.1 Aqueous samples are extracted with dichloromethane (methylene chloride) into base/neutral and acid fractions. The base/neutral and acid fractions are concentrated and combined for quantitative and/or qualitative capillary GC/MS analysis.

2.2 Solid samples are extracted with an appropriate solvent. The extract is concentrated and then analyzed by capillary GC/MS for quantitative and/or qualitative identification.

3. Comments

3.1 All deviations made from this SOP should be approved by the supervisor and be well documented.

3.2 All reusable glassware should be solvent rinsed prior to use.

3.3 Care should be taken not to introduce contamination into the samples. Particular attention should be paid to selecting a protective glove that does not contribute contamination during sample preparation.

3.4 pH adjustments required for the preparation of aqueous samples are critical. Analyte recoveries can be adversely affected if the pH of the sample is not sufficiently basic or acidic.

4. Safety Issues

4.1 The toxicity or carcinogenicity of each chemical used in this procedure has not been precisely defined, however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals should be reduced to the lowest possible.

4.2 A Material Safety Data Sheet (MSDS) is available for all laboratory standard and reagent chemicals. The appropriate MSDS must be read before handling the chemical(s).

4.3 All laboratory personnel should be thoroughly familiar with the laboratory Safety Manual and Hazard Communication Standard before undertaking any laboratory work.

5. Sample Collection, Preservation, Containers and Holding Times

5.1 Grab samples must be collected in glass containers. Conventional sampling practices should be followed, except that the bottle must not

be pre-rinsed with sample before collection. Automatic sampling equipment must be as free as possible of Tygon tubing and other potential sources of contamination. Solid samples should be collected in wide mouth glass jars.

5.2 All samples must be iced or refrigerated at 4°C from the time of collection until extraction.

5.3 All aqueous samples must be extracted within 7 days of collection and completely analyzed within 40 days of extraction. Soil/waste samples must be extracted within 10 days of collection and completely analyzed within 40 days of extraction.

5.4 Refer to Enseco, Inc. QA Program Plan for Environmental Chemical Monitoring Rev. 3.3, Sections 4-5 and Appendix I for more information.

6. Apparatus

6.1 Separatory funnel - 2 L, with Teflon stopcock.

6.2 Filtering Funnel - 75 mm top diameter.

6.3 Pyrex glass wool

6.4 Disposable 1 mL pipet

6.5 Concentrator tube, Kuderna-Danish - 10 mL graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test.

6.6 Evaporative flask, Kuderna-Danish - 500 mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with springs.

6.7 Snyder column, Kuderna/Danish - Three ball macro (Kontes K-503000-0121 or equivalent).

6.8 Vials - 2 mL capacity with Teflon-lined screw cap suitable for GC autosampler.

6.9 Boiling chips - Approximately 10/40 mesh.

6.10 Water bath - Heated, with concentric ring over cover, capable of temperature control (± 2°C). The bath should be used in a hood.

6.11 Toploading balance capable of accurately weighing 0.01 g.

6.12 Analytical balance capable of accurately weighing 0.0001 g.

6.13 Spatula - stainless steel or teflon

6.14 Sonicator - A horn-type sonicator equipped with a titanium tip should be used.

6.15 Sonabox - Recommended with above disrupters for decreasing cavitation sound.

6.16 Gas Chromatograph for screening - A capillary chromatography system suitable for splitless injection and all required accessories such as syringes, analytical columns, gases, flame ionization detector, and recording device. A data system is recommended for data acquisition, integration and output.

6.17 Nitrogen evaporation device equipped with a water bath that can be maintained at 35-40°C.

6.18 Continuous liquid-liquid extractors-equipped with Teflon or glass connecting joints and stopcocks requiring no lubrication.

6.19 Capillary column. One of the following or an approved substitute should be used.

6.19.1 J&W 30 m DB-5, 0.25 mm id, 0.25 um film thickness

6.19.2 Quadrex 25 m Methy Phenyl (5%) Silicone, 0.25 mm id, 0.5 um film thickness.

6.19.3 Restec 30 m RTX-5, 0.32 mm id, 1.0 um film thickness.

6.20 Syringes and/or micro pipets - 10 uL, 25 uL, 50 uL, 100 uL, 500 uL and 1 mL, as appropriate for making injections, adding internal standard and preparing sample dilutions.

6.21 Gas Chromatograph/Mass Spectrometer

6.21.1 Gas chromatograph: An analytical system that includes a temperature-programmable gas chromatograph suitable for splitless injection including all required accessories such as syringes, columns, and gases.

6.21.2 Mass spectrometer: Capable of scanning from 35 to 500 amu every 1 sec or less, using 70 volts (nominal) electron energy in the electron impact ionization mode.

6.21.3 GC/MS interface: An interface that allows direct coupling of the capillary column and adequate analyte response.

6.21.4 Data system: A computer system must be interfaced to the mass spectrometer which will allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the analytical run. The software should be capable of producing extracted ion profiles and integrating these abundances. The EPA/NIH (NBS) Mass Spectral Library should also be available on the system.

7. Reagents and Standards

7.1 Reagent water - Carbon filtered house distilled water. Interferents not observed at or above the reporting limit of the parameters of interest.

7.2 Sodium hydroxide solution (10 N)-Dissolve 40 g NaOH in reagent water and dilute to 100 mL.

7.3 Sulfuric Acid (1+1)-Slowly add 50 mL of H_2SO_4 (ACS sp. gr. 1.84) to 50 mL of reagent water.

7.4 Acetone, methanol, methylene chloride - Pesticide quality or equivalent.

7.5 Sodium sulfate-(ACS), anhydrous. Granulated for drying extracts, powdered for extracting soil samples.

7.6 Surrogate standard spiking solution. (See Appendix B)

7.7 Laboratory Control Sample standard spiking solution. (See Appendix B).

7.8 GC calibration standard. (See Appendix B)

7.9 Internal standard solution. (See Appendix B)

7.10 Calibration standard solutions. (See Appendix B)

8. Procedure

8.1 Scheduling Samples for Extraction

8.1.1 Samples will be scheduled for extraction by the section supervisor. Samples should be scheduled for preparation according to the following priorities:

a) Meet holding times

b) Meet promised due date

8.2 Preparation of Water Samples

NOTE: ALL GLASSWARE MUST BE SOLVENT RINSED BEFORE-USE. RINSE WITH ACETONE AND METHYLENE CHLORIDE.

8.2.1 Emulsion test. This test is performed if a sample contains particulate matter. It is used to determine if the sample will be extracted using a separatory funnel or a continuous liquid-liquid extractor.

8.2.1.1 3-4 mL of sample is transferred to a 12 mL screw top glass vial with a Teflon liner.

8.2.1.2 Approximately 1 mL of CH_2Cl_2 is added. The vial should be shaken vigorously for about 30 seconds and the layers allowed to separate.

8.2.1.3 If no emulsion forms, the prep should proceed with the shakeout technique in Section 8.2.2.

8.2.1.4 If an emulsion forms, the prep should proceed with the continuous extractor technique in Section 8.2.3.

8.2.2 Separatory Funnel Extraction (Shakeout Technique)

8.2.2.1 Samples that are clearly described as ground waters in the project folder are decanted from any sediment in the bottle, all others are mixed by shaking the bottle.

8.2.2.2 If the sample is in a larger bottle or requires decanting, a 1 L graduated cylinder is used to measure the sample volume. The sample is poured into a 2 L separatory funnel.

8.2.2.3 If the sample does not require decanting and is in a 1 L or smaller bottle, the water meniscus is marked on the side of the sample bottle and used to measure the sample volume. The sample is then poured into a 2 L separatory funnel. After the sample bottle is rinsed with methylene chloride (Section 8.2.2.11), the sample volume used is measured by adding tap water to the bottle to the marked level. The volume added is measured with a graduated cylinder.

8.2.2.4 The volume of each sample is recorded on the benchsheet.

8.2.2.5 For blanks and quality control samples, 1 L of carbon filtered water is poured into the separatory funnel.

8.2.2.6 For matrix specific QC, 1 L of the appropriate sample required for each QC sample is poured into a separatory funnel. If insufficient sample is available to use at least 500 mL per aliquot, the supervisor is consulted. The volume of sample used for each portion is recorded on the benchsheet.

8.2.2.7 1 mL of the surrogate standard spiking solution is added to each sample and QC sample using a 1 mL disposable pipet, making sure the pipet tip is below the surface of the sample while the standard is being added. The sample is mixed immediately by stoppering and shaking the separatory funnel. The addition of the surrogate spiking solution is noted on the bench sheet, including who added it and the date and by whom the standard was made.

8.2.2.8 0.5 mL of the DCS or matrix standard spiking solution is added if appropriate, using the procedure described in 8.2.2.7.

8.2.2.9 The pH is checked by removing a small aliquot of the sample with a pasteur pipet, placing enough of the sample on a pH paper to thoroughly soak the paper. The pH paper is compared with the chart on the container and the initial pH is recorded on the prep sheet.

8.2.2.10 The pH is adjusted to >12 with 10 N NaOH and the adjusted pH is noted on the prep sheet.

8.2.2.11 For samples that were mixed before pouring, 60 mL methylene chloride (CH_2Cl_2) is added to the sample container to rinse the inner walls. The solvent is then transferred to the separatory funnel. For samples that were decanted, the first aliquot of CH_2Cl_2 is added directly to the separatory funnel.

8.2.2.12 The sample is extracted by shaking it for two minutes with frequent ventilation. The layers are allowed to separate. If there is an emulsion it is broken up using one of several techniques. The optimum technique will depend upon the sample, and may include: stirring, centrifugation, filtration through glass wool or other physical methods. If the emulsion cannot be broken, the sample must be transferred to a continuous extractor. See section 8.2.3.

8.2.2.13 After separation, the bottom layer (CH_2Cl_2) is drained into a 400 mL beaker.

8.2.2.14 The extraction is repeated twice more using a 60 mL aliquot of CH_2Cl_2 each time. The solvent is collected in the same beaker described in 8.2.2.13.

8.2.2.15 The pH is then adjusted to < 2.

NOTE: THIS pH ADJUSTMENT IS CRITICAL. PHENOL RECOVERIES MAY BE LOW IF THE pH IS NOT LESS THAN 2.

8.2.2.16 The extraction is repeated another three times at the acidic pH using a 60 mL aliquot of CH_2CL_2 each time. The solvent is collected in the same beaker described in 8.2.2.13.

8.2.2.17 A plug of glass wool is placed in a funnel and the funnel is filled about 2/3 full with granular Na_2SO_4. The funnel and Na_2SO_4 are rinsed with 30-40 mL of CH_2Cl_2 which is discarded after being used for rinsing.

8.2.2.18 The sample extract is poured through the Na_2SO_4 into a 500 mL Kuderna-Danish (K-D) evaporative concentrator. The beaker which contained the extract and the Na_2SO_4 in the funnel are rinsed with small amounts of CH_2Cl_2. These rinses are added to the K-D.

8.2.2.19 A boiling chip is added to the K-D and a 3- ball Snyder column is attached to the top. The column is prewet by adding about 1 mL of CH_2Cl_2 to the top of the Snyder column.

NOTE: THE CONCENTRATION STEP IS CRITICAL; LOSSES OF TARGET COMPOUNDS CAN OCCUR IF CARE IS NOT TAKEN.

8.2.2.20 The K-D is placed in a 95°C hot water bath so that the receiver tube is immersed in hot water below the joint and the entire lower rounded surface is bathed in steam. At a proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume reaches 5-10 mL, the K-D is removed from the bath and allowed to cool completely.

8.2.2.21 When the K-D has cooled, the column and flask are rinsed with a small amount of CH_2Cl_2. The extract is transferred to a calibrated 15 mL centrifuge tube, rinsing with a small amount of CH_2Cl_2. It is important to rinse all of the ground glass joints of the K-D well, as compounds collect on the ground glass.

8.2.2.22 The extract is carefully concentrated to 1.0 mL under a gentle stream of nitrogen using the N-evap apparatus. The temperature of the water bath should be 35-40°C. If the extract is highly colored, forms a precipitate, or stops evaporating, the supervisor is consulted before the procedure is continued.

8.2.2.23 The extract is transferred to a labelled autosampler vial with Teflon lined cap. The meniscus is marked and the extract is placed on a tray in the BNA extract refrigerator with any other samples having the same QC Lot number. The tray number is recorded on the outside of the prep folder containing the bench sheets.

8.2.2.24 As the final step, the prep sheet is completed for the extraction and concentration steps.

8.2.3 Continuous Extractor

8.2.3.1 The continuous extractor is set up in a hood or well-ventilated area.

8.2.3.2 250 mL of CH_2Cl_2 is placed in a round bottom flask and a few boiling chips are added. 300 mL of CH_2Cl_2 is added to the extractor flask.

8.2.3.3 500 mL of carbon filtered water is carefully poured into the extractor, minimizing the disturbance of the solvent layer and preventing water from entering into either sidearm by pouring the water down the back of the extractor.

8.2.3.4 The pH of the sample is checked by removing a small aliquot with a pasteur pipet, placing enough of the sample on a pH paper to thoroughly soak the paper. The pH paper is compared with the chart on the container and the initial pH is recorded on the prep sheet.

8.2.3.5 Samples that are clearly described as ground waters in the project folder are decanted from any sediment in the bottle, all others are mixed by shaking the bottle.

8.2.3.6 If the sample is in a larger bottle or requires decanting, a 1 L graduated cylinder is used to measure the sample volume. The sample is-then carefully poured into the extractor flask.

8.2.3.7 If the sample does not require decanting and is in a 1 L or smaller bottle, the water meniscus is marked on the side of the sample bottle and later used to measure the sample volume. The sample is carefully poured into the extractor flask. After the sample bottle is rinsed with methylene chloride (section 8.2.2.11), the sample volume used is measured by adding tap water to the bottle to the marked level. The volume added is measured with a graduated cylinder.

8.2.3.8 The volume of each sample is recorded on the benchsheet.

8.2.3.9 For blanks and quality control samples, 1 L of carbon filtered water is poured into the extractor flask.

8.2.3.10 For matrix specific QC, 1 L of the appropriate sample required for each QC sample is measured into the extractor flask. If insufficient sample is available to use at least 500 mL per aliquot, the supervisor is consulted. The volume of sample used for each portion is recorded on the bench sheet.

8.2.3.11 Sufficient carbon filtered water is added to the extractor to allow the solvent in the removable sidearm to begin to drip into the round bottom flask. It is important that the extractor stays in a strictly vertical position.

8.2.3.12 1 mL of the surrogate spiking solution is added to each sample and QC sample using a 1 mL disposable pipet, making sure that the tip of the pipet is below the surface of the liquid in the extractor as the standard is being added. The sample is mixed immediately using a glass stirring rod. The addition of the surrogate spiking solution is noted on the bench sheet, including who added it and the date and by whom the standard was made.

8.2.3.13 0.5 mL of DCS or matrix spike mix is added if appropriate, using the procedure described in 8.2.3.12

8.2.3.14 The pH is adjusted to >12 with 10 N NaOH, stirring the sample carefully with a glass rod. If an excessive amount of NaOH is used (more than about 15 mL), this is recorded along with the adjusted pH on the prep sheet.

8.2.3.15 The condenser is wiped clean with solvent at the lower joint and lip and then placed on the top of the extractor. The water chiller (set at 10°C) is turned on and flow indicators checked.

8.2.3.16 The heating mantle beneath the round bottom (rb) flask is turned on and the extraction starting time is recorded on the bench sheet. The extractor is checked after 15 minutes to ensure it is operating correctly and that there are no leaks.

8.2.3.17 The extraction is allowed to proceed for a minimum of eighteen hours.

8.2.3.18 The heating mantle is turned off and the apparatus is allowed to cool (30-60 minutes) with water flowing through the condenser.

8.2.3.19 The solvent contained in the round bottom flask is the base/neutral extract. The flask is removed, capped, refrigerated and replaced with a clean round bottom flask containing 250 mL CH_2Cl_2 and a few boiling chips.

8.2.3.20 The condenser is removed and the pH is adjusted to < 2.

NOTE: THIS pH ADJUSTMENT IS CRITICAL. PHENOL RECOVERIES MAY BE LOW IF THE pH IS NOT LESS THAN 2.

8.2.3.21 Steps 8.2.3.16-8.2.3.19 are then repeated for the acid extraction.

8.2.3.22 The solvent contained in the round bottom flask is the acid fraction. The round bottom flask is removed, capped and refrigerated.

8.2.3.23 The contents of the extractor are poured into a separatory funnel to separate the water from the methylene chloride. The water layer from the extractor flask is discarded into the sink with plenty

of cold tap water. The solvent layer from the extractor flask is discarded into a contaminated waste bottle.

8.2.3.24 The drying and concentration steps are similar to those described in section 8.2.2.17 to 8.2.2.18. Due to the larger volume of solvent the base/neutral fraction must be concentrated first to an apparent volume of 10-15 mL. Then a new boiling chip is added and the acid fraction is poured through Na_2SO_4 into the same K-D. The concentration is then completed as described in sections 8.2.2.19 to 8.2.2.23.

8.2.3.25 As the final step, the prep sheet is completed for the extraction and concentration steps.

8.3 Preparation of Soil Samples

8.3.1 Percent Moisture Determination (optional)

5-10 g of the sample is added to a tared weighing dish. The wet weight of the sample is recorded. The weighed sample is allowed to dry overnight at 105°C and then reweighed. The dry weight of the sample is recorded, correcting for weight of the weighing dish. The percent moisture of the sample is then calculated using equation 10.2.7.

8.3.2 pH Determination (optional)

50 g of sample is transferred to a 100 mL disposable beaker. 50 mL of water and a teflon-coated magnetic stirring bar are added and the beaker is stirred for one hour on a magnetic stir plate. The pH of the sample is determined with glass electrode and pH meter while stirring. The pH value is reported on appropriate data sheets.

8.3.3 Extraction by Sonication Technique

NOTE: ALL GLASSWARE MUST BE SOLVENT RINSED BEFORE USE. RINSE WITH ACETONE AND METHYLENE CHLORIDE.

8.3.3.1 Any water layer on a sediment sample is discarded. Samples are mixed thoroughly, discarding any foreign objects such as sticks, leaves and rocks.

8.3.3.2 Approximately 30 g (to the nearest 0.1 g) of sample is weighed into a 250 mL bottle. 60 g of anhydrous powdered sodium sulfate is added and mixed well. 100 mL of 1:1 methylene chloride-acetone is added to the sample, followed by 0.5 mL of surrogate spiking solution. The addition of the surrogate spiking solution is noted on the bench sheet, including who added it and the date and by whom the standard was made.

8.3.3.3 1.0 mL of the DCS or matrix spike mix is added, if appropriate, using the procedure described in Section 8.3.3.2.

8.3.3.4 The bottom surface of the tip of the sonicator horn is placed about 1/2 inch below the surface of the solvent but above the sediment layer.

8.3.3.5 The sample is sonicated for 1 1/2 minutes.

8.3.3.6 The solvent layer is decanted into a rinsed 400 mL beaker.

8.3.3.7 The extraction is repeated twice more using 100 mL aliquots of 1:1 CH_2Cl_2-acetone for each extraction making certain that the sodium sulfate is free flowing and not a consolidated mass. As required, large lumps are broken up with a clean spatula. These extracts are collected in the the same beaker described in 8.3.3.6.

8.3.3.8 A funnel is plugged with glasswool and filled 2/3 full with Na_2SO_4.

8.3.3.9 A boiling chip is added to the K-D and a 3- ball Snyder column attached to the top. The column is pre-wet by adding about 1 mL of CH_2Cl_2 to the top of the Snyder column.

NOTE: THE CONCENTRATION STEP IS CRITICAL; LOSSES OF TARGET COMPOUNDS CAN OCCUR IF CARE IS NOT TAKEN.

8.3.3.10 The K-D is placed in a heated water bath set at 95°C so that the receiver tube is immersed in hot water below the joint and the entire lower rounded surface is bathed in steam. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume reaches 5-10 mL, the K-D is removed from the bath and allowed to completely cool.

8.3.3.11 After the K-D has cooled, the Snyder column and flask are rinsed with a small amount of methylene chloride. The extract is transferred to a calibrated 15 mL centrifuge tube, rinsing the K-D flask with a small amount of methylene chloride. It is important to rinse all of the ground glass joints of the K-D well, as compounds collect on the ground glass.

8.3.3.12 The extract is carefully concentrated to 1.0 mL under a gentle stream of nitrogen using the N-evap apparatus. The temperature of the water bath should be 35-40°C. If the extract is highly colored, forms a precipitate, or stops evaporating, a supervisor is consulted before the procedure is continued.

8.3.3.13 The extract is transferred to a labelled autosampler vial with Teflon lined cap. The meniscus is marked and the extract is placed on a tray in the BNA extract refrigerator with any other samples having the same QC Lot number. The tray number is recorded on the outside of the prep folder containing the bench sheets.

8.3.3.14 The prep sheet is completed for the extraction and concentration steps.

8.3.3.15 If GPC cleanup is required, see section 8.5.

8.3.4 Other Extraction Techniques

8.4 Preparation of Sludge Samples

8.4.1 Approximately 10 g of sample is weighed into a pre-weighed aluminum weigh boat and allowed to air-dry overnight. The percent moisture is determined using equation 10.2.7.

8.4.2 A continuous extractor is set-up as described in Sections 8.2.3.1 to 8.2.3.4.

8.4.3 Weigh the bottle containing the sample and carefully transfer {insert approximate amount of sample used} g of the sample to the extractor flask. Re-weigh the bottle, calculate the difference, and record it as the amount extracted on the prep sheet.

8.4.4 The continuous extractor method is followed as described in Sections 8.2.3.9 to 8.2.3.26.

8.5 Screening Extracts

8.5.1 Chromatographic conditions.

8.5.1.1 Column: 15 m DB-5, 0.25 mm id, 0.25 um; or equivalent

8.5.1.2 Temperature Program: 50°(1)-20°/min-280°C

8.5.2 Inject 1 uL of the GC Calibration Standard on the screening GC. This solution contains 50 ug/mL of phenol, phenanthrene, and di-n-octyl phthalate. The response of phenanthrene should be approximately 50% full scale deflection (FSD). The response of di-n-octyl phthalate should be > 25% FSD.

8.5.3 For water samples screen only if the extract is colored. If the extracts were not combined during the concentration step, screen both fractions. If there are no non-surrogate peaks greater than 400% of the peak height of the phenanthrene peak in either fraction, submit as is. If either fraction has peaks greater than 400% of the peak height of the phenanthrene peak, dilute both fractions so that the largest peak is approximately four times the peak height of phenanthrene, i.e., both fractions will always have the same dilution factor. Note the dilution factor on the benchsheet.

8.5.4 If acid and base/neutral fractions were not combined in the concentration step, combine 0.5 mL aliquots of each fraction in one autosampler vial. Clearly label as BNA with the sample number.

8.5.5 For solid samples screen the extract at an appropriate volume (i.e. a clear extract may be screened at 1 mL; a dark extract at 10 mL.) Adjust final volume such that the largest non-surrogate peak is between 200% and 400% of the phenanthrene peak. Note the dilution factor on the benchsheet.

8.5.6 Put aliquot of any dilutions performed in a labeled autosampler vial. If any of the undiluted extract remains, label and store this autosampler vial with the dilution.

8.6 Instrument Set-Up for the GC/MS

8.6.1 Chromatographic Conditions

8.6.1.1 Column: See section 6.19.

8.6.1.2 Temperature Program:

Initial Temperature:	30°C
Initial hold time:	1 minute
Temperature ramp:	10°C/min
Final temperature:	325°C
*Final hold time:	10 minutes

*The final hold time should be adjusted according to the retention time of the final compound in the standard.

8.6.1.3 These parameters are verified and/or changed using the DESC procedure for the Finnigan 4500's or GC method for the Finnigan Incos 50 instruments.

8.6.2 Mass Spectrometer Parameters:

8.6.2.1 Mass range: 35-500 amu

8.6.2.2 Scanning rate: 0.75 second/scan

8.6.2.3 Zone temperatures:

Injection port:	250°C
Transfer line:	275°C
Manifold temperature:	100°C
Source temperature: (if applicable)	150°C

8.6.3 Acquisition Parameters (Program ACQU)

Consult your supervisor if your acquisition parameters differ from the following:

minimum peak width	= 3
minimum area	= 30
threshold	= 1

8.7 File Naming Conventions

For each semivolatiles instrument, the filenames for DFTPP, calibration standards, QC samples and client samples acquisitions are based on the use of common prefix abbreviations followed by instrument letter designation and a consecutive integer progression from 001 to 999. This numeric portion of the filename is commonly referred to as the "run number". When the run number on a particular instrument reaches 999, the progression begins again with 001.

Filenames are recorded in the instrument log book at the time of data acquisition along with other pertinent sample information (e.g. project & sample number, extract dilution, and analyst's initials and date of acquisition).

8.7.1 The filename for DFTPP uses the prefix DF followed by instrument letter designation and run number (next consecutive integer - up to 999 - in instrument log book).

For Example:

DFU006 is the filename for DFTPP analyzed on instrument U at run number 006 in the consecutive integer count maintained in the log book for instrument U.

8.7.2 The filenames for standards use the prefix STD followed by instrument letter designation and run number (next consecutive integer - up to 999 - in instrument log book).

For Example:

STDU007 is the filename for a standard analyzed on instrument U at run number 007 in the consecutive integer count maintained in the log book for instrument U.

8.7.3 The filenames for QC samples (Refer to Section 9. for a discussion of DCS and Blank QC Samples) use either the prefix DCS or Blank, depending on the type of QC sample, followed by instrument letter designation and run number (next consecutive integer - up to 999 - in the instrument log book).

For Example:

DCSU008 is the filename for a Duplicate Control Sample (DCS) analyzed on instrument U at run number 008 in the consecutive integer count maintained in the log book for instrument U.

8.7.4 The filenames for client samples use the prefix S followed by the RMA project number, instrument letter designation and run number (next consecutive integer - up to 999 - in the instrument log book).

For Example:

S6450U009 is the filename for a sample belonging to RMA project number 6450, run on instrument U at run number 009 in the consecutive integer count maintained in the log book for instrument U.

8.8 Instrument Calibration

8.8.1 DFTPP

8.8.1.1 Each GC/MS **MUST** be hardware tuned to meet the EPA ion abundance criteria for DFTPP. The instrument tune **MUST** be verified at the start of every 12 hours of operation or as specified by client contract. Ion abundance criteria are listed in Appendix C, Table C-1.

8.8.1.2 50 ng of DFTPP is directly injected into the GC using the following chromatographic conditions:

Initial temperature:	175°C
Initial hold:	1 min
Rate:	35°C/min
Final temperature:	250°C

DFTPP will generally elute between scans 250-450.

8.8.1.3 The spectrum of DFTPP is evaluated for overall intensity and ion abundances. Ion abundance criteria given in Table C-1 must be met. Averaging scans across the peak and reasonable background subtraction is acceptable. Manipulations which will distort the spectrum, such as excessive background subtraction, are not acceptable.

8.8.1.4 If the ion abundance criteria are not met, the instrument must be retuned and DFTPP reinjected.

8.8.1.5 If the ion abundance criteria are met, daily instrument calibration may proceed. Hardcopies of the DFTPP spectrum, mass list and ion abundance summary are kept on file for QC purposes.

8.8.2 Initial Calibration

An initial calibration must be performed before analysis of sample extracts can begin. This consists of a five point calibration curve for all of the analytes listed in Appendix A. Standard solutions are prepared at concentrations of 20 ug/mL, 50 ug/mL, 80 ug/mL, 120 ug/mL and 160 ug/mL.

Certain compounds have been selected to monitor instrument performance. These compounds are identified as System Performance Check Compounds (SPCC) and Calibration Check Compounds (CCC).

The average response factor for each System Performance Check Compound (SPCC) MUST be greater than 0.05.

The percent relative standard deviation (%RSD) for or equal to each Calibration Check Compound (CCC) MUST be less than or equal to 30%. The %RSD for all compounds should also be reviewed. Any values greater than 50% should be evaluated before analyses are continued.

If samples are NOT being analyzed for these specific compounds, the criteria for these compounds need not be met. The fact that these compounds are not in the analyte set should be documented.

See Table C-2 for a summary of CCC and SPCC criteria.

8.8.2.1 The following five standard solutions are analyzed: 50 ug/mL, 20 ug/mL, 80 ug/mL, 120 ug/mL and 160 ug/mL.

For each standard to be analyzed, a 1-2 uL injection is made under the conditions specified in 8.7.

8.8.2.2 Immediately following the analysis of the 50 ug/mL standard, the data acquired should be processed and evaluated to ensure that certain criteria are met before proceeding with initial calibration.

8.8.2.2.1 To minimize saturation in the higher level standards, the peak area of the quantitation ion for the 1st internal standard, d4-1,2-dichlorobenzene, should be between 10,000 and 30,000 counts, depending on the instrument. If the peak area is outside these limits, the analyst should adjust the electron multiplier and re-inject the 50 ug/mL standard. (DFTPP criteria must first be met under the new instrument operating conditions. See Section 8.9.1).

8.8.2.2.2 The SPCC limits **MUST** be verified. If the response factor of an SPCC compound is <0.05, a supervisor should be consulted to determine if corrective action will be taken.

8.8.2.3 The following target compounds are at 100 ug/mL in the 50 ug/mL standard:

Benzidine
3,3'-Dichlorobenzidine

8.8.2.4 The concentration of acid surrogate compounds is 100 ug/mL. The concentration of base/neutral surrogate compounds is 50 ug/mL. The concentration of internal standard compounds is 40 ug/mL.

8.8.2.5 The quantitation list is reviewed to ensure that the internal standards are correctly identified and that the analytes are properly quantitated. The following compounds should be checked for proper integration:

Benzyl Alcohol
Benzoic Acid
4-Nitrophenol
4-Nitroaniline
Benzo(b &/or k)fluoranthene
Indeno(1,2,3-cd)pyrene
Dibenzo(a,h)anthracene
Benzo(g,h,i)perylene

These compounds do not chromatograph well, particularly as a column is used.

8.8.2.6 Structural isomer pairs are checked for correct peak assignment. Identification is based on elution order, as they appear in the library. These pairs are:

2,4,6-/2,4,5-Trichlorophenol
Anthracene/Phenanthrene
Chrysene/Benzo(a)anthracene
Benzo(b)/Benzo(k)fluoranthene

8.8.2.7 If the standard evaluation criteria have been met, the component libraries are updated with the response factors and retention times of the 50 ug/mL standard just analyzed.

8.8.2.8 Each of the other standards analyzed should be evaluated in the same manner as the 50 ug/mL standard with particular attention focused on the "troublesome" compounds noted in Sections 8.9.2.5 and 8.9.2.6. Some additional compounds in the 120 and 160 ug/mL standards may need to be manually quantitated due to poor chromatography at higher concentrations.

8.8.2.9 Following analysis and evaluation of the 5 calibration standards, a table which lists each standard's response factors, the average RF and % RSD's for all compounds is generated using the RMA program RFAC. Refer to Appendix E for examples of the initial

calibration table and a continuing calibration table. The % RSD's for CCC compounds and the average response factors for SPCC compounds are then evaluated. Compounds having % RSD's greater than 30% are checked to verify accuracy of quantitation for each standard. Individual response factors which differ greatly from the compound average are also investigated.

8.8.2.10 If all CCC and SPCC criteria are met, the data are stored in an initial calibration file. This file is then referred to during continuing calibration. Quan lists are then copied to unit 1.

8.8.3 Continuing Calibration

The 50 ug/mL standard is used as the continuing calibration standard. It MUST be analyzed for each 12 hour period immediately following a successful DFTPP analysis.

The response factor of each CCC compound should be compared to the initial calibration average response factor. A table which lists the initial calibration average response factors, continuing calibration response factors and % differences for all compounds is generated for comparison using the RMA procedure RFAC. See Appendix E for an example of this table. The percent difference for each CCC compound should be less than or equal to 25% for analysis to proceed. Deviations from the specified criteria for continuing calibration are allowed, provided a supervisor is informed and rationale is well documented. Response factors for all analytes should be reviewed. Any significant deviations from the initial calibration should be evaluated before analyses are continued. (e.g. deviations of greater than 50%)

All SPCC criteria should be met (i.e. RF > 0.05).

8.8.3.1 A 1 or 2 uL injection of the 50 ug/mL standard is made under the conditions specified in Section 8.7.

8.8.3.2 The 50 ug/mL continuing calibration standard should be evaluated and quantitation checked as outlined in Sections 8.9.2.2 through 8.9.2.6.

8.8.3.3 The continuing calibration standard is compared to the initial calibration curve by generating the table described in Section 8.9.3. The CCC and SPCC compounds are evaluated as outlined in Section 8.9.3. If any large percent differences between initial and continuing calibration response factors are noted, the peak assignment and integration for these compounds should be checked.

8.8.3.4 If the standard evaluation criteria are NOT met, and analyses to be performed require meeting SPCC & CCC criteria then corrective action must be taken. Corrective action may include replacement of injection port liner, cutting a portion off the front end of the column, or replacing the column. If corrective action is taken, the instrument must be recalibrated beginning with injection of DFTPP (See Section 8.9.1.2 through 8.9.1.3). Following successful analysis of DFTPP, the 50 ug/mL continuing calibration standard is reinjected. If deviations between initial and continuing calibrations still exist, a new initial calibration may need to be performed (See Section 8.9.2).

8.8.3.5 If these criteria are not met and the decision is made to continue analysis, the rationale must be documented by the individual making that decision. This documentation must be kept on file with the continuing calibration record.

8.8.3.6 If the standard evaluation criteria have been met or the decision has been made to continue, the component libraries are updated with the response factors and retention times of the continuing calibration standard just analyzed.

8.9 Sample Analysis

8.9.1 Scheduling

8.9.1.1 Analyses are scheduled by the supervisor or a senior operator and assigned to analysts on a daily basis.

8.9.1.2 A LIMS generated semivolatile sample backlog is used for prioritizing sample analyses and for recording the status of analyses, (e.g. prep completed, analysis in progress, analysis completed). The analytical test (i.e. target compound list) required is indicated on the sample backlog. Modifications to the normal analytical protocol, such as low detection limits, special compounds or special criteria are also flagged on the backlog and pertinent information is accessed by the analyst or supervisor via LIMS.

8.9.2 Extract Preparation

8.9.2.1 Extracts are located in numbered trays stored in the BNA extract refrigerator. The tray number for a given set of samples is recorded on the project folder for those samples.

8.9.2.2 Appropriate sample extract vials are located by the analyst and placed in the semivolatiles hood for aliquoting. Acid and B/N fractions are combined if necessary and the internal standard solution (400 ug/mL) is added to each extract aliquot at a ratio of 1:10 resulting in I.S. concentration of 40 ug/mL in the extract. The vial is then marked with the letters "WIS" (with internal standard) to indicate the addition of internal standard.

8.9.2.3 The syringe is rinsed with CH_2Cl_2 between each use.

8.9.2.4 The prepared extracts are then transferred to the appropriate instrument for analysis. If the samples are to be acquired using an autosampler (rather than being injected and acquired manually by the analyst), the vials are placed in the autosampler (either HP or Varian) rack as per the manufacturer's instructions.

8.9.3 Data Acquisition

8.9.3.1 Samples are analyzed using the same instrument parameters as the calibration standards. (conditions specified in Section 8.7)

8.9.3.2 Sample data can be acquired manually as a single run (analysis) or as a series of runs in an automated sequence of samples. Two custom RMA procedures programs which utilize the Finnigan ACQU command are used for data acquisition. The RMA procedure DOIT allows the analyst to set acquisition parameters and input sample header information for manual acquisition of a single sample. The RMA procedure ATAQ enables the analyst to set acquisition parameters and input sample header information for a series of samples. The ATAQ procedure automatically coordinates sample acquisition with the autosampler, the GC ready condition, and sample header information and filename. The analyst is responsible for making sure extract vials are placed in the autosampler rack in the order intended for analysis and that sample header information and filenames are loaded into the computer in the correct sequence as well.

8.9.3.3 If a limited list of compounds is being used, the analysis time may be shortened, as appropriate to the compounds being analyzed.

8.9.3.4 Filenames, sample identification information (project number and sample number, e.g. 6450-02), extract dilution, analyst's initials and date of analysis are recorded by the analyst in the instrument log book.

8.9.4 Analysis Sequence

8.9.4.1 QC samples (refer to Section 9 for a discussion of DCS and blank samples) should be analyzed and QC criteria met before client samples from that QC Lot number can be analyzed. See Section 9 for QC criteria for DCS and blank samples.

8.9.4.2 If QC criteria are met, client samples will be analyzed according to scheduling priority. Matrix spikes, matrix spike duplicates, and sample duplicates when available are analyzed in conjunction with the associated samples.

8.9.4.3 If the QC criteria are not met, the supervisor is informed and the decision to reprep samples from the QC lot or to continue analysis is made and documented. (See Section 9.)

8.10 Data Processing & Reduction & Data Package Assembly

"The analyst who generates the analytical data has the prime responsibility for the correctness and completeness of the data." Quote from Enseco, Inc. QAPP for Env. Chem. Monitoring, Rev. 3.3.

For a general overview of Enseco data reduction, validation and reporting guidelines from a quality assurance standpoint refer to Enseco, Inc. QAPP for Env. Chem. Monitoring, Rev. 3.3, Section 8.

8.10.1 Following sample data acquisition, each sample data file is processed using the RMAL procedure TC which utilizes Finnigan AUTOQUAN software and semivolatile component libraries developed by RMAL. A number of "Library Lists" - subsets of the semivolatile libraries - reside on each instrument computer and are selected by the analyst for use in conjunction with the procedure TC to generate a data package for each sample that specifically covers the target compound list (analytical test) requested by the client. In addition to processing one data file at a time using TC, a batch of data files can be processed

automatically using the RMAL procedure AUTOB. For more detailed instructions on using the RMAL procedure "TC" to process data files, refer to "RMAL GC/MS Software Manual" (Ver. 6/28/88).

8.10.2 The data package generated contains the following print outs:

8.10.2.1 **Log Page**

Included on this page are the header information, GC conditions, scan parameters, and instrument tuning parameters that were used at the time of data acquisition.

8.10.2.2 **Chromatogram**

Included on this page is the header information and the reconstructed ion chromatogram displaying the range of scans used during data acquisitions.

8.10.2.3 **Diagnostic Report**

Included on this page is a table of library entry numbers and chromatographic and library search information used in the computerized target compound evaluation. In addition, if any peak found is saturated, this is flagged on the diagnostic report.

8.10.2.4 **Quantitation Report**

Included in this section is the list of target compounds, internal standards and surrogate standards used for processing the datafile and their corresponding library entry numbers. This section contains (1) chromatographic information such as retention times (scan numbers) and relative retention times and (2) quantitation information such as peak areas, updated library response factors, and quantitated amounts for the library entries.

8.10.2.5 **Quantitation Summary**

This summary page lists the internal standards in elution order, followed by base/neutral surrogates in elution order, acid surrogates in elution order, and then all target compounds searched for by the processing routine, also in elution order. Tabulated with each compound are the library entry number, quantitation mass, indication of reference internal standard, and for those compounds with amounts calculated to be > 1.0 ug/mL, peak area, scan number (directly related to R.T.) at which peak maximized, calculated response factor and quantitated amount. Those compounds not found by the processing routine or with amounts calculated to be < 1.0 ug/mL are flagged with the words "not found".

8.10.3 For each processed data file the following steps should be taken:

8.10.3.1 Internal standard retention times should be reviewed and compared to the daily (continuing calibration) standard. No internal standard retention time should vary more than 30 seconds from the daily standard. If this occurs, the chromatographic system should be checked and corrections made as necessary. Affected samples will be reanalyzed.

8.10.3.2 Internal standard areas should be reviewed and compared to the daily standard. An internal standard comparison report should be generated using the RMA program "ISCHK" and placed permanently into the log book. The areas should not vary by more than a factor of two (50% to 200% of daily standard internal standard area) from the daily standard. If the areas do vary by more than a factor of two, the analyst should first check to see if the peak has been correctly integrated. If the area is still outside

the limits, the system should be checked and corrected if necessary. The sample should be reanalyzed once problems with the GC/MS system have been corrected. If, upon verification that the instrument is operating properly, the areas are still outside of the limits both analyses will be submitted with the data package and the actions taken documented by the analyst.

8.10.3.3 Surrogate standard amounts should be reviewed to verify acceptable surrogate recoveries. If any one acid and/or base/neutral surrogate falls outside the acceptable recovery limits, the quantitation should be checked.

8.10.3.3.1 If the quantitation is correct, the unacceptable surrogate recovery and affected sample(s) should be shown to a supervisor. Once a decision is made to use the data or submit for a reprep, all action should be documented on the anomaly sheet.

8.10.3.3.2 If more than one acid and/or base/neutral surrogate is outside the acceptable recovery limits, the quantitation should be checked. If the quantitation is correct, the analyst should inform a supervisor and the affected sample(s) may be submitted for repreparation. The analyst should document all action taken on the anomaly sheet used for analysis.

8.10.3.4 The same general guidelines outlined in sections 8.9.2.5 and 8.9.2.6 regarding "troublesome" compounds and structural isomers should be followed whenever any of these compounds are found in a sample by the processing routine.

8.10.3.5 Target compound amounts should be reviewed to verify that the quantitated amounts are within the upper linear range of calibration (160 ug/mL +/- 10%).

8.10.3.5.1 If any target compound amounts appear to be greater than the upper limit, the quantitation should be checked by the analyst. If quantitation is correct, the sample extract must be diluted appropriately and reanalyzed for the target compound involved. The initial run may be used for any target compound data which fall within the linear range of calibration (10 ug/mL - 160 ug/mL).

8.10.3.5.2 The diagnostic report (see Section 8.11.2.3) should also be reviewed for any peak saturation of target compounds. If any target compound peak is saturated, the quantitation should be verified by the analyst and the sample extract diluted appropriately and reanalyzed.

8.10.4 Spectra Verification

The data processing routine (TC) generates a "scan list" file for each data file which contains the scan numbers for all target compounds found in the sample at amounts > 1.0 ug/mL (i.e. all target compounds listed on the data processing quantitation summary, Section 8.11.2.5). Following initial processing of a datafile, the RMAL procedure NDUAL is used to hardcopy the unenhanced and enhanced spectra for all scans in the scan list. In addition to hardcopying spectra for one data file at a time using NDUAL, a batch of data files' spectra can be hardcopied automatically using the RMAL procedure AUTOB.

Prior to qualitative identification of target compounds in a sample, "standard" mass spectra of known identity should be obtained on the instrument(s) used for sample analysis. Hardcopies of mass spectra from a calibration standard (e.g. 50 ug/mL continuing calibration standard) run on the instrument under similar tuning conditions would serve this purpose.

8.10.4.1 Each mass spectrum generated for a sample data file should be compared to the standard spectrum. EPA criteria for qualitative identification by comparison of mass spectra should be followed. The EPA criteria for comparing spectra are:

8.10.4.1.1 All ions present in the standard spectrum at a relative intensity greater than 10% of the base peak MUST be present in the sample spectrum.

8.10.4.1.2 The relative intensities of the ions specified in (1) must agree within plus or minus 20% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%.)

8.10.4.1.3 Ions greater than 10% in the sample spectrum, but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. If the analyst can easily identify a co-eluting compound (e.g. an alkane or a surrogate spike compound) then the sample spectrum should be labeled with the contaminant.

8.10.4.2 When a spectra meets the criteria listed in Section 8.11.4.1 the compound name in the header should be circled. All spectra that do not meet the EPA criteria should have the compound name crossed out in the header, and the corresponding entries on the quantitation summary should be crossed out (line out the amounts calculated for those compounds which are not "hits") by the analyst making the judgment. If a compound cannot be verified by all of the criteria listed in 8.11.4.1, but in the technical judgment of the analyst the identification is correct, the compound should be reported. If the analyst is uncertain of an identification, a senior operator or a supervisor should be consulted.

8.10.5 Summary Sheets

A number of summary sheets are computer generated at the instrument following data file processing and pulling (hardcopying) target compound spectra. These summaries include:

8.10.5.1 **Target Compound Summary**
(also referred to as "sum sheets" or "sums")

This one-or two page summary is generated with the RMAL procedure "SUM1" and tabulates pertinent information about a sample analysis including header information (e.g. sample number, dilution used for analysis, sample matrix), date analyzed, analyst's initials, run factor (refer to calculations in Section 10.1.1), final reporting units, surrogate % recoveries, target compounds listed in elution order, and target compound CAS #'s when available. For those target compounds with amounts > 1.0 ug/mL in the extract the scan number, amount listed on quantitation summary (i.e. concentration in the analyzed extract ug/mL) and concentration in sample (expressed in final reporting units, usually ug/l for waters and ug/kg for soils) are tabulated.

8.10.5.2 **DCS Summary**
(Refer to Section 9 for discussion of DCS QC samples)

This one-page summary is generated with the RMA procedure DCS and tabulates pertinent information about the DCS samples representing a specific QC lot. The information tabulated includes QC lot number, matrix (soil or water), date analyzed, analyst's initials, DCS data file numbers, list of samples belonging to the QC lot, names of spike components and QC limits for spike % recoveries and RPD's, calculated amount and percent recovery for each spike compound in both DCS samples, and calculated RPD between the two

DCS recoveries for each spike component. Percent recoveries and RPD's that fall outside the acceptable QC limits are flagged with asterisks and require further action by the analyst (see Section 9). When generating the summary sheet it is imperative that the analyst input the complete list of samples belonging to the QC lot for QC tracking purposes. This information can be found in the QC summary notebook kept in the prep lab.

8.10.5.3 SCS Summary

(Also referred to as "SCS Summary"- see section 9 for discussion of SCS QC samples)

This one page summary tabulates pertinent information about the SCS sample representing a specific analytical lot (also called "QC" or "SCS" lot) of client samples including QC lot number, matrix (soil or water), date analyzed, analyst's initials, SCS data file number, list of samples belonging to the SCS lot, names of surrogates and QC limits for surrogate % recoveries, and calculated amount and % recovery for each surrogate. Percent recoveries that fall outside the acceptable QC limits are flagged with asterisks and require further action by the analyst (see Section 9). When generating this summary sheet, it is imperative that the analyst input the complete list of samples associated with the SCS sample for QC tracking and data reporting (blank subtraction) purposes. This information can be found in the QC summary notebook kept in the sample preparations laboratory.

8.10.6 Library Searches (Optional)

Some data packages will include mass spectral library searches on non-target compounds (excluding internal standards, surrogates and spikes) as outlined in contract requirements or requested by the client. If the library searches (also referred to as "TIC's" or Tentatively Identified Compounds) are

necessary to the data package, the 20 largest unidentified peaks that are greater than 10% of the peak (RIC) area of the nearest uninterfered internal standard will be searched against the NBS library for possible matches.

8.10.6.1 The generation of TIC data is partially automated through the use of several RMA procedures which perform the following functions:

1. Select non-target, unidentified peaks from the chromatogram for tentative identification,

2. Quantitate unknown peaks using RIC area of closest uninterfered internal standard as reference peak and a response factor of 1.0 (See calculations, Section 10.2.11),

3. Perform and generate hardcopy mass spectral library searches on the unknown peaks (searching against the NBS library of mass spectra),

4. Select preliminary "Best Match" spectra from the library searches and tabulate them with quantitation information for all unknowns,

5. Allow editing of computer's final selections for compound names,

6. Allow manual quantitation of unusually shaped peaks,

7. Generate final summary with amounts calculated to reflect concentration in sample (i.e., run factor applied).

8.10.6.2 Listed below, in the order that they are generally used by the analyst, are the RMA procedures used for TIC generation and their main functions:

TICP - Used to verify and/or change peak selection criteria (e.g. can specify to search all unknown peaks >10% internal standard RIC area.

TIC1 - Generates TIC data (hardcopies of library search spectra and quantitation information page) for one data file.

TIC - Generates TIC data (hardcopies of library search spectra and quantitation information page) for a batch of data files.

TICQS- Allows for manual quantitation by the analyst.

TICE - Allows the analyst to edit the names of compounds selected and generates final TIC summary page after input from the analyst.

8.10.6.3 After reviewing each library search spectrum, the analyst should indicate the compound name selected by labelling the spectrum or circling the correct name (if it appears on the library search spectrum as one of the computer's selections). If the compound is to be reported as "Unknown" (See Section 8.11.6.5) the spectrum should be labelled as such.

8.10.6.4 The EPA criteria to be used for making these decisions for tentative identifications are as follows:

8.10.6.4.1 Relative intensities of major ions present in the library spectrum at a relative intensity greater than 10% of the base peak should be present in the sample spectrum.

8.10.6.4.2 The relative intensities of the ions specified in (1) must agree within plus or minus 20% between the library and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance should be between 30% and 70%.)

8.10.6.4.3 Molecular ions present in the library spectrum should be present in the sample spectrum.

8.10.6.4.4 Ions present in the sample spectrum, but not present in the standard spectrum should be considered and accounted for by the analyst making the comparison. If the analyst can easily identify a co-eluting compound (e.g. an alkane or a surrogate spike compound) then the sample spectrum should be labelled with the contaminant.

8.10.6.4.5 Ions present in the library spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting compounds.

8.10.6.5 If in the technical judgment of the analyst, no valid tentative identification can be made, the compound should be reported as "Unknown." If possible, assign additional compound class information (e.g. unknown hydrocarbon). The analyst should consult with a more experienced analyst or a supervisor if there are questions concerning compound identification.

8.10.6.6 For more detailed instructions on the generation of TIC data using the RMA TIC procedures, refer to "RMA GC/MS Software Manual", version 6/28/88.

8.10.7 Reconstructed Ion Chromatogram

The RIC generated by data file processing should be labelled to identify internal standards, surrogates, spikes if applicable, target compounds and tentatively identified compounds (if library searches are performed - see Section 8.11.6). For industrial, non-contract work, the abbreviations IS - internal standard, SS - surrogate standard, SP or SC - spike compound, TC - target compound, and TIC - tentatively identified compound, are sufficient. In contract work the internal standards and surrogates are usually labelled with the compound names; the abbreviations SC, TC and TIC are normally sufficient for contract work for spike compounds, target compounds and tentatively identified compounds provided a legend is clearly indicated on each chromatogram page. Refer to Appendix F for more specific criteria.

8.10.8 Anomaly Sheet

Each data package submitted by the analyst for secondary review must include an anomaly sheet summarizing any problems or unusual circumstances incurred during sample analysis. If no problems were encountered during analysis, the analyst must still submit an anomaly sheet to that effect. All anomaly sheets must be singed by a supervisor.

8.10.9 QC Summary Sheets (DCS & SCS)

Each data package submitted for secondary review must include copies of DCS and SCS summary sheets for all QC related to the sample set in the data package.

8.10.10 Summary of Data Package Requirements

When initial data review has been completed by the analyst, the data package should be assembled to include the following:

- Anomaly Sheet
- DCS Summary Sheet
- SCS Summary Sheet
- SCS Target Compound Summary Sheet, TIC Summary and Data
- Out-of-Control forms (if necessary - See Section 9)
- Sample Raw Data
- Sample Target Compound Summaries (Attached to sample raw data)
- TIC Data & Summary (when requested)

In some cases, contract requirements or client requests will necessitate including tuning and calibration information (i.e. DFTPP), daily standard raw data and initial & continuing calibration response factor tables. Refer to Appendix F for specific data package criteria.

8.10.11 Upon completion of the data package assembly and review, the data package is passed on to the data review specialist for secondary review.

9. QA/QC Requirements (See SOP no.: M-EQA-002)

9.1 DCS. Laboratory Control Samples are extracted and analyzed for every 20 samples. DCS components and spike concentrations are given in Appendix D. For aqueous samples, DCS components are spiked into organic-free water. For solid samples, the DCS components are spiked into the extraction solvent. No solid matrix (sand or Celite) is incorporated since there is no true representative solid matrix.

9.2 SCS. A Surrogate Control Sample is analyzed with every analytical lot. This sample serves as the method blank. An analytical lot for extractable organics is defined as samples extracted or prepared at the same time up to a maximum of 20 samples. An SCS sample is also referred to as an SCS.

SCS components and spike concentrations are given in Appendix D.

9.3 Blank. For this method the blank and the SCS are one and the same. In order for data associated with an SCS to be acceptable, blank values for target compounds must be acceptable. If the blank (SCS) contains target compounds at values above the reporting limit, the supervisor should be consulted.

If the blank value lies between the reporting limit and three times the reporting limit, the reporting limit is adjusted to the level found in the blank.

9.4 Surrogates spiked into samples. Surrogate compounds are required to be spiked into all samples and QC samples for this method. Surrogate spike mix concentrations are given in Appendix D.

9.5 Matrix specific QC. Matrix spike and matrix spike duplicates are performed at the request of the client. Matrix spike compounds will be the same as those used for the DCS. See Appendix D. These compounds will be spiked into aliquots of the sample specified by the client at the same concentration level as the DCS.

9.6 Data acceptability is initially based upon the results of the Duplicate Control Samples. If DCS criteria is not in control, consult a supervisor and document any corrective action and/or alternative data acceptability criteria on an Out-of Control form. QC data must fall within established control limits in order for the laboratory to be considered "in control" when samples from that QC lot were analyzed.

9.6.1 At least 80% of the DCS recovery data, and at least 80% of the DCS precision data and at least 80% of the SCS recovery data must be within established control limits in order for the laboratory to be considered "in control". Blank values must be acceptable. (See Section 9.3) For this method the maximum number of values permitted outside control limits are

	recovery	precision
DCS:	4	2
SCS:	1	

9.7 Control Limits: Control limits will be determined as described in SOP No.: M-EQA-002. At a minimum, CLP limits must be met. These limits are listed in Table D-2. Current historical limits generated by the laboratory will be available through LDMS.

10. Calculations

10.1 Calculations required to reduce data

10.1.1 Acid and B/N run factors (rf):

$$rf = \frac{Vf\ (mL)}{Vi\ (L\ or\ kg)} \times \frac{100\%}{(\%\ Dilution\ of\ extract\ used\ for\ analysis)} \times (Column\ Clean\ up\ Factor)$$

Where Vf = final extract volume as prepped
Vi = initial sample volume (or weight) used for extraction

$$Column\ Clean\ up\ Factor = \frac{Volume\ available\ for\ clean\ up}{Volume\ put\ through\ column}$$

10.1.2 Expected (theoretic) Acid and B/N surrogate and/or spike concentrations in extract (TSC):

$$TSC = \frac{C_s\ (ug/_{mL}) \times Vs\ (mL)}{Vf\ (mL)} \times (\%\ Dilution\ of\ extract\ used\ for\ analysis) \times \frac{1}{(Column\ Clean\ up\ Factor)}$$

Where TSC = theoretic surrogate or spike concentration in extract

Cs = concentration of surrogate or spike in mix

Vs = volume of surrogate mix or spike mix spiked into sample during extraction procedure

10.2 Calculations required to verify automated data reduction

10.2.1 Response factors (RF):

$$RF = \frac{A_x \times Cis\ (ug/mL)}{A_{is} \times C_x\ (ug/mL)}$$

where A_x = area of characteristic (quantitation) ion for the compound to be measured

A_{is}= area of characteristic (quantitation) ion for the reference internal standard

C_{is}= concentration of the internal standard

C_x = concentration of the compound to be measured

10.2.2 Average Response Factor (RF_{ave}):

$$RF_{ave} = \frac{(RF_{20} + RF_{50} + RF_{80} + RF_{120} + RF_{160})}{5}$$

Where RF_x = response factor of a compound in each of the calibration standards

10.2.3 Percent Relative Standard Deviation (%RSD):

$$\%RSD = \frac{SD}{RF_{ave}} \times 100$$

Where SD = standard deviation of individual initial calibration response factors (per compound)

and where

$$SD = \sum_{i=1}^{N} \sqrt{\frac{(X_i - X)^2}{N-1}}$$

10.2.4 Percent Deviation from average response factor (%D):

$$\%D = \frac{RF_I - RF_C}{RF_I} \times 100$$

where RF_I = average response factor from initial calibration

RF_C = response factor from current calibration daily standard

10.2.5 Percent Recovery (%Rec):

Spikes -

$$\%Rec = \frac{SSR\ (ug/mL) - SR\ (ug/mL)}{TSC\ (ug/mL)} \times 100$$

Where SSR = spiked sample results
SR = sample results
TSC = theoretic spike concentration

Surrogates -

$$\%Rec = \frac{SR\ (ug/mL)}{TSC\ (ug/mL)} \times 100$$

Where SR = surrogate concentration (results in sample)

TSC = theoretic surrogate concentration

10.2.6 Relative Percent Difference (RPD):

$$RPD = \frac{D_1 - D_2}{(D_1 + D_2)/2} \times 100$$

Where D_1 = first sample value

D_2 = second sample value (e.g. duplicate or matrix spike duplicate)

10.2.7 Percent moisture

$$\%moisture = \frac{\text{Wet wt (sample plus dish)} - \text{Dry wt (sample plus dish)}}{\text{Wet wt (sample plus dish)}} \times 100$$

10.2.8 Analyte concentration in an aqueous sample

$$\text{Concentration } \left(\frac{ug}{L}\right) = \frac{A_x \times C_{is}\ (ug/mL)}{A_{is} \times RF} \times rf \left(\frac{mL}{L}\right)$$

Where A_x = area of quantitation ion for compound being measured

A_{is} = area of quantitation ion for reference internal standard

C_{is} = concentration of internal standard in sample extract

RF = response factor for compound being measured from ongoing (continuing) calibration standard

rf = run factor (See 10.1.1)

10.2.9 Analyte concentration in a soil sample (wet weight)

$$\text{Concentration } \left(\frac{ug}{kg}\right) = \frac{A_x \times C_{is}\ (ug/mL)}{A_{is} \times RF} \times rf\ \left(\frac{mL}{kg}\right)$$

Where A_x, A_{is}, C_{is}, RF, rf = same as given for water in 10.2.8

10.2.10 Analyte concentration in a soil sample (dry weight)

$$\text{Concentration } \left(\frac{ug}{kg}\right) = \frac{A_x \times C_{is}\ (ug/mL)}{A_{is} \times RF} \times \frac{1}{\left(1-\frac{\%moisture}{100}\right)} \times rf\left(\frac{mL}{kg}\right)$$

Where A_x, A_{is}, C_{is}, RF, rf = same as given for water in 10.2.8

and % moisture as defined in 10.2.7

10.2.11 Tentatively identified compound concentration in sample

$$\text{Concentration } \left(\frac{ug}{L} \text{ or } \frac{ug}{kg}\right) = \frac{RIC_x \times C_{is}\ (ug/mL)}{RIC_{is} \times 1.0} \times rf\ \left(\frac{mL}{L} \text{ or } \frac{mL}{kg}\right)$$

Where RIC_x = area of reconstructed ion chromatogram for unknown compound being measured

RIC_{is} = area of reconstructed ion chromatogram for closest uninterfered internal standard

C_{is} = concentration of internal standard in sample extract

rf = run factor for sample extract as analyzed

11. Reporting requirements

11.1 Units: Aqueous samples will be reported in ug/L. Solid and sludge samples will be reported in ug/kg on a wet weight basis, unless requested otherwise by the client.

11.2 Reporting limits:

See Appendix A

11.3 Significant Figures:

All data <10 ppb will be reported with one significant figure. All other data will be reported with two significant figures.

11.4 LDMS data entry

12. References

12.1 Method Source: Method 8270, SW-846 Third Edition, USEPA, Office of Solid Waste and Emergency Response, Washington, DC 20460, September 1986.

12.2 Deviations from Source Methods

12.2.1 Daily (continuing) calibration is considered to be valid for 12 hours from time of injection of DFTPP.

12.2.2 Deviations from the specified criteria for initial and continuing calibration are allowed, provided the rationale is documented.

12.2.3 An internal QA/QC program has been adopted by Enseco, Inc. This is described in Enseco's "Quality Assurance Program Plan for Environmental Chemical Monitoring" (Rev. 3.3). Specific elements of the QC program are described in SOP NO. M-EQA-002: "Internal QC Checks--Laboratory Performance QC." The Laboratory Control Samples generated in this program are used to monitor method performance. Matrix spikes are performed at the request of the client.

12.2.4 Additional Sources

12.2.4.1 USEPA Contract Laboratory Program Statement of Work 10/86 Rev:1/88, 2/88, 7/88

12.2.4.2 RMAL GC/MS Software Manual (Ver. 6/28/88)

12.2.4.3 Enseco, Inc. Quality Assurance Program Plan for Environmental Chemical Monitoring (Rev 3.2).

APPENDIX A Analytes & Detection Limits

TABLE A-1 HSL LIST

Analyte	Detection Limit Water (ug/mL)	Detection Limit Soil (ug/mL)
Phenol	10	330
bis(2-Chloroethyl)ether	10	330
2-Chlorophenol	10	330
1,3-Dichlorobenzene	10	330
1,4-Dichlorobenzene	10	330
Benzyl alcohol	10	330
1,2-Dichlorobenzene	10	330
2-Methylphenol	10	330
bis(2-Chloroisopropyl)ether	10	330
4-Methylphenol	10	330
N-Nitroso-di-n-propylamine	10	330
Hexachloroethane	10	330
Nitrobenzene	10	330
Isophorone	10	330
2-Nitrophenol	10	330
2,4-Dimethylphenol	10	330
Benzoic acid	50	1600
bis(2-Chloroethoxy)methane	10	330
2,4-Dichlorophenol	10	330

TABLE A-1 HSL LIST (Cont.)

Analyte	Detection Limit Water (ug/mL)	Detection Limit Soil (ug/mL)
1,2,3-Trichlorobenzene	10	330
Naphthalene	10	330
4-Chloroaniline	10	330
Hexachlorobutadiene	10	330
4-Chloro-3-methylphenol	10	330
2-Methylnaphthalene	10	330
Hexachlorocyclopentadiene	10	330
2,4,6-Trichlorophenol	10	330
2,4,5-Trichlorophenol	50	1600
2-Chloronaphthalene	10	330
2-Nitroaniline	50	1600
Dimethyl phthalate	10	330
Acenaphthylene	10	330
3-Nitroaniline	50	1600
Acenaphthene	10	330
2,4-Dinitrophenol	50	1600
4-Nitrophenol	50	1600
Dibenzofuran	10	330
2,4-Dinitrotoluene	10	330
2,6-Dinitrotoluene	10	330
Diethyl phthalate	10	330
4-Chlorophenylphenyl ether	10	330
Fluorene	10	330
4-Nitroaniline	50	1600
4,6-Dinitro-2-methylphenol	50	1600
N-Nitrosodiphenylamine	10	330
4-Bromophenylphenyl ether	10	330
Hexachlorobenzene	10	330
Pentachlorophenol	50	1600
Phenanthrene	10	330
Anthracene	10	330
Di-n-butyl phthalate	10	330
Fluoranthene	10	330
Pyrene	10	330

Butyl benzyl phthalate	10	330
3,3'-Dichlorobenzidine	20	660
Benzo(a)anthracene	10	330
bis(2-Ethylhexyl)phthalate	10	330
Chrysene	10	330
Di-n-octyl phthalate	10	330
Benzo(b)fluoranthene	10	330
Benzo(k)fluoranthene	10	330
Benzo(a)pyrene	10	330
Indeno(1,2,3-c,d)pyrene	10	330
Dibenzo(a,h)anthracene	10	330
Benzo(g,h,i)perylene	10	330

TABLE A-2 PRIORITY POLLUTANT LIST

Analyte	Detection Limit Water (ug/mL)	Detection Limit Soil (ug/mL)
Phenol	10	330
bis(2-Chloroethyl)ether	10	330
2-Chlorophenol	10	330
1,3-Dichlorobenzene	10	330
1,4-Dichlorobenzene	10	330
1,2-Dichlorobenzene	10	330
bis(2-Chloroisopropyl)ether	10	330
N-Nitroso-di-n-propylamine	10	330
Hexachloroethane	10	330
Nitrobenzene	10	330
Isophorone	10	330
2-Nitrophenol	10	330
2,4-Dimethylphenol	10	330
bis(2-Chloroethoxy)methane	10	330
2,4-Dichlorophenol	10	330
1,2,4-Trichlorobenzene	10	330
Naphthalene	10	330
Hexachlorobutadiene	10	330
4-Chloro-3-methylphenol	10	330
Hexachlorocyclopentadiene	10	330
2,4,6-Trichlorophenol	10	330
2-Chloronaphthalene	10	330
Dimethyl phthalate	10	330
Acenaphthylene	10	330
Acenaphthene	10	330
2,4-Dinitrophenol	50	1600
4-Nitrophenol	50	1600
2,4-Dinitrotoluene	10	330
2,6-Dinitrotoluene	10	330
Diethyl phthalate	10	330

TABLE A-2 PRIORITY POLLUTANT LIST (Cont.)

Analyte	Detection Limit Water (ug/mL)	Detection Limit Soil (ug/mL)
4-Chlorophenylphenyl ether	10	330
Fluorene	10	330
4,6-Dinitro-2-methylphenol	50	1600
1,2-Diphenylhydrazine	10	330
N-Nitrosodiphenylamine	10	330
4-Bromophenylphenyl ether	10	330
Hexachlorobenzene	10	330
Pentachlorophenol	50	1600
Phenanthrene	10	330
Anthracene	10	330
Di-n-butyl phthalate	10	330
Fluoranthene	10	330
Pyrene	10	330
Butyl benzyl phthalate	10	330
3,3'-Dichlorobenzidine	20	660
Benzo(a)anthracene	10	330
bis(2-Ethylhexyl)phthalate	10	330
Chrysene	10	330
Di-n-octyl phthalate	10	330
Benzo(b)fluoranthene	10	330
Benzo(k)fluoranthene	10	330
Benzo(a)pyrene	10	330
Indeno(1,2,3-c,d)pyrene	10	330
Dibenzo(a,h)anthracene	10	330
Benzo(g,h,i)perylene	10	330

APPENDIX B Standards Solutions

TABLE B-1 SURROGATE STANDARD SPIKING SOLUTION

Compound	Concentration in Mix (ug/mL)
Nitrobenzene - D_5	100
2-Fluorobiphenyl	100
Terphyenyl - D_{14}	100
Phenol - D_5	200
2-Fluorophenol	200
2,4,6-Tribromophenol	200

TABLE B-2 DCS STANDARD SPIKING SOLUTION
(Matrix Spiking Solution)

Compound	Concentration in Mix (ug/mL)
1,2,4-Trichlorobenzene	100
Acenaphthene	100
2,4-Dinitrotoluene	100
Pyrene	100
N-Nitroso-Di-n-propylamine	100
1,4-Dichlorobenzene	100
Pentachlorophenol	200
Phenol	200
2-Chlorophenol	200
4-Chloro-3-Methylphenol	200
4-Nitrophenol	200

TABLE B-3 GC CALIBRATION STANDARD
(for Screening)

BNA-SCS/IS Working Solution

Component	Concentration (ug/mL)
Dichlorobenzene-D4	8
Naphthalene-D8	8
Acenaphthene-D10	8
Phenanthrene-D10	8
Chrysene-D12	8
Perylene-D12	8
2-Fluorophenol	10
Phenol-D5	10
Tribromophenol	10
Nitrobenzene-D5	5
2-Fluorobiphenyl	5
Terphenyl-D14	5

TABLE B-4 INTERNAL STANDARD SOLUTION

Compound	Concentration in Mix (ug/mL)
D_4-1,4-Dichlorobenzene	400
D_8-Naphthalene	400
D_{10}-Acenaphthene	400
D_{10}-Phenanthrene	400
D_{12}-Chrysene	400
D_{12}-Perylene	400

TABLE B-5 CALIBRATION STANDARD SOLUTIONS

1) MS-BNAHSL-CSB

Compound	Concentration in Mix (ug/mL)
Nitrobenzene-D_5	50
2-Fluorobiphenyl	50
Terphenyl-D_{14}	50
D_5-Phenol	100
2-Fluorophenol	100
2,4,6-Tribromophenol	100
N-Nitrosodimethylamine	50
Aniline	50
Phenol	50
bis(2-Chloroethyl)ether	50
2-Chlorophenol	50
1,3-Dichlorobenzene	50
1,4-Dichlorobenzene	50
Benzyl Alcohol	50
1,2-Dichlorobenzene	50

TABLE B-5 CALIBRATION STANDARD SOLUTIONS (Cont.)

1) MS-BNAHSL-CSB

Compound	Concentration in Mix (ug/mL)
2-Methylphenol	50
bis(2-Chloroisopropyl)ether	50
4-Methylphenol	50
N-Nitroso-di-n-propylamine	50
Hexachloroethane	50
Nitrobenzene	50
Isophorone	50
2-Nitrophenol	50
2,4-Dimethylphenol	50
bis(2-Chloroethoxy)methane	50
2,4-Dichlorophenol	50
Benzoic Acid	50
1,2,4-Trichlorobenzene	50
Naphthalene	50
4-Chloroanaline	50
Hexachlorobutadiene	50
4-Chloro-3-Methylphenol	50
2-Methylnaphthalene	50
Hexachlorocyclopentadiene	50
2,4,6-Trichlorophenol	50
2,4,5-Trichlorophenol	50
2-Chloronaphthalene	50
2-Nitroanaline	50
Dimethylphthalate	50
Acenaphthylene	50
2,6-Dinitrotoluene	50
3-Nitroaniline	50
Acenaphthene	50
2,4-Dinitrophenol	50
Dibenzofuran	50

TABLE B-5 CALIBRATION STANDARD SOLUTIONS

1) MS-BNAHSL-CSB

Compound	Concentration in Mix (ug/mL)
4-Nitrophenol	50
2,4-Dinitrotoluene	50
Diethylphthalate	50
Fluorene	50
4-Chlorophenyl-phenyl Ether	50
4-Nitroanaline	50
4,6-Dinitro-2-Methylphenol	50
N-Nitrosodiphenylamine	50
Azobenzene	50
4-Bromophenylphenyl Ether	50
Hexachlorobenzene	50
Pentachlorophenol	50
Phenanthrene	50
Anthracene	50
Di-n-butylphthalate	50
Fluoranthene	50
Benzidine	100
Pyrene	50
Butylbenzylphthalate	50
Benzo(a)anthracene	50
3,3'-Dichlorobenzidine	100
Chrysene	50
bis(2-Ethylhexyl)phthalate	50
Di-n-octyl phthalate	50
Benzo(b)fluoranthene	50
Benzo(k)fluoranthene	50
Benzo(a)pyrene	50
Indeno(1,2,3-c,d)pyrene	50
Dibenz(a,h)anthracene	50
Benzo(g,h,i)perylene	50

The other 4 points in the 5 pt. calibration standard set contain the above list of compounds at 20 ug/mL, 80 ug/mL, 120 ug/mL and 160 ug/mL, with the exception of acid surrogates, benzidine and 3,3'-Dichlorobenzidine which are in each mix at twice the concentration. 1 or 2 uLs of each standard is injected.

APPENDIX C

TABLE C-1

DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
51	30.0 - 60.0 percent of mass 198
68	less than 2.0 percent of mass 69
70	less than 2.0 percent of mass 69
127	40.0 - 60.0 percent of mass 198
197	less than 1.0 percent of mass 198
198	base peak, 100 percent relative abundance
199	5.0 - 9.0 percent of mass 198
275	10.0 - 30.0 percent of mass 198
365	greater than 1.00 percent of mass 198
441	present but less than mass 443
442	greater than 40.0 percent of mass 198
443	17.0 - 23.0 percent of mass 442

TABLE C-2

CALIBRATION CHECK COMPOUNDS

Initial Calibration: The percent relative standard deviation for the each compound listed below should be ≤30%.

Continuing Calibration: The percent deviation from the average response factor determined in the initial calibration should be ≤25% for each compound listed below.

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-Methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
N-Nitrosodinphenylamine	Phenol
Di-n-octylphthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

SYSTEM PERFORMANCE CHECK COMPOUNDS

Initial calibration: The average response factor for each compound listed below should be >0.05.

Continuing calibration: The response factor for each compound listed below should be >0.05.

Base/Neutral Fraction	Acid Fraction
N-Nitroso-di-n-propylamine	2,4-Dinitrophenol
Hexachlorocyclopentadiene	4-Nitrophenol

APPENDIX D

TABLE D-1
SPIKE LEVELS FOR LABORATORY CONTROL SAMPLES

	Spike Level Aqueous (ug/L)	Spike Level Solid* (ug/Kg)	Concentration in mix (ug/ml)
DCS			
Pentachlorophenol	100	6,670	200
Phenol	100	6,670	200
2-Chlorophenol	100	6,670	200
4-Chloro-3-Methylphenol	100	6,670	200
4-Nitrophenol	100	6,670	200
1,2,4-Trichlorobenzene	50	3,330	100
Acenaphthene	50	3,330	100
2,4-Dinitrotoluene	50	3,330	100
Pyrene	50	3,330	100
N-Nitroso-di-n-propylamine	50	3,330	100
1,4-Dichlorobenzene	50	3,330	100
SCS			
Phenol-d_5	200	3,330	200
2-Fluorophenol	200	3,330	200
2,4,6-Tribromophenol	200	3,330	200
Nitrobenzene-d_5	100	1,670	100
2-fluorobiphenyl	100	1,670	100
Terphenyl-d_{14}	100	1,670	100

*Assumes 30g sample (wet weight)

TABLE D-2

CLP RECOVERY & RPD CONTROL LIMITS

	Minimum Control Limit			
	WATER		SOIL	
DCS:				
	DCS	RPD	DCS	RPD
Pentachlorophenol	9-103	50	17-109	47
Phenol	12-89	42	26-90	35
2-Chlorophenol	27-123	40	25-102	50
4-Chloro-3-Methylphenol	23-97	42	26-103	33
4-Nitrophenol	10-80	50	11-114	50
1,2,4-Trichlorobenzene	39-98	28	38-107	23
Acenaphthene	46-118	31	31-137	19
2,4-Dinitrotoluene	24-96	38	28-89	47
Pyrene	26-127	31	35-142	36
N-Nitroso-di-n-propylamine	41-116	38	41-126	38
1,4-Dichlorobenzene	36-97	28	28-104	27

SCS	WATER	SOIL
Phenol-d_5	10-94	24-113
2-Fluorophenol	21-100	25-121
2,4,6-Tribromophenol	10-123	19-122
Nitrobenzene-d_5	35-114	23-120
2-Fluorobiphenyl	43-116	30-115
Terphenyl-d_{14}	33-141	18-137

APPENDIX A ANALYTICAL TEST REFERENCES

Table of Contents

1 HAZARDOUS SUBSTANCE LIST (HSL)

2 PRIORITY POLLUTANT (PP OR 625)

3 EPA/CLP

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7 TCLP (TOXICITY CHARACTERISTIC LEACHING PROCEDURE)

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11 PAH - PART PER BILLION/FULL SCAN

Subject or Title:

GC/MS Analysis of Semivolatile Organics

SOP No.: LM-RMA-3013 BNA 625	Revision No.: 1.0	Effective Date: 12/01/89

Supersedes:

Note About Analytical Test References

The following test references have been written utilizing commands as described in the GC/MS Software Manual. Procedure names and functions are subject to change and therefore most have not been written into this SOP.

See Appendix B for a summary of current procedures.

GC/MS Analysis of Semivolatile Organics

SOP No.: LM-RMA-3013 BNA 625	Revision No.: 1.0	Effective Date: 12/01/89

Supersedes:

Analytical Test Reference No. 1

HAZARDOUS SUBSTANCE LIST (HSL)

(INDUSTRIAL, NON-CONTRACT CLIENTS)

I. Instrumentation

A. Calibration

1) 4500's

a) Turn on CAL gas button

b) Run "TUNE" procedure - automatically sets scan conditions for CAL gas acquisition

c) Use "ACQU" to acquire CAL gas scans, e.g.
> ACQU MMDDYYC* <CR>
where "*" is instrument letter designation

d) Run procedures "CALI" & "FIT"

B. GC Descriptors

1) 4500's

a) For DFTPP load descriptor QD using procedure "QD" (also loaded by the DFTPP acquisition procedure "ROAD") - initial temp is 175°C and is displayed by LED on GC

b) For standards and samples, load descriptor ST using procedure "ST" (also loaded by the sample acquisition procedures "ATAQ" and "DOIT") - initial temp is 30°C and is displayed by LED on GC

C. Standards

1) Daily Calibration (i.e., continuing calibration)

a) MS - BNAHSL - CSB @ 50 ug/mL

2) Initial Calibration

a) 5 point curve using 20 ug/mL, 50 ug/mL, 80 ug/mL, 120 ug/mL & 160 ug/mL standards

3) Internal Standard

a) MS - BNA - IS @ 400 ug/mL

b) Spike 100 ul into 1 mL extract

D. Instrument Commands

1) "ROAD" is used to acquire DFTPP.

2) "DOIT" is used on 4500's to acquire data manually.

3) Autoroutines for Autoruns

a) Sample filenames go in "TA.NL"

b) Header information goes in "HEDR"

c) Step through "AUTO" to input both acquisition information and processing information

Example: Acquisition information (in AUTO):

AUTO: SA1 <CR> (Allows input for sample 1)
AUTO: ATAQ <CR> (Specifies acquisition for sample 1)
AUTO: CA1,2,X <CR> (Copies acquisition information for samples 1 to samples 2 through X, X being number of samples in autorun)

Example: Processing information (in AUTO):

AUTO: SB1 <CR> (Allows input for sample 1)
AUTO: TC,, BNAHSLSTD; NDUAL; TIC1 <CR> (Specifies processing for sample 1)
AUTO: CB1,2,X <CR> (Same function as CA1,2,X above)

II. QC

A. Must meet DFTPP criteria (Appendix C).

B. Evaluate CCC and SPCC compounds for daily standard and consult supervisor if outside limits

C. Enseco QC - DCS's and blanks

D. For samples, one surrogate each fraction is allowed out if present above 10%.

E. Document analytical problems on anomaly sheet

III. Data

A) Processing

1) Standard, samples and QC processed using AUTOQUAN and BNAHSLSTD.LL, i.e.

TC filename,, BNAHSLSTD <CR>

2) Pull spectra for samples and blanks.

B) Data Package Includes

1) DFTPP summary or RFAC summary required; do not tear pages apart

2) Processed data with chromatogram marked with "IS, SS, TC" in pen

3) Target compound summary sheets attached to front of each sample

4) Labeled spectra attached to back of each sample

5) DCS and SCS summary sheets (and target compound summary for SCS)

6) Anomaly sheet

7) Preparations laboratory benchsheets.

GC/MS Analysis of Semivolatile Organics

SOP No.: LM-RMA-3013 BNA 625	Revision No.: 1.0	Effective Date: 12/01/89

Supersedes:

Analytical Test Reference No. 2

PRIORITY POLLUTANT (PP OR 625)

(INDUSTRIAL, NON-CONTRACT CLIENTS)

I. Instrumentation

A. Calibration

1) 4500's

a) Turn on CAL gas button

b) Run "TUNE" procedure - automatically sets scan conditions for CAL gas acquisition

c) Use "ACQU" to acquire CAL gas scans, e.g.
> ACQU MMDDYYC* <CR>
where "*" is instrument letter designation

d) Run procedures "CALI" & "FIT"

B. GC Descriptors

1) 4500's

a) For DFTPP load descriptor QD using procedure "QD" (also loaded by the DFTPP acquisition procedure "ROAD") - initial temp is 175°C and is displayed by LED on GC

b) For standards and samples, load descriptor ST using procedure "ST" (also loaded by the sample acquisition procedures "ATAQ" and "DOIT") - initial temp is 30°C and is displayed by LED on GC

C. Standards

1) Daily Calibration (i.e., continuing calibration)

a) MS - BNAHSL - CSB @ 50 ug/mL

2) Initial Calibration

   a) 5 point curve using 20 ug/mL, 50 ug/mL, 80 ug/mL, 120 ug/mL & 160 ug/mL standards

3) Internal Standard

   a) MS - BNA - IS @ 400 ug/mL

   b) Spike 100 ul into 1 mL extract

D. Instrument Commands

1) "ROAD" is used to acquire DFTPP.

2) "DOIT" is used on 4500's to acquire data manually.

3) Autoroutines for Autoruns

   a) Sample filenames go in "TA.NL"

   b) Header information goes in "HEDR"

   c) Step through "AUTO" to input both acquisition information and processing information

Example: Acquisition information (in AUTO):

```
AUTO:   SA1 <CR> (Allows input for sample 1)
AUTO:   ATAQ <CR> (Specifies acquisition for sample 1)
AUTO:   CA1,2,X <CR> (Copies acquisition information for
        samples 1 to samples 2 through X, X being number
        of samples in autorun)
```

Example: Processing information (in AUTO):

```
AUTO:   SB1 <CR> (Allows input for sample 1)
AUTO:   TC,, BNAHSL; NDUAL; TIC1 <CR> (Specifies
        processing for sample 1)
AUTO:   CB1,2,X <CR> (Same function as CA1,2,X above)
```

II. QC

A. Must meet DFTPP criteria (Appendix C).

B. Evaluate CCC and SPCC compounds for daily standard and consult supervisor if outside limits

C. Enseco QC - DCS's and blanks

D. In samples, one surrogate each fraction is allowed out if present above 10%.

E. Document analytical problems on anomaly sheet

III. Data

A) Processing

1) Standard, samples and QC processed using AUTOQUAN and BNAHSLSTD.LL, i.e.

TC filename,, BNAHSLSTD

2) Pull spectra for samples and blanks.

B) Data Package Includes

1) DFTPP summary or RFAC summary required; do not tear pages apart

2) Processed data with chromatogram marked with "IS, SS, TC" in pen

3) Target compound summary sheets attached to front of each sample

4) Labeled spectra attached to bank of each sample

5) DCS and SCS summary sheets (and target compound summary for SCS)

6) Anomaly sheet

7) Preparations Laboratory benchsheet

GC/MS Analysis of Semivolatile Organics

SOP No.: LM-RMA-3013 BNA 625	Revision No.: 1.0	Effective Date: 12/01/89

Supersedes:

Analytical Test Reference No. 3

INDUSTRIAL, NON-CONTRACT CLIENTS

INDUSTRIAL CLP (RAS)

I. Instrumentation

A. Calibration

1) 4500's

a) Turn on CAL gas button

b) Run "TUNE" procedure - automatically sets scan conditions for CAL gas acquisition

c) Use "ACQU" to acquire CAL gas scans, e.g.
> ACQU MMDDYYC* <CR>
where "*" is instrument letter designation

d) Run procedures "CALI" & "FIT"

B. GC Descriptors

1) 4500's

a) For DFTPP load descriptor QD using procedure "QD" (also loaded by the DFTPP acquisition procedure "ROAD") - initial temp is 175°C and is displayed by LED on GC

b) For standards and samples, load descriptor ST using procedure "ST" (also loaded by the sample acquisition procedures "ATAQ" and "DOIT") - initial temp is 30°C and is displayed by LED on GC

C. Standards

NOTE: A special header format is required for each analysis (i.e., standard, blanks, samples and QC samples). See attached sheet for instruction on header format before analyzing standard.

1) Daily Calibration (i.e., continuing calibration)

   a) MS - BNAHSL - CSB @ 50 ug/mL

2) Initial Calibration

   a) 5 point curve using 20 ug/mL, 50 ug/mL, 80 ug/mL, 120 ug/mL & 160 ug/mL standards

3) Internal Standard

   a) MS - BNA - IS @ 400 ug/mL

   b) Spike 100 ul into 1 mL extract

D. Instrument Commands

1) "ROAD" is used to acquire DFTPP.

2) "DOIT" is used on 4500's to acquire data manually.

3) Autoroutines for Autoruns

   a) Sample filenames go in "TA.NL"

   b) Header information goes in "HEDR"

   c) Step through "AUTO" to input both acquisition information and processing information

   Example: Acquisition information (in AUTO):

   AUTO: SA1 <CR> (Allows input for sample 1)
   AUTO: ATAQ <CR> (Specifies acquisition for sample 1)
   AUTO: CA1,2,X <CR> (Copies acquisition information for samples 1 to samples 2 through X, X being number of samples in autorun)

   Example: Processing information (in AUTO):

AUTO: SB1 <CR> (Allows input for sample 1)
AUTO: TC,, BNAHSLSTD; DUAL; TIC1 <CR> (Specifies processing for sample 1)
AUTO: CB1,2,X <CR> (Same function as CA1,2,X above)

II. QC

A. Must meet DFTPP criteria (Appendix C)

B. Must meet EPA criteria for CCC and SPCC compounds in daily calibration standard and initial calibration

C. Calibration is valid for 12 hours from time of injection of DFTPP

D. QC samples as prepped (e.g. method blanks, matrix spikes and matrix spike dups) with each project

E. Repreps are required for any samples (blanks not included) having 2 or more surrogates either fraction outside limits; 1 surrogate each fraction is allowed outside limits provided recovery is > 10%.

F. Document analytical problems on anomaly sheet

III. Data

A) Processing

1) Standard processed using AUTOQUAN and BNAHSLSTD.LL

2) Samples and QC processed using AUTOQUAN and BNACLP.LL

3) Pull spectra for samples and QC samples, <u>except</u> no spectra required for spike compounds in QC samples

4) Pull TIC's for 25 largest unknown peaks > 10% peak area of closest uninterfered I.S.

5) Capture target compound data and TIC data for foremaster using procedures SCAP and TICCAP respectively.

B) Data Package Includes

1) All raw data including

DFTPP spectrum
DFTPP mass list
DFTPP summary
DFTPP "Form V" (which lists datafiles and time of acquisition)
Daily standard raw data
RFAC ongoing calibration summary
RFAC initial calibration summary
Sample, blank and QC sample raw data

NOTE: All pages must be torn apart at perforation so copies can be made

2) Sample, blank and QC sample chromatograms labeled (stamped) with I.S. names, S.S. names, "TC, TIC and SC" for target compounds, TIC's and spike compounds respectively, and a legend defining any symbols used (i.e. TC, TIC and SC)

3) Target compound summary sheets attached to front of each sample

4) Labeled spectra attached to back of each sample (No spectra required for QC sample spike compounds)

5) TIC library searches (labeled) and summary.

6) Data generated by capturing target compound and TIC data.

7) Floppy disk on which data is captured

8) Anomaly sheet

9) Spike recovery forms for matrix spikes and matrix spike dups

GC/MS Analysis of Semivolatile Organics

SOP No.: LM-RMA-3013 BNA 625	Revision No.: 1.0	Effective Date: 12/01/89

Supersedes:

Analytical Test Reference No. 4

REFINERY (REF)

(INDUSTRIAL, NON-CONTRACT CLIENTS)

I. Instrumentation

A. Calibration

1) 4500's

a) Turn on CAL gas button

b) Run "TUNE" procedure - automatically sets scan conditions for CAL gas acquisition

c) Use "ACQU" to acquire CAL gas scans, e.g.
> ACQU MMDDYYC* <CR>
where "*" is instrument letter designation

d) Run procedures "CALI" & "FIT"

B. GC Descriptors

1) 4500's

a) For DFTPP load descriptor QD using procedure "QD" (also loaded by the DFTPP acquisition procedure "ROAD") - initial temp is 175°C and is displayed by LED on GC

b) For standards and samples, load descriptor ST using procedure "ST" (also loaded by the sample acquisition procedures "ATAQ" and "DOIT") - initial temp is 30°C and is displayed by LED on GC

C. Standards

1) Daily Calibration (i.e., continuing calibration)

a) MS - SKBNA - CSB @ 50 ug/mL

2) Initial Calibration

   a) 3 point curve using 10 ug/mL, 50 ug/mL & 150 ug/mL standards

3) Internal Standard

   a) MS - BNA - IS @ 400 ug/mL
   b) Spike 100 ul into 1 mL extract

D. Instrument Commands

1) "ROAD" is used to acquire DFTPP
2) "DOIT" is used on 4500's to acquire data manually; "HEDR" is used on INCOS 50
3) Autoroutines for Autoruns

   a) Sample filenames go in "TA.NL"

   b) Header information goes in "AASK"

   c) Step through "AUTO" to input both acquisition information and processing information

   Example: Acquisition information (in AUTO):

   AUTO: SA1 <CR> (Allows input for sample 1)
   AUTO: ATAQ <CR> (Specifies acquisition for sample 1)
   AUTO: CA1,2,X <CR> (Copies acquisition information for samples 1 to samples 2 through X, X being number of samples in autorun)

   Example: Processing information (in AUTO):

   AUTO: SB1 <CR> (Allows input for sample 1)
   AUTO: TC,, BNAREF; NDUAL; TIC1 <CR> (Specifies processing for sample 1)
   AUTO: CB1,2,X <CR> (Same function as CA1,2,X above)

II. QC

A. Must meet DFTPP criteria (Appendix C).

B. Evaluate CCC and SPCC compounds for daily calibration and consult supervisor if outside limits

C. Enseco QC - DCS's and SCS's

D. Document analytical problems on anomaly sheet

E. One surrogate each fraction allowed out for samples if detected above 10%

III. Data

A) Processing

1) Standard and QC samples processed using AUTOQUAN and BNAREFSTD.LL

2) Samples processed using AUTOQUAN and BNAREF.LL.

3) Pull spectra for samples and blanks

B) Data Package Includes

1) DFTPP summary or RFAC summary required; do not tear pages apart

2) Processed data with chromatogram marked with "IS, SS, TC" in pen

3) Target compound summary sheets attached to front of each sample

4) Labeled spectra attached to back of each sample

5) DCS and SCS summary sheets (and target compound summary for SCS)

6) Anomaly sheet

7) Sample preparations laboratory benchsheet

Analytical Test Reference No. 5

APPENDIX IX (AP9)

(INDUSTRIAL, NON-CONTRACT CLIENTS)

I. Instrumentation

A. Calibration

1) 4500's

a) Turn on CAL gas button

b) Run "TUNE" procedure - automatically sets scan conditions for CAL gas acquisition

c) Use "ACQU" to acquire CAL gas scans, e.g.
> ACQU MMDDYYC* <CR>
where "*" is instrument letter designation

d) Run procedures "CALI" & "FIT"

B. GC Descriptors

1) 4500's

a) For DFTPP load descriptor QD using procedure "QD" (also loaded by the DFTPP acquisition procedure "ROAD") - initial temp is 175°C and is displayed by LED on GC

b) For standards and samples, load descriptor ST using procedure "ST" (also loaded by the sample acquisition procedures "ATAQ" and "DOIT") - initial temp is 30°C and is displayed by LED on GC

C. Standards

NOTE: Turn filament on ~ 50 scans earlier than for HSL or Refinery work (edit ATAQ.PR); 1st AP9 compound is Methylmethacrylate, S1#10, followed by Pyridine, S1#20

1) Daily Calibration (i.e., continuing calibration)

   a) MS - BNAHSL - CSB @ 50 ug/mL

   b) MS - AP9BNA - CSB @ 50 ug/mL

2) Initial Calibration is identical to HSL or PP Initial Calibration

3) Internal Standard

   a) MS - BNA - IS @ 400 ug/mL

   b) Spike 100 ul into 1 mL extract

D. Instrument Commands

1) "ROAD" is used to acquire DFTPP.

2) "DOIT" is used on 4500's to acquire data manually.

3) Autoroutines for Autoruns

   a) Sample filenames go in "TA.NL"

   b) Header information goes in "HEDR"

   c) Step through "AUTO" to input both acquisition information and processing information

   Example: Acquisition information (in AUTO):

   AUTO: SA1 <CR> (Allows input for sample 1)
   AUTO: ATAQ <CR> (Specifies acquisition for sample 1)
   AUTO: CA1,2,X <CR> (Copies acquisition information for samples 1 to samples 2 through X, X being number of samples in autorun)

Example: Processing information (in AUTO):

AUTO: SB1 <CR> (Allows input for sample 1)
AUTO: TC,, BNAAP9; NDUAL; TIC1 <CR> (Specifies processing for sample 1)
AUTO: CB1,2,X <CR> (Same function as CA1,2,X above)

II. QC

A. Must meet DFTPP criteria (Appendix C).

B. Evaluate CCC and SPCC compounds for HSL standard and consult supervisor if outside limits

C. Enseco QC - DCS's and blanks

D. One surrogate each fraction allowed out for samples if detected above 10%

E. Document analytical problems on anomaly sheet

III. Data

A) Processing

1) HSL standard processed using AUTOQUAN and BNAHSLSTD.LL

2) AP9 Standard processed using AUTOQUAN and BNAAP9STD.LL

a) AA - Dimethylphenethylamine typically has extremely poor peak shape and needs to be manually quantitated.
b) Hexachlorophene is usually not detected at 50 ug/mL and as a general rule is subtracted from the AP9 STD QUAN list prior to updating the libraries to preserve the existing retention time data for this compound.

3) Samples and QC processed using AUTOQUAN and BNAAP9.LL

4) Famphur degrades in the standard and needs to be searched for by mass chro for all samples.
5) Pull spectra for samples and blanks.

B) Data Package Includes

1) DFTPP summary or RFAC summary required; do not tear pages apart

2) Processed data with chromatogram marked with "IS, SS, TC" in pen

3) Target compound summary sheets attached to front of each sample

4) Labeled spectra attached to back of each sample

5) DCS and SCS summary sheets (and target compound summary for SCS)

6) Anomaly sheet

7) Sample preparations laboratory benchsheets

Analytical Test Reference No. 6

APPENDIX VIII (AP8)

(INDUSTRIAL, NON-CONTRACT CLIENTS)

I. Instrumentation

A. Calibration

1) 4500's

a) Turn on CAL gas button

b) Run "TUNE" procedure - automatically sets scan conditions for CAL gas acquisition

c) Use "ACQU" to acquire CAL gas scans, e.g.
> ACQU MMDDYYC* <CR>
where "*" is instrument letter designation

d) Run procedures "CALI" & "FIT"

B. GC Descriptors

1) 4500's

a) For DFTPP load descriptor QD using procedure "QD" (also loaded by the DFTPP acquisition procedure "ROAD") - initial temp is 175°C and is displayed by LED on GC

b) For standards and samples, load descriptor ST using procedure "ST" (also loaded by the sample acquisition procedures "ATAQ" and "DOIT") - initial temp is 30°C and is displayed by LED on GC

C. Standards

NOTE: Turn filament on ~ 50 scans earlier than for HSL or Refinery work (edit ATAQ.PR); 1st AP9 compound is Methylmethacrylate, S1#10, followed by Pyridine, S1#20

1) Daily Calibration (i.e., continuing calibration)

   a) MS - BNAHSL - CSB @ 50 ug/mL

   b) MS - AP9BNA - CSB @ 50 ug/mL

2) Initial Calibration is identical to HSL or PP Initial Calibration

3) Internal Standard

   a) MS - BNA - IS @ 400 ug/mL

   b) Spike 100 ul into 1 mL extra

D. Instrument Commands

1) "ROAD" is used to acquire DFTPP.

2) "DOIT" is used on 4500's to acquire data manually.

3) Autoroutines for Autoruns

   a) Sample filenames go in "TA.NL".

   b) Header information goes in "HEDR".

   c) Step through "AUTO" to input both acquisition information and processing information

   Example: Acquisition information (in AUTO):

   AUTO: SA1 <CR> (Allows input for sample 1)
   AUTO: ATAQ <CR> (Specifies acquisition for sample 1)
   AUTO: CA1,2,X <CR> (Copies acquisition information for samples 1 to samples 2 through X, X being number of samples in autorun)

Example: Processing information (in AUTO):

AUTO: SB1 <CR> (Allows input for sample 1)
AUTO: TC,, BNAAP9; NDUAL; TIC1 <CR> (Specifies processing for sample 1)
AUTO: CB1,2,X <CR> (Same function as CA1,2,X above)

II. QC

A. Must meet DFTPP criteria (Appendix C).

B. Evaluate CCC and SPCC compounds for HSL standard and consult supervisor if outside limits

C. Enseco QC - DCS's and blanks

D. One surrogate each fraction allowed out for samples (above 10% rec.).

E. Document analytical problems on anomaly sheet

III. Data

A) Processing

1) HSL standard processed using AUTOQUAN and BNAHSLSTD.LL

2) AP9 Standard processed using AUTOQUAN and BNAAP9STD.LL

a) AA - Dimethylphenethylamine typically has extremely poor peak shape and needs to be manually quantitated.

b) Hexachlorophene is usually not detected at 50 ug/mL and as a general rule is subtracted from the AP9 STD QUAN list prior to updating the libraries to preserve the existing retention time data for this compound.

3) Samples and QC processed using AUTOQUAN and BNAAP9.LL

4) Famphur degrades in the standard and needs to be searched by mass chro for all samples.

5) Pull spectra for samples and blanks.

6) Pull library "E8" searches on all unknown peaks > I.S. height and quantitate any peaks that match library spectra using a response factor of 1.

B) Data Package Includes

1) DFTPP summary or RFAC summary required; do not tear pages apart

2) Processed data with chromatogram marked with "IS, SS, TC" in pen

3) Target compound summary sheets attached to front of each sample

4) Labeled spectra attached to back of each

5) DCS and SCS summary sheets (and target compound summary for SCS)

6) Anomaly sheet

7) Sample preparations laboratory benchsheet.

GC/MS Analysis of Semivolatile Organics

SOP No.: LM-RMA-3013 BNA 625	Revision No.: 1.0	Effective Date: 12/01/89

Supersedes:

Analytical Test Reference No. 7

TCLP

(INDUSTRIAL, NON-CONTRACT CLIENTS)

I. Instrumentation

A. Calibration

1) 4500's

a) Turn on CAL gas button

b) Run "TUNE" procedure - automatically sets scan conditions for CAL gas acquisition

c) Use "ACQU" to acquire CAL gas scans, e.g.
> ACQU MMDDYYC* <CR>
where "*" is instrument letter designation

d) Run procedures "CALI" & "FIT"

B. GC Descriptors

1) 4500's

a) For DFTPP load descriptor QD using procedure "QD" (also loaded by the DFTPP acquisition procedure "ROAD") - initial temp is 175°C and is displayed by LED on GC

b) For standards and samples, load descriptor ST using procedure "ST" (also loaded by the sample acquisition procedures "ATAQ" and "DOIT") - initial temp is 30°C and is displayed by LED on GC

C. Standards

NOTE: For convenience TCLP samples should be scheduled on an instrument running AP9 work on same day.

NOTE: Turn filament on ~ 50 scans earlier than for HSL or Refinery work (edit ATAQ.PR); 1st AP9 compound is Methylmethacrylate, S1#10, followed by Pyridine, S1#20

1) Daily Calibration (i.e., continuing calibration)

a) MS - BNAHSL - CSB @ 50 ug/mL

b) MS - AP9BNA - CSB @ 50 ug/mL - the AP9 STD is necessary to the TCLP analysis for retention time and response factor data for the following 2 compounds:

Pyridine
2,3,4,6-Tetrachlorophenol

Consult a supervisor prior to injecting AP9 STD if AP9 samples have not been scheduled on the instrument the day TCLP samples are to be analyzed.

2) Initial Calibration is identical to HSL or PP Initial Calibration

3) Internal Standard

a) MS - BNA - IS @ 400 ug/mL

b) Spike 100 ul into 1 mL extract

D. Instrument Commands

1) "ROAD" is used acquire DFTPP.

2) "DOIT" is used on 4500's to acquire data manually.

3) Autoroutines for Autoruns

a) Sample filenames go in "TA.NL"

b) Header information goes in "HEDR"

c) Step through "AUTO" to input both acquisition information and processing information

Example: Acquisition information (in AUTO):

AUTO: SA1 <CR> (Allows input for sample 1)
AUTO: ATAQ <CR> (Specifies acquisition for sample 1)
AUTO: CA1,2,X <CR> (Copies acquisition information for samples 1 to samples 2 through X, X being number of samples in autorun)

Example: Processing information (in AUTO):

AUTO: SB1 <CR> (Allows input for sample 1)
AUTO: TC,, BNAHSL; NDUAL; TIC1 <CR> (Specifies processing for sample 1)
AUTO: CB1,2,X <CR> (Same function as CA1,2,X above)

II. QC

A. Must hit DFTPP

B. Evaluate CCC and SPCC compounds for HSL standard and consult supervisor if outside limits

C. Enseco QC - DCS's and blanks

D. One surrogate, >10%, allowed out for each fraction

E. Document analytical problems on anomaly sheet

F. Leach blanks are analyzed as regular samples

III. Data

A) Processing

1) HSL standard processed using AUTOQUAN and BNAHSLSTD.LL

2) AP9 Standard processed using AUTOQUAN and BNAAP9STD.LL (See Analytical Test Reference No. 5 (AP9) for processing hints)

3) Samples and QC processed using AUTOQUAN and BNATCLP.LL

4) Pull spectra for samples and SCS's

B) Data Package Includes

1) DFTPP summary or RFAC summary required; do not tear pages apart

2) Processed data with chromatogram marked with "IS, SS, TC" in pen

3) Target compound summary sheets attached to front of each sample

4) Labeled spectra attached to back of each sample

5) DCS and SCS summary sheets (and target compound summary for SCS)

6) Anomaly sheet

7) Preparations laboratory benchsheet.

Subject or Title:

GC/MS Analysis of Semivolatile Organics

SOP No.: LM-RMA-3013 BNA 625	Revision No.: 1.0	Effective Date: 12/01/89

Supersedes:

Analytical Test Reference No. 8

AFIRP Version 1

I. Instrumentation

A. Calibration

1) 4500's

a) Turn on CAL gas button

b) Run "TUNE" procedure - automatically sets scan conditions for CAL gas acquisition

c) Use "ACQU" to acquire CAL gas scans, e.g.
> ACQU MMDDYYC* <CR>
where "*" is instrument letter designation

d) Run procedures "CALI" & "FIT"

B. GC Descriptors

1) 4500's

a) For DFTPP load descriptor QD using procedure "QD" (also loaded by the DFTPP acquisition procedure "ROAD") - initial temp is 175°C and is displayed by LED on GC

b) For standards and samples, load descriptor ST using procedure "ST" (also loaded by the sample acquisition procedures "ATAQ" and "DOIT") - initial temp is 30°C and is displayed by LED on GC

C. Standards

1) Daily Calibration (i.e., continuing calibration)

a) MS - BNAHSL - CSB @ 50 ug/mL

2) Initial Calibration

a) 5 point curve using 20 ug/mL, 50 ug/mL, 80 ug/mL, 120 ug/mL & 160 ug/mL standards

3) Internal Standard

a) MS - BNA - IS @ 400 ug/mL

b) Spike 100 ul into 1 mL extract

D. Instrument Commands

1) "ROAD" is used to acquire DFTPP.

2) "DOIT" is used on 4500's to acquire data manually.

3) Autoroutines for Autoruns

a) Sample filenames go in "TA.NL"

b) Header information goes in "HEDR"

c) Step through "AUTO" to input both acquisition information and processing information

Example: Acquisition information (in AUTO):

AUTO: SA1 <CR> (Allows input for sample 1)
AUTO: ATAQ <CR> (Specifies acquisition for sample 1)
AUTO: CA1,2,X <CR> (Copies acquisition information for samples 1 to samples 2 through X, X being number of samples in autorun)

Example:. Processing information (in AUTO):

AUTO: SB1 <CR> (Allows input for sample 1)
AUTO: TC,, BNAHSL; DUAL; TIC1 <CR> (Specifies processing for sample 1)
AUTO: CB1,2,X <CR> (Same function as CA1,2,X above)

II. QC

A. Must meet DFTPP criteria (Appendix C)

B. Must meet EPA criteria for CCC and SPCC compounds for daily standard

C. Calibration is valid for 12 hours from time of injection of DFTPP

D. Enseco QC - DCS's and blanks

E. One surrogate >10% allowed out for each fraction

F. Document analytical problems on anomaly sheet

III. Data

A) Processing

1) Standard and QC processed using AUTOQUAN and BNAHSLSTD.LL

2) Samples processed using AUTOQUAN and BNAHSLSTD.LL

3) Pull spectra for samples and SCS's

4) Pull TIC's for 20 largest unknown peaks > 10% peak area of closest uninterfered I.S.

B) Data Package Includes

1) DFTPP summary and RFAC summary are required; do not tear pages apart

2) Processed data with chromatogram marked with "IS, SS, TC, TIC" in pen

3) Target compound summary sheets attached to front of each sample

4) Labeled spectra attached to back of each sample

5) TIC library searches and summary attached to back of each sample

6) DCS and SCS summary sheets (and target compound summary for SCS)

7) Anomaly sheet

8) Copies of DCS and blank sample raw data

GC/MS Analysis of Semivolatile Organics

SOP No.: LM-RMA-3013 BNA 625	Revision No.: 1.0	Effective Date: 12/01/89

Supersedes:

Analytical Test Reference No. 9

AFIRP Version 2

I. Instrumentation

A. Calibration

1) 4500's

a) Turn on CAL gas button

b) Run "TUNE" procedure - automatically sets scan conditions for CAL gas acquisition

c) Use "ACQU" to acquire CAL gas scans, e.g.
> ACQU MMDDYYC* <CR>
where "*" is instrument letter designation

d) Run procedures "CALI" & "FIT"

B. GC Descriptors

1) 4500's

a) For DFTPP load descriptor QD using procedure "QD" (also loaded by the DFTPP acquisition procedure "ROAD") - initial temp is 175°C and is displayed by LED on GC

b) For standards and samples, load descriptor ST using procedure "ST" (also loaded by the sample acquisition procedures "ATAQ" and "DOIT") - initial temp is 30°C and is displayed by LED on GC

C. Standards

NOTE: Turn filament on ~ 50 scans earlier than for HSL or Refinery work (edit ATAQ.PR); 1st AP9 compound is Methylmethacrylate, S1#10, followed by Pyridine, S1#20

1) Daily Calibration (i.e., continuing calibration)

   a) MS - BNAHSL - CSB @ 50 ug/mL

   b) MS - AP9BNA - CSB @ 50 ug/mL

2) Initial Calibration is identical to HSL or PP Initial Calibration

3) Internal Standard

   a) MS - BNA - IS @ 400 ug/mL

   b) Spike 100 ul into 1 mL extract

D. Instrument Commands

1) "ROAD" is used to acquire DFTPP.

2) "DOIT" is used on 4500's to acquire data manually.

3) Autoroutines for Autoruns

   a) Sample filenames go in "TA.NL"

   b) Header information goes in "HEDR"

   c) Step through "AUTO" to input both acquisition information and processing information

   Example: Acquisition information (in AUTO):

   AUTO: SA1 <CR> (Allows input for sample 1)
   AUTO: ATAQ <CR> (Specifies acquisition for sample 1)
   AUTO: CA1,2,X <CR> (Copies acquisition information for samples 1 to samples 2 through X, X being number of samples in autorun)

Example: Processing information (in AUTO):

AUTO: SB1 <CR> (Allows input for sample 1)
AUTO: TC,, BNAAFIR; NDUAL; TIC1 <CR> (Specifies processing for sample 1)
AUTO: CB1,2,X <CR> (Same function as CA1,2,X above)

II. QC

A. Must hit DFTPP

B. Must meet EPA criteria for CCC and SPCC compounds for daily standard (HSL)

C. Calibration is valid for 12 hours from time of injection of DFTPP

D. Enseco QC - DCS's and blanks

E. One surrogate >10% allowed out for each fraction

F. Document analytical problems on anomaly sheet

III. Data

A) Processing

1) HSL standard processed using AUTOQUAN and BNAHSLSTD.LL

2) AP9 Standard processed using AUTOQUAN and BNAAP9STD.LL

3) Samples and QC processed using AUTOQUAN and BNAAFIR.LL

4) Pull spectra for samples and SCS's

5) Pull TIC's for 20 largest unknown peaks > 10% peak area of closest uninterfered I.S.

6) Search for Dibenz(a,j)acridine by mass chromatogram.

B) Data Package Includes

1) DFTPP summary and RFAC summary are required; DFTPP "Form V" (lists samples and time of acquisition) is required; do not tear pages apart

2) Processed data with chromatogram marked with "IS, SS, TC, TIC" in pen

3) Target compound summary sheets attached to front of each sample

4) Labeled spectra attached to back of each sample

5) TIC library searches and summary attached to back of each sample

6) DCS and SCS raw data

7) Anomaly sheet

GC/MS Analysis of Semivolatile Organics

SOP No.: LM-RMA-3013 BNA 625	Revision No.: 1.0	Effective Date: 12/01/89

Supersedes:

Analytical Test Reference No. 10

USGS USGS

(CONTRACTUAL)

I. Instrumentation

A. Calibration

1) 4500's

a) Turn on CAL gas button

b) Run "TUNE" procedure - automatically sets scan conditions for CAL gas acquisition

c) Use "ACQU" to acquire CAL gas scans, e.g.
> ACQU MMDDYYC* <CR>
where "*" is instrument letter designation

d) Run procedures "CALI" & "FIT"

B. GC Descriptors

1) 4500's

a) For DFTPP load descriptor QD using procedure "QD" (also loaded by the DFTPP acquisition procedure "ROAD") - initial temp is 175RC and is displayed by LED on GC

b) For standards and samples, load descriptor ST using procedure "ST" (also loaded by the sample acquisition procedures "ATAQ" and "DOIT") - initial temp is 30RC and is displayed by LED on GC

C. Standards

1) Daily Calibration (i.e., continuing calibration)

a) MS - USGSBNA - CSB @ 50 ug/mL

2) Initial Calibration

a) 5 point curve using 20, 50, 80, 120, and 160 ug/mL standards.

3) Internal Standard

a) MS - BNA - IS @ 400 ug/mL

b) spike 100 ul into 1 mL extract

D. Instrument Commands

1) "ROAD" is used to acquire DFTPP.

2) "DOIT" is used on 4500's to acquire data manually.

3) Autoroutines for Autoruns

a) Sample filenames go in "TA.NL"

b) Header information goes in "HEDR"

c) Step through "AUTO" to input both acquisition information and processing information

Example: Acquisition information (in AUTO):

AUTO: SA1 <CR> (Allows input for sample 1)
AUTO: ATAQ <CR> (Specifies acquisition for sample 1)
AUTO: CA1,2,X <CR> (Copies acquisition information for samples 1 to samples 2 through X, X being number of samples in autorun)

Example: Processing information (in AUTO):

AUTO: SB1 <CR> (Allows input for sample 1)
AUTO: TC,, BNAHSL; DUAL; TIC1 <CR> (Specifies processing for sample 1)
AUTO: CB1,2,X <CR> (Same function as CA1,2,X above)

II. QC

A. Must meet DFTPP criteria (Appendix C)

B. Must meet EPA criteria for CCC and SPCC compounds in daily calibration standard (when compared to initial calibration curve average response factors)

C. Calibration is valid for 12 hours from time of injection of DFTPP

D. QC samples as prepped - usually a blank and blank spike for each project or analytical lot

E. All surrogate recoveries must fall within recovery limits set by the USGS; if any surrogates are outside limits a reprep of the sample involved is requested.

F. Document analytical problems on anomaly sheet

III. Data

A) Processing

1) Standard, samples and QC processed using AUTOQUAN and BNAUSGS.LL

2) Pull spectra for samples and QC samples

3) Pull "LCOMPS", i.e., Library Comparison Spectra, for samples and QC samples target compounds

4) When requested pull TIC's for all unknown peaks >10% peak area of closest uninterfered I.S.

B) Data Package Includes

1) All raw data (torn apart at perforations) including

DFTPP spectrum
DFTPP mass list

DFTPP summary
DFTPP "Form V" (which lists datafiles and time of acquisition)
Daily Standard Raw Data -
RFAC ongoing calibration summary
RFAC initial calibration summary
Sample, blank and blank spike raw data

2) Sample, blank and blank spike chromatograms labeled with I.S. names, S.S. names, "TC, TIC and SC" for target compounds, TIC's and spike compounds respectively, and a legend defining any symbols used (i.e. TC, TIC, SC)

3) USGS target compound cover sheets attached to front of each sample

4) Labeled spectra and LCOMP spectra attached to back of each sample

5) TIC library searches (when requested) and summary attached to back of each sample

6) USGS blank summary forms

7) USGS blank spike recovery forms

8) USGS continuing calibration forms (or initial calibration forms if appropriate)

9) USGS surrogate recovery summary form

10) Anomaly sheet

GC/MS Analysis of Semivolatile Organics

SOP No.: LM-RMA-3013 BNA 625	Revision No.: 1.0	Effective Date: 12/01/89

Supersedes:

Analytical Test Reference No. 11

PAH - part per billion Full Scan

I. Instrumentation

A. Calibration

1) 4500's

a) Turn on CAL gas button

b) Run "TUNE" procedure - automatically sets scan conditions for CAL gas acquisition

c) Use "ACQU" to acquire CAL gas scans, e.g.
> ACQU MMDDYYC* <CR>
where "*" is instrument letter designation

d) Run procedures "CALI" & "FIT"

B. GC Descriptors

1) 4500's

a) For DFTPP load descriptor QD using procedure "QD" (also loaded by the DFTPP acquisition procedure "ROAD") - initial temp is 175°C and is displayed by LED on GC

b) For standards and samples, load descriptor ST using procedure "ST" (also loaded by the sample acquisition procedures "ATAQ" and "DOIT") - initial temp is 30°C and is displayed by LED on GC

5. Then "START" (GC control section)

C. Standards

1) Daily Calibration (i.e., continuing calibration)

a) 50 ug/mL PAH standard

2) Initial Calibration

a) 5 point curve using 20 ug/mL, 50 ug/mL, 80 ug/mL, 120 ug/mL & 160 ug/mL standards

3) Internal Standard

a) MS - PAH - IS @ 400 ug/mL

b) Spike 100 ul into 1 mL extract

D. Instrument Commands

1) "ROAD" is to acquire DFTPP.

2) "DOIT" is used on 4500's to acquire data manually.

3) Autoroutines for Autoruns

a) Sample filenames go in "TA.NL".

b) Header information goes in "HEDR".

c) Step through "AUTO" to input both acquisition information and processing information

Example: Acquisition information (in AUTO):

AUTO: SA1 <CR> (Allows input for sample 1)
AUTO: ATAQ <CR> (Specifies acquisition for sample 1)
AUTO: CA1,2,X <CR> (Copies acquisition information for samples 1 to samples 2 through X, X being number of samples in autorun)

Example: Processing information (in AUTO):

AUTO: SB1 <CR> (Allows input for sample 1)
AUTO: TC,, BNAPAHSTD; NDUAL; TIC1 <CR> (Specifies processing for sample 1)
AUTO: CB1,2,X <CR> (Same function as CA1,2,X above)

II. QC

A. Must meet DFTPP criteria (Appendix C)

B. CCC compounds must be within ± 25% of initial calibration curve average

C. Calibration is valid for 12 hours from time of injection of DFTPP

D. QC samples as prepped

E. Samples having 1 or more surrogates outside limits are reanalyzed for verification of original data, if consistent with first analysis surrogate problem is then assessed by the analyst and/or supervisor.

F. Document analytical problems on anomaly sheet

III. Data

A) Processing

1) Standard samples and QC processed using autoquan

e.g., TC filename,, BNAPAHSTD

2) Pull spectra for samples and QC samples

3) No TIC's

B) Data Package Includes

1) All raw data including

DFTPP spectrum
DFTPP mass list
DFTPP summary
DFTPP "Form V" (which lists datafiles and time of acquisition)
Daily standard raw data
RFAC ongoing calibration summary
RFAC initial calibration summary
Sample, blank and QC sample raw data

NOTE: All pages must be torn apart at perforation so copies can be made

2) Sample, blank and QC sample chromatograms labeled (stamped) with I.S. names, S.S. names, "TC and SC" for target compounds and spike compounds respectively, and a legend defining any symbols used (i.e. TC and SC)

3) Target compound summary sheets attached to front of each sample

4) Labeled spectra attached to back of each sample

5) Spike recovery forms for matrix spikes and matrix spike duplicates

6) Surrogate recovery forms

7) Method blank summary sheet

8) Anomaly sheet

APPENDIX B

Higher Level Procedures

```
HEDR FILES:     HSL AP9 REF PAH PYR ABL SBL ADCS1 ADCS2 SDCS1 SDCS2

LIBRARIES:      BNAHSLSTD       HSL ONLY
                BNAAP9STD       AP9 ONLY
                BNAAP9          HSL + AP9
                BNAREFSTD       ALL REF
                BNAREF          REF - SPIKE COMPOUNDS
                BNAPAHSTD       PAH ONLY
                BNAPYRSTD       PYR ONLY
                BNAPYR          HSL + PYR
                BNACLP          HSL - 2 COMPOUNDS
                SPIKE           SPIKE COMPOUNDS
                BNAAFIR         HSL + AP9 - SOME COMPOUNDS

TICS:           IBL=RUN TICP(/SB;E)             IBL1=RUN TICP(/SB;E)
                   =TIC IBL                         =TIC1

                ISAMP=RUN TICP(/SS;E)           ISAMP1=RUN TICP(/SS;E)
                     =TIC ISAMP                       =TIC1

                CBL=RUN TICP(/SB;E)             CBL1=RUN TICP(/SB;E)
                   =TICCLP CBL                      =TICCL1

                CSAMP=RUN TICP(/SS;E)           CSAMP1=RUN TICP(/SS;E)
                     =TICCLP CSAMP                    =TICCL1

SUMSHEETS:      [S][MATRIX][LIBR][LEVEL/SPECIAL]

                SA=CHK;SET8 $1;RUN SUM1(/BE;L;H;E)
                SS=CHK;SET8 $1;RUN SUM1(/BF;L;H;E)
                SAPAH=CHK;SET8 $1;RUN SUM1(/PA;L;H;E)
                SAPYR=CHK;SET8 $1;RUN SUM1(/AP;L;H;E)           (INCLUDES HSL)
                SSPYRM=CHK;SET8 $1;RUN SUM1(/SP;X,2;L;H;E)      (INCLUDES HSL)
                SSPYRL=CHK;SET8 $1;RUN SUM1(/SP;L;H;E)          (INDLUDES HSL)
                SSCLP=CHK;SET8 $1;RUN SUM1(/CL;L;H;E)
                SAPYRO=CHK;SET8 $1;RUN SUM1(/SA;L;H;E)          (PYR ONLY)

        PARAMETERS:     PA      PAH SURROGATE RECOVERIES
                        AP      AQUEOUS HSL/PYR SURROGATE RECOVERIES
                        SP      SOIL HSL/PYR SURROGATE RECOVERIES
                        SA      AQUEOUS PYR SURROGATE RECOVERY

STANDARDS:      [LIBRARY][STD]

                HSLSTD=TCIND,,BNAHSLSTD;CCHSL/H
                AP9STD=TCIND,,BNAAP9STD
                REFSTD=TCIND,,BNAREFSTD;CCREF/H
                PAHSTD=TCIND,,BNAPAHSTD;CCPAHB/H
                PYRSTD=TCIND,,BNAPYRSTD;CCPYR/H

QC:             [MATRIX][BL/DCS]

                ABL=TCIND,,BNAHSLSTD;NDUAL;IBL1;SA
                SBL=TCIND,,BNAHSLSTD;NDUAL;IBL1;SS
                ADCS=TCIND,,SPIKE;SA
                SDCS=TCIND,,SPIKE;SS

INDUSTRIAL:     [MATRIX][LIBR][LEVEL/SPECIAL]

                AHSL=TCIND,,BNAHSLSTD;NDUAL;SA
                SHSL=TCIND,,BNAHSLSTD;NDUAL;SS
                AAP9=TCIND,,BNAAP9;NDUAL;SA;FAM;DBAJ
                SAP9=TCIND,,BNAAP9;NDUAL;SS;FAM;DBAJ
                AREF=TCIND,,BNAREF;NDUAL;SA
                SREF=TCIND,,BNAREF;NDUAL;SS
                APAH=TCIND,,BNAPAHSTD;NDUAL;SAPAH
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            APYRO=TCIND,,BNAPYRSTD;NDUAL;SAPYRO     (PYR ONLY)
            AAFIR=TCIND,,BNAAFIR;NDUAL;SA;DBAJ
            SAFIR=TCIND,,BNAAFIR;NDUAL;SS;DBAJ
            ATCLP=TCIND,,BNATCLP;NDUAL;SA

P:          [MATRIX][LIBR][LEVEL/SPECIAL]

            ACLP=TCCLP,,BNACLP;SDUAL;SA
            SCLP=TCCLP,,BNACLP;SDUAL;SSCLP
            APYR=TCIND,,BNAPYR;SDUAL;SAPYR          (INCLUDES HSL)
            SPYRL=TCIND,,BNAPYR;SDUAL;SSPYRL        (INCLUDES HSL)
            SPYRM=TCIND,,BNAPYR;SDUAL;SSPYRM        (INCLUDES HSL)

MISC:       NEWCOL=QUAN NEWHSL(S;T;E)
                  =QUAN NEWAP9(S;T;E)
                  =QUAN NEWREF(S;T;E)

            CAL=ATAQ1
               =ACQU/O(G10;E)

            GAS=CALI#3
               =FIT

            FIA=SET4 S1#1                           (QUANTITATES IS#1)
               =CHRO($;#;&;R;A1,1;N1,1;D-50,50;E)

            LIA=SET4 S6#1                           (QUANTITATES IS#6)
               =CHRO($;#;&;R;A1,1;N1,1;D-50,50;E)

            QION=SETL BNAHSLSTD     (DISPLAYS QUAN ION FOR ALL ENTRIES
                =QION2               IN THE SPECIFIED LIBRARY)
            QION2=GETL
                 =CHRO($;#;&;R;D-50,50)
                 =LOOP

            SCUP #SCAN,,#SCAN
                    UPDATES "FILAMENT ON" AND "FIRST SURROGATE"
                    IN FOLLOWING:   TCIND
                                    TCCLP
                                    TIC
                                    TIC1
                                    TICCLP
                                    TICCLI

            SCL     CHECKS SCUP UPDATED SCANS

            DTB=EDNL TB(-;W;E)

            F0=FILE(H 0:*.TI;E)

            F2=FILE(H 2:*.TI;E)

            F3=FILE(H 3:*.TI;E)

            METHCH=PRIN(T;METHYL CHRYSENE;C;E)
                  =CHRO(D1800,2600;121;239;242;243;H;E)

            FAM=PRIN(T;FAMPHUR;C;E)
               =CHRO(D1800,2200;93;125;218;H;E)

            DBAJ=PRIN(T;DIBENZ-A,J-ACRIDINE;C;E)
                =CHRO(D2300,3100;277;278;279;280;H;E)

            DBAH=PRIN(T;DIBENZ-A,H-ACRIDINE;C;E)
                =CHRO(D2300,3100;277;278;279;280;H;E)
```

B12 A

FIXED / Updated

Enseco Rocky Mountain Analytical Laboratory

BNA ANALYSIS QUANITATION REPORT

Data file : /chem/T.i/101392B.b/t0382.d
Lab. Id. :
Inj Date : 13-OCT-92 18:45 Autotune Date: 09-Oct-92 09:30:3
Operator : da Inst ID: T.i
Smp Info : 50 ppm AP9std V#6493
Misc Info :
Comment :
Method : /chem/T.i/101392B.b/AP9smpl.m
Meth Date : 13-Oct-1992 19:14
Cal Date : 13-OCT-92 18:45 Cal File: t0382.d
Als bottle: 1 Continuing Calibration Sample
Dil Factor: 1.000 Target Version: Target 2.00
Integrator: HP RTE Compound Sublist: AP9std.sub
Rpt Date : 13-Oct-1992 19:57 Sample Type: WATER

RT (REL RT)	MASS	RESPONSE	CONCENTRATIONS ON-COL (ug/ml)	FINAL (ug/L)	TARGET RANGE	RATIO
2 Ethylmethacrylate					CAS #: 97-63-2	
4.844(0.567)	41	485825	57.6	57.6		100.00
4.844(0.567)	69	827351			150.30- 190.30	170.30
4 2-Picoline					CAS #: 109-06-8	
5.329(0.624)	93	1062103	54.4	54.4		100.00
5.329(0.624)	66	469427			24.20- 64.20	44.20
5.329(0.624)	78	166036			0.00- 35.63	15.63
5 N-Nitrosomethylethylamine					CAS #: 10595-95-6	
5.534(0.648)	42	267940	56.0	56.0		100.00
5.534(0.648)	88	412000			133.77- 173.77	153.77
5.534(0.648)	43	214584			60.09- 100.09	80.09
6 Methylmethanesulfonate					CAS #: 66-27-3	
6.032(0.707)	80	353158	53.6	53.6		100.00
6.032(0.707)	79	400533			93.41- 133.41	113.41
6.032(0.707)	65	96312			7.27- 47.27	27.27
7 N-Nitrosodiethylamine					CAS #: 55-18-5	
6.684(0.783)	102	442981	51.6	51.6		100.00
6.684(0.783)	57	165932			17.46- 57.46	37.46
6.684(0.783)	56	154856			14.96- 54.96	34.96
9 Ethylmethanesulfonate					CAS #: 62-50-0	
7.195(0.843)	79	634770	50.5	50.5		100.00
7.195(0.843)	97	165929			6.14- 46.14	26.14
7.195(0.843)	109	480091			55.63- 95.63	75.63
10 Pentachloroethane					CAS #: 76-01-7	
8.026(0.940)	117	299986	53.3	53.3		100.00

RT (REL RT)	MASS	RESPONSE	CONCENTRATIONS ON-COL (ug/ml)	FINAL (ug/L)	TARGET RANGE	RATIO
8.026(0.940)	119	300900			80.30- 120.30	100.30
8.026(0.940)	167	230661			56.89- 96.89	76.89
* 19 1 4-Dichlorobenzene-d4					CAS #:	
8.537(1.000)	152	426862	40.0	40.0		100.00
8.524(1.000)	150	701897			144.43- 184.43	164.43
8.524(1.000)	115	266235			42.37- 82.37	62.37
17 N-Nitrosopyrrolidine					CAS #: 930-55-2	
9.240(1.082)	100	401053	49.4	49.4		100.00
9.227(1.081)	41	206202			31.42- 71.42	51.42
9.227(1.081)	43	89269			2.26- 42.26	22.26
18 Acetophenone					CAS #: 98-86-2	
9.278(1.087)	105	802607	50.8	50.8		100.00
9.278(1.087)	78	75308			0.00- 29.38	9.38
9.278(1.087)	120	297655			17.09- 57.09	37.09
20 N-Nitrosomorpholine					CAS #: 59-89-2	
9.317(1.091)	56	251335	49.1	49.1		100.00
9.317(1.091)	86	113792			25.28- 65.28	45.28
9.317(1.091)	116	184558			53.43- 93.43	73.43
22 O-Toluidine					CAS #: 95-53-4	
9.342(1.094)	106	1065034	51.4	51.4		100.00
9.342(1.094)	107	826553			57.61- 97.61	77.61
9.342(1.094)	77	136649			0.00- 32.83	12.83
27 N-Nitrosopiperidine					CAS #: 100-75-4	
9.879(0.904)	42	300539	51.1	51.1		100.00
9.879(0.904)	55	177590			39.09- 79.09	59.09
9.879(0.904)	114	381641			106.99- 146.99	126.99
33 O,O,O-Triethylphosphorothio					CAS #: 126-68-1	
10.428(0.954)	121	288990	51.8	51.8		100.00
10.428(0.954)	198	195606			47.69- 87.69	67.69
10.428(0.954)	93	206830			51.57- 91.57	71.57
34 AA-Dimethylphenethylamine					CAS #: 122-09-8	
10.773(0.986)	58	1315825	63.2	63.2		100.00(M)
10.760(0.985)	91	116922			0.00- 26.19	8.89
* 44 Naphthalene-d8					CAS #:	
10.927(1.000)	136	1382629	40.0	40.0		100.00
10.914(1.000)	68	127855			0.00- 29.25	9.25
39 2,6-Dichlorophenol					CAS #: 87-65-0	
11.093(1.015)	162	452995	43.5	43.5		100.00
11.093(1.015)	164	282851			42.44- 82.44	62.44
11.080(1.014)	53	18207			0.00- 24.02	4.02

RT (REL RT)	MASS	RESPONSE	CONCENTRATIONS ON-COL (ug/ml)	FINAL (ug/L)	TARGET RANGE	RATIO
41 Hexachloropropene					CAS #: 1888-71-7	
11.144(1.020)	213	170382	47.4	47.4		100.00
11.144(1.020)	211	105295			41.80- 81.80	61.80
11.144(1.020)	117	86752			30.92- 70.92	50.92
47 p-Phenylenediamine					CAS #: 106-50-3	
11.757(1.076)	108	509660	46.0	46.0		100.00
11.757(1.076)	80	150367			9.50- 49.50	29.50
11.757(1.076)	54	70069			0.00- 33.75	13.75
50 Safrole					CAS #: 94-59-7	
12.179(1.115)	162	406280	44.6	44.6		100.00
12.179(1.115)	131	188596			26.42- 66.42	46.42
12.179(1.115)	104	142599			15.10- 55.10	35.10
51 1,2,4,5-Tetrachlorobenzene					CAS #: 95-94-3	
12.665(0.879)	216	297882	49.2	49.2		100.00
12.665(0.879)	214	243808			61.85- 101.85	81.85
12.665(0.879)	218	140307			27.10- 67.10	47.10
54 Isosafrole (#1)					CAS #: 120-58-1	
12.754(0.886)	162	44759	50.2	50.2		100.00
12.754(0.886)	131	20304			25.36- 65.36	45.36
12.754(0.886)	104	15792			15.28- 55.28	35.28
56 1-Chloronaphthalene					CAS #: 90-13-1	
13.342(0.926)	162	829222	52.5	52.5		100.00(M)
13.329(0.925)	127	366959			29.48- 69.48	44.25
13.342(0.926)	164	255695			10.84- 50.84	30.84
59 1,4-Naphthoquinone					CAS #: 130-15-4	
13.648(0.948)	158	339388	50.9	50.9		100.00
13.648(0.948)	130	120140			15.40- 55.40	35.40
13.648(0.948)	104	154461			25.51- 65.51	45.51
61 m-Dinitrobenzene					CAS #: 99-65-0	
13.943(0.968)	168	160226	50.0	50.0		100.00
13.943(0.968)	76	100355			42.63- 82.63	62.63
13.930(0.967)	50	72373			25.17- 65.17	45.17
64 Isosafrole (#2)					CAS #: 120-58-1	
13.201(0.917)	162	374880	50.7	50.7		100.00
13.201(0.917)	131	157029			21.89- 61.89	41.89
13.201(0.917)	104	123065			12.83- 52.83	32.83
* 72 Acenaphthene-d10					CAS #:	
14.403(1.000)	164	497957	40.0	40.0		100.00
14.403(1.000)	162	484802			77.36- 117.36	97.36
14.403(1.000)	160	206267			21.42- 61.42	41.42
67 Pentachlorobenzene					CAS #: 608-93-5	

RT (REL RT)	MASS	RESPONSE	CONCENTRATIONS ON-COL (ug/ml)	FINAL (ug/L)	TARGET RANGE	RATIO
14.735(1.023)	250	260775	47.5	47.5		100.00
14.735(1.023)	215	50451			0.00- 39.35	19.35
14.735(1.023)	108	60338			3.14- 43.14	23.14
69 1-Naphthylamine					CAS #: 134-32-7	
14.977(1.040)	143	896970	53.8	53.8		100.00
14.977(1.040)	115	328253			16.60- 56.60	36.60
71 2,3,4,6-Tetrachlorophenol					CAS #: 58-90-2	
15.080(1.047)	232	147768	45.6	45.6		100.00
15.080(1.047)	230	113738			56.97- 96.97	76.97
15.067(1.046)	131	81457			35.12- 75.12	55.13
73 2-Naphthylamine					CAS #: 91-59-8	
15.144(1.051)	143	854621	51.5	51.5		100.00
15.131(1.051)	115	326399			18.19- 58.19	38.19
77 Thionazin					CAS #: 297-97-2	
15.489(1.075)	97	180846	56.3	56.3		100.00
15.489(1.075)	96	162645			69.94- 109.94	89.94
15.489(1.075)	143	91434			30.56- 70.56	50.56
80 5-Nitro-O-Toluidine					CAS #: 99-55-8	
15.540(1.079)	152	268767	47.2	47.2		100.00
15.540(1.079)	106	177687			46.11- 86.11	66.11
15.540(1.079)	77	157015			38.42- 78.42	58.42
82 Sulfotep					CAS #: 3689-24-5	
16.128(0.927)	97	117830	59.4	59.4		100.00
16.141(0.928)	322	119801			81.67- 121.67	101.67
16.128(0.927)	65	80327			48.17- 88.17	68.17
86 sym-Trinitrobenzene					CAS #: 99-35-4	
16.320(0.938)	213	59761	48.2	48.2		100.00
16.308(0.937)	75	100566			148.28- 188.28	168.28
16.320(0.938)	120	37153			42.17- 82.17	62.17
88 Phorate					CAS #: 298-02-2	
16.384(0.942)	121	189495	57.4	57.4		100.00
16.384(0.942)	75	502455			245.15- 285.15	265.15
16.397(0.943)	260	49019			5.87- 45.87	25.87
89 Phenacetin					CAS #: 62-44-2	
16.423(0.944)	108	347380	51.8	51.8		100.00
16.423(0.944)	137	165849			27.74- 67.74	47.74
16.423(0.944)	179	176458			30.80- 70.80	50.80
93 Dimethoate					CAS #: 60-51-5	
16.717(0.961)	87	375379	53.7	53.7		100.00
16.717(0.961)	125	162269			23.23- 63.23	43.23
16.717(0.961)	229	14013			0.00- 23.73	3.73

RT (REL RT)	MASS	RESPONSE	CONCENTRATIONS ON-COL (ug/ml)	FINAL (ug/L)	TARGET RANGE	RATIO
94 4-Aminobiphenyl					CAS #: 92-67-1	
17.037(0.979)	169	653877	51.3	51.3		100.00
17.037(0.979)	168	145518			2.25- 42.25	22.25
17.037(0.979)	115	48333			0.00- 27.39	7.39
95 Pentachloronitrobenzene					CAS #: 82-68-8	
17.049(0.980)	237	45644	54.6	54.6		100.00
17.049(0.980)	249	35467			57.70- 97.70	77.70
17.037(0.979)	142	52566			95.17- 135.17	115.17
96 Pronamide					CAS #: 23950-58-5	
17.190(0.988)	173	259985	50.2	50.2		100.00
17.190(0.988)	175	163546			42.91- 82.91	62.91
17.190(0.988)	145	87395			13.62- 53.62	33.62
99 2-secbutyl-4,6-Dinitropheno					CAS #: 88-85-7	
17.421(1.001)	211	109861	50.8	50.8		100.00
17.421(1.001)	163	72046			45.58- 85.58	65.58
17.421(1.001)	147	50843			26.28- 66.28	46.28
100 Disulfoton					CAS #: 0298-04-4	
17.446(1.003)	88	444816	57.0	57.0		100.00
17.446(1.003)	60	104297			3.45- 43.45	23.45
17.446(1.003)	89	174872			19.31- 59.31	39.31
* 102 Phenanthrene-d10					CAS #:	
17.395(1.000)	188	622612	40.0	40.0		100.00
17.395(1.000)	94	135952			1.84- 41.84	21.84
104 Methyl Parathion					CAS #: 298-00-0	
18.188(1.046)	109	284534	54.3	54.3		100.00
18.188(1.046)	125	215100			55.60- 95.60	75.60
18.188(1.046)	263	102305			15.96- 55.96	35.96
107 Parathion					CAS #: 56-38-2	
18.993(1.092)	97	146312	55.0	55.0		100.00
18.993(1.092)	291	72253			29.38- 69.38	49.38
18.993(1.092)	109	149562			82.22- 122.22	102.22
108 4-Nitroquinoline-1-oxide					CAS #: 56-57-5	
19.006(1.093)	190	74708	44.6	44.6		100.00
19.006(1.093)	160	28319			17.91- 57.91	37.91
19.006(1.093)	89	39965			33.49- 73.49	53.50
109 Methapyrilene					CAS #: 91-80-5	
19.211(1.104)	58	321780	53.7	53.7		100.00
19.211(1.104)	72	93314			9.00- 49.00	29.00
19.211(1.104)	97	246721			56.67- 96.67	76.67
112 p-Dimethylaminoazobenzene					CAS #: 60-11-7	
20.847(0.933)	120	341045	54.8	54.8		100.00

RT (REL RT)	MASS	RESPONSE	CONCENTRATIONS ON-COL (ug/ml)	FINAL (ug/L)	TARGET RANGE	RATIO
20.847(0.933)	225	160808			27.15- 67.15	47.15
20.847(0.933)	77	116715			14.22- 54.22	34.22
115 3,3'-Dimethylbenzidine					CAS #: 119-93-7	
21.435(0.959)	212	229981	45.5	45.5		100.00
21.435(0.959)	106	61810			6.88- 46.88	26.88
117 2-Acetylaminofluorene					CAS #: 53-96-3	
21.844(0.977)	181	263031	49.3	49.3		100.00
21.844(0.977)	223	129601			29.27- 69.27	49.27
21.844(0.977)	152	67854			5.80- 45.80	25.80
* 122 Chrysene-d12					CAS #:	
22.356(1.000)	240	404840	40.0	40.0		100.00
22.356(1.000)	236	95588			3.61- 43.61	23.61
22.343(1.000)	120	133289			12.92- 52.92	32.92
124 7,12-Dimethylbenz(a)anthrac					CAS #: 57-97-6	
23.877(0.978)	256	277894	52.6	52.6		100.00
23.877(0.978)	241	150300			34.09- 74.09	54.09
* 129 Perylene-d12					CAS #:	
24.415(1.000)	264	440888	40.0	40.0		100.00
24.415(1.000)	260	97341			2.08- 42.08	22.08
24.415(1.000)	265	94770			1.50- 41.50	21.50
130 3-Methylcholanthrene					CAS #: 56-49-5	
24.850(1.112)	268	305969	50.0	50.0		100.00
24.850(1.112)	252	117496			18.40- 58.40	38.40
24.850(1.112)	126	154774			30.58- 70.58	50.58
46 N-Nitroso-Di-N-Butylamine					CAS #: 924-16-3	
11.757(1.076)	84	306090	46.4	46.4		100.00
11.757(1.076)	57	187406			41.23- 81.23	61.23
11.770(1.077)	116	109062			15.63- 55.63	35.63

QC Flag Legend

M - Compound response manually integrated.

B-12A

FIXED / Updated

Enseco Rocky Mountain Analytical Laboratory

BNA ANALYSIS QUANITATION REPORT

Data file : /chem/T.i/101392B.b/t0381.d
Lab. Id. :
Inj Date : 13-OCT-92 17:56 Autotune Date: 09-Oct-92 09:30:3
Operator : da Inst ID: T.i
Smp Info : 50 ppm HSLstd V#6531
Misc Info :
Comment :
Method : /chem/T.i/101392B.b/AP9smpl.m
Meth Date : 13-Oct-1992 19:14
Cal Date : 13-OCT-92 17:56 Cal File: t0381.d
Als bottle: 15 Continuing Calibration Sample
Dil Factor: 1.000 Target Version: Target 2.00
Integrator: HP RTE Compound Sublist: HSL.sub
Rpt Date : 13-Oct-1992 20:12 Sample Type: WATER

RT (REL RT)	MASS	RESPONSE	CONCENTRATIONS ON-COL (ug/ml)	FINAL (ug/L)	TARGET RANGE	RATIO
1 N-Nitrosodimethylamine					CAS #: 62-75-9	
4.141(0.485)	74	894655	58.5	58.5		100.00
4.128(0.484)	42	366870			21.01- 61.01	41.01
3 Pyridine					CAS #: 110-86-1	
4.154(0.487)	79	1272083	57.5	57.5		100.00
4.141(0.485)	52	865354			48.03- 88.03	68.03
4.141(0.485)	78	150864			0.00- 31.86	11.86
$ 8 2-Fluorophenol					CAS #:	
6.275(0.735)	112	2397104	120	120		100.00
6.275(0.735)	64	1045326			23.61- 63.61	43.61
6.275(0.735)	92	604085			5.20- 45.20	25.20
$ 11 Phenol-d5					CAS #:	
7.872(0.922)	99	2149248	113	113		100.00
7.872(0.922)	71	420610			0.00- 39.57	19.57
12 Phenol					CAS #: 108-95-2	
7.898(0.925)	94	1218748	56.3	56.3		100.00
7.898(0.925)	66	309866			5.42- 45.42	25.42
13 Aniline					CAS #: 62-53-3	
7.949(0.931)	93	1391245	54.6	54.6		100.00
7.949(0.931)	92	160178			0.00- 31.51	11.51
14 bis(-2-Chloroethyl)Ether					CAS #: 111-44-4	
8.076(0.946)	93	1172796	58.4	58.4		100.00
8.076(0.946)	63	680793			38.05- 78.05	58.05
15 2-Chlorophenol					CAS #: 95-57-8	

RT (REL RT)	MASS	RESPONSE	CONCENTRATIONS ON-COL (ug/ml)	FINAL (ug/L)	TARGET RANGE	RATIO
8.153(0.955)	128	1151441	57.0	57.0		100.00
8.153(0.955)	130	357321			11.03- 51.03	31.03
16 1 3-Dichlorobenzene					CAS #: 541-73-1	
8.434(0.988)	146	1086608	55.9	55.9		100.00
8.434(0.988)	148	689519			43.46- 83.46	63.46
8.434(0.988)	111	442550			20.73- 60.73	40.73
* 19 1 4-Dichlorobenzene-d4					CAS #:	
8.536(1.000)	152	515361	40.0	40.0		100.00
8.536(1.000)	150	967729			167.78- 207.78	187.78
8.536(1.000)	115	316632			41.44- 81.44	61.44
21 1 4-dichlorobenzene					CAS #: 106-46-7	
8.575(1.004)	146	1015806	53.8	53.8		100.00
8.575(1.004)	148	652155			44.20- 84.20	64.20
8.575(1.004)	111	399440			19.32- 59.32	39.32
23 Benzyl Alcohol					CAS #: 100-51-6	
8.792(1.030)	108	671722	57.1	57.1		100.00
8.792(1.030)	79	656237			77.69- 117.69	97.69
8.792(1.030)	107	465483			49.30- 89.30	69.30
24 1 2-Dichlorobenzene					CAS #: 95-50-1	
8.843(1.036)	146	1008052	54.6	54.6		100.00
8.843(1.036)	148	633330			42.83- 82.83	62.83
8.843(1.036)	111	429723			22.63- 62.63	42.63
25 2-Methylphenol					CAS #: 95-48-7	
8.984(1.052)	108	881579	57.6	57.6		100.00
8.984(1.052)	107	749884			65.06- 105.06	85.06
26 bis(2-Chloroisopropyl)ether					CAS #: 108-60-1	
9.047(1.060)	45	1042859	55.2	55.2		100.00(M)
9.047(1.060)	77	116314			0.00- 31.15	11.15
28 4-Methylphenol					CAS #: 106-44-5	
9.277(1.087)	108	844792	55.4	55.4		100.00
9.277(1.087)	107	1014063			100.04- 140.04	120.04
29 N-nitroso-Di-n-propylamine					CAS #: 621-64-7	
9.303(1.090)	70	535786	53.7	53.7		100.00
9.303(1.090)	43	376100			50.20- 90.20	70.20
9.303(1.090)	130	199289			17.20- 57.20	37.20
30 Hexachloroethane					CAS #: 67-72-1	
9.469(1.109)	117	413293	58.9	58.9		100.00
9.469(1.109)	201	228660			35.33- 75.33	55.33
9.469(1.109)	199	145431			15.19- 55.19	35.19
$ 31 Nitrobenzene-d5					CAS #:	

RT (REL RT)	MASS	RESPONSE	CONCENTRATIONS ON-COL (ug/ml)	FINAL (ug/L)	TARGET RANGE	RATIO
9.559(0.875)	82	763217	58.3	58.3		100.00
9.559(0.875)	54	325426			22.64- 62.64	42.64
9.559(0.875)	128	550704			52.16- 92.16	72.16
32 Nitrobenzene					CAS #: 98-95-3	
9.597(0.878)	77	753383	58.1	58.1		100.00
9.597(0.878)	123	578400			56.77- 96.77	76.77
9.597(0.878)	51	328890			23.66- 63.66	43.66
35 Isophorone					CAS #: 78-59-1	
10.057(0.920)	82	1427821	56.1	56.1		100.00
10.057(0.920)	138	307566			1.54- 41.54	21.54
36 2-Nitrophenol					CAS #: 88-75-5	
10.197(0.933)	139	644840	55.6	55.6		100.00
10.197(0.933)	65	174836			7.11- 47.11	27.11
10.197(0.933)	109	87825			0.00- 33.62	13.62
37 2 4-Dimethyphenol					CAS #: 105-67-9	
10.287(0.942)	107	598571	51.4	51.4		100.00
10.287(0.942)	122	625485			84.50- 124.50	104.50
10.287(0.942)	77	130833			1.86- 41.86	21.86
38 Benzoic Acid					CAS #: 65-85-0	
10.530(0.964)	122	492187	51.4	51.4		100.00(M)
10.543(0.965)	105	531426			87.97- 127.97	107.97
10.543(0.965)	77	316070			44.22- 84.22	64.22
40 bis(-2-Chloroethoxy)Methane					CAS #: 111-91-1	
10.479(0.959)	93	964562	57.1	57.1		100.00
10.479(0.959)	63	512077			33.09- 73.09	53.09
10.479(0.959)	95	303270			11.44- 51.44	31.44
42 2 4-Dichlorophenol					CAS #: 120-83-2	
10.657(0.975)	162	596967	51.8	51.8		100.00
10.657(0.975)	164	377564			43.25- 83.25	63.25
10.657(0.975)	98	231025			18.70- 58.70	38.70
43 1 2 4-Trichlorobenzene					CAS #: 120-82-1	
10.824(0.991)	180	586351	51.2	51.2		100.00
10.824(0.991)	182	545905			73.10- 113.10	93.10
10.824(0.991)	145	202780			14.58- 54.58	34.58
* 44 Naphthalene-d8					CAS #:	
10.926(1.000)	136	1704215	40.0	40.0		100.00
10.926(1.000)	68	144872			0.00- 28.50	8.50
45 Naphthalene					CAS #: 91-20-3	
10.977(1.005)	128	1997417	57.8	57.8		100.00
10.977(1.005)	129	222399			0.00- 31.13	11.13
10.977(1.005)	102	125226			0.00- 26.27	6.27

RT (REL RT)	MASS	RESPONSE	CONCENTRATIONS ON-COL (ug/ml)	FINAL (ug/L)	TARGET RANGE	RATIO
48 4-Chloroaniline					CAS #: 106-47-8	
11.079(1.014)	127	1083469	56.2	56.2		100.00
11.079(1.014)	129	317115			9.27- 49.27	29.27
49 Hexachlorobutadiene					CAS #: 87-68-3	
11.220(1.027)	225	221881	50.8	50.8		100.00
11.220(1.027)	190	96388			23.44- 63.44	43.44
11.220(1.027)	118	85244			18.42- 58.42	38.42
52 4-Chloro-3-Methylphenol					CAS #: 59-50-7	
12.037(1.102)	107	620626	56.7	56.7		100.00
12.037(1.102)	142	564605			70.97- 110.97	90.97
53 2-Methylnaphthalene					CAS #: 91-57-6	
12.331(1.129)	142	1188868	55.6	55.6		100.00
12.331(1.129)	141	1012213			65.14- 105.14	85.14
55 Hexachlorocyclopentadiene					CAS #: 77-47-4	
12.651(0.878)	237	250200	54.3	54.3		100.00
12.651(0.878)	239	159350			43.69- 83.69	63.69
12.651(0.878)	235	155902			42.31- 82.31	62.31
57 2 4 6-Trichlorphenol					CAS #: 88-06-2	
12.894(0.894)	196	250880	49.8	49.8		100.00
12.894(0.894)	198	239680			75.54- 115.54	95.54
58 2 4 5-Trichlorophenol					CAS #: 95-95-4	
12.957(0.899)	196	297349	52.4	52.4		100.00
12.957(0.899)	198	279876			74.12- 114.12	94.12
$ 60 2-Fluorobiphenyl					CAS #:	
13.073(0.907)	172	1056839	57.4	57.4		100.00
13.085(0.908)	171	354061			13.50- 53.50	33.50
62 2-Chloronaphthalene					CAS #: 91-58-7	
13.303(0.923)	162	1041780	57.7	57.7		100.00
13.303(0.923)	164	327798			11.47- 51.47	31.47
13.303(0.923)	127	431182			21.39- 61.39	41.39
63 2-Nitroaniline					CAS #: 88-74-4	
13.507(0.937)	65	299193	59.2	59.2		100.00
13.507(0.937)	138	506077			149.15- 189.15	169.15
13.507(0.937)	92	233199			57.94- 97.94	77.94
65 Dimethyl Phthalate					CAS #: 131-11-3	
13.916(0.965)	163	981678	51.2	51.2		100.00
13.903(0.965)	77	155250			0.00- 35.81	15.81
66 2 6-Dinitrotoluene					CAS #: 606-20-2	
14.018(0.973)	165	284683	52.7	52.7		100.00
14.018(0.973)	89	95702			13.62- 53.62	33.62

RT (REL RT)	MASS	RESPONSE	CONCENTRATIONS ON-COL (ug/ml)	FINAL (ug/L)	TARGET RANGE	RATIO
68 Acenaphthylene					CAS #: 208-96-8	
14.121(0.980)	152	1570795	58.6	58.6		100.00
14.121(0.980)	151	311918			0.00- 39.86	19.86
* 72 Acenaphthene-d10					CAS #:	
14.414(1.000)	164	597451	40.0	40.0		100.00
14.414(1.000)	162	575582			76.34- 116.34	96.34
14.414(1.000)	160	238261			19.88- 59.88	39.88
74 Acenaphthene					CAS #: 83-32-9	
14.478(1.004)	153	965976	56.8	56.8		100.00
14.478(1.004)	152	433922			24.92- 64.92	44.92
14.478(1.004)	154	911179			74.33- 114.33	94.33
70 3-Nitroaniline					CAS #: 99-09-2	
14.338(0.995)	138	363463	55.7	55.7		100.00
14.338(0.995)	92	297351			61.81- 101.81	81.81
75 2 4-Dinitrophenol					CAS #: 51-28-5	
14.555(1.010)	184	126676	56.5	56.5		100.00
14.542(1.009)	107	41861			13.05- 53.05	33.05
14.555(1.010)	91	53431			22.18- 62.18	42.18
76 4-Nitrophenol					CAS #: 100-02-7	
14.683(1.019)	109	72489	56.9	56.9		100.00
14.683(1.019)	139	285433			373.76- 413.76	393.76
14.683(1.019)	65	148263			184.53- 224.53	204.53
78 2 4-Dinitrotoluene					CAS #: 121-14-2	
14.823(1.028)	165	319415	55.5	55.5		100.00
14.823(1.028)	63	114252			15.77- 55.77	35.77
79 Dibenzofuran					CAS #: 132-64-9	
14.823(1.028)	168	1211025	55.8	55.8		100.00
14.823(1.028)	139	363124			9.98- 49.98	29.98
81 Diethylphthalate					CAS #: 84-66-2	
15.347(1.065)	149	990009	55.8	55.8		100.00
15.347(1.065)	177	197687			0.00- 39.97	19.97
15.347(1.065)	150	114237			0.00- 31.54	11.54
83 4-Chlorophenyl-phenylether					CAS #: 7005-72-3	
15.539(1.078)	204	268155	50.1	50.1		100.00
15.539(1.078)	206	87143			12.50- 52.50	32.50
15.539(1.078)	141	186607			49.59- 89.59	69.59
84 Fluorene					CAS #: 86-73-7	
15.513(1.076)	166	882682	56.0	56.0		100.00
15.513(1.076)	165	802794			70.95- 110.95	90.95
85 4-Nitroaniline					CAS #: 100-01-6	

RT (REL RT)	MASS	RESPONSE	CONCENTRATIONS ON-COL (ug/ml)	FINAL (ug/L)	TARGET RANGE	RATIO
15.565(1.080)	138	280062	52.5	52.5		100.00
15.565(1.080)	92	86894			11.03- 51.03	31.03
15.565(1.080)	108	75215			6.86- 46.86	26.86
87 4 6-Dinitro-2-methylphenol					CAS #: 534-52-1	
15.642(0.899)	198	154243	56.3	56.3		100.00
15.642(0.899)	105	69815			25.26- 65.26	45.26
90 N-nitrosodiphenylamine					CAS #: 86-30-6	
15.782(0.907)	169	599765	56.1	56.1		100.00
15.782(0.907)	168	400917			46.85- 86.85	66.85
15.782(0.907)	167	207362			14.57- 54.57	34.57
91 Azobenzene					CAS #: 103-33-3	
15.859(0.912)	77	1001905	62.1	62.1		100.00
15.859(0.912)	182	261284			6.08- 46.08	26.08
15.859(0.912)	105	278583			7.81- 47.81	27.81
$ 92 2 4 6-Tribromophenol					CAS #:	
16.000(1.110)	330	300613	100	100		100.00
16.000(1.110)	332	288779			76.06- 116.06	96.06
16.000(1.110)	141	79332			6.39- 46.39	26.39
97 4-Bromophenyl-phenylether					CAS #: 101-55-3	
16.524(0.950)	248	206681	52.7	52.7		100.00
16.524(0.950)	250	198618			76.10- 116.10	96.10
16.511(0.949)	141	170099			62.30- 102.30	82.30
98 Hexachlorobenzene					CAS #: 118-74-1	
16.626(0.956)	284	261395	53.3	53.3		100.00
16.613(0.955)	142	103008			19.41- 59.41	39.41
101 Pentachlorophenol					CAS #: 87-86-5	
17.010(0.978)	266	156148	51.2	51.2		100.00
17.010(0.978)	264	99103			43.47- 83.47	63.47
17.010(0.978)	268	101084			44.74- 84.74	64.74
* 102 Phenanthrene-d10					CAS #:	
17.394(1.000)	188	780942	40.0	40.0		100.00
17.394(1.000)	94	161749			0.71- 40.71	20.71
103 Phenanthrene					CAS #: 85-01-8	
17.445(1.003)	178	1059814	58.0	58.0		100.00
17.445(1.003)	176	197585			0.00- 38.64	18.64
105 Anthracene					CAS #: 120-12-7	
17.547(1.009)	178	1022489	56.0	56.0		100.00
17.547(1.009)	176	181447			0.00- 37.75	17.75
110 Di-n-Butylphthalate					CAS #: 84-74-2	
18.659(1.073)	149	1510062	63.8	63.8		100.00

RT (REL RT)	MASS	RESPONSE	CONCENTRATIONS ON-COL (ug/ml)	FINAL (ug/L)	TARGET RANGE	RATIO
18.659(1.073)	150	138890			0.00- 29.20	9.20
111 Fluoranthene					CAS #: 206-44-0	
19.834(1.140)	202	932528	57.9	57.9		100.00
19.834(1.140)	101	234145			5.11- 45.11	25.11
114 Pyrene					CAS #: 129-00-0	
20.269(0.906)	202	964118	51.4	51.4		100.00
20.269(0.906)	101	280606			9.10- 49.10	29.10
113 Benzidine					CAS #: 92-87-5	
20.115(0.899)	184	16641	10.2	10.2		100.00
$ 116 Terphenyl-d14					CAS #:	
20.601(0.921)	244	619646	48.8	48.8		100.00
20.588(0.921)	122	161828			6.12- 46.12	26.12
118 Butylbenzylphthalate					CAS #: 85-68-7	
21.483(0.961)	149	682807	56.2	56.2		100.00
21.483(0.961)	91	371912			34.47- 74.47	54.47
21.483(0.961)	206	125489			0.00- 38.38	18.38
119 3 3'-Dichlorobenzidine					CAS #: 91-94-1	
22.314(0.998)	252	254298	49.3	49.3		100.00
22.314(0.998)	254	161894			43.66- 83.66	63.66
120 bis(2-ethylhexyl)Phthalate					CAS #: 117-81-7	
22.429(1.003)	149	993335	61.0	61.0		100.00
22.429(1.003)	167	361624			16.41- 56.41	36.41
121 Benzo(a)Anthracene					CAS #: 56-55-3	
22.339(0.999)	228	788265	53.2	53.2		100.00
22.339(0.999)	229	151851			0.00- 39.26	19.26
22.339(0.999)	226	199504			5.31- 45.31	25.31
* 122 Chrysene-d12					CAS #:	
22.365(1.000)	240	523455	40.0	40.0		100.00
22.365(1.000)	236	126056			4.08- 44.08	24.08
22.365(1.000)	120	130248			4.88- 44.88	24.88
123 Chrysene					CAS #: 218-01-9	
22.404(1.002)	228	722264	53.0	53.0		100.00
22.404(1.002)	226	193111			6.74- 46.74	26.74
22.404(1.002)	229	132507			0.00- 38.35	18.35
125 Di-n-octyl Phthalate					CAS #: 117-84-0	
23.376(0.957)	149	1677797	64.6	64.6		100.00
23.376(0.957)	150	164834			0.00- 29.82	9.82
126 Benzo(b)fluoranthene					CAS #: 205-99-2	
23.887(0.978)	252	920586	55.3	55.3		100.00

RT	(REL RT)	MASS	RESPONSE	CONCENTRATIONS ON-COL (ug/ml)	FINAL (ug/L)	TARGET RANGE	RATIO
23.887	(0.978)	253	193629			1.03- 41.03	21.03
127 Benzo(k)fluoranthene						CAS #: 207-08-9	
23.926	(0.980)	252	746050	51.3	51.3		100.00(H)
23.926	(0.980)	253	157577			1.12- 41.12	21.12
128 Benzo(a)pyrene						CAS #: 50-32-8	
24.348	(0.997)	252	743133	52.9	52.9		100.00
24.348	(0.997)	253	164253			2.10- 42.10	22.10
* 129 Perylene-d12						CAS #:	
24.425	(1.000)	264	626282	40.0	40.0		100.00
24.425	(1.000)	260	132937			1.23- 41.23	21.23
24.425	(1.000)	265	129512			0.68- 40.68	20.68
131 Indeno(1 2 3-cd)pyrene						CAS #: 193-39-5	
26.150	(1.071)	276	832382	51.7	51.7		100.00
26.163	(1.071)	138	527474			43.37- 83.37	63.37
132 Dibenzo(a h)anthracene						CAS #: 53-70-3	
26.176	(1.072)	278	687709	53.1	53.1		100.00
26.176	(1.072)	279	156003			2.68- 42.68	22.68
26.176	(1.072)	139	242001			15.19- 55.19	35.19
133 Benzo(g h i)perylene						CAS #: 191-24-2	
26.662	(1.092)	276	744930	51.4	51.4		100.00
26.662	(1.092)	138	424197			36.94- 76.94	56.94
26.662	(1.092)	277	176858			3.74- 43.74	23.74
106 Carbazole						CAS #: 86-74-8	
17.879	(1.028)	167	702533	61.8	61.8		100.00
17.879	(1.028)	166	136447			0.00- 39.42	19.42

QC Flag Legend

M - Compound response manually integrated.
H - Operator selected an alternate compound hit.

SOP Number: LM-RMA-3001
Title: GC/MS Analysis of Volatile Organics

This addendum is specific to the Chemical Waste Management, Vickery, Ground Water Monitoring Plan QAPjP. Procedural changes or clarification statements presented herein are not applicable to Enseco SOPs for other projects, and are made here in response to EPA letters dated March 17, 1992 and September 15, 1992 to CWM containing comments (with additional attachments) regarding the CWM-Vickery GWMP QAPjP.

Section 1.2: See QAPjP Appendix A-10 for the project specific "reporting limits".

Section 7.3 to Section 7.6: Referenced SOPs are included in QAPjP Appendix B-13A.

Section 8.3: Referenced SOP LM-RMA-3022 is included ia QAPjP Appendix B-13A.

Section 8.4: All samples are homogenized prior to analysis. Aqueous samples only will be analyzed for this project, percent moisture determination is not applicable.

Section 8.7.1.2: Ion Abundance Criteria for BFB is found in Table C-1 of the SOP Appendix C, which is present near the end of QAPjP Appendix B-13.

The wording of the section's second and third paragraphs remain as stated, and are intended to be instructive to the analyst.

Section 8.7.2: For the purpose of this project, the next-to-last paragraph of the section is deleted. Specific compounds being analyzed for are listed in the project parameter listings in Section 7 of the GWMP.

Section 8.9: Dilutions of samples are performed within accepted EPA guidelines in SW-846 and the CLP methods. All samples are screened prior to analysis. The screening results are used to optimize the sample analysis to achieve the lowest possible reporting limits. Dilutions are only performed relative to compounds that would interfere with the analysis. Thus, a high concentration of inorganic ions such as sodium would not result in a dilution being performed for volatile organics.

Section 8.9.3.9 to 8.9.3.10: Normal acceptance control limits for matrix spikes, duplicate control sample spike compounds, surrogate control samples and surrogate spike recovery are located immediately following this addendum page. For this project however, drilling mud matrix samples taken during drilling will have no recovery control limit requirements. For any ground water matrix samples, control limits of 80% - 120% will be an objective, but not a requirement. After one year of experience working with the ground water matrix, control limit reqirements will be set. Limits will be specified in the raw data.

Section 11.2: Project specific reporting limits were listed in QAPjP Appendix A-10.

SOP Appendix A: Target compounds are provided in this appendix. The relative retention times for individual compounds per instrument conditions is indicated in the screening calibration data found within the latter portions of QAPjP Appendix B-13B.

GC-MS VOA
MATRIX SPIKE CONTROL LIMITS

Reduced by :_____ Date:_____
Reviewed by:_____ Date:_____

Mass Spectrometry
Spike / Spike Duplicate Report

Comment:
Sample :

	Data File	Run Factor	Extract Vol
Sample:	>L7628	1.00	
Spike :	>L7629	1.00	.0050
Spike Dup:	>L7630	1.00	.0050

Compound	Spike Amt UG	Measured Conc Sample UG/L	RPD	QC Limit
C045 1,1-DICHLOROETHENE	.250	0.00	3	20
C150 TRICHLOROETHENE	.250	0.00	4	13
C165 BENZENE	.250	0.00	3	12
C230 TOLUENE	.250	0.00	0	13
C235 CHLOROBENZENE	.250	0.00	2	10

Spike

Compound	Concentration UG/L Spiked	Measured	% Recovery	QC limits
C045 1,1-DICHLOROETHENE	50.0	42.6	85	56-138
C150 TRICHLOROETHENE	50.0	46.9	94	76-109
C165 BENZENE	50.0	48.2	96	78-119
C230 TOLUENE	50.0	49.2	98	82-114
C235 CHLOROBENZENE	50.0	51.6	103	84-117

Spike Duplicate

Compound	Concentration UG/L Spiked	Measured	% Recovery	QC limits
C045 1,1-DICHLOROETHENE	50.0	41.2	82	56-138
C150 TRICHLOROETHENE	50.0	45.1	90	76-109
C165 BENZENE	50.0	47.0	94	78-119
C230 TOLUENE	50.0	49.1	98	82-114
C235 CHLOROBENZENE	50.0	52.9	106	84-117

GC-MS VOA
DCS CONTROL LIMITS FOR SPIKE COMPOUNDS

QC lot #: Date Analyzed: 10/14/92

Method:
Mass Spectrometry
DCS (Duplicate Control Samples)
ID1FILE HSL/API/BU INST. L

	Data File	Run Factor
Analyst:	LCS #1 >L7629	1.00
Instrument:	LCS# 2 >L7630	1.00
Matrix:		

Sample #	Sample #	Sample #	Sample #	Sample #
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

Compound	Spike Conc. UG/L	Measured Concentration UG/L LCS #1	LCS #2	Average
C045 1,1-DICHLOROETHENE	50.0	42.6	41.2	41.9
C150 TRICHLOROETHENE	50.0	46.9	45.1	46.0
C165 BENZENE	50.0	48.2	47.0	47.6
C230 TOLUENE	50.0	49.2	49.1	49.2
C235 CHLOROBENZENE	50.0	51.6	52.9	52.3

Compound	Accuracy: Average % Recovery	Accuracy: Control Limits	Precision: RPD	Precision: Control Limit
C045 1,1-DICHLOROETHENE	84	56-138	3	20
C150 TRICHLOROETHENE	92	76-109	4	13
C165 BENZENE	95	78-119	3	12
C230 TOLUENE	98	82-114	0	13
C235 CHLOROBENZENE	105	84-117	2	10

Note: 80% of values must be within control limits

100% of Average Percent Recoveries are within control limits
100% of RPD are within control limits

Supervisor Approval:

GC-MS VOA
SURROGATE CONTROL LIMITS FOR BLANKS

QC lot #: Date Analyzed: 10/14/92

Method:
Mass Spectrometry
SCS (Surrogate Control Samples)
ID1FILE HSL/API/BU INST. L

	Data File	Run Factor
Analyst:	>L7628	1.00
Instrument:		
Matrix:		

Sample #	Sample #	Sample #	Sample #	Sample #

Compound	Spike Conc. UG/L	Measured Conc. UG/L	Accuracy Percent Recovery	Accuracy Control Limits
CS15 1,2-DICHLOROETHANE-	50.0	51.1	102	82-112
CS10 BROMOFLUOROBENZENE	50.0	47.1	94	83-113
CS05 TOLUENE-D8	50.0	51.6	103	90-112

Note: 80% Analytes must be within control limits

100% of Average Percent Recoveries are within control limits

Supervisor Approval:

GC-MS VOA
SAMPLE SURROGATE CONTROL LIMITS

educed by : _____ Date: _____ Data File: >L7628
eviewed by: _____ Date: _____ Page: 1

Enseco Mass Spectrometry
Target Compound Data Summary Sheet

Sample:
Misc : 5ML/5ML ANALYST:AF INST:L
Injected : 10/14/92 16:28 Units: UG/L
Analyst: LUEY1 Run Factor: 1.000
ID File: IDBUL Surrogate vol: .005
Quant list threshold: 1.00

Surrogate Spike Recoveries

Compound	Surrogate Amount (ug) Spiked	Measured	% Recovery Measured	QC limits
CS15 1,2-DICHLOROETHANE-D4	.2500	.2557	102	76 114
CS10 BROMOFLUOROBENZENE	.2500	.2356	94.2	86 115
CS05 TOLUENE-D8	.2500	.2579	103	88 110

Target Compounds: IDBUL

R.T.	Concentration Quant List UG/L	Sample UG/L		Compound
	BDL		V001	DICHLORODIFLUOROMETHANE
	BDL		C010	CHLOROMETHANE
	BDL		C020	VINYL CHLORIDE
	BDL		C015	BROMOMETHANE
	BDL		C025	CHLOROETHANE
	BDL		V007	TRICHLOROFLUOROMETHANE
	BDL		V003	DICHLOROFLUOROMETHANE
	BDL		C045	1,1-DICHLOROETHENE
	BDL		V005	ACROLEIN
	BDL		V004	IODOMETHANE
	BDL		C040	CARBON DISULFIDE
	BDL		V015	TRICHLORO-TRIFLUOROETHANE
167	93.867	94	V040	ETHANOL
	BDL		C035	ACETONE
	BDL		C030	METHYLENE CHLORIDE
	BDL		VJNK	trans-1,2-DICHLOROETHENE
	BDL		V006	ACRYLONITRILE
	BDL		VJNK	TERT-BUTYL METHYL ETHER
	BDL		V009	HEXANE
	BDL		C050	1,1-DICHLOROETHANE
	BDL		V011	cis-1,2-DICHLOROETHENE
	BDL		C053	1,2 DICHLOROETHENE TOTAL
	BDL		C110	2-BUTANONE
	BDL		V013	TETRAHYDROFURAN
	BDL		C060	CHLOROFORM
	BDL		C065	1,2-DICHLOROETHANE
	BDL		V016	DIBROMOMETHANE

ADDENDUM TO CWM Vickery GWMP QAPjP Appendix B-20
SOP Number: LM-RMA-1100
Title: Total, Fixed and Volatile Solids

This addendum is specific to the Chemical Waste Management, Vickery, Ground Water Monitoring Plan QAPjP. Procedural changes or clarification statements presented herein are not applicable to Enseco SOPs for other projects, and are made here in response to an EPA letter dated March 17, 1992 to CWM containing comments (with additional attachment) regarding the CWM-Vickery GWMP QAPjP.

Section 1.1: Only the total solids of samples from the Brassfield monitor well will be determined using this method, within the scope of this project. Total solids will not be determined for Knox-Kerbel monitor well samples.

Section 2: Only Sub-Section 2.1 (total solids) is applicable to this project.

Section 8.2: Only aqueous samples are involved in this project. Sample containers with the tops in place will be inverted rapidly three times to homogenize the sample before drawing the aliquot for analysis.

Section 9.1.3: Duplicate analyses at a frequency of one per batch of 20 or fewer samples will be performed. There is currently no guidance for control limits for duplicate analyses. Both MCAWW method 160.3 and method 2504G in the 17th Edition of Standard Methods do not have control limits for these analyses. Laboratory acceptance control limits are matrix specific, and will be determined and reported with the raw data.

TEXAS WORLD OPERATIONS, INC.

DATA VALIDATION SERVICES
QUALITY ASSURANCE PROJECT PLAN

Prepared By:

QuantaLex, Inc.
300 Union Boulevard
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Lakewood, Colorado 80228
(303) 763-8881

October 1992

Controlled Copy No. [ ]

TEXAS WORLD OPERATIONS, INC.
DATA VALIDATION SERVICES
QUALITY ASSURANCE PROJECT PLAN

Developed By: _____ Date: _____

Ken Schroeder
Quality Assurance Manager
QuantaLex, Inc.

Approved By: _____ Date: _____

Keith Wegner
President
QuantaLex, Inc.

Concurrence: _____ Date: _____

Robert Thielke
Technical Operations Manager
QuantaLex, Inc.

October 1992
Version 0.0

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APPENDICES

Appendix I Texas World Operations, Inc. Subcontract Statement of Work

1.0 INTRODUCTION

QuantaLex, Inc. (QuantaLex) is committed to delivering high quality, usable work products which are scientifically sound and technically defensible. All work products developed by QuantaLex are prepared under comprehensive quality assurance/quality control (QA/QC) protocols and procedures. The QA/QC protocols described in this plan are consistent with the QuantaLex QA Program Plan, contract requirements, and applicable quality requirements and standards.

This QuantaLex Quality Assurance Project Plan (QA Project Plan) governs all quality-related and quality-affecting activities conducted under the Texas World Operations, Inc. (Texas World) subcontract. The QA Project Plan has been developed by the QA manager with the assistance of the project manager and the president.

1.1 Purpose

This QA Project Plan defines and describes the specific policies and procedures by which QuantaLex assures that the work products developed, services provided, applications performed, and reports delivered are of sound technical, legal, and scientific quality; are usable; satisfy all contract requirements; and are supported by an adequate record of their evolution. The QA Project Plan also defines the work product standards adopted by QuantaLex to ensure that products and services delivered will meet or exceed client expectations in terms of completeness, accuracy, consistency, and comparability.

1.2 Scope

The QA Project Plan encompasses the project management procedures by which QuantaLex controls the development, adaptation, application, and delivery of its products and services to meet contract requirements. The plan specifically provides the QA/QC guidance for the development, adaptation, application, and production of contract deliverables. These deliverables include:

- Reduced and full data validation worksheets
- Reduced and full data validation reports, specifying deficiencies and recommendations for qualifiers
- Analysis Data Sheets (Form 1) or a summary of results with appropriate qualifiers

Ad hoc deliverables will be developed consistent with the requirements applied to standard deliverables and the protocols contained herein.

1.3 Applicable QA Requirements and Standards

The following quality assurance requirements and standards apply to the Texas World subcontract work and the applicable portions are incorporated into this QA Project Plan:

- "Guidelines and Specifications for Preparing Quality Assurance Project Plans;" USEPA-Quality Assurance Management Staff (EPA QAMS) 005/80; 10/80.
- "QuantaLex Quality Assurance Program Plan;" QuantaLex, Inc., Version 2.0; 8/92.

The applicable requirements of "EPA QAMS (005/80)" are cross-referenced to this QA Project Plan in Table 1-1. Applicability of the requirements was determined by the president.

1.4 Definitions

Key terms used in this QA Project Plan are defined below.

Quality Assurance - The overall system or program of organizational and procedural checks and balances and associated standards which assure the quality and reliability of products and services. Quality assurance also defines the organizational and procedural authority under which quality controls are implemented, conducted, and evaluated.

Quality Control - The systematic performance of checks and reviews of work products according to prescribed SOPs to identify any deviations from applicable work product standards. Quality control is the implementation of the QA Program as applied to specific project tasks, activities, and work products.

Work Product Standards - Those *objective* measures of correctness such as compliance to guidelines, spelling, diction, grammar, arithmetic, internal consistency, accurate translation/extraction and style (e.g., format, organization, content). Work product standards also include *subjective* measures of correctness such as completeness, clarity, logic, responsiveness, content related to professional judgments, inferences, analyses, and conclusions which are adopted by QuantaLex for its work products. Work product standards are developed specific to each program or project and are defined in the QA project plans.

Standard Operating Procedure - A document which describes in detail performance of a task or activity whose mechanisms are standardized and/or commonly accepted as the methods for performing these tasks or activities.

Independent Review - Quality control process whereby a review of a work product is conducted by a staff member who was not directly involved in the development or execution of the work product.

Peer Review - Quality control process whereby an independent staff member with appropriate expertise and experience performs a review of a work product based on subjective and objective work product standards and project requirements.

Management Review - Quality control process whereby a manager with appropriate expertise and experience performs a review of a work product based on subjective and objective work product standards and program, project, and/or contractual requirements.

Production Review - Quality control process whereby an independent staff member performs a review of a work product based on objective work product standards.

Technical Review - Quality control process whereby an independent, qualified, staff member performs a verification activity to determine the technical quality of the work product.

Reduced Data Validation - A shortened version of the data validation process wherein only specified QC criteria are evaluated by the data validator. Typically the data package contains report forms and little, if any, raw data.

Full Data Validation - The standard data validation process wherein all QC criteria are evaluated by the data validator. The data package contains the report forms and all raw data.

Audit - A formal, systematic inspection and evaluation of the status and effectiveness of a program and its elements and controls. The evaluation also includes documented identification and correction of existing or potential deficiencies.

Surveillance - A systematic inspection and evaluation of a particular process, project, or specific program activity. The surveillance is typically a documented, informal QA evaluation activity.

TABLE 1-1
QA REQUIREMENTS CROSS-REFERENCE
(EPA QAMS TO QA PROJECT PLAN)

EPA QAMS Requirements	Applicable (Yes/No)	QA Project Plan Reference Section
1. Title Page	Yes	Title Page
2. Table of Contents	Yes	Table of Contents
3. Project Description	Yes	Section 2.0
4. Project Organization	Yes	Section 4.0
5. Quality Assurance Objectives	Yes	Section 7.0
6. Sampling Procedures	No	- -
7. Sample Custody	No	- -
8. Calibration Procedures & Frequency	No	- -
9. Analytical Procedures	No	- -
10. Data Analysis, Validation, and Reporting	Yes	Sections 6.0, 8.0
11. Internal Quality Control Checks	Yes	Section 9.0
12. Performance and System Audits	Yes	Section 11.0
13. Preventative Maintenance	No	- -
14. Procedures for Data Assessment	Yes	Section 8.0
15. Corrective Action	Yes	Section 11.0
16. Quality Assurance Reports to Management	Yes	Section 13.0

2.0 STATEMENT OF WORK

The Statement of Work describes the scope of services to be delivered under the subcontract and specific requirements with respect to tasks, data validation, and deliverables. A copy of the Texas World data validation subcontract Statement of Work is included in Appendix I.

The provisions of this QA Project Plan are designed to address all quality-related or quality-affecting aspects of the subcontract Statement of Work. The QA Project Plan requirements and procedures are designed to ensure that QuantaLex will meet or exceed all contractual obligations regarding quality assurance and quality control.

3.0 QA LEVEL DESIGNATION

Quality assurance objectives define the levels of quality needed by users of QuantaLex products and services to support a particular program, decision, or activity. QuantaLex recognizes two levels of controls for assuring quality: "QA Level One" and "QA Level Two." "QA Level One" consists of the minimum QA requirements applicable to all QuantaLex activities. "QA Level Two" is applied to products and services which must meet the highest levels of performance and work product standards. This quality level is typically associated with products and services which may impact human health, safety, or the environment; that is, substantive failure of the work product would adversely impact or jeopardize human health, safety, or the environment.

QuantaLex will prepare all products and services for this contract under "QA Level Two" requirements. The QA Level Two requirements are described below.

3.1 QA Level Two

"QA Level Two" consists of the maximum QA requirements applicable to all QuantaLex activities and project (client) work. This level of QA is intensive and is reserved for programs and projects requiring the highest standards of accuracy, completeness, consistency, and comparability. The elements of "QA Level Two" are described in Sections 3.1.1 - 3.1.5.

3.1.1 *Document Control*

All documents (including correspondence, reports, printouts, etc.) received or generated by QuantaLex are to be logged into and out of company document control systems as specified in QuantaLex's document control and security SOPs.

3.1.2 *Peer Review*

Documents and reports generated may be subject to documented peer review. Peer reviews are conducted at the discretion of the principal author consistent with the nature and type of work product under development.

3.1.3 *Production Review*

Documents and reports generated may be subject to documented production review. Production reviews are conducted at the discretion of the principal author consistent with the nature and type of work product under development.

3.1.4 *Technical Review*

Each data element, item, or group in databases and spreadsheets developed by QuantaLex and distributed outside of QuantaLex are subject to 100 percent verification during documented technical reviews unless otherwise specified by the client or contractual arrangement.

Reports and documents generated by QuantaLex will be reviewed for conformance to the highest attainable standards for accuracy, completeness, consistency, and comparability.

3.1.5 *Management Review*

All documents and reports generated are subject to documented management review.

4.0 PROGRAM ORGANIZATION

Separate organizations for program administration and quality assurance administration have been established. The corporate QA Program organization is described in Section 5.0. The program organization for managing the activities under the Texas World subcontract is described below.

4.1 Program Organization

The QuantaLex Program Organization Chart for the Texas World Data Validation Program is illustrated in Figure 4-1.

4.2 Duties and Responsibilities

The quality-related and quality-affecting duties and responsibilities of primary program positions are described below:

Texas World Program Manager - The Texas World program manager will provide the technical direction for the work conducted under the subcontract. The program manager will ensure that draft work products are reviewed and approved prior to acceptance. The program manager will provide overall technical guidance to QuantaLex.

Principal - The principal is responsible for determining the QA level for the subcontract; ensuring adequate commitment of budgetary resources and allocating these resources; ensuring that contractual obligations for the subcontract are met; providing periodic guidance, training, and updates regarding changes in policies and procedures that affect project procedures and work products; requesting QA audits and surveillances of program activities as necessary or appropriate; responding to QA audit reports and taking corrective action for any problems or deficiencies identified; and ensuring that the QA project plan is developed and updated. The principal may perform peer, technical, or management reviews of work products.

Quality Assurance Manager - The QA manager for the contract is responsible for developing and approving the QA project plan, ensuring that the QA project plan provisions and corrective actions are effectively implemented, and that quality controls are operative and effective. The QA manager also provides periodic training, orientation, and updates in quality assurance and quality control procedures to new employees and program participants. The QA manager performs periodic documented surveillances of specific programs, projects, or activities.

Document Control Officer (DCO) - The document control officer is responsible for ensuring that a document control program is in place and is effectively implemented. The DCO conducts periodic training, orientation, and updates in document control procedures for new employees and program participants. The DCO is responsible for monitoring access to Texas World documents maintained by QuantaLex and for seeing that appropriate mechanisms are in place to ensure the integrity and security of documents and data.

QUANTALEX, INC.
TEXAS WORLD DATA VALIDATION
PROGRAM ORGANIZATION CHART

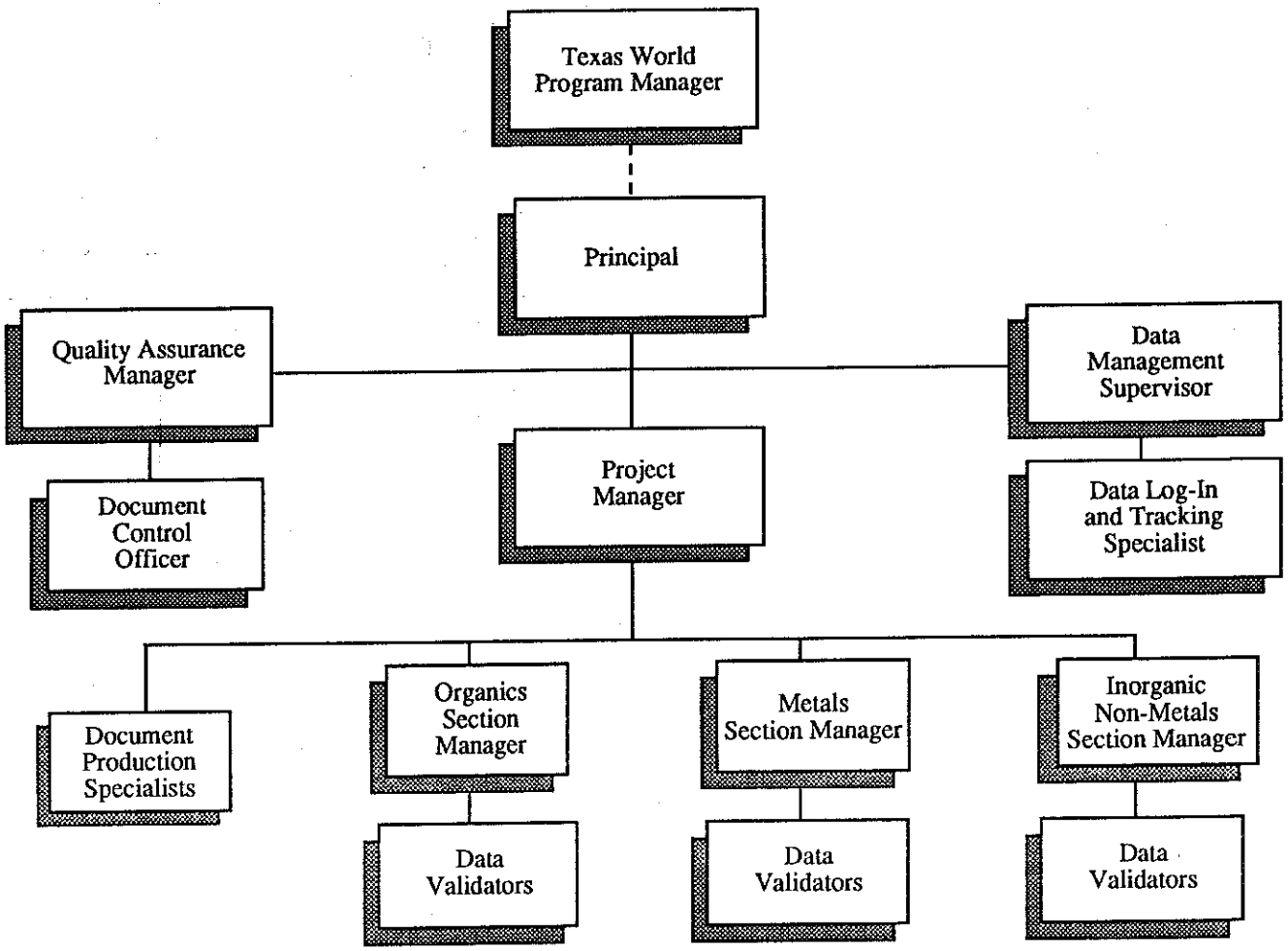

Figure 4 - 1

Project Manager - The project manager is responsible for reviewing and approving work products, responding to *ad hoc* project requests, maintaining daily liaison between the Texas World project manager and QuantaLex, establishing procedures and standards for work products, ensuring that QA program and document control security program procedures are implemented and operative, ensuring that training programs are implemented and operative, and assisting the principal in ensuring that corrective action is taken to resolve any problems or deficiencies identified. The project manager may perform peer, technical, or management reviews of work products.

Chemistry Section Managers - The chemistry section managers are responsible for coordinating personnel resources on project work to ensure that project task activities are adequately staffed to meet scheduled completion dates and quality control requirements, reviewing and approving work products, conducting routine and specialized training for staff, and directing corrective action to resolve any identified problems or deficiencies. Chemistry section managers may perform peer, technical, or management reviews of work products.

Data Validators - The data validators are responsible for developing and/or producing work products, conducting peer reviews, conducting routine and specialized training for project staff, ensuring that quality assurance and quality control procedures are followed and requirements are met, ensuring that project tasks are completed on schedule, and taking corrective action to resolve any problems or deficiencies identified in project work or work products.

Data Management Supervisor - The data management supervisor is responsible for developing database work products and ensuring that database systems being used for the program are functional, are adequately safeguarded from inadvertent erasure or degradation, and are periodically backed-up electronically and with appropriate documentation.

Data Log-in and Tracking Specialist - The data log-in and tracking specialist is responsible for receiving data packages and entering data package information into the data validation tracking database system. This specialist is responsible for ensuring that information in the tracking system is current, accurate, and retrievable.

Document Production Specialists - Document production specialists are responsible for providing accurate reports and work products. They ensure that objective work product standards are met, including spell check, style characteristics, and completeness.

4.3 Qualification Requirements

All personnel performing data validation activities should be adequately trained and have the appropriate educational background to perform the work. Personnel training and qualifications will be adequately documented in the QA records file.

All QuantaLex data validation personnel performing data validation in conjunction with this Texas World subcontract will have the following qualifications:

- A minimum of a bachelor's degree in a physical science, mathematics, or engineering.
- Completion of QuantaLex in-house validation training. Validator qualification records and documentation of additional training will be maintained in the QA records file.

5.0 CORPORATE QA PROGRAM ORGANIZATION

The corporate QA organization is described in this section. QuantaLex, as a subsidiary company of TechLaw, Inc., actively participates in this QA organization.

5.1 Organization

The corporate QA organization is designed to ensure that persons performing QA functions with respect to a task are, to the extent possible, separated from any other responsibility for work being performed in that task.

QuantaLex's participation in the corporate QA organization is illustrated in Figure 5-1. The purpose of the corporate QA organization is to ensure that a generally uniform program approach to quality assurance is effectively implemented throughout the TechLaw companies.

5.2 Duties and Responsibilities

The duties and responsibilities associated with the corporate QA program organization are described below:

President, TechLaw - The president ensures that a corporate QA program is developed and implemented for all operating units and subsidiaries. The president also ensures that the effectiveness of the QA program is evaluated regularly and that corrective action to remedy deficient areas is implemented.

Director of QA - The director of QA is responsible for developing and implementing the QA Program. The director of QA is also responsible for administering the QA organization, assessing the effectiveness of QA programs, determining the need for corrective action, and regularly reporting the progress of QA activities to the president.

Subsidiary Presidents - Subsidiary presidents are responsible for ensuring that QA programs are developed and implemented for governing the quality-related and quality-affecting activities of their subsidiary operations. Subsidiary presidents are also responsible for ensuring that implemented QA programs are evaluated periodically for effectiveness and that any corrective actions required are implemented.

Program/Project Managers - The program and projects managers are responsible for ensuring that QA project plans are developed and updated for programs and projects. Program/project managers are also responsible for periodic reporting of quality progress.

Subsidiary QA Managers - Subsidiary QA managers are responsible for assisting in preparation of QA project plans, monitoring the effectiveness of QA programs, training and orientation of staff, and assisting the director of QA in performing audits and surveillances.

TechLaw Corporate QA Program Organization Chart

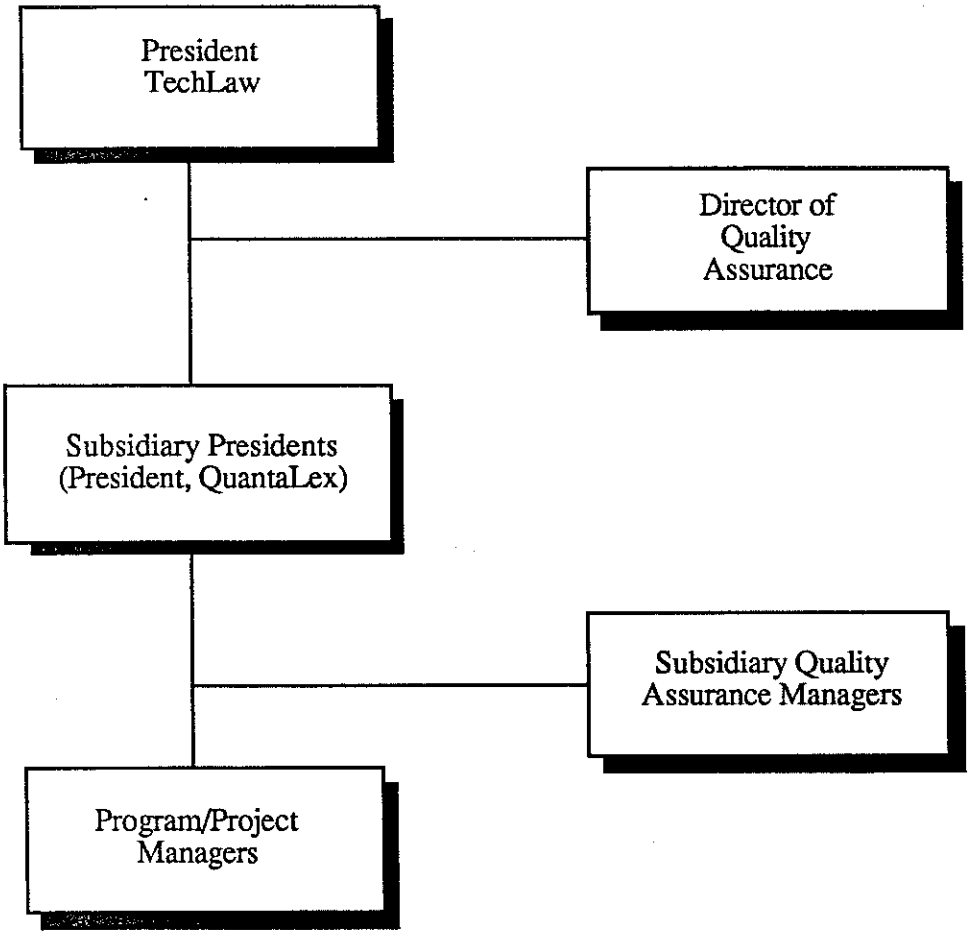

Figure 5-1

6.0 REPORTING REQUIREMENTS

The following reporting requirements are for designated deliverables and are designed so that project and program progress may be monitored. These reporting requirements also ensure that all work products are delivered to Texas World on schedule and that project and contract requirements are met.

6.1 Reduced Data Validation

For each data summary package, QuantaLex will provide the following deliverables within 30 calendar days of receipt of the data summary package.

- Reduced data validation worksheets
- Reduced data validation reports, specifying deficiencies and recommendations for qualifiers.
- Analysis Data Sheets (Form I) with appropriate qualifiers or a summary of results with appropriate qualifiers.

For each of the QC criteria that exceeds the control limits, the report shall describe the problems and how they affect the sample data. All data validation worksheets and reports generated by the data validator will be reviewed by a QuantaLex technical reviewer and managerial reviewer before delivery to Texas World.

Data packages will be returned to Texas World within 30 days of receipt of the above deliverables.

6.2 Full Data Validation

For each data package, QuantaLex will provide the following deliverables within 30 calendar days of receipt of the data package.

- Full data validation worksheets
- Full data validation reports, specifying deficiencies and recommendations for qualifiers.
- Analysis Data Sheets (Form I) with appropriate qualifiers or a summary of results with appropriate qualifiers.

Full data validation reports will discuss in detail each of the QC criteria listed in the SOW (see Appendix I). For each QC criterion, the report should describe any problems noted and how they affect the sample data. If no problems are found, that should be noted as well. All data validation worksheets and reports generated by the data validators will be reviewed by a QuantaLex technical reviewer before delivery to Texas World.

Data packages will be returned to Texas World within 30 days of receipt of the above deliverables.

6.3 Rush Turnarounds

Upon request from Texas World, QuantaLex will provide either cursory or full data validation deliverables on a "rush" basis. Those deliverables requested as a "rush" will be due within an agreed-upon time frame.

6.4 Contact Reports and Data Requests

Phone calls to Texas World and requests for data are documented on contact records (see Figure 6-1). Data requests are summarized in memoranda.

6.5 Control of Non-Conforming Data Packages

Data packages received by QuantaLex may be considered to be non-conforming for any of the following reasons:

- Data packages are missing information necessary to complete the validation process. Such information may include, but is not limited to:

  - Forms that summarize detailed calculation.

  - Raw data necessary to substantiate results or QC analyses.

  - Chain-of-custody records.

- Data packages that contain grossly inaccurate information. Typically this would be organics results that are quantified from an incorrect calibration.

Data packages will not be subject to data validation until they are considered to be complete and conforming. Texas World will pre-screen all data packages before sending them to QuantaLex. However, should any information be deemed missing after receipt by QuantaLex, the QuantaLex project manager will notify the Texas World technical contact immediately. Texas World will either contact the laboratory or authorize QuantaLex to contact the laboratory directly to obtain the necessary information. Turnaround times will stop on the date Texas World is initially notified and start upon receipt of a written notification that all information has been provided and received by QuantaLex. (The clock does not start over at this point.)

QUANTALEX Contact Record

Person Initiating Contract: _____ Project Name: _____

Date: _____ Time: _____ Project Number: _____

Person Receiving Contract: _____ Title: _____

Company Name: _____ Phone: _____

Street: _____

City: _____ State: _____ Zip: _____

Means of Communication:

Phone Call: _____ Letter: _____ Personal Visit: _____

Other: _____

Action:

Initiated: _____ Received: _____ Return Call: _____

Other, explain _____

Summary: _____

QUANTALEX Contact Record

Figure 6-1

7.0 QUALITY ASSURANCE OBJECTIVES AND WORK PRODUCT STANDARDS

The quality assurance objectives presented in this QA project plan describe the standards of quality necessary to meet or exceed all requirements and obligations of the contract. Work product standards define the QA objectives for each work product. Guidelines for program functions which are quality-related or quality-affecting are included in the QA project plan.

7.1 Quality Assurance Objectives

Quality assurance objectives define the levels of quality required for work products developed by QuantaLex. The QA objectives are formulated from quality standards contained in QuantaLex's QA Program Plan and applicable contract requirements. The QA objectives are expressed quantitatively where possible.

7.1.1 *Definitions*

The following terms are used in formulating the QA objectives:

Accuracy - A measure of the degree of agreement of information included in a work product (report, database, etc.) compared to the source from which the information was obtained.

Completeness - A measure of the amount of information obtained from an activity (database, audit, etc.) compared to the amount that was expected to be obtained under correct procedures and normal conditions.

Consistency - A measure of mutual agreement among information elements having similar properties or characteristics and obtained under similar conditions.

Comparability - A measure of the confidence with which one information set can be compared to another.

7.1.2 *Quality Assurance Objectives for the Texas World Subcontract*

Overall QA objectives have been derived from the quality standards defined in the QA program plan, the QA level designators for this subcontract, and applicable subcontract-specific QA standards and practices. The overall QA objectives for this subcontract are defined below:

Accuracy - The degree of agreement of information contained in work products will compare to the source from which the information was obtained to a frequency greater than 99 percent. Errors will not exceed one percent.

Completeness - The amount of information contained in work products compared to the amount that was expected to be included (under correct procedures and normal conditions) will exceed 99 percent. Omissions will not exceed one percent.

Consistency - The mutual agreement among information elements contained in work products having similar properties or characteristics and obtained under prescribed similar conditions will exceed 99 percent. Discrepancies and inconsistencies will not exceed one percent.

Comparability - The confidence that information sets contained within similar work products can be compared to each other will exceed 99 percent. Dissimilarities will not exceed one percent.

7.2 Work Product Standards

Work product standards for contract deliverables are formulated from the QA objectives presented in Section 7.1.2. The work product standards provide specific quality assurance goals for deliverables developed under this contract. SOPs and guidelines for specific deliverables (e.g., data validation) provide the mechanisms for ensuring that work product standards are met or exceeded.

7.2.1 *Data Validation Reports*

Data validation is the process by which data quality is determined with respect to data quality criteria defined in project and laboratory quality control programs and analytical methods. Data validation can be described as a systematic, rigorous, and documented verification activity which involves substantive professional judgment and discretion.

The data validation process consists of assessing the acceptability or validity of project data with respect to stated project goals and requirements for data usability. Data validation consists of data editing, screening, checking, certification, review, and interpretation. The purpose of data validation is to define and document analytical data quality and determine if the data quality is sufficient for the intended use(s) of the data.

Data validation will include the review of the QC criteria specified in the subcontract SOW (see Appendix I) and other QC criteria as required by the analytical protocols (e.g., EPA SW-846).

In addition, QuantaLex data validation guidelines will be developed to assist the validators with reviewing specialty analyses. These guidelines will be modeled after the EPA functional guidelines and will incorporate the QC criteria specified in the subcontract SOW and in the analytical method.

The analytical quality of the data which has been validated will be assessed by determining the degree to which the data have been compromised by any deviation from protocol, QA/QC problems, lack of analytical control, etc. that occurred during the analytical process. Data with no qualification necessary is assessed as "Acceptable"; estimated data with bias unknown or data with specified high or low bias is assessed as Estimated (J); unusable data is assessed as "Rejected" (R). Other qualification methods and qualifiers may be specified by Texas World.

The data packages submitted to QuantaLex under this subcontract will require either reduced or full data validation. In either case, each data point will continue through the validation process and will be qualified and requalified as many times as it fails to meet the established criteria. The data qualifiers will be applied to the analysis data sheets (Form I) and will also appear in the data validation report where specific deficiencies are discussed.

7.2.2 *Quality Assurance Objectives for Data Validation Reports*

Quality assurance objectives for data validation reports are formulated in terms of accuracy, completeness, consistency, and comparability.

- **Accuracy** - All statements in the report are factual, objective, and can be traced to a specific source; all findings are based on established data validation requirements and guidelines. The degree of agreement of information contained in the data validation report will compare to the uncorrected data package results to a frequency greater than 99 percent. Errors will not exceed one percent per report.

- **Completeness** - All data validated during the course of the validation process are addressed in the report; quality assessment elements are addressed during the course of the validation process; all worksheets are signed and dated. The content of data validation reports compared to the required quality assessment elements in the functional guidelines and specifications will exceed 99 percent. Omissions will not exceed one percent per report.

- **Consistency** - Reports are written in the current standard format. All findings are based on established data validation requirements. Information elements contained in data validation reports will be mutually consistent with the functional guideline requirements for 99 percent of the elements. Inconsistencies and discrepancies will not exceed one percent.

- **Comparability** - Reports for similar analyses and parameters are prepared using similar assumptions, data quality flags, and notations based on the guidelines in effect at the time the reports were generated. Information contained in reports for sample analyses and matrices will be mutually comparable to the extent of 99 percent. Dissimilarities will not exceed one percent.

7.2.3 *Data Validation Guidelines*

Data validation guidelines developed by the EPA for the Contract Laboratory Program will be employed for data validation where such guidelines are available and applicable to the analyses that Texas World requires. QuantaLex will develop data validation guidelines for non-CLP analyses.

For those guidelines that QuantaLex develops, the general approach, format, and techniques will be similar to those in the EPA's guidelines. Guidelines developed by QuantaLex will be subject to approval by Texas World prior to use.

Where appropriate, and to the extent that the methodologies allow, reviewer latitude, discretion and professional judgment for data qualification will prevail over rigorous adherence to method performance standards. Although every attempt will be made to parallel the EPA versions, internally-developed guidelines may reflect more latitude than conventional EPA guidelines. Successive versions of guidelines will be numbered and dated so that any data validation report generated during a specific time period is developed according to the requirements of the current guideline.

7.2.4 *Quality Assurance Objectives for Internally-Developed Guidelines*

Quality assurance objectives for internally-developed guidelines are formulated in terms of accuracy, completeness, consistency and comparability.

- **Accuracy** - Requirements contained in the guidelines reflect, to the extent known, those expectations of data quality reasonably attainable by the methods prescribed under the conditions and matrices actually encountered.

- **Completeness** - All relevant data assessment elements are addressed in the guideline.

- **Consistency** - The format, content, and organization of the guideline is similar to those same characteristics in the EPA guidelines.

- **Comparability** - Data assessment elements, evaluation techniques, qualifiers, and reporting requirements among the different guidelines will be in agreement to the extent feasible.

8.0 STANDARD OPERATING PROCEDURES

Standard operating procedures (SOPs) have been developed for document control and security; laboratory auditing; data validation; receipt, verification, and transmittal of electronic deliverables; and software quality assurance. Implemented SOPs are approved, dated, and are identified with the current version numbers. Copies of previous SOP versions are maintained for historical continuity.

The SOPs applicable to this subcontract are discussed below.

8.1 Document Control and Security

A formalized document control and security program is in place to ensure that project documents and quality assurance records are secure and retrievable.

Document control and security procedures are documented in the current version of "Document Control and Security Standard Operating Procedures." All quality assurance and quality control records related to the program activities covered by this QA Project Plan are maintained according to the procedures described in this document.

The following types of documents should be maintained in a QA records file:

- QA Project Plans
- SOPs and guidelines
- Auditor and validator training records
- Internal and external audit reports
- Corrective action reports
- Corrective action log
- Quality-related correspondence

8.2 Data Validation

The data validation will be performed using the appropriate internal and external SOPs and guidelines for Texas World work. The following guidelines will be used:

- "Guidelines and Specifications for Preparing Quality Assurance Project Plans;" USEPA-Quality Assurance Management Staff (EPA QAMS) 005/80; 10/80.

- "Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses," 7/88.

- "Laboratory Data Validation Functional Guidelines for Evaluating Organic Analyses," 2/88.

- In addition, method specific guidelines will be developed for non-CLP methods as needed.

Worksheets to be used for validation will be prepared by QuantaLex to accommodate any contractual requirements.

8.3 Software Quality Assurance

The SOP entitled "Software Quality Assurance Program Standard Operating Procedure" was developed to document QuantaLex's QA policies and procedures related to software development. Any software developed as a deliverable to Texas World under this contract will be documented and developed using these software QA SOPs.

9.0 QUALITY CONTROL

Quality control procedures are used to ensure that the quality of work products meets or exceeds the QA objectives established for this program. Quality control procedures consist of a series of systematic and documented reviews. These reviews are described below.

9.1 Peer Review

All documents and reports may be subject to documented peer review at the discretion of the author. The individual reviewing the document or report will have an understanding of the document subject. This review is completed prior to technical review.

The purpose of peer review is to ensure that QuantaLex QA standards and contractual requirements are met. Peer review consists of the following items:

- **Completeness** - Review work products to verify that all applicable information has been included.
- **Accuracy** - Review the grammatical accuracy of narrative material and review data for easily discernable errors.
- **Consistency** - Review both the narrative material and data for consistency of similar information.
- **Comparability** - Review the work product to verify that the format and organization are comparable with similar work products.

9.2 Production Review

All documents and reports may be subject to document production review at the discretion of the author. Production review is normally performed by the technical editor and is usually completed prior to technical review. Production review consists of the following items:

- **Accuracy** - Review the grammatical accuracy of the narrative material.
- **Comparability** - Review the work product to verify that the format and organization are comparable with similar work products.

9.3 Technical Review

All work products will be subjected to documented technical review to ensure that they are technically correct and scientifically sound. This review is completed prior to management review. Technical review consists of the following items:

- **Accuracy and Completeness** - Every item and data element in a report or database is examined to ensure the accuracy and completeness of information.
- **Consistency** - Every item and data element will be qualified and evaluated in a manner consistent with the applicable SOPs and guidelines.

After a work product is developed, the following QC steps at a minimum will be undertaken to ensure that the QA objectives for the work product are achieved.

First Pass QC - Every element, item, or group in a database or report will be compared to the original source of the information to determine if all results were transcribed correctly and that all data qualifier flags are appropriate.

Second Pass QC - Every element, item, or group in a database will be compared to the information on the edited QC document to ensure that all of the changes in the first pass QC document were properly made.

Corrective Action - Additional QC passes will be made if problems that require corrective action are identified during the QC phase of the project. Additional passes will be conducted until it is determined that problems have been satisfactorily resolved.

9.4 Management Review

All documents and reports will be subject to documented management review. The purpose of management review is to ensure that QA standards and contractual requirements are met. At a minimum, management review should consist of the following items:

- **Completeness** - Review the work product to determine that the information requested by the client has been addressed. Review the work product to ensure that all results, qualifier flags, and explanations have been included.
- **Accuracy** - Review the grammatical and factual accuracy of narrative material. Review data for transcription errors.
- **Consistency** - Review data for consistency of similar information. Review for consistency of information in validation report. Review for agreement of information between the validation worksheet and the validation reports.
- **Comparability** - Review the work product to verify that the format and organization are comparable with similar work products.

9.5 Quality Control Documentation

The Report Review Tracking Form will be completed at each step of the review process and will serve as a record of peer, production, technical, and management review (see Figure 9-1).

QuantaLex Report Review
Tracking Form

Document Initiation Date _____ Typist Initials _____

Author _____ Filename/Disk _____

Project Number _____ Title _____

Document Description _____

_____

_____

_____

_____

	Document Received		Document Relinquished	
	Date	Initials	Date	Initials
Peer Review Comments:				
Technical Review Comments:				
Management Review Comments:				

Figure 9-1

10.0 FACILITIES, EQUIPMENT, AND SUPPLIES

This section describes the facilities, equipment, and supplies to be used for this program.

10.1 Facilities

Project work for this contract is primarily conducted in the QuantaLex offices. The QuantaLex office is located at 300 Union Boulevard, Suite 600, Lakewood, Colorado. This office is equipped with designated secure areas. The building complex is locked after business hours and is accessible only to building tenants. In addition, the entrance to the QuantaLex secure area is locked after business hours.

During business hours, the main entrance is monitored by a receptionist who requires all visitors to sign in and will not allow a visitor to enter the secured area unless they are accompanied by a QuantaLex employee.

10.2 Equipment and Supplies

Equipment and supplies are present in sufficient quantity and condition and are operationally consistent with their intended use.

All equipment owned or leased by QuantaLex and requiring continuous maintenance (such as copiers, computers, and microfilm equipment) will normally have documented maintenance criteria in the form of maintenance agreements. All equipment has documented operating criteria in the form of operating manuals.

Computer equipment owned or leased by QuantaLex is functional and able to produce, store, and retrieve all data produced by QuantaLex. Equipment is present in sufficient quantity and condition and is operationally consistent with its intended use.

11.0 QUALITY ASSURANCE AUDITS

QA audits are an integral part of the overall QA program. Audits are performed as a management function to verify compliance with the following:

- QA Program Plan
- QA Project Plans
- Contracts and Subcontracts
- Program Operations, Policies, and Procedures

The primary purpose of conducting QA audits is to determine the effectiveness of the QA program as implemented. A secondary purpose is to improve the quality of work products and improve the effectiveness of quality-related and quality-affecting activities.

11.1 Scope

Audits of the QA program will consist of review of the QA program requirements, QA project plans and procedures, QC records and documentation, and interviews of project participants. Surveillances may be conducted periodically for specific projects, specific technical work, or particular areas of the QA program.

11.2 Procedures

Audits will be conducted by the corporate director of QA. Audits will be scheduled with the knowledge and concurrence of the subsidiary president and affected program management. Surveillances will be conducted by the QuantaLex QA manager or his/her designee. Surveillances may be scheduled or unannounced depending on the timing and reasons for conducting them.

Audits will be conducted using documented checklists and SOPs for inspecting, reviewing, and collecting information. Observations and conclusions will be documented and based on objective evidence and results of interviews with personnel. Surveillances will be conducted using the "QuantaLex Surveillance Report" as a guide (see Figure 11-1).

11.3 Performance

The corporate director of QA will perform internal audits of the QA program according to the corporate audit schedule. Audits will be guided by checklists, and objective evidence will be examined to the depth necessary to determine the effectiveness of the QA program as implemented for the program and projects included in the scope of the audit. Audits will include review of objective evidence to determine adherence to QA project plans.

QUANTALEX SURVEILLANCE REPORT FOR DATA VALIDATION PACKAGES

Section: _____ Date Received: _____
Project No. _____ Date Missing Data Received: _____
Client: _____ Date Tables Received: _____
Case No.: _____ Date Sent: _____
Batch No.: _____ Turnaround Time (days): _____
Lab: _____ Samples/Matrix: _____
SDG No.: _____ QLI No.: _____
Parameters: _____

Package Contents (Y/N)
Cover Letter: _____
Summary Report Form:
Typed _____ Handwritten_____
Report Review Tracking Form:_____
Tables: Typed_____ Handwritten_____
Worksheets: _____
Data Completeness Checklist: _____
Raw Data: _____
Other: _____

Error Summary
No. of Typing Errors: _____
No. of Transferring Errors: _____
No. of Punctuation and Grammar Errors:____

Validator: _____ Date: _____
Tech. Rev: _____ Date: _____
Mgr. Rev: _____ Date: _____
Typist: _____

Data Package Log-in/out Check:
_____
_____
_____
_____

Data Tracking Sheet Check:
_____
_____
_____

Findings: _____

Observations: _____

Comments: _____

Post-surveillance meeting (with) _____ Date:_____
Corrective Action Report required? (Y/N) _____
Surveillance Completed (by) _____ Date:_____
QA Manager

Figure 11-1

Checklists will be developed and approved prior to performance of audits. The director of QA will approve the checklists. Audits will include a pre-audit briefing and a post-audit conference with audit participants. Management personnel responsible for the areas being audited are expected to make themselves available during the audits and are to attend the pre-audit briefings and post-audit conferences.

The QuantaLex QA manager will perform surveillances of specific projects, sections, or work products within the company. The "QuantaLex Surveillance Report" provides a guide during the surveillance process as well as documentation of findings, observations, and comments.

External audits may be conducted by Texas World or its designee as arranged with QuantaLex.

11.4 Reporting Requirements

Internal audit results will be documented in formal audit reports submitted to the president of TechLaw. The audit reports will contain the following information:

- Programs/projects audited
- Date
- Audit team members
- Audit participants
- Statement of QA program effectiveness
- Audit summary
- Findings
- Corrective actions needed
- Recommendations
- Copies of audit workpapers

Deficient conditions identified during the conduct of audits are reported as audit findings. Findings may be based solely, or in part, on QA program requirements, contract requirements, QA project plan requirements, program policies and procedures, and good management practices. (Good management practices are based on the judgement and experience of the auditor.) Audit reports will be filed in the QA records file.

Surveillances will be reported using similar criteria except that a statement of the QA program effectiveness may be limited to the specific program, project, or activity examined. Surveillance reports will be filed in the QuantaLex QA manager's office.

11.5 Corrective Action

Corrective action needed to complete the audit process will be taken by management responsible for correcting the cited deficiencies. Corrective action will be in the form of a response to the audit report and will describe the actions to be taken to correct the deficient condition and preclude recurrence. Responses will be evaluated by the corporate director of QA who will determine whether the responses effectively complete the audit process. Responses deemed insufficient may be appealed to the president of TechLaw for resolution.

Corrective action for external audits will be in the form of resolving any identified deficiencies by responding in writing to the audit report.

Corrective action responses are developed in the following format:

1. Findings as stated in the audit report.
2. Requirement(s) cited.
3. Recommendation(s) made.
4. Corrective action taken to correct the deficiency and action taken to preclude recurrence.

Where applicable, each corrective action shall include a schedule or target date for completion.

For surveillances, corrective action involves the completion of a "QuantaLex Corrective Action Report" (see Figure 11-2). Upon completion, this form is filed in the QA records file, with a copy filed in the appropriate project file.

11.6 Audit Schedule

A schedule of planned audits and surveillances is developed periodically by the corporate director of QA and submitted to the president of TechLaw. The schedule typically includes the audit dates, duration, budget, and scope of each audit or surveillance. The audit schedule will be approved and filed in the corporate QA records file.

Affected managers of programs selected for audit are notified at least 30 days in advance of a pending audit.

QUANTALEX CORRECTIVE ACTION REPORT

ORIGINATION

Requirement (Technical/Contractual):

Deficiency (Description):

Recommended Action(s):

RESPONSE

Cause:

Remedial/Investigative Action(s): Scheduled Implementation Date_____

Action(s) to Prevent Recurrence: Scheduled Implementation Date_____

Responder Signature/Date:

EVALUATION

RESPONSE COMMENTS:
_____Accept _____Amend _____Reject
QA Manager Signature/Date:

CORRECTIVE ACTIONS COMPLETED COMMENTS:
Implementor Signature/Date:

AMENDED RESPONSE COMMENTS:
_____Accept _____Reject
QA Manager Signature/Date:

VERIFICATION COMMENTS:
_____Satisfactory _____Unsatisfactory
QA Manager Signature/Date:

DATE OF CLOSURE IF:_____ (if applicable)

Figure 11-2

12.0 QUALITY IMPROVEMENT

QuantaLex endorses the concept of continuous quality improvement in our client programs and corporate operations, policies, and procedures.

To the extent that quality progress is measurable, periodic self-assessments will be performed, in addition to the routine audit and surveillance process.

Each program will be assessed periodically (usually annually), and a written report of problems encountered and how they were solved will be developed. Suggestions for quality improvement in processes and work products will be solicited from QuantaLex program/project participants and be included in these written reports.

In addition, suggestions for quality improvement will be obtained from Texas World by means of solicitation or by means of feedback after delivery of completed work products. These suggestions will also be included in these written reports. The reports will be periodically reviewed and operations upgraded as warranted.

Formal corrective action may be implemented as applicable in response to any external or internal suggestions or observations. All formal corrective action will be documented on a "QuantaLex Corrective Action Report" form. The original will be filed in the QC records file and a copy will be filed in the project file.

APPENDIX I

TEXAS WORLD OPERATIONS, INC. SUBCONTRACT

STATEMENT OF WORK

STANDARD OPERATING PROCEDURE

PREPARING TEXAS WORLD OPERATIONS, INC. DATA VALIDATION REPORTS

Prepared By:

QuantaLex, Inc.
300 Union Boulevard
Lakewood, Colorado 80228
(303) 763-8881

Approved for Use:_____
Texas World Operations, Inc.

October 1992

STANDARD OPERATING PROCEDURE

PREPARING TEXAS WORLD OPERATIONS, INC. DATA VALIDATION REPORTS

Developed By: _____ Date _____
Richard Kantrowitz
Project Manager
QuantaLex, Inc.

Approved By: _____ Date _____
Ken Schroeder
Quality Assurance Manager
QuantaLex, Inc.

Concurrence: _____ Date _____
Robert J. Thielke
Technical Operations Manager
QuantaLex, Inc.

October 1992
Version 0.0

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Appendix I Data Validation Reports

1.0 INTRODUCTION

The purpose of this Standard Operating Procedure (SOP) is to describe the methods and procedures used by QuantaLex to document the results of the validation of Texas World Operations, Inc. (Texas World) data and prepare the associated data validation report. This SOP consists of the following sections:

- Scope and Purpose
- Responsibilities
- Report Format
- Report Preparation
- Report Review
- Report Delivery
- Validation Quality Control

2.0 PURPOSE AND SCOPE

The purpose of this SOP is to provide specific instruction to QuantaLex personnel in preparing data validation reports. This SOP is to be used in conjunction with the "Quality Assurance Project Plan" and the appropriate validation guidelines.

The scope of this SOP is all data validation reports prepared for Texas World Operations, Inc.

3.0 RESPONSIBILITIES

The responsibility for the consistent, complete, and accurate preparation and delivery of Texas World data validation reports rests primarily with the data validator, data reviewer, chemistry section manager, technical operations manager, and quality assurance manager.

3.1 Data Validator

The data validator is responsible for initiating data validation reports that are based upon the results of the data evaluation of a data package. The data validator is responsible for preparing the report, utilizing the guidance set forth in this SOP, the "Quality Assurance Project Plan", the appropriate validation guideline, standard comment language, and other guidance issued by Texas World or QuantaLex.

3.2 Data Reviewers

The data reviewers are responsible for performing technical and management reviews of the data validation report and ensuring that data validation reports are prepared within 30 working days of the data package receipt. The reviews are to be performed using the guidance set forth in this SOP, the "Quality Assurance Project Plan", the appropriate validation guideline, standard comment language, and other guidance issued by Texas World or QuantaLex.

3.3 Chemistry Section Manager

The Chemistry Section Manager is responsible for ensuring that all data validation reports are prepared and delivered to Texas World within 30 working days of complete data package receipt. The Chemistry Section Manager is responsible for ensuring that the quality of the data validation report is maintained by reviewing a percentage of all reports prior to release. The Chemistry Section Manager is responsible for maintaining standard comments language, coordinating validation and review priorities, informing validators and reviewers of issues that may affect the validation process, and informing the Technical Operations Manager of any proposed changes to the validation process.

3.4 Technical Operations Manager

The Technical Operations Manager is primarily responsible for ensuring that all data validation reports are prepared using current revisions of guidelines and SOPs and ensuring that any validation issues or changes are communicated to and from the client.

3.5 Quality Assurance Manager

The Quality Assurance Manager is responsible for performing periodic data validation report surveillances to determine if corporate data quality objectives are met. The Quality Assurance Manager is responsible for setting the Chemistry Section Manager review percentage based upon the observed data validation report quality.

4.0 REPORT FORMAT

The data validation report format for Texas World consists of the following sections:

Data Validation Memo
Action Items and Comments
Data Summary Tables

Examples of completed reports for several sections are presented in Appendix I of this SOP.

4.1 Data Validation Memo

The data validation memo serves as the cover sheet for the data validation report and provides the data users with an overview of the quality of the associated data. The following fields will be filled in for each data assessment form.

File No. (Radiochemistry) or Batch No. (all except Radiochemistry) - The file number/batch number are assigned by QuantaLex. This number represents the year and month of receipt by QuantaLex, a one letter laboratory designation, and a set number. For example, 9201G102.

SDG No. - The SDG number is the number provided by the laboratory to identify the data package. This number may vary between laboratories.

Site - The site is the operable unit or program associated with the samples in a data package.

Laboratory - The specific name of the laboratory should be indicated. If more than one laboratory analyzed the samples, the primary laboratory (the laboratory that prepared the case narrative) should be listed first. The laboratory name should be indicated by the laboratory name, a slash, and the branch name. Multiple laboratory names should be separated by a comma.

Number of Samples/Matrix - The number of samples include all natural samples taken in the field and all field quality control samples, including but not limited to:

Field blanks
Trip blanks
Field replicates

The matrix is typically either soil or water, although matrices such as air or biota may be encountered.

The number of samples and matrices are indicated by separating the number and matrix with a slash, for example, 5/Soil. Multiple matrices should be separated by a comma, for example, 5/Soil, 3/Water.

Sample Numbers - The sample numbers should appear as indicated on the chain-of-custody records from the field subcontractor.

4.2 Quality Control Categories

The individual quality control assessment categories for non-radiochemistry validation represent the quality of the data based upon each quality control category. The assessment of V (valid), A (acceptable), or R (reject) should reflect the majority of individual data point qualifiers assigned due to the quality control results.

4.3 Overall Data Assessment

The overall data assessment represents the quality of the majority of the data. It can only be assigned once the data validation process is complete. At this point, each data point for each analytical result has been assigned a data qualifier based upon deficiencies in one or more of the QC categories (i.e., holding times, calibration, etc.). All results have been qualified. Determine which qualifier has been assigned to the majority of the analytical results and assign that qualifier as the overall data assessment qualifier.

4.4 Action Items and Comments

The action items and comments are a narrative accounting of all the problems associated with the data.

Action items describe all the problems that cause data points to be qualified.

Comments are problems that did not cause data to be qualified, but that if left uncorrected, may cause data to be qualified in the future. Comments may also be irregularities or unusual occurrences that impeded the validation process or that may be of use to any data users.

5.0 ORGANIC AND INORGANIC DATA VALIDATION

Data validation procedures are distributed to all validation personnel to provide step-by-step instruction for validating organic and inorganic data to ensure a thirty (30) day turn around time. The discussions at the beginning of each section explain the requirements for completion of the review process for each area considered.

5.1 Data Screening

The data validator will sign-out a data package by initialing and dating the Tracking Sheet (Figure 5-1) under the "Validated by" column for the appropriate data package. The tracking sheets for current data packages are located in the Document Control room. Tracking sheet notebooks are maintained for Organic and Inorganic data packages for each client. Data packages for each analysis must be signed-out separately.

The data validator initiates the Report Review Tracking Form (Figure 5-2) by completing the following items:

Author
Document initiation date
Project number
Document description

The data validator inspects the data package to determine if all information necessary to validate the data has been provided.

Data package problems or missing data will be brought to the attention of the project manager. The project manager will be responsible for preparing a letter to request the missing data according to Texas World's procedures.

Upon receipt of the requested data, the appropriate data validation manager will distribute the data to the case file and note on the appropriate Summary Sheet the date of receipt of requested data. This will alert data validation or review personnel that validation can be resumed.

5.2 Data Validation

The validation process is performed using the following steps (in the order specified) to ensure that the validation process is accurate, complete, consistent, and comparable.

Check from three to five calculations for each quality control analysis or for each calculation step. If problems are encountered or if unusual quality control results are found, a greater percentage may be verified (up to 100%).

07/20/92 QuantaLex, Inc. Data Tracking System Summary Page 1

Summary of Batches Received the Week of 07/17/92

Batch Number	SDG Number	QLI Number	Type	Table Rec Date	Missing Data Req	Missing Data Rec	Validator	Reviewer
9205P273	P273	O00022	VOA-CLP	_____	_____	_____	_____	_____
9205P273	P273	O00023	BNA-CLP	_____	_____	_____	_____	_____
9205P273	P273	O00024	PCB'S/PEST.-CLP	_____	_____	_____	_____	_____
9205P275	P275	O00021	VOA-CLP	_____	_____	_____	_____	_____
9206G075	9206G075	O00029	VOA-CLP	_____	_____	_____	_____	_____
9206G075	9206G075	O00030	BNA-CLP	_____	_____	_____	_____	_____
9206G846	9206G846	O00025	VOA-CLP	_____	_____	_____	_____	_____
9206G846	9206G846	O00026	BNA-CLP	_____	_____	_____	_____	_____
9206G846	9206G846	O00027	PCB'S/PEST.-CLP	_____	_____	_____	_____	_____
9206G846	9206G846	O00028	HERB	_____	_____	_____	_____	_____
9206G915	9205P273	O00031	VOA-CLP	_____	_____	_____	_____	_____

Figure 5-1 Data Tracking Sheet

QuantaLex Report Review Tracking Form

Document Initiation Date _____ Typist Initials _____

Author _____ Filename/Disk _____

Project Number _____ Title _____

Document Description _____

_____

_____

_____

_____

	Document Received		Document Relinquished	
	Date	Initials	Date	Initials
Peer Review Comments:				
Technical Review Comments:				
Management Review Comments:				
Resolution of Client Comments:				

Figure 5-2 Report Review Tracking Form

If the laboratory does not provide diskettes, transcribe all sample results from the appropriate reporting form or raw data (if necessary) to the appropriate Data Summary Report Table.

Perform the data validation according to the appropriate functional guideline, SOW, or data validation procedure. The following procedures and guidelines are currently being used:

1. Organic Data Validation Guidelines, EPA SW-846 Method 8240
2. Organic Data Validation Guidelines, EPA SW-846 Method 8270
3. Organic Data Validation Guidelines, EPA SW-846 Method 8150
4. Organic Data Validation Guidelines, EPA SW-846 Method 8080
5. Inorganic Data Validation Guidelines, EPA SW-846 Methods
6. Water Quality Parameters Data Validation Guidelines

All validation results and subsequent qualifications will be documented on the appropriate worksheets.

When applying data qualifiers to sample results, the following hierarchy is used:

R - A quality control result that causes a sample value to be rejected (R) will have the highest priority.
UJ - A quality control result that causes a sample value to be estimated and undetected (UJ) will have the next highest priority.
J - A quality control result that causes a sample value to be estimated (J) will have the lowest priority.

Action Items are written for each quality control result that caused sample values to be qualified: R, UJ, or J. An individual result should only have one Action Item for that qualification.

The completed data validation report will be submitted to typing.

6.0 REPORT REVIEW

All Texas World data validation reports are subject to fully documented technical and management review. Technical review or management review may not be performed by the individual that validated the data package. Technical and management review should ideally be performed by different individuals. However, it is acceptable to have one individual perform both reviews.

6.1 Technical Review

The technical review should be performed by comparing the validation worksheets and validation report to the associated raw data. At a minimum, the following tasks should be performed:

The quality control results from the raw data will be examined to determine if all failed quality control criteria were applied correctly to the data.

Determine that every qualified data point is explained in an Action Item found in the validation report and in at least one reason code found in the Data Summary Table. Determine that Action Items correspond to qualified data points.

Determine that the Data Validation Memo, Action Items, and Comments are grammatically correct, all required information is completed, and all information is in acceptable format.

After all corrections have been made, the reviewer signs as the technical reviewer on the Report Review Tracking Form and returns the report to the appropriate word processor for corrections, if any.

6.2 Management Review

The management review should be performed by comparing the final validation report to the draft validation report. At a minimum, the following tasks should be performed:

Determine that all corrections in the draft report were made.

Determine that the failed quality control criteria indicated were applied correctly to the data.

Determine that every qualified data point is explained in an Action Item found in the validation report and in at least one reason code found in the Data Summary Table.

Read the report to ensure that the report is formatted correctly and that the report is grammatically correct.

Read the report to ensure that all contractual obligations are met.

After all corrections have been made, the reviewer signs as the management reviewer on the Report Review Tracking Form and returns the report to the appropriate word processor for corrections, if any. The individual performing management review will check the report after it has been returned by the word processor to determine that all changes were made.

The validator and technical reviewer will sign each report after all reviews have been completed.

7.0 REPORT DELIVERY

All Texas World validation reports and EDDs will be sent to a specific set of individuals. Your section manager should have the current list of individuals and the deliverables they receive.

The appropriate laboratory contact at each laboratory should receive a copy of the validation reports related to their laboratory. However, these reports must not be sent until the original reports have been delivered to Texas World.

Diskettes are typically sent within one week of report transmittal. In an effort to facilitate the process of preparing the diskettes, the following procedures should be followed:

1. As soon as possible, the transmittal letter author should notify the support staff responsible for producing the data validation reports of all the reports that will be transmitted.

2. The support staff should copy the reports to be transmitted onto disks within one or two days of the notification and give the disks to Ms. Carla Cummins.

3. The disks should be sent to Texas World within five days of the data validation report transmittal.

8.0 VALIDATION QUALITY CONTROL

The quality of data validation reports are continually verified through section manager review and validation report surveillance. The percentage of validation reports reviewed is determined by the results of the validation report surveillance.

8.1 Section Manager Review

A percentage of all data validation reports (up to 100%) require a review by the appropriate Chemistry Section Manager. A minimum of 10% of all reports are reviewed. The reports are picked at random and reports are reviewed for every report delivery group sent to Texas World. The reviewer may review more reports from validators or reviewers who are experiencing difficulties. The review consists of the following steps:

Read the report for clarity of presentation (grammar, punctuation, etc.).

Determine that all sections of the report are completed and technically correct.

Ensure the report is signed and dated.

Ensure that Action Items reflect Data Summary Table results.

All errors should be corrected prior to the release of the reports, and major systematic errors may require review of additional reports. It is the responsibility of the Chemistry Section Manager to discuss the problems that were found with the validator whose reports are in error, and supply additional training as necessary.

The actual percentage of reports reviewed is determined by the Quality Assurance Manager based upon the results of the validation report surveillance.

8.2 Validation Report Surveillance

The Quality Assurance Manager periodically (quarterly, at a minimum) performs validation report surveillances for all chemistry sections. The reports are selected at random for each section. The surveillance consists of the following steps:

Read the report for clarity of presentation (grammar, punctuation, etc.).

Determine if the report reviews were documented.

Determine if the reports are signed and dated.

Determine if the reports are prepared in accordance with applicable SOPs and guidelines.

Determine that Action Items reflect the Data Summary results.

Based upon the results of the validation report surveillance, the Quality Assurance Manager determines what percentage of the validation reports will be subject to section manager review.

Appendix I

Data Validation Reports

EXAMPLE VOLATILES DATA VALIDATION REPORT

October 14, 1992

Mr. James E. Sandt
Texas World Operations, Inc.
520 Post Oak Boulevard
Houston, TX 77027

RE: Chem Waste Management
Site: Vickery, Ohio
Lab: ENSECO - RMA
Volatiles: 2/Soil
Samples: 418, 419

Dear Mr. Sandt:

A full validation was performed on the analytical data from two level soil samples out of a data package containing 16 soil and two water samples which were submitted to ENSECO - RMA for Volatile analysis. The data were evaluated based on the following parameters:

* Data Completeness
* Holding Times
* GC/MS Tuning
  Calibration
* Blanks
* Surrogate Recoveries
* Matrix Spike/Matrix Spike Duplicate
* Internal Standard Performance
* Compound Identification
* Compound Quantitation
  Tentatively Identified Compounds
* System Performance

* All criteria were met for this parameter

Validation was conducted in accordance with the EPA document "Laboratory Data Validation Functional Guidelines for Organic Analyses" (February 1, 1988).

Data Completeness

All data were submitted in full.

Holding Times

The samples were analyzed within the required holding times.

GC/MS Tuning

All BFB tuning criteria were met.

Calibration

The %RSD for 2-hexanone in the 8/4/92 initial calibration and the %D for acetone in the 8/6/92 continuing calibration exceeded criteria. No action is necessary because there were no positive results for these compounds in the associated samples.

Blanks

No contaminants were found in the method blank.

Surrogate Recoveries

All surrogate recovery criteria were met.

Matrix Spike/Matrix Spike Duplicate

All matrix spike/matrix spike duplicate (MS/MSD) criteria for the MS/MSD analyses of sample 419 were met.

Internal Standard Performance

All internal standard areas and retention times were within criteria.

Compound Identification

No problems were observed.

Compound Quantitation

No calculation errors were found.

Tentatively Identified Compounds

One TIC was found in sample 419.

System Performance

No problems were observed.

Overall Assessment

The data are acceptable.

No calculation or transcription errors were found.

Sincerely,
QuantaLex, Inc.

Jane Doe
Staff Associate

Mary Jones
Associate Consultant

Enclosures

IF:0212-004

EXAMPLE SEMIVOLATILES DATA VALIDATION REPORT

October 14, 1992

Mr. James E. Sandt
Texas World Operations, Inc.
520 Post Oak Boulevard
Houston, TX 77027

RE: Chem Waste Management

Site:	Vickery, Ohio
Lab:	ENSECO - RMA
Semivolatiles:	2/Soil
Samples:	416, 417

Dear Mr. Sandt:

A full validation was performed on the analytical data from two low level soil samples out of a data package containing 15 soil samples and one water sample which were submitted to ENSECO - RMA for Semivolatile analysis. The data were evaluated based on the following parameters:

* Data Completeness
  Holding Times
* GC/MS Tuning
  Calibration
  Blanks
* Surrogate Recoveries
  Matrix Spike/Matrix Spike Duplicate
  Internal Standard Performance
* Compound Identification
  Compound Quantitation
* Tentatively Identified Compounds
* System Performance

* All criteria were met for this parameter

Validation was conducted in accordance with the EPA document "Laboratory Data Validation Functional Guidelines for Organic Analyses" (February 1, 1988).

Data Completeness

All data were submitted in full.

Holding Times

Sample 416 was extracted beyond the required holding time. Because the holding time was grossly exceeded, all non-detected results in this sample are rejected (R). (No positive results were found.)

GC/MS Tuning

All DFTPP tuning criteria were met.

Calibration

The %RSDs and %Ds for several compounds exceeded criteria in the various calibrations. No action is necessary as there were no positive results for these compounds in the associated samples.

Blanks

Bis(2-ethylhexyl)phthalate was reported in both method blanks, MB001 and MB002. Benzo(a)pyrene was also found in MB002. No action is necessary because there were no positive results for these blank contaminants in the associated samples.

The concentration for bis(2-ethylhexyl)phthalate in method blank MB001 was well above 5x the CRQL; however, the sample data were not affected.

Surrogate Recoveries

All surrogate recovery criteria were met.

Matrix Spike/Matrix Spike Duplicate

The percent recoveries for pentachlorophenol, n-nitroso-di-n-propylamine, 4-nitrophenol and 2,4-dinitrotoluene were below QC limits in the matrix spike and matrix spike duplicate (MS/MSD) analyses of sample 417. (The recoveries for 4-nitrophenol and pentachlorophenol were less than 10% in these analyses.) No action is taken because sample results are not qualified solely on MS/MSD data.

Internal Standard Performance

The areas for the internal standards 1,4-dichlorobenzene-d4 and naphthalene-d8 in method blank EBS094 were below QC limits. Although the blank was not reanalyzed, no action is required because the sample data were not affected.

All remaining internal standard areas and retention times were within QC limits.

Compound Identification

No problems were observed.

Compound Quantitation

The results in method blank MB001 were incorrectly calculated. The relative response factors (RRFs) from the 9/2/92 (0002) rather than the 9/3/92 (0959) continuing calibration were used for surrogate and compound quantitation. The correct values for bis(2-ethylhexyl)phthalate and benzo(a)pyrene should be 8600 ug/kg and 130 ug/kg, respectively. The correct surrogate recoveries are still within QC limits. Sample results were not affected.

Tentatively Identified Compounds

Two TICs were found in method blank, MB002, and several other TICs were found in the associated sample, 416. The TICs were correctly reported.

System Performance

No problems were observed.

Overall Assessment

All results in sample 416 are rejected (R) due to grossly exceeded holding times.

All results in sample 417 are acceptable.

Sincerely,
QuantaLex, Inc.

Goldie Locks
Staff Associate

Cindy Rella
Associate Consultant

Enclosures
IF: 0555-004

svol-tex

EXAMPLE PESTICIDES/PCBs DATA VALIDATION REPORT

October 14, 1992

Mr. James E. Sandt
Texas World Operations, Inc.
520 Post Oak Boulevard
Houston, TX 77027

RE: Chem Waste Management

Site:	Vickery, Ohio
Lab:	ENSECO - RMA
Pesticide/PCBs:	1/Soil
Sample:	DEF12

Dear Mr. Sandt:

A full validation was performed on the analytical data from one low level soil sample out of a data package containing three low level soil samples which were submitted to ENSECO - RMA for Pesticide/PCBs analysis. The data were evaluated based on the following parameters:

Data Completeness
* Holding Times
Calibration
Blanks/Blank Spike
Surrogate Recoveries
Matrix Spike/Matrix Spike Duplicate
Pesticide Instrument Performance
* Compound Identification
Compound Quantitation

* All criteria were met for this parameter

Validation was conducted in accordance with the EPA document "National Functional Guidelines for Organic Data Review" (Draft 6/91).

Data Completeness

Form 1s for instrument blanks were not provided by the laboratory. No action is taken because the data was not adversely affected.

Holding Times

The samples were extracted and analyzed within holding times.

Calibration

Various %RSDs exceeded 20% in the initial calibrations. No action is necessary because there were no positive results for these compounds in the associated sample.

Blanks/Blank Spike

The result for blank spike reported on Form 3c differed from Form 1 and Form 10. In addition, the results on Form 3c were reported in ng/ml. No action is taken.

Surrogate Recoveries

The percent recoveries for three of the four surrogates were less than QC limits in sample DEF12. The positive result for alpha-chlordane in sample DEF12 is estimated (J).

Matrix Spike/Matrix Spike Duplicate

Matrix spike/matrix spike duplicate criteria were met.

Pesticide Instrument Performance

Various resolutions could not be verified using the provided raw data. No action is taken.

Compound Identification

All positive results above CRQL were correctly identified.

Compound Quantitation

The positive result for gamma-chlordane was not reported by the laboratory. It was found above one-half the method detection limit by QuantaLex, Inc. The positive result was reported and estimated (J) because the %D comparison between the columns was greater than 25%.

CRQLs were reported with three significant figures instead of two. No action is taken.

Overall Assessment

The majority of the data are acceptable.

The positive alpha-chlordane result in sample DEF12 is estimated (J) due to surrogate percent recoveries outside criteria.

The positive result for gamma-chlordane is estimated due to column comparison criteria not being met.

Sincerely,
QuantaLex, Inc.

Maggie Simpson
Staff Associate

George Jetson
Associate Consultant

Enclosures

IF: 0555-004

EXAMPLE METALS DATA VALIDATION REPORT

October 14, 1992

Mr. James E. Sandt
Texas World Operations, Inc.
520 Post Oak Boulevard
Houston, TX 77027

RE: Chem Waste Management
Site Location: Vickery, Ohio
Metals: 2/Soil
Lab: ENSECO - RMA
Samples: ABC123, ABC124

Dear Mr. Sandt:

A full validation was performed on the analytical data from 2 low level soil samples out of a data package containing 17 samples which were submitted to ENSECO - RMA for inorganic analysis. The data were evaluated based on the following parameters:

* Holding times
* Calibration verification
  Laboratory and field blank analyses
* ICP interference check sample
  Matrix spike recoveries
  Laboratory and field duplicates
  Laboratory control sample results
* Graphite furnace atomic absorption QC
* Serial dilution results
  Sample result verification

* - All criteria were met for this parameter

Validation was conducted in accordance with the EPA document "Laboratory Data Validation Guidelines for Evaluating Inorganic Analyses" (July 1, 1988).

met-tex/texas

Holding Times

All holding time criteria were met.

Calibration Verification

All calibration verification criteria were met.

Laboratory Blank Analysis

Analyte	Maximum Conc./Units	Action Level
V	4.6 mg/Kg	23.0 mg/Kg
K	140.4 mg/Kg	702.0 mg/Kg

Value > IDL, and < Action Level = Report value U.
Value > IDL and > Action Level = Report value unqualified.

The vanadium result for ABC123 is made a non-detect due to blank contamination.

The potassium result for ABC123 is made a non-detect due to blank contamination.

ICP Interference Check Sample

All ICP interference check sample criteria were met.

Matrix Spike Recoveries

Analyte	SSR	SR	S	%R
Sb	51.5 mg/Kg	9.9 U mg/Kg	103.0 mg/Kg	50.0
Cu	94.3 mg/Kg	18.5 mg/Kg	51.5 mg/Kg	147.2
Mn	416.1 mg/Kg	362.5 mg/Kg	103.0 mg/Kg	52.0
Hg	0.59 mg/Kg	0.22 mg/Kg	0.51 mg/Kg	72.5

Estimate (J) positive results and estimate (UJ) non-detects when pre-digestion matrix spike recovery is between 30%-74%.
Estimate (J) positive results when pre-digestion matrix spike recovery is greater than 125%.

The antimony non-detects for ABC123 and ABC124 are estimated (UJ) due to poor matrix spike recovery.

The copper, manganese, and mercury results for ABC123 and ABC124 are estimated (J) due to poor matrix spike recoveries.

Laboratory Duplicates

Analyte	Sample Result	Duplicate Result	RPD%
Ca	13182 mg/Kg	6920 mg/Kg	62.3
Pb	40.4 mg/Kg	9.88 mg/Kg	121.4

Estimate (J) positive results when relative percent difference (RPD) is greater than 35%.

The calcium and lead results for ABC123 and ABC124 are estimated (J) due to poor laboratory duplicate precision recovery.

Field Duplicates

The field duplicates were identified as ABC123 and ABC124.

No field duplicate criteria have been established for EPA Region 9.

Laboratory Control Sample Results

Analyte	Matrix	True Value	Found Value	Low Limit	High Limit
Al	Soil	9510.0 mg/Kg	5480.0 mg/Kg	5710.0 mg/Kg	13300.0 mg/Kg

Estimate (J) positive results and estimate (UJ) non-detects when the soil LCS recovery is less than the control windows.

The aluminum results for ABC123 and ABC124 are estimated (J) due to poor laboratory control sample recovery.

Graphite Furnace Atomic Absorption (GFAA) QC

All graphite furnace atomic absorption criteria were met.

ICP Serial Dilution Results

All ICP serial dilution criteria were met.

Sample Result Verification

Due to poor copy quality, it was difficult to read the left hand side of the GFAA raw data. No action was taken.

Overall Assessment

The majority of the data are acceptable.

The antimony non-detects for ABC123 and ABC124 are estimated (UJ) due to poor matrix spike recovery.

The copper, manganese, and mercury results for ABC123 and ABC124 are estimated (J) due to poor matrix spike recoveries.

The calcium and lead results for ABC123 and ABC124 are estimated (J) due to poor laboratory duplicate precision recovery.

The aluminum results for ABC123 and ABC124 are estimated (J) due to poor laboratory control sample recovery.

The vanadium and potassium detection limits are raised due to blank contamination.

The chain of custody does not indicate preservation of the soil samples to 4°C.

Sincerely,
QUANTALEX, INC.

John Q. Adams
Staff Associate

Howard Taft
Associate Consultant

Enclosures

IF: 0555-004

TEXAS WORLD OPERATIONS, INC.
ORGANIC DATA VALIDATION GUIDELINES
EPA SW-846 METHOD 8240

VOLATILE ORGANICS
BY
GAS CHROMATOGRAPHY/MASS SPECTROMETRY
(GC/MS):
PACKED COLUMN TECHNIQUE
(SEPTEMBER, 1986)

Prepared By:

QuantaLex, Inc.
300 Union Boulevard, Suite 600
Lakewood, Colorado 80228
(303) 763-8881

October 1992

TEXAS WORLD OPERATIONS, INC.
ORGANIC DATA VALIDATION GUIDELINES
EPA SW-846 METHOD 8240

VOLATILE ORGANICS
BY
GAS CHROMATOGRAPHY/MASS SPECTROMETRY
(GC/MS):
PACKED COLUMN TECHNIQUE
(SEPTEMBER, 1986)

Developed By: _____ Date _____

William T. Fear
Associate Consultant
QuantaLex, Inc.

Approved By: _____ Date _____

Ken Schroeder
Quality Assurance Manager
QuantaLex, Inc.

Concurrence: _____ Date _____

Robert Thielke
Deputy Program Manager
QuantaLex, Inc.

October 1992
Version 1.0

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APPENDICES

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Appendix B Data Qualifier Definitions

1.0 INTRODUCTION

This document is designed to offer guidance in laboratory data evaluation and validation of volatile organics in water, soil, and solid waste sample analyses. In some areas, specific guidelines and criteria are available. In other, less objective areas, only general guidance is offered due to the complexities and uniqueness of data relative to specific samples. The reviewer has a responsibility to inform the user concerning data quality and limitations, based on his/her technical judgment and evaluation, to assist the user in the proper use of the data.

Those areas where specific criteria are included are primarily areas in which definitive method performance requirements are established. These requirements are concerned with specifications that are not strictly sample dependent. They specify performance requirements normally under a laboratory's control. These specific areas include GC/MS tuning calibration standards, blanks, matrix spikes, and surrogate recoveries. Calculation and transcription errors must be rectified by laboratory resubmission of corrected data sheets if verification of the reported result is in doubt.

2.0 HOLDING TIMES

2.1 Objective

The objective is to ascertain the validity of results based on the holding time of the sample from the time of collection to the time of analysis.

2.2 Criteria

All water and soil samples must be iced or refrigerated at 4°C. Sodium thiosulfate should be added to water samples containing residual chlorine. Concentrated hydrochloric acid should be added to all water samples. Concentrated waste samples need no preservation (including refrigeration). (Although not used for qualification, these criteria are noteworthy.)

All samples must be analyzed within fourteen days of collection. If the collection or sampling episode spans multiple days, the day of completion is used as the collection date.

2.3 Evaluation Procedure

Actual holding times are established by comparing the collection date on the chain-of-custody form with the date of analysis found in the laboratory raw data. Holding times are determined as follows:

Analysis Holding Time (Days) = Analysis Date - Collection Date

2.4 Action

1. If water samples are not preserved with hydrochloric acid and the analysis holding time exceeds seven days but is less than or equal to fourteen days, estimate (J) positive results or (UJ) non-detected results for aromatic compounds.

2. If the fourteen-day analysis holding time is exceeded, estimate (J) positive results and (UJ) non-detected results.

3. If the fourteen-day analysis holding time is grossly exceeded, estimate (J) positive results and reject (R) non-detected results. Grossly exceeded is defined as twice the acceptable holding time.

3.0 GC/MS TUNING

3.1 Objective

Instrument tuning and performance criteria are established to ensure mass resolution, identification,and to some extent, sensitivity. Although these criteria are not sample specific, conformance is determined by the laboratory using standard materials. These criteria must be met in all circumstances.

3.2 Criteria

All tuning criteria must be met and demonstrated during each working shift prior to the initial calibration and prior to the daily (continuing) calibration and sample analyses. These criteria must be demonstrated every 12 hours. The performance criteria for 4-bromofluorobenzene (BFB) key ion abundances are listed below:

BFB ION ABUNDANCE CRITERIA	
MASS (m/z)	Relative Abundance
50	15.0% to 40.0% of mass 95
75	30.0% to 60.0% of mass 95
95	Base peak, 100.0% relative abundance
96	5.0% to 9.0% of mass 95
173	< 1.0% of mass 95
174	> 50.0% of mass 95
175	5.0% to 9.0% of mass 174
176	> 95.0% but < 101.0% of mass 174
177	5.0% to 9.0% of mass 176

3.3 Evaluation Procedure

1. Verify from the raw data that the mass calibration is correct.

2. Compare the mass listings submitted in the raw data to the reported relative abundances.

3. Ensure the following:

   a. The GC/MS tune for each working shift, including the tune associated with each initial calibration, is present in the data package.

   b. The laboratory has not made any transcription or calculation errors.

4. If possible, verify that spectra were generated using appropriate background subtraction techniques.

5. Verify that the sample analyses occur within twelve hours of the preceding tune.

3.4 Action

1. If the ion abundance criteria are not met, reject (R) all results.

2. If the reviewer has reason to believe that tuning criteria were achieved using techniques that distorted or skewed the spectra, full documentation on the tuning quality control should be provided in the data package by the laboratory. If the anomalies are not adequately explained or documented, reject (R) all results.

3. If the sample is analyzed more than twelve hours after the tune, use professional judgment to qualify the data. At a minimum, provide a comment in the validation report.

4.0 CALIBRATION

4.1 Objective

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. This may be done by using response factors (RFs).

Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents satisfactory performance of the instrument over specific time periods during sample analysis.

4.2 Criteria

1. Initial Calibration

   a. Calibration standards are prepared and analyzed at the following concentrations: 20 ug/L, 50 ug/L, 100 ug/L, 150 ug/L, and 200 ug/L. The internal standard calibration technique is used.

   b. The percent relative standard deviations (%RSDs) of the RFs for all compounds must be less than or equal to 30%.

   c. The RFs for all compounds must be greater than or equal to 0.05.

   d. There are five system performance check compounds (SPCCs): bromoform, chlorobenzene, chloromethane, 1,1-dichloroethane, and 1,1,2,2-tetrachloroethane. The RFs for these SPCCs should be greater than or equal to 0.300 except the RF for bromoform which should be greater than or equal to 0.250.

   e. There are six calibration check compounds (CCCs): 1,1,-dichloroethene, chloroform, 1,2-dichloropropane, toluene, ethylbenzene, and vinyl chloride. The %RSDs for these compounds should be less than or equal to 30%.

2. Continuing Calibration

   a. The working calibration curve must be verified every 12 hours by the analysis of one 50 ug/L standard.

   b. The percent difference (%D) of the response factors for all compounds must be less than or equal to 25%.

   c. The RFs for all compounds must be greater than or equal to 0.05.

d. The RFs for the SPCCs (mentioned above) should be greater or equal to than 0.300 except the RF for bromoform which should be greater than or equal to 0.250.

e. The %Ds for the CCCs (mentioned above) should be less than or equal to 25%.

4.3 Evaluation Procedure

1. Initial Calibration

   a. Check the raw data and recalculate 10-20% of the RFs and %RSDs.

   b. Verify that the %RSDs are less than or equal to 30%.

   c. Verify that the RFs for all compounds are greater than or equal to 0.05.

   d. Verify that the SPCCs and CCCs meet criteria.

   e. If errors are detected, pursue the explanation via more comprehensive recalculation.

2. Continuing Calibration

   a. Check the raw data and recalculate 10-20% of the RFs and %Ds.

   b. Verify that the %Ds are less than or equal to 25%.

   c. Verify that the RFs are greater than or equal to 0.05.

   d. Verify that the SPCCs and CCCs meet criteria.

   e. If errors are detected, pursue the explanation via more comprehensive recalculation.

3. Calculations

$$RF = \frac{A_x \times C_{is}}{A_{is} \times C_x}$$

where:

A_x = Response for the characteristic ion for the analyte to be measured, units area counts.
C_{is} = Concentration of the internal standard, ug/L.
A_{is} = Response for the characteristic ion for the internal standard, units area counts.
C_x = Concentration of the analyte to be measured, ug/L.

$$\%RSD = \frac{SD}{\bar{x}} \times 100$$

$$SD = \sqrt{\sum_{i=1}^{n} \frac{(X_i - \bar{x})^2}{(n-1)}}$$

where:

$\bar{x}$ = Mean of 5 response factors.

$$\%D = \frac{R_1 - R_2}{R_1} \times 100$$

R_1 = Average response factor from initial calibration.
R_2 = Response factor from continuing calibration.

4.4 Action

1. Initial Calibration

   a. Estimate (J) positive results for those compounds whose %RSDs exceed 30% in the associated initial calibration.

   b. Estimate (J) positive results and (UJ) non-detected results for those compounds whose %RSDs exceed 50% in the associated initial calibration.

   c. Estimate (J) positive results and reject (R) non-detected results for those compounds whose %RSDs exceed 100% in the associated initial calibration.

   d. Estimate (J) positive results for those compounds whose RFs are less than 0.05.

   e. Reject (R) non-detected results for those compounds whose RFs are less than 0.05.

   f. If the SPCCs and CCCs do not meet criteria, provide a comment in the data validation report. Qualify the associated results for these compounds according to a. through e.

2. Continuing Calibration

   a. Estimate (J) positive results for those compounds whose %Ds exceed 25% in the associated continuing calibration.

   b. Estimate (J) positive results and (UJ) non-detected results for those compounds whose %Ds exceed 50% in the associated continuing calibration.

   c. Estimate (J) positive results and reject (R) non-detected results for those compounds whose %Ds exceed 100% in the associated continuing calibration.

   d. Estimate (J) positive results for those compounds whose RFs are less than 0.05.

   e. Reject (R) non-detected results for those compounds whose RFs are less than 0.05.

   f. If the SPCCs and CCCs do not meet criteria, provide a comment in the data validation report. Qualify the associated results for these compounds according to a. through e.

5.0 BLANKS

5.1 Objective

The assessment of method blank analysis results is to determine the existence and magnitude of contamination problems. If problems with any method blank exist, all data associated with the method blank must be carefully evaluated to determine whether or not there is an inherent variability in the data for the batch, or if the problem is an isolated occurrence not affecting other data. ("Associated" refers to those samples analyzed with a particular method blank.)

5.2 Criteria

A laboratory reagent water blank (method blank) must be analyzed before each set of samples (one every 12 hour analysis sequence), when there is a change in reagents, and following any concentrated sample that has saturated ions from a compound. No contaminants should be present in the blank(s) at or above the practical quantitation limit (PQL). The blank samples should be carried through all stages of the sample preparation and measurement steps.

5.3 Evaluation Procedure

1. Review the results of all associated blank(s) and raw data (chromatograms, mass spectra, quantitation reports, or data system printouts).

2. Verify that method blank analysis has been reported per matrix, per concentration level, and per 12 hour analysis sequence to analyze samples.

5.4 Action

1. If a target compound is found in the method blank but not in the samples, no action is taken.

2. If a target compound is found in the method blank and is also found in the sample, apply the following (the common contaminants are listed below):

    a. If the sample concentration is less than 5 times the blank concentration (10 times for common contaminants) and less than the PQL: Report the PQL followed by (UJ).

    b. If the sample concentration is less than or equal to 5 times the blank concentration (10 times for common contaminants) and greater than or equal to the PQL: Report the value followed by (UJ).

    c. If the sample concentration is greater than 5 times the blank concentration (10 times for common contaminants) and greater than the PQL: Report the value unqualified.

3. If an associated method blank exhibits gross contamination, reject (R) results for all affected compounds.

4. If an associated method blank was not analyzed for the samples, estimate (J) positive results.

5. If an associated method blank was not analyzed for the samples, and there are positive results for the same target compound in all samples, reject (R) the positive results for that target compound in all samples.

Common Contaminants :

1. acetone
2. 2-butanone
3. methylene chloride
4. toluene

6.0 SURROGATES

6.1 Objective

Laboratory performance on individual samples is established by means of spiking activities. The evaluation of the results of these surrogate spikes is not necessarily straightforward. The sample itself may produce effects due to such factors as interferences and high concentrations of analytes. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the review and validation of data based on specific sample results is frequently subjective and demands analytical experience and professional judgment. Accordingly, this section consists primarily of guidelines.

6.2 Criteria

1. Surrogates must be added at 50 ppb to all samples and blanks prior to analysis. (Surrogate concentrations will differ for medium-concentration soil analyses.)

2. The surrogate compounds 4-bromofluorobenzene, 1,2-dichloroethane-d4, and toluene-d8 are recommended. Surrogate recoveries must fall within the control limits set by the laboratory. The laboratory control limits must fall within the following ranges:

Surrogate Compound	Low/Medium Water	Low/Medium Soil/Sediment
4-bromofluorobenzene	86-115	74-121
1,2-dichloroethane-d4	76-114	70-121
toluene-d8	88-110	81-117

3. If surrogate recoveries are outside the control limits and the instrument is at fault, the instrument problem must be resolved and the samples reanalyzed.

4. If surrogate recoveries are outside the control limits and the instrument is not at fault, the samples must be reextracted (for medium-concentration) and reanalyzed.

5. If one surrogate recovery is outside control limits in the method blank, the samples and blank should be reanalyzed.

6.3 Evaluation Procedure

1. Verify the calculations and recoveries using the raw data (i.e., chromatograms, quantitation reports, etc.).

2. Verify the transcription of the recoveries from the raw data.

3. If recoveries are not within the control limits, check the raw data for possible interferences.

4. Check to verify that appropriate reanalyses or reextractions were performed.

5. If multiple analyses of the same sample are submitted, determine which analysis should be reported by assessing which has better overall QC. The QC categories used to make this assessment are: holding times, internal standards, surrogate recoveries, and presence and absence of positive results.

6.4 Action

1. If the recovery of any one surrogate is greater than the control limits, estimate (J) positive results. (Determine that high bias was not due to interference with the surrogate compound only.)

2. If the recovery of any one surrogate is less than the laboratory control limits but greater than 10%, estimate (J) positive results and (UJ) non-detected results.

3. If the recovery of any one surrogate is less than 10%, estimate (J) positive results and reject (R) non-detected results.

4. If appropriate reanalyses or reextractions were not performed, provide a comment in the validation report.

7.0 MATRIX SPIKE ANALYSIS

7.1 Objective

These data are generated to determine long-term precision and accuracy of the analytical method on various matrices. These data alone cannot be used to evaluate the precision and accuracy of individual samples.

7.2 Criteria

The laboratory is required to collect a portion of their samples to monitor spike recoveries. The frequency of spiked sample analyses must be one matrix spike (MS) and one matrix spike duplicate (MSD) per analysis batch (up to 20 samples). The matrix spiking solution contains the following compounds : 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. The concentration for each compound is 50 ppb for low-level water and soil samples. The laboratory control limits should fall within the following CLP acceptance criteria:

	Percent Recovery		RPD	
Compound	Water	Soil/Sediment	Water	Soil
1,1-dichloroethene	61-145	59-172	14	22
trichloroethene	71-120	62-137	14	24
benzene	76-127	66-142	11	21
toluene	76-125	59-139	13	21
chlorobenzene	76-130	60-133	13	21

7.3 Evaluation Procedure

1. Verify the calculations, recoveries, and RPDs using the raw data (i.e., chromatograms, quantitation reports, etc.).

2. Verify the transcription of the recoveries and RPDs from the raw data.

3. Verify that the MS/MSD recoveries and RPDs, as applicable, fall within the control limits set by the laboratory.

$$RPD = \frac{D_1 - D_2}{(D_1 + D_2)/2} \times 100$$

D_1 = MS Concentration.
D_2 = MSD Concentration.

7.4 Action

No action is taken on MS/MSD data alone to qualify an entire batch. However, using informed professional judgment the data reviewer may use the MS/MSD results in conjunction with other QC criteria and determine the need for some qualification of data.

8.0 INTERNAL STANDARDS

8.1 Objective

Internal standards (IS) performance criteria ensure that GC/MS sensitivity and response are stable during every run.

8.2 Criteria

1. Internal standards must be added at 50 ppb to all samples, standards, and blanks prior to analysis.

2. The IS compounds bromochloromethane, 1,4-difluorobenzene, and chlorobenzene-d5 are recommended.

3. The IS area counts in the samples must not vary by more than a factor of two (-50% to +100%) from the associated calibration standard.

4. The IS retention times in the samples must not vary by more than +/- 30 seconds from the associated calibration standard.

8.3 Evaluation Procedure

1. Check raw data (i.e., chromatograms, quantitation reports, etc.) to verify the area counts and retention times.

2. Verify that all IS area counts and retention times are acceptable.

8.4 Action

1. If the area counts for any IS are outside the control limits (-50% to +100%), estimate (J) positive results and (UJ) non-detected results for compounds quantitated using that IS.

2. If the area counts for any IS are extremely low (i.e. less than 50% of the lower control limit), or if performance exhibits a major abrupt drop off, reject (R) non-detected results and estimate (J) positive results for compounds quantitated using that IS.

3. If any IS retention time varies by more than 30 seconds, examine the chromatographic profile to determine if any false positives or false negatives exist. If identification problems are evident, reject (R) affected results.

4. If any IS retention time varies by a large magnitude, reject (R) all results.

9.0 ADDITIONAL QUALITY CONTROL

9.1 Objective

Laboratory control samples are required to establish acceptable bias and precision prior to any sample analyses. (Data are not likely to be included as a deliverable.)

9.2 Criteria

It is not clear that the laboratory control sample data will be included in the data package. If provided, the laboratory control sample recoveries should be comparable to the data from Section 7.2 of these guidelines.

The maximum number of values that can fall outside the control limits are one percent recovery and one RPD.

9.3 Evaluation Procedure

Evaluate the accuracy of the data.

Verify that the recoveries and RPDs are within control limits.

9.4 Action

Professional judgment is required to determine the impact of outliers on the sample data.

10.0 COMPOUND IDENTIFICATION

10.1 Objective

The objective of the criteria for GC/MS qualitative analysis is to minimize the number of erroneous identifications of compounds.

10.2 Criteria

1. The mass spectra for standard reference should be obtained on the instrument used for sample analysis.

2. The sample compound relative retention time (RRT) must compare within plus or minus 0.06 RRT units of the standard component. The reference standard must be run within the same 12 hours as the sample.

3. All ions present in the standard mass spectra (ions of interest) at a relative intensity greater than 10% must be present in the sample spectrum. In addition, all ions meeting the above criteria must agree within plus or minus 20% between the sample and standard spectra.

10.3 Evaluation Procedure

1. Verify the transcription of all results from the quantitation report to the Form I or equivalent results summary.

2. Verify that RRT of reported compounds is within 0.06 RRT units of the reference standard.

3. Verify that the ions of interest present in the standard spectra are also present in the sample compound spectra at the appropriate intensities.

4. Review the spectra for ions present in the sample spectrum but not in the reference spectrum. This may indicate possible background contamination or presence of coeluting compounds.

5. Review the spectra for ions present in the reference spectrum but not in the sample spectrum. This may indicate possible subtraction from the sample spectrum because of background contamination or coeluting compounds.

6. The reviewer should be aware of situations (e.g. high concentration samples preceding low concentration samples) when sample carry-over is a possibility and should use judgment to determine if carry-over has occurred.

10.4 Action

1. The application of qualitative criteria for GC/MS analysis of target compound list (TCL) compounds requires professional judgment. If it is determined that an incorrect false positive exists, the result should be reported as non-detected (U). If it is determined that a false negative exists, reject (R) the non-detected result.

2. Professional judgment should be used to qualify data in which carry-over is evident.

11.0 COMPOUND QUANTITATION

11.1 Objective

The objective is to ensure that the reported quantitation results and PQLs are accurate.

11.2 Criteria

1. Compound quantitation, as well as the adjustment of the PQLs, must be calculated according to the method. Compound quantitation shall be performed using the internal standard nearest in retention time.

2. If the response for the peak exceeds the calibration range of the system, the sample must be diluted and reanalyzed. All dilutions should keep the response of the major constituents in the upper half of the calibration range of the system.

11.3 Evaluation Procedure

1. Examine the raw data to verify the correct calculation of all sample results. Quantitation reports, chromatograms, sample preparation logs, and dilutions are compared to the reported sample results and quantitation limits.

2. Verify that if the response for any target compound exceeded the calibration range of the system, the sample was diluted appropriately and reanalyzed.

3. Calculations:

Water and water-miscible waste:

$$\text{Concentration (ug/L)} = \frac{A_x \times I_s \times D}{A_{is} \times RF \times V_o}$$

where:

A_x = Response of the characteristic ion for the analyte in the sample, area counts.
I_s = Amount of internal standard injected, ng.
D = Dilution factor.
A_{is} = Response of the characteristic ion for the internal standard, area counts.
RF = Response factor for the analyte from the appropriate calibration standard (see page 4-2).
V_o = Volume of water purged, mL.

Sediment/soil, sludge, and waste:

High-concentration:

$$\text{Concentration (ug/Kg)} = \frac{A_x \times I_s \times V_t}{A_{is} \times RF \times V_i \times W_s}$$

Low-concentration:

$$\text{Concentration (ug/Kg)} = \frac{A_x \times I_s}{A_{is} \times RF \times W_s \times P_s}$$

where:

A_x, I_s, A_{is}, RF = Same as in water and water-miscible waste above.

V_t = Volume of total extract, uL.

V_i = Volume of extract added for purging, uL.

W_s = Weight of sample extracted or purged, g.

P_s = Percent solids.

For tentatively identified compound (TIC) calculations:

A_x = Total area or peak height for analyte of interest.

A_{is} = Total area or peak height of nearest internal standard free of interference.

RF = One.

11.4 Action

1. If major discrepancies are found which prevent determination of the correct result, resubmittal will be necessary to resolve the problem.

2. If the response for any target compound exceeds the linear range of the system and the sample was not diluted and reanalyzed, the value should be qualified with an (E).

3. If the diluted sample analysis fails to keep the response of the major constituents in the upper half of the calibration range, use professional judgment to qualify the data. At a minimum, provide a comment in the validation report.

12.0 SYSTEM PERFORMANCE

12.1 Objective

A thorough review of on-going data acquisition and examination of the raw data can yield indicators of instrument performance.

12.2 Criteria

Instrument performance indicators are as follows:

1. Abrupt, discrete baseline changes possibly indicating gain or threshold changes.

2. Poor chromatographic performance characterized by high background levels, shifts in absolute retention times of internal standards, large extraneous peak interferences, excessive baseline rise at elevated temperatures, loss of peak resolution, and peak tailing or peak splitting which could result in inaccurate quantitation.

12.3 Action

Based on the performance indicators discussed previously, the data reviewer must decide if the system has degraded to the point of affecting data quality or validity. If data quality may have been affected, data should be qualified based on the reviewer's technical judgment and subjective evaluation.

13.0 TENTATIVELY IDENTIFIED COMPOUNDS

13.1 Objective

Chromatographic peaks which are not TCL compounds, surrogates, or internal standards are potential tentatively identified compounds (TICs). TICs may be qualitatively identified by GC/MS library search and the identifications assessed by the data reviewer.

13.2 Criteria

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification of the 10 or 15 largest unidentified peaks. The necessity to perform this type of identification will be determined by the type of analyses being conducted. Guidelines for making tentative identification are:

1. Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum. In addition, all ions meeting the above criteria should agree within plus or minus 20%.

2. Molecular ions present in the reference spectrum should be present in the sample spectrum.

13.3 Evaluation Procedure

1. Check the raw data to verify that a library search has been generated for all required peaks in the chromatograms.

2. Examine blank chromatograms to verify that TICs present in the samples are not present in the associated blanks.

3. Examine the mass spectra to verify that the best match was chosen based upon the guidelines for identification listed above.

4. Review the spectra for ions present in the sample spectrum but not in the reference spectrum. This may indicate possible background contamination or presence of coeluting compounds.

5. Review the spectra for ions present in the reference spectrum but not in the sample spectrum. This may indicate possible subtraction from the sample spectrum because of background contamination or coeluting compounds.

4. Verify that none of the TICs reported is actually a TCL compound.

13.4 Action

1. Estimate (J) all TIC results. (The reported results should already be estimated by the laboratory.)

2. If all contractually required peaks were not library searched, request resubmittal data.

3. If a TIC result in the sample is not sufficiently above the level in the blank, the TIC result should not be reported. (Generally, the 5X blank rule is applied here.)

4. If it is determined that the TIC identification is not acceptable, the TIC should be changed to an unknown or an appropriate class of compound.

5. In general, TIC identification is dependant on professional judgment which incorporates factors such as boiling point. In addition, if a TIC spectral match is poor but other samples have the same TIC present with good spectral match, similar retention time, and the same ions, the identification may be inferred from the other sample TIC results.

14.0 OVERALL DATA ASSESSMENT

The overall data assessment represents the quality of the majority of the data. It can only be assigned once the data validation process is complete. At this point, each data point for each analytical result has been assigned a data qualifier based upon deficiencies in one or more of the QC categories (i.e. holding times, calibration, etc.). All results have been qualified. Determine which qualifier has been assigned to the majority of the analytical results and assign that qualifier as the overall data assessment qualifier.

APPENDIX A
ACRONYM LIST

ACRONYM LIST

BFB	Bromofluorobenzene
%D	Percent Difference
IS	Internal Standard
MS	Matrix Spike
MSD	Matrix Spike Duplicate
PQL	Practical Quantitation Limit
RF	Response Factor
RPD	Relative Percent Difference
RRT	Relative Retention Time
%RSD	Percent Relative Standard Deviation
SD	Standard Deviation
TCL	Target Compound List
TIC	Tentatively Identified Compound

APPENDIX B

DATA QUALIFIER DEFINITIONS

DATA QUALIFIER DEFINITIONS

A Data are acceptable, with qualifications.

E The associated value exceeds calibration range. Dilute and reanalyze.

J The associated value is an estimated quantity.

R Data are rejected.

U The compound was analyzed for but not detected at or above the method detection limit.

UJ The compound was analyzed for but not detected at or above the method detection limit. The associated value is an estimated quantity.

V Data are valid.

TEXAS WORLD OPERATIONS, INC
ORGANIC DATA VALIDATION GUIDELINES
EPA SW-846 METHOD 8270

SEMIVOLATILE ORGANICS
BY
GAS CHROMATOGRAPHY/MASS SPECTROMETRY
(GC/MS):
CAPILLARY COLUMN TECHNIQUE
(SEPTEMBER, 1986)

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October 1992

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EPA SW-846 METHOD 8270

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1.0 INTRODUCTION

This document is designed to offer guidance in laboratory data evaluation and validation of semivolatile organics in extracts prepared from water, soil, and solid waste samples. In some areas, specific guidelines and criteria are available. In other, less objective areas, only general guidance is offered due to the complexities and uniqueness of data relative to specific samples. The reviewer has a responsibility to inform the user concerning data quality and limitations, based on his/her technical judgment and evaluation, to assist the user in the proper use of the data.

Those areas where specific criteria are included are primarily areas in which definitive method performance requirements are established. These requirements are concerned with specifications that are not strictly sample dependent. They specify performance requirements normally under a laboratory's control. These specific areas include GC/MS tuning calibration standards, blanks, matrix spikes, and surrogate recoveries. Calculation and transcription errors must be rectified by laboratory resubmission of corrected data sheets if verification of the reported result is in doubt.

2.0 HOLDING TIMES

2.1 Objective

The objective is to ascertain the validity of results based on the holding time of the sample from the time of collection to the time of extraction and from the time of extraction to the time of analysis.

2.2 Criteria

All water and soil samples must be iced or refrigerated at 4°C. Sodium thiosulfate should be added to water samples containing residual chlorine. Concentrated waste samples need no preservation (including refrigeration). (Although not used for qualification, these criteria are noteworthy.)

Water samples must be extracted within seven days of collection and the extracts must be analyzed within 40 days of extraction. If the collection or sampling episode spans multiple days, the day of completion is used as the collection date.

Soil samples and concentrated waste samples must be extracted within 14 days of collection and the extracts must be analyzed within 40 days of extraction.

2.3 Evaluation Procedure

Actual holding times are established by comparing the collection date on the chain-of-custody form with the extraction date found in the laboratory extraction logs (if available) or on Form 1 and 4 and with the date of analysis found in the laboratory raw data. Holding times are determined as follows:

Extraction Holding Time (Days) = Extraction Date - Collection Date
Analysis Holding Time (Days) = Analysis Date - Extraction Date

2.4 Action

1. If any holding time is exceeded, estimate (J) positive results and (UJ) non-detected results.

2. If the extraction or analysis holding time is grossly exceeded, estimate (J) positive results and reject (R) non-detected results. Grossly exceeded is defined as twice the acceptable holding time.

3.0 GC/MS TUNING

3.1 Objective

Instrument tuning and performance criteria are established to ensure mass resolution, identification, and to some extent, sensitivity. Although these criteria are not sample specific, conformance is determined by the laboratory using standard materials. These criteria must be met in all circumstances.

3.2 Criteria

All tuning criteria must be met and demonstrated during each working shift prior to the initial calibration and prior to the daily (continuing) calibration and sample analyses. These criteria must be demonstrated every 12 hours. The performance criteria for decafluorotriphenylphosphine (DFTPP) key ion abundances are listed below:

DFTPP ION ABUNDANCE CRITERIA	
MASS (m/z)	Relative Abundance
51	30.0% to 60.0% of mass 198
68	< 2.0% of mass 69
70	< 2.0% of mass 69
127	40.0-60.0% of mass 198
197	< 1.0% of mass 198
198	Base peak, 100% relative abundance
199	5.0% to 9.0% of mass 198
275	10.0-30.0% of mass 198
365	> 1.0% of mass 198
441	Present but less than mass 443
442	> 40.0% of mass 198
443	17.0-23.0% of mass 442

If pesticide analyses are included with this method, the breakdown of DDT to DDE and DDD should not exceed 20%. Refer to the Data Validation Guidelines for method 8080 for calculations and guidance.

3.3 Evaluation Procedure

1. Verify from the raw data that the mass calibration is correct.

2. Compare the mass listings submitted in the raw data to the reported relative abundances.

3. Ensure the following:

   a. The GC/MS tune for each 12-hour period, including the tune associated with each initial calibration, is present in the data package.

   b. The laboratory has not made any transcription or calculation errors.

4. If possible, verify that spectra were generated using appropriate background subtraction techniques.

5. Verify that the sample analyses occur within twelve hours of the preceding tune.

3.4 Action

1. If the ion abundance criteria are not met, reject (R) all results.

2. If the reviewer has reason to believe that tuning criteria were achieved using techniques that distorted or skewed the spectra, full documentation on the tuning quality control should be provided in the data package by the laboratory. If the anomalies are not adequately explained or documented, reject (R) all results.

3. If the sample is analyzed more than twelve hours after the tune, use professional judgment to qualify the data. At a minimum, provide a comment in the validation report.

4.0 CALIBRATION

4.1 Objective

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. This may be done by using response factors (RFs).

Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents satisfactory performance of the instrument over specific time periods during sample analysis.

4.2 Criteria

1. Initial Calibration

   a. A five point calibration curve is generated using the calibration standards at the following concentrations: 20 ug/mL, 50 ug/mL, 80 ug/mL, 120 ug/mL, and 160 ug/mL. The internal standard calibration technique is used and the concentration of the internal standards should be 40 ug/mL.

   b. The percent relative standard deviations (%RSDs) of the RFs for all compounds must be less than or equal to 30%.

   c. The RFs for all compounds must be greater than or equal to 0.05.

   d. There are four system performance check compounds (SPCCs): n-nitroso-di-n-propylamine, hexachlorocyclopentadiene, 2,4-dinitrophenol, and 4-nitrophenol. The RFs for these SPCCs should be greater than or equal to 0.05.

   e. There are thirteen calibration check compounds (CCCs): acenaphthene, 1,4-dichlorobenzene, hexachlorobutadiene, n-nitrosodiphenylamine, di-n-octylphthalate, fluoranthene, benzo(a)pyrene, 4-chloro-3-methylphenol, 2,4-dichlorophenol, 2-nitrophenol, phenol, pentachlorophenol, and 2,4,6-trichlorophenol. The %RSDs for these compounds must be less than or equal to 30%.

2. Continuing Calibration

   a. The working calibration curve must be verified every 12 hours by the analysis of the 50 ug/mL standard, containing each compound of interest. (Note: The acid surrogate compounds and the compounds benzidine and 3,3-dichlorobenzidine are at 100 ug/mL in this standard.)

b. The percent difference (%D) of the response factors for all compounds must be less than or equal to 25%.

c. The RFs for all compounds must be greater than or equal to 0.05.

d. The RFs for the SPCCs (mentioned above) must be greater or equal to 0.05.

e. The %Ds for the CCCs (mentioned above) must be less than or equal to 25%.

f. Isomeric pairs must be properly separated and properly identified based on elution order. These pairs are:

2,4,6- / 2,4,5-trichlorophenol
anthracene/phenanthrene
chyrsene/benzo(a)anthracene
benzo(b)/benzo(k)fluoranthene

4.3 Evaluation Procedure

1. Initial Calibration

a. Check the raw data and recalculate 10-20% of the RFs and %RSDs.

b. Verify that the %RSDs are less than or equal to 30%.

c. Verify that the RFs for all compounds are greater than or equal to 0.05.

d. Verify that the SPCCs and CCCs meet criteria.

e. If errors are detected, pursue the explanation via more comprehensive recalculation.

2. Continuing Calibration

a. Check the raw data and recalculate 10-20% of the RFs and %Ds.

b. Verify that the %Ds are less than or equal to 25%.

c. Verify that the RFs are greater than or equal to 0.05.

d. Verify that the SPCCs and CCCs meet criteria.

e. Verify that the isomeric pairs are identified by the correct elution order.

f. If errors are detected, pursue the explanation via more comprehensive recalculation.

3. Calculations

$$RF = \frac{A_x \times C_{is}}{A_{is} \times C_x}$$

where:

A_x = Response for the characteristic ion for the analyte to be measured, units area counts.
C_{is} = Concentration of the internal standard, ug/mL.
A_{is} = Response for the characteristic ion for the internal standard, units area counts.
C_x = Concentration of the analyte to be measured, ug/mL.

$$\%RSD = \frac{SD}{x} \times 100$$

$$SD = \sqrt{\sum_{i=1}^{n} \frac{(X_i - \bar{x})^2}{(n-1)}}$$

where:

x = Mean of 5 response factors.

$$\%D = \frac{R_1 - R_2}{R_1} \times 100$$

R_1 = Average response factor from initial calibration.
R_2 = Response factor from continuing calibration.

4.4 <u>Action</u>

1. Initial Calibration

a. Estimate (J) positive results for those compounds whose %RSDs exceed 30% in the associated initial calibration.

b. Estimate (J) positive results and (UJ) non-detected results for those compounds whose %RSDs exceed 50% in the associated initial calibration.

c. Estimate (J) positive results and reject (R) non-detected results for those compounds whose %RSDs exceed 100% in the associated initial calibration.

d. Estimate (J) positive results for those compounds whose RFs are less than 0.05.

e. Reject (R) non-detected results for those compounds whose RFs are less than 0.05.

f. If the SPCCs and CCCs do not meet criteria, provide a comment in the data validation report. Qualify the associated results for these compounds according to a. through e.

2. Continuing Calibration

a. Estimate (J) positive results for those compounds whose %Ds exceed 25% in the associated continuing calibration.

b. Estimate (J) positive results and (UJ) non-detected results for those compounds whose %Ds exceed 50% in the associated continuing calibration.

c. Estimate (J) positive results and reject (R) non-detected results for those compounds whose %Ds exceed 100% in the associated continuing calibration.

d. Estimate (J) positive results for those compounds whose RFs are less than 0.05.

e. Reject (R) non-detected results for those compounds whose RFs are less than 0.05.

f. If the SPCCs and CCCs do not meet criteria, provide a comment in the data validation report. Qualify the associated results for these compounds according to a. through e.

g. If the isomeric pairs were not properly identified, professional judgment must be applied to assess the impact on the data.

5.0 BLANKS

5.1 Objective

The assessment of method blank analysis results is to determine the existence and magnitude of contamination problems. If problems with any method blank exist, all data associated with the method blank must be carefully evaluated to determine whether or not there is an inherent variability in the data for the batch, or if the problem is an isolated occurrence not affecting other data. ("Associated" refers to those samples extracted with a particular method blank.)

5.2 Criteria

A laboratory reagent water blank (method blank) must be extracted with each set of samples (a set may consist of up to 20 samples). A solvent blank should be analyzed following any concentrated sample that has saturated ions from a compound. No contaminants should be present in the blank(s) at or above the practical quantitation limit (PQL). The method blank should be carried through all stages of the sample preparation and measurement steps.

5.3 Evaluation Procedure

1. Review the results of all associated blank(s) and raw data (chromatograms, mass spectra, quantitation reports, or data system printouts).

2. Verify that method blank analysis has been reported per matrix, per concentration level, and per extraction batch.

5.4 Action

1. If a target compound is found in the method blank but not in the samples, no action is taken.

2. If a target compound is found in the method blank and is also found in the sample, apply the following :

   a. If the sample concentration is less than 5 times the blank concentration (10 times for common phthalate esters) and less than the PQL: Report the PQL followed by (UJ).

   b. If the sample concentration is less than or equal to 5 times the blank concentration (10 times for common phthalate esters) and greater than or equal to the PQL: Report the value followed by (UJ).

   c. If the sample concentration is greater than 5 times the blank concentration (10 times for common contaminants) and greater than the PQL: Report the value unqualified.

3. If an associated method blank exhibits gross contamination, reject (R) results for all affected compounds.

4. If an associated method blank was not extracted or analyzed for the samples, estimate (J) positive results.

5. If an associated method blank was not extracted or analyzed for the samples, and there are positive results for the same target compound in all samples, reject (R) the positive results for that target compound in all samples.

6.0 SURROGATES

6.1 Objective

Laboratory performance on individual samples is established by means of spiking activities. The evaluation of the results of these surrogate spikes is not necessarily straightforward. The sample itself may produce effects due to such factors as interferences and high concentrations of analytes. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the review and validation of data based on specific sample results is frequently subjective and demands analytical experience and professional judgment. Accordingly, this section consists primarily of guidelines.

6.2 Criteria

1. Surrogates must be added to all samples and blanks just prior to extraction. (Surrogate concentrations will differ depending on matrix or sample type, clean-up, and concentration steps.)

2. The surrogate compounds used are phenol-d6, 2-fluorophenol, 2,4,6-tribromophenol, nitrobenzene-d5, 2-fluorobiphenyl, and p-terphenyl-d14. Surrogate recoveries must fall within the following control limits:

Surrogate Compound	Low/High Water	Low/High Soil/Sediment
Base/neutrals		
nitrobenzene-d5	35-114	23-120
2-fluorobiphenyl	43-116	30-115
p-terphenyl-d14	33-141	18-137
Acids		
phenol-d6	10-94	24-113
2-fluorophenol	21-100	25-121
2,4,6-tribromophenol	10-123	19-122

3. If surrogate recoveries are outside the control limits and the instrument is at fault, the instrument problem must be resolved and the samples reanalyzed.

4. If surrogate recoveries are outside the control limits and the instrument is not at fault, the samples must be re-extracted and reanalyzed.

5. If more than one surrogate recovery falls outside the control limits in the method blank, the samples and blanks should be and re-extracted and reanalyzed.

6.3 Evaluation Procedure

1. Verify the calculations and recoveries using the raw data (i.e., chromatograms, quantitation reports, etc.).

2. Verify the transcription of the recoveries from the raw data.

3. If recoveries are not within the control limits, check the raw data for possible interferences.

4. Check to verify that appropriate reanalyses or re-extractions were performed.

5. If multiple analyses of the same sample are submitted, determine which analysis should be reported by assessing which has better overall QC. The QC categories used to make this assessment are: holding times, internal standards, surrogate recoveries, and presence and absence of positive results.

6.4 Action

1. If the recoveries of two surrogates in the acid fraction or two surrogates in the base/neutral are greater than the control limits, estimate (J) positive results for that fraction of the sample. (Determine that high bias was not due to interference with the surrogate compound only.)

2. If the recoveries of two surrogates in the acid fraction or two surrogates in the base/neutral fraction are less than the laboratory control limits but greater than 10%, estimate (J) positive results and (UJ) non-detected results for that fraction of the sample.

3. If the recovery of any one surrogate in the acid fraction or base/neutral fraction is less than 10%, estimate (J) positive results and reject (R) non-detected results for that fraction.

4. If appropriate reanalyses or re-extractions were not performed, provide a comment in the validation report.

5. If the sample is analyzed at a high dilution and the surrogates are diluted out professional judgment is required to determine qualification of the data.

7.0 MATRIX SPIKE ANALYSIS

7.1 Objective

These data are generated to determine long-term precision and accuracy of the analytical method on various matrices. These data alone cannot be used to evaluate the precision and accuracy of individual samples.

7.2 Criteria

The laboratory is required to collect a portion of their samples to monitor spike recoveries. The frequency of spiked sample analyses must be one matrix spike (MS) and one matrix spike duplicate (MSD) per extraction batch (up to 20 samples). Matrix spiking compounds are added to the sample prior to extraction. The matrix spiking solution compounds and their acceptance criteria are as follows :

	Percent Recovery		RPD	
Compound	Water	Soil/Sediment	Water	Soil/Sediment
1,2,4-trichlorobenzene	39-98	38-107	28	23
acenaphthene	46-118	31-137	31	19
2,4-dinitrotoluene	24-96	28-89	38	47
pyrene	26-127	35-142	31	36
n-nitroso-di-n-propylamine	41-116	41-126	38	38
1,4-dichlorobenzene	36-97	28-104	28	27
pentachlorophenol	9-103	17-109	50	47
phenol	12-89	26-90	42	35
2-chlorophenol	27-123	25-102	40	50
4-chloro-3-methylphenol	23-97	26-103	42	33
4-nitrophenol	10-80	11-114	50	50

7.3 Evaluation Procedure

1. Verify the calculations, recoveries, and RPDs using the raw data (i.e., chromatograms, quantitation reports, etc.).

2. Verify the transcription of the recoveries and RPDs from the raw data.

3. Verify that the MS/MSD recoveries and RPDs, as applicable, fall within the control limits set by the laboratory.

$$RPD = \frac{|D_1 - D_2|}{(D_1 + D_2)/2} \times 100$$

D_1 = MS Concentration.
D_2 = MSD Concentration.

7.4 Action

No action is taken on MS/MSD data alone to qualify an entire batch. However, using informed professional judgment the data reviewer may use the MS/MSD results in conjunction with other QC criteria and determine the need for some qualification of data.

8.0 INTERNAL STANDARDS

8.1 Objective

Internal standards (IS) performance criteria ensure that GC/MS sensitivity and response are stable during every run.

8.2 Criteria

1. Internal standards must be added to achieve a concentration of 40 ug/mL in all samples, standards, and blanks just prior to analysis.

2. The IS compounds are 1,4-dichlorobenzene-d4, naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12, and perylene-d12.

3. The IS area counts in the samples must not vary by more than a factor of two (-50% to +100%) from the associated calibration standard.

4. The IS retention times in the samples must not vary by more than +/- 30 seconds from the associated calibration standard.

8.3 Evaluation Procedure

1. Check raw data (i.e., chromatograms, quantitation reports, etc.) to verify the area counts and retention times.

2. Verify that all IS area counts and retention times are acceptable.

8.4 Action

1. If the area counts for any IS are outside the control limits (-50% to +100%), estimate (J) positive results and (UJ) non-detected results for compounds quantitated using that IS.

2. If the area counts for any IS are extremely low (i.e. less than 50% of the lower control limit), or if performance exhibits a major abrupt drop off, reject (R) non-detected results and estimate (J) positive results for compounds quantitated using that IS.

3. If any IS retention time varies by more than 30 seconds, examine the chromatographic profile to determine if any false positives or false negatives exist. If identification problems are evident, reject (R) affected results.

4. If any IS retention time varies by a large magnitude, reject (R) all results.

9.0 ADDITIONAL QUALITY CONTROL

9.1 Objective

Laboratory control samples are required to establish acceptable bias and precision prior to any sample analyses. (Data are not likely to be included as a deliverable.)

9.2 Criteria

It is not clear if the laboratory control sample data will be included in the data package. If provided, the laboratory control sample recoveries should be comparable to the percent recoveries and RPDs listed in section 7.2 of these guidelines. The maximum number of values allowed to fall outside the control limits are four recoveries and two RPDs.

9.3 Evaluation Procedure

Evaluate the accuracy of the data. Verify that the percent recoveries and RPDs are within the control limits and that no more than four recoveries and two RPDs are outside these limits.

9.4 Action

Professional judgment should be used to evaluate the impact of outliers on the data.

10.0 COMPOUND IDENTIFICATION

10.1 Objective

The objective of the criteria for GC/MS qualitative analysis is to minimize the number of erroneous identifications of compounds.

10.2 Criteria

1. The mass spectra for standard reference should be obtained on the same instrument used for sample analysis.

2. The sample compound relative retention time (RRT) must compare within plus or minus 0.06 RRT units of the standard component. The reference standard must be run within the same 12 hours as the sample.

3. All ions present in the standard mass spectra (ions of interest) at a relative intensity greater than 10% must be present in the sample spectrum. In addition, all ions meeting the above criteria must agree within plus or minus 20% between the sample and standard spectra.

10.3 Evaluation Procedure

1. Verify the transcription of all results from the quantitation report to the Form I or equivalent results summary.

2. Verify that RRT of reported compounds is within 0.06 RRT units of the reference standard.

3. Verify that the ions of interest present in the standard spectra are also present in the sample compound spectra at the appropriate intensities.

4. Review the spectra for ions (>10%) present in the sample spectrum but not in the reference spectrum. This may indicate possible background contamination or presence of collating compounds.

5. Review the spectra for ions present in the reference spectrum but not in the sample spectrum. This may indicate possible subtraction from the sample spectrum because of background contamination or coeluting compounds.

6. The reviewer should be aware of situations (e.g. high concentration samples preceding low concentration samples) when sample carry-over is a possibility and should use judgment to determine if carry-over has occurred.

10.4 Action

1. The application of qualitative criteria for GC/MS analysis of target compound list (CT) compounds requires professional judgment. If it is determined that an incorrect false positive exists, the result should be reported as non-detected (U). If it is determined that a false negative exists, reject (R) the non-detected result.

2. Professional judgment should be used to qualify data in which carry-over is evident.

11.0 COMPOUND QUANTITATION

11.1 Objective

The objective is to ensure that the reported quantitation results and PSALMS are accurate.

11.2 Criteria

1. Compound quantitation, as well as the adjustment of the PSALMS, must be calculated according to the method. Compound quantitation shall be performed using the internal standard nearest in retention time.

2. If the response for the peak exceeds the calibration range of the system, the sample must be diluted and reanalyzed. All dilutions should keep the response of the major constituents in the upper half of the calibration range of the system.

11.3 Evaluation Procedure

1. Examine the raw data to verify the correct calculation of all sample results. Quantitation reports, chromatograms, sample preparation logs, and dilutions are compared to the reported sample results and quantitation limits.

2. Verify that if the response for any target compound exceeded the calibration range of the system, the sample was diluted appropriately and reanalyzed.

3. Calculations:

Water and water-miscible waste:

$$\text{Concentration (UGLY)} = \frac{A_x \times I_s \times V_t}{A_{is} \times RF \times V_o \times V_i}$$

where:

A_x = Response of the characteristic ion for the analyte in the sample, area counts.

I_s = Amount of internal standard injected, ng.

V_t = Final Volume of total extract, uL (if half the base/neutral extract and half the acid extract are combined, 2000 uL is used for the final volume; if the base/neutral and acid fractions were combined, 1000uL is used for the final volume.)

V_i = Volume of extract injected, uL

A_{is} = Response of the characteristic ion for the internal standard, area counts.
RF = Response factor for the analyte from the appropriate calibration standard (see page 4-3).
V_o = Volume of water extracted, mL.

Sediment/soil, sludge, and waste:

$$\text{Concentration (ug/Kg)} = \frac{A_x \times I_s \times V_t}{A_{is} \times RF \times V_i \times W_s \times D}$$

where:

A_x, I_s, A_{is}, RF = Same as in water and water-miscible waste above.
V_t = Final volume of extract, uL.
V_i = Volume of extract injected, uL.
W_s = Weight of sample extracted, g.
D_s = percent solids

For tentatively identified compound (TIC) calculations:

A_x = The total area or peak height for the analyte of interest.
A_{is} = Total area or peak height for the nearest internal standard free of interference.
RF = One.

11.4 Action

1. If major discrepancies are found which prevent determination of the correct result, resubmittal will be necessary to resolve the problem.

2. If the response for any target compound exceeds the linear range of the system and the sample was not diluted and reanalyzed, the value should be qualified with an (E).

3. If the diluted sample analysis fails to keep the response of the major constituents in the upper half of the calibration range, use professional judgment to qualify the data. At a minimum, provide a comment in the validation report.

12.0 SYSTEM PERFORMANCE

12.1 Objective

A thorough review of on-going data acquisition and examination of the raw data can yield indicators of instrument performance.

12.2 Criteria

Instrument performance indicators are as follows:

1. Abrupt, discrete baseline changes possibly indicating gain or threshold changes.

2. Poor chromatographic performance characterized by high background levels, shifts in absolute retention times of internal standards, large extraneous peak interferences, excessive baseline rise at elevated temperatures, loss of peak resolution, and peak tailing or peak splitting which could result in inaccurate quantitation.

12.3 Action

Based on the performance indicators discussed previously, the data reviewer must decide if the system has degraded to the point of affecting data quality or validity. If data quality may have been affected, data should be qualified based on the reviewer's technical judgment and subjective evaluation.

13.0 TENTATIVELY IDENTIFIED COMPOUNDS

13.1 Objective

Chromatographic peaks which are not TCL compounds, surrogates, or internal standards are potential tentatively identified compounds (TICs). TICs may be qualitatively identified by GC/MS library search and the identifications assessed by the data reviewer.

13.2 Criteria

For samples containing components not associated with the calibration standards, a library search is performed for the purpose of tentative identification of the 20 largest unidentified peaks. Guidelines for making tentative identification are:

1. Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum. In addition, all ions meeting the above criteria should agree within plus or minus 20%.

2. Molecular ions present in the reference spectrum should be present in the sample spectrum.

13.3 Evaluation Procedure

1. Check the raw data to verify that a library search has been generated for all required peaks in the chromatograms.

2. Examine blank chromatograms to verify that TICs present in the samples are not present in the associated blanks.

3. Examine the mass spectra to verify that the best match was chosen based upon the guidelines for identification listed above.

4. Review the spectra for ions present in the sample spectrum but not in the reference spectrum. This may indicate possible background contamination or presence of coeluting compounds.

5. Review the spectra for ions present in the reference spectrum but not in the sample spectrum. This may indicate possible subtraction from the sample spectrum because of background contamination or coeluting compounds.

4. Verify that none of the TICs reported are actually TCL compounds.

13.4 Action

1. Estimate (J) all TIC results. (The reported results should already be estimated by the laboratory.)

2. If all contractually required peaks were not library searched, request resubmittal data.

3. If a TIC result in the sample is not sufficiently above the level in the blank, the TIC result should not be reported. (Generally, the 5X blank rule is applied here.)

4. If it is determined that the TIC identification is not acceptable, the TIC should be changed to an unknown or an appropriate class of compound.

5. In general, TIC identification is dependant on professional judgment which incorporates factors such as boiling point. In addition, if a TIC spectral match is poor but other samples have the same TIC present with good spectral match, similar retention time, and the same ions, the identification may be inferred from the other sample TIC results.

14.0 OVERALL DATA ASSESSMENT

The overall data assessment represents the quality of the majority of the data. It can only be assigned once the data validation process is complete. At this point, each data point for each analytical result has been assigned a data qualifier based upon deficiencies in one or more of the QC categories (i.e. holding times, calibration, etc.). All results have been qualified. Determine which qualifier has been assigned to the majority of the analytical results and assign that qualifier as the overall data assessment qualifier.

APPENDIX A
ACRONYM LIST

ACRONYM LIST

DFTPP	Decafluorotriphenylphosphine
%D	Percent Difference
IS	Internal Standard
MS	Matrix Spike
MSD	Matrix Spike Duplicate
PQL	Practical Quantitation Limit
RF	Response Factor
RPD	Relative Percent Difference
RRT	Relative Retention Time
%RSD	Percent Relative Standard Deviation
SD	Standard Deviation
TCL	Target Compound List
TIC	Tentatively Identified Compound

APPENDIX B
DATA QUALIFIER DEFINITIONS

DATA QUALIFIER DEFINITIONS

A	Data are acceptable, with qualifications.
E	The associated value exceeds calibration range. Dilute and reanalyze.
J	The associated value is an estimated quantity.
R	Data are rejected.
U	The compound was analyzed for but not detected at or above the method detection limit.
UJ	The compound was analyzed for but not detected at or above the method detection limit. The associated value is an estimated quantity.
V	Data are valid.

TEXAS WORLD OPERATIONS, INC.
ORGANIC DATA VALIDATION GUIDELINES

EPA SW-846 METHOD 8080
Organochlorine Pesticides and PCBs Analysis
(SEPTEMBER, 1986)

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October 1992

TEXAS WORLD OPERATIONS, INC.
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APPENDICES

Appendix A Acronym List

Appendix B Data Qualifier Definitions

1.0 INTRODUCTION

This document is designed to offer guidance in laboratory data evaluation and validation of organochlorine pesticides and PCBs in water, soil, and solid waste sample analyses. In some areas, specific guidelines and criteria are available. In other, less objective areas, only general guidance is offered due to the complexities and uniqueness of data relative to specific samples. The reviewer has a responsibility to inform the user concerning data quality and limitations, based on his/her technical judgment and evaluation, to assist the user in the proper use of the data.

Those areas where specific criteria are included are primarily areas in which definitive method performance requirements are established. These requirements are concerned with specifications that are not strictly sample dependent. They specify performance requirements normally under a laboratory's control. These specific areas include calibration standards, blanks, matrix spikes, and surrogate recoveries. Calculation and transcription errors must be rectified by laboratory resubmission of corrected data sheets if verification of the reported result is in doubt.

2.0 HOLDING TIMES

2.1 Objective

The objective is to ascertain the validity of results based on the holding time of the sample from the time of collection to the time of extraction and analysis.

2.2 Criteria

All samples and extracts must be iced or refrigerated at 4 ^{o}C. Sodium thiosulfate should be added to water samples containing residual chlorine. (Although not used for qualification, these criteria are noteworthy.)

Water:
All samples must be extracted within seven days of collection and completely analyzed within 40 days of extraction.

Soil:
All samples must be extracted within fourteen days of collection and completely analyzed within 40 days of extraction.

2.3 Evaluation Procedure

Actual holding times are established by comparing the collection date on the Chain-of-Custody form with the dates of extraction and analysis found in the laboratory raw data. Holding times are determined as follows:

Extraction Holding Time (Days) = Extraction Date - Collection Date
Analysis Holding Time (Days) = Analysis Date - Extraction Date

2.4 Action

1. If the seven-day water extraction holding time, the fourteen-day soil extraction holding time, or the 40-day analysis holding time (after extraction) is exceeded, estimate (J) positive results and (UJ) non-detected results.

2. If the seven-day water extraction holding time, the fourteen-day soil extraction holding time, or the 40-day analysis holding time (after extraction) is grossly exceeded, estimate (J) positive results and reject (R) non-detected results. Grossly exceeded is defined as twice the acceptable holding time.

3.0 CALIBRATION

3.1 Objective

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents satisfactory performance of the instrument over specific time periods during sample analysis.

This may be done by calibration factors (CFs) or by the use of the calibration curve. If the curve is used, professional judgment is employed to assess validity.

3.2 Criteria

1. Initial Calibration

   a. External calibration standards for all analytes except the multi-response analytes (PCBs, toxaphene, and chlordane) must be prepared and analyzed at a minimum of five concentrations. One of the standards should be near but above the method detection limit which the laboratory has established. The other concentrations should define the range of the detector used.

      An external standard must be prepared at one concentration for the multi-response analytes. The standard should be near but above the method detection limit which the laboratory has established.

   b. The percent relative standard deviations (%RSDs) of the calibration factors (CFs) must be less than or equal to 20%, if the average calibration factor is to be used in place of the calibration curve for calculations.

   c. Both a and b apply to each GC column and instrument used for quantitation.

2. Continuing Calibration

   a. The working calibration curve or calibration factor must be verified on each working day, every ten samples in the analysis sequence, and at the end of the analysis sequence by measuring a mid-level standard. The response for any analyte must be within ±15% of the initial calibration response on the quantitation column or else the test must be repeated using a fresh calibration standard, or a new calibration curve or calibration factor must be generated. In addition, the retention time of any analyte should fall within the retention time window of that analyte from the initial calibration.

In addition, the response for any analyte must be within ±20% of the initial calibration response on the confirmation column.

3.3 Evaluation Procedure

1. Initial Calibration

   a. Check the raw data and recalculate 10-20% of the CFs and %RSDs.

   b. Verify that the %RSDs are less than or equal to 20% if the average calibration factor is used for quantitation.

   c. If errors are detected, pursue the explanation via more comprehensive recalculation.

2. Continuing Calibration

   a. Check the raw data and recalculate 10-20% of the CFs and %Ds.

   b. Verify that the %Ds are less than or equal to 15% or 20% for the quantitation and confirmation columns, respectively.

   c. If errors are detected, pursue the explanation via more comprehensive recalculation.

$$CF = \frac{\text{total area of peak}}{\text{nanograms injected}}$$

$$\%RSD = \frac{SD}{\bar{x}} \times 100$$

$$SD = \sqrt{\sum_{i=1}^{n} \frac{(X_i - \bar{x})^2}{(n-1)}}$$

where:

$\bar{x}$ = Mean of 5 calibration factors.

$$\%D = \frac{R_1 - R_2}{R_1} \times 100$$

R_1 = Calibration factor from first analysis.
R_2 = Calibration factor from subsequent analysis.

3.4 Action

1. Initial Calibration

   a. Estimate (J) positive results for those compounds whose %RSDs exceed 20% in the associated initial calibration.

   b. Estimate (J) positive results and (UJ) non-detected results for those compounds whose %RSDs exceed 50% in the associated initial calibration.

   c. Estimate (J) positive results and reject (R) non-detected results for those compounds whose %RSDs exceed 100% in the associated initial calibration.

2. Continuing Calibration

   a. Estimate (J) positive results for those compounds whose %Ds on the quantitation column exceed 15% in the associated continuing calibration.

   b. Estimate (J) positive results and (UJ) non-detected results for those compounds whose %Ds on the quantitation column exceed 25% in the associated continuing calibration.

   c. Estimate (J) positive results and reject (R) non-detected results for those compounds whose %Ds on the quantitation column exceed 50% in the associated continuing calibration.

   d. Provide a comment in the data validation report for those positive result whose %Ds on the confirmation column exceed 20% in the associated continuing calibration.

4.0 BLANKS

4.1 Objective

The assessment of method blank analysis results is to determine the existence and magnitude of contamination problems. If problems with any method blank exist, all data associated with the method blank must be carefully evaluated to determine whether or not there is an inherent variability in the data for the batch, or if the problem is an isolated occurrence not affecting other data. ("Associated" refers to those samples extracted and analyzed with a particular method blank.)

4.2 Criteria

A laboratory reagent blank (method blank) must be extracted and analyzed with each set of samples extracted (one every twenty samples) and when there is a change in reagents. No contaminants should be present in the blank(s).

4.3 Evaluation Procedure

1. Review the results of all associated blank(s) and raw data (chromatograms, mass spectra, quantitation reports, or data system printouts).

2. Verify that method blank analysis has been reported per matrix, per concentration, per GC system used to analyze samples, and per extraction batch.

4.4 Action

1. If a target compound is found in the method blank but not in the samples, no action is taken.

2. If a target compound is found in the method blank and is also found in the sample, apply the following:

   a. If the sample concentration is less than 5 times the blank concentration and less than the MDL: Report the MDL followed by (UJ).

   b. If the sample concentration is less than or equal to 5 times the blank concentration and greater than or equal to the MDL: Report the value followed by (UJ).

   c. If the sample concentration is greater than 5 times the blank concentration and greater than the MDL: Report the value unqualified.

3. If an associated method blank was not extracted and analyzed for the samples, estimate (J) positive results.

4. If an associated method blank was not extracted and analyzed for the samples, and there are positive results for the same target compound in all samples, reject (R) the positive results for that target compound in all samples.

5.0 SURROGATES

5.1 Objective

Laboratory performance on individual samples is established by means of spiking activities. The evaluation of the results of these surrogate spikes is not necessarily straightforward. The sample itself may produce effects due to such factors as interferences and high concentrations of analytes. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the review and validation of data based on specific sample results is frequently subjective and demands analytical experience and professional judgment. Accordingly, this section consists primarily of guidelines.

5.2 Criteria

1. Surrogate recoveries must fall within the control limits set by the laboratory. Dibutylchlorendate (DBC) is the primary surrogate and should be used whenever possible. However, DBC is subject to interference as well as acid and base degradation. An alternate surrogate, 2,4,5,6-tetrachloro-meta-xylene, may be used instead of DBC for evaluation.

2. Surrogates must be added to the samples prior to the extraction process.

3. Samples in which surrogate recoveries are outside the control limits must be reanalyzed.

4. Samples reanalyzed due to failed surrogate recoveries which fail again must be reextracted and reanalyzed or the data must be "flagged as estimated concentration".

5.3 Evaluation Procedure

1. Check raw data (i.e., chromatograms, quantitation list, etc.) to verify the recoveries.

2. If recoveries are not within the control limits, check the raw data for possible interferences.

3. Check to verify that appropriate reanalyses or reextractions were performed.

5.4 Action

1. If the surrogate recovery is greater than the laboratory control limits, estimate (J) positive results. (Determine that high bias was not due to interference with the surrogate compound only.)

2. If the surrogate recovery is less than the laboratory control limits but greater than 10%, estimate (J) positive results and (UJ) non-detected results.

3. If the surrogate recovery is greater than zero but less than 10%, estimate (J) positive results and reject (R) non-detected results.

4. If zero pesticide surrogate recovery is reported, the reviewer should examine the sample chromatogram to determine if the surrogate may be present, but slightly outside its retention time window. If this is the case, in addition to assessing surrogate recovery for quantitative bias, the overriding consideration is to investigate the qualitative validity of the extraction and analysis. If the surrogate is not present, estimate (J) positive results and reject (R) non-detected results.

5. If appropriate reanalyses or reextractions were not performed, provide a comment in the validation report.

6.0 MATRIX SPIKE ANALYSIS

6.1 Objective

These data are generated to determine long-term precision and accuracy of the analytical method on various matrices. These data alone cannot be used to evaluate the precision and accuracy of individual samples.

6.2 Criteria

The laboratory is required to collect a portion of their samples to monitor spike recoveries. The frequency of spiked sample analyses must be one matrix spike (MS) and one matrix spike duplicate (MSD) per extraction batch (up to 20 samples). Samples are spiked with the following compounds: lindane, heptachlor, and aldrin at 0.2 ug/L; and dieldrin, endrin, and 4,4'-DDT at 0.5 ug/L. The acceptance criteria are :

	Percent Recovery			
Compound	Water	RPD	Soil/Sediment	RPD
lindane	56-123	15	46-127	50
heptachlor	40-131	20	35-130	31
aldrin	40-120	22	34-132	43
dieldrin	52-126	18	31-134	38
endrin	56-121	21	42-139	45
4,4'-DDT	38-127	27	23-134	50

6.3 Evaluation Procedure

1. Inspect results for the MS/MSD percent recoveries and relative percent differences (RPDs).

2. Verify transcriptions from raw data and verify calculations.

3. Verify that the MS/MSD recoveries and RPD fall within the control limits set by the laboratory.

$$RPD = \frac{D_1 - D_2}{(D_1 + D_2)/2} \times 100$$

D_1 = MS Concentration.
D_2 = MSD Concentration.

6.4 Action

No action is taken on MS/MSD data alone to qualify an entire batch. However, using informed professional judgment the data reviewer may use the MS/MSD results in conjunction with other QC criteria and determine the need for some qualification of data.

7.0 ADDITIONAL QUALITY CONTROL

7.1 Objective

Laboratory control samples are required to establish acceptable accuracy and precision prior to any sample analyses and if any compound is outside its recovery range in the matrix spike analyses. (Data are not likely to be included as a deliverable.)

7.2 Criteria

It is not likely that the laboratory control sample data will be included in the data package. If provided, the laboratory control sample recovery should be comparable to the data from Tables 3 and 4 within the method.

7.3 Evaluation Procedure

Evaluate the accuracy of the data.

7.4 Action

Discuss any areas of concern in the comments portion of the report.

8.0 COMPOUND IDENTIFICATION

8.1 Objective

The objective of the criteria for GC qualitative analysis is to minimize the number of erroneous identifications of compounds.

8.2 Criteria

1. The laboratory must establish retention time windows for each analyte on each GC column used for sample analysis. This is done by making three injections of all compounds throughout a 72 hour period, and calculating three times the standard deviation for each analyte (using only one major peak for all multiple compounds). Daily retention time windows are then established using the initial mid-level standard of the sequence as the midpoint of the window. All succeeding standards in an analysis sequence must fall within this daily retention time window. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

2. Tentative identification of an analyte occurs when a peak falls within the daily retention time window. Normally, second column confirmation is required. GC/MS confirmation may also be done for any compound at sufficient concentration that has been confirmed on both columns.

3. If peak detection and identification are prevented due to interferences, further cleanup is required. Interferences are a major problem in pesticide determinations.

8.3 Evaluation Procedure

1. Review Form I or the equivalent results summary, the associated raw data (chromatograms and data system printouts), and Form X or the equivalent summary. Confirm the reported positive results and verify the reported non-detected results using appropriate retention times and retention time windows.

2. Verify that positive identifications have dissimilar column analysis or GC/MS analysis.

3. Verify that cleanup techniques were employed for samples with interference.

8.4 Action

1. If second column confirmation data are not provided and a positive result is evident in the primary data, reject (R) the positive result.

2. If the criteria for positive identification (i.e. peak within its window on both columns or any evident shifts explained) are not met, and it is clear that an error in identification has occurred, change the result to non-detected (U).

3. If the criteria for identification are not met, and the misidentified peak(s) cause interference with potential detection of the target compound, then the value should be reported as an estimated quantitation limit (UJ).

4. If identification of any target compound is obscured by interference, professional judgment should be applied to estimate (UJ) the quantitation limits. (If no cleanup techniques were employed, provide a comment in the validation report.)

9.0 COMPOUND QUANTITATION

9.1 Objective

The objective is to ensure that the reported quantitation results and detection limits are accurate. Quantitations of the multi-response compounds (PCBs, toxaphene, and chlordane) are often difficult. Suggestions for calculations may be found in the method.

9.2 Criteria

1. Compound quantitation, as well as the adjustment of the method detection limit (MDL), must be calculated according to the method.

2. If the response for the peak exceeds the linear range of the system, the extract must be diluted and reanalyzed.

9.3 Evaluation Procedure

1. Examine the raw data to verify the correct calculation of all sample results. Quantitation lists, chromatograms, sample preparation logs, dilutions, and cleanups are compared to the reported sample results and quantitation limits.

2. Verify that responses for target compounds and standard peaks were measured consistently, i.e., all values were determined by either integrated areas or peak heights, not both.

3. Verify that if the response for any target compound exceeded the linear range of the system, the sample was diluted and reanalyzed.

   If the external standard calibration procedure was used, the concentration in the sample can be calculated as follows:

$$\text{Concentration (ug/L or ug/Kg)} = \frac{A_x \times A \times V_t \times D}{A_s \times V_i \times (V_s \text{ or } W)}$$

where:

A_x = Response for the analyte in the sample, using peak area or height.
A = Amount of standard injected, ng.
V_t = Volume of total extract, uL.
D = Dilution factor.
A_s = Response for external standard, using same as A_x.
V_i = Volume of extract injected, uL.
V_s = Volume of water extracted, mL.
W = Weight of soil extracted, g.

9.4 Action

1. If major discrepancies are found which prevent determination of the correct result, resubmittal will be necessary to resolve the problem.

2. If the response for any target compound exceeds the linear range of the system and the sample was not diluted and reanalyzed, the value should be qualified with an (E).

10.0 SYSTEM PERFORMANCE

10.1 Objective

A thorough review of on-going data acquisition and examination of the raw data can yield indicators of instrument performance.

10.2 Criteria

Instrument performance indicators are as follows:

1. 4,4'-DDT and endrin can degrade easily in the injection port. Degradation problems will be checked at the beginning of every sequence by the injection of a standard containing 4,4'-DDT and endrin. If degradation of either 4,4'-DDT or endrin exceeds 20%, corrective action must be taken before proceeding.

2. To ensure adequate separation of compounds, the retention time of 4,4'-DDT should be greater than 12 minutes.

3. Abrupt, discrete baseline changes indicating electronic or detector changes.

4. Poor chromatographic performance characterized by high background levels, shifts in absolute retention times of standards, large extraneous peak interferences, baseline rise at elevated temperatures, loss of peak resolution, and peak tailing or peak splitting which could result in inaccurate quantitation.

10.3 Evaluation Procedure

1. Using area counts, calculate the degradation (% breakdown) as follows:

$$\text{\% breakdown for 4,4'-DDT} = \frac{\text{Peak area (DDE + DDD)}}{\text{Peak area (DDT + DDE + DDD)}} \times 100$$

$$\text{\% breakdown for endrin} = \frac{\text{Peak area (endrin aldehyde + endrin ketone)}}{\text{Peak area (endrin + endrin aldehyde + endrin ketone)}} \times 100$$

2. Check the raw data to verify that the 4,4'-DDT retention time is greater than 12 minutes and that there is adequate separation of the compounds.

10.4 Action

If 4,4'-DDT or endrin breakdown is greater than 20%, beginning with the samples following the last in-control standard:

1. Estimate (J) positive results for 4,4'-DDT, 4,4'-DDE, and 4,4'-DDD.

2. Reject (R) non-detected results for 4,4'-DDT if there are positive results for 4,4'-DDE and 4,4'-DDD.

3. Estimate (J) positive results for endrin, endrin aldehyde, and endrin ketone.

4. Reject (R) non-detected results for endrin if there are positive results for endrin aldehyde and endrin ketone.

If 4,4'-DDT has a retention time exceeding 12 minutes:

1. Closely examine the standard chromatograms to determine if adequate separation of individual compounds was achieved. If not, reject (R) the results for all affected compounds.

11.0 OVERALL DATA ASSESSMENT

The overall data assessment represents the quality of the majority of the data. It can only be assigned once the data validation process is complete. At this point, each data point for each analytical result has been assigned a data qualifier based upon deficiencies in one or more of the QC categories (i.e. holding times, calibration, etc.). All results have been qualified. Determine which qualifier has been assigned to the majority of the analytical results and assign that qualifier as the overall data assessment qualifier.

APPENDIX A
ACRONYM LIST

ACRONYM LIST

CF	Calibration Factor
%D	Percent Difference
MDL	Method Detection Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
RF	Response Factor
RPD	Relative Percent Difference
%RSD	Percent Relative Standard Deviation
SD	Standard Deviation

APPENDIX B
DATA QUALIFIER DEFINITIONS

DATA QUALIFIER DEFINITIONS

A Data are acceptable, with qualifications.

E The associated value exceeds calibration range. Dilute and reanalyze.

J The associated value is an estimated quantity.

R Data are rejected.

U The compound was analyzed for but not detected at or above the method detection limit.

UJ The compound was analyzed for but not detected at or above the method detection limit. The associated value is an estimated quantity.

V Data are valid.

TEXAS WORLD OPERATIONS, INC.

INORGANIC DATA VALIDATION GUIDELINES

EPA SW-846 METHODS

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INORGANIC DATA VALIDATION GUIDELINES

EPA SW-846 METHODS

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APPENDICES

Appendix I Acronym List

Appendix II Data Qualifier Definitions

1.0 INTRODUCTION

The validation guidelines established in this document were developed specifically to evaluate and validate data generated using EPA Test Methods for Evaluating Solid Waste SW-846, November 1990, Revision 1, Third Edition. These guidelines utilize the quality control procedures specified in the following chapters and methods of SW-846:

1. Chapter One - Quality Control

   Defines the minimum quality control requirements. These requirements are method-wide.

2. Chapter Three - Metallic Analytes

   Defines the requirements for sampling, sample preservation, sample preparation, and sample holding times. These requirements are also method-wide.

3. Method 6010A - Inductively Coupled Plasma-Atomic Emission Spectroscopy

   Defines instrumentation and quality control procedures required for Inductively Coupled Plasma (ICP) analyses. These requirements are method-specific.

4. Method 7000A - Atomic Absorption Methods

   Defines instrumentation and quality control procedures required for Graphite Furnace Atomic Absorption (GFAA), Flame Atomic Absorption (Flame AA), and Cold Vapor Atomic Absorption (CVAA) Methods. These requirements are method-specific.

5. Method 7060A - Arsenic (GFAA)
6. Method 7740 - Selenium (GFAA)
7. Method 7421 - Lead (GFAA)
8. Method 7841 - Thallium (GFAA)
9. Method 7471A - Mercury in Solid or Semisolid Waste (CVAA)
10. Method 7470A - Mercury in Liquid Waste (CVAA)

1.1 Quality Control Requirements

As described in Chapter One, the words "must" and "shall", which appear throughout SW-846, are mandatory requirements for any RCRA related sampling activity. The words "may" and "should" are recommended requirements that may become mandatory for a specific project. Professional judgment should be used in

determining whether these "may" and "should" requirements are mandatory. Also, whenever conflicting control windows or control limits exist between method-wide criteria and method-specific criteria, the more stringent control limits will be used to evaluate data.

1.2 Definitions

Due to the lack of consistency in terminology used throughout SW-846, this section will define the terms used in these guidelines and also give any alternate terms used in SW-846. Definitions were taken directly from SW-846.

Analytical batch
A group of samples which behave similarly with respect to the procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

Calibration standards
A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve).

Continuing calibration verification (CCV)
Used to assure calibration accuracy during each analysis run. It must be run for each analyte at a frequency of 10% or every two hours during the run, whichever is more frequent. It must also be analyzed after the last analytical sample. Its concentration must be at or near the mid-range levels of the calibration curve.

Also referred to as instrument check standard for ICP, mid-range standard for GFAA, and reference standard for GFAA.

Initial calibration blank (ICB) and continuing calibration blank (CCB)
A volume of Type II water acidified with the same amounts of acids as the standards and samples.

Also referred to as calibration blank.

Initial calibration verification (ICV) standard
A certified (EPA or other) or independently prepared solution used to verify the accuracy of the initial calibration. For ICP analysis, it must be run at each wavelength used in the analysis.

Also referred to as calibration check standard for GFAA.

Reporting Limit
The instrument detection limits (IDL) established by the laboratory.

Interference check sample (ICS)
A solution containing both interfering and analyte elements of known concentration that can be used to verify background and interelement correction factors.

Also referred to as interference check solution.

Laboratory duplicate
An interlaboratory split sample which is used to document the precision of a method in a given sample matrix. The laboratory duplicate is brought through the whole sample preparation and analytical process in duplicate.

Also referred to as matrix duplicate and replicate sample.

Linear range
The concentration range over which the analytical curve remains linear.

Also referred to as linear dynamic range.

Matrix spike duplicate
Interlaboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis.

Also referred to as spiked replicate samples.

Method of standard addition
The standard-addition technique involves the use of the unknown and the unknown plus a known amount of standard.

Post digestion matrix spike
A known amount of analyte added to a pre-determined sample after sample digestion. The concentration added should bring the concentration of the analyte from two to five times the original concentration.

Also referred to as analytical spike.

Pre-digestion matrix spike
An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit.

Preparation blank
A volume of Type II water processed through each sample preparation procedure. The preparation blank must contain the same acid concentration in the final solution as the sample solution used for analysis.

Also referred to as method blank and reagent blank.

Sample holding time
The storage time allowed between the completion of sample collection and the date of sample analysis.

Split samples
Aliquots of sample taken from the same container and analyzed independently. In cases where aliquots of samples are impossible to obtain, field duplicate samples must be taken for the matrix duplicate analysis. These are usually taken after mixing or compositing and are used to document intra-or interlaboratory precision.

1.3 Analytical Methods

Table 1-1 gives a list of instruments and the analytes for which each instrument is applicable. It should be noted that the Method 7000A series also includes Flame AA and Gaseous Hydride Methods. These methods may be applicable for analysis of other analytes in certain situations. Specific methods in the SW-846 should be consulted when these techniques are used. All quality control criteria outlined in these guidelines for GFAA are also required for Flame AA.

TABLE 1-1 ANALYTICAL INSTRUMENTS AND ASSOCIATED ANALYTES

ICP:	Aluminum	Copper	Potassium
	Antimony	Iron	Selenium *
	Arsenic *	Lead *	Silver
	Barium	Lithium	Sodium
	Beryllium	Magnesium	Strontium
	Cadmium	Manganese	Thallium *
	Calcium	Molybdenum	Vanadium
	Chromium	Nickel	Zinc
	Cobalt	Phosphorus	

GFAA: Arsenic, Lead, Selenium, Thallium

Cold-Vapor AA: Mercury

* These analytes are run by GFAA when lower detection limits are required.

2.0 HOLDING TIMES AND SAMPLE PRESERVATION

2.1 Objective

The objective is to ascertain the validity of results based on the holding time and preservation of the sample.

NOTE: The holding time is based on the date when collection was completed, rather than verified time of sample receipt (VTSR).

2.2 Criteria

Technical requirements for sample holding times and sample preservation are listed in Table 2-1.

TABLE 2-1 HOLDING TIME AND PRESERVATION CRITERIA

Analyte	Holding Time	Preservative	
		Solid matrix:	Aqueous matrix:
Metals	180 days	Storage at 4°C	pH < 2 w/HNO_3
Mercury	28 days	Storage at 4°C	pH < 2 w/HNO_3

2.3 Evaluation Procedure

Actual holding times are established by comparing the sampling date on the EPA Sample Traffic Report (or Chain-of-Custody) with dates of analysis found in the laboratory raw data and on the data summary report. Holding times are determined as follows:

Holding Time = Analysis Date - Collection Date

Verify samples were properly preserved by examining the Chain-of-Custody (COC) and the sample preparation logs.

2.4 Action

1. If holding times are exceeded, all positive results are estimated (J) and non-detects are estimated (UJ).

2. If holding times are grossly exceeded (greater than or equal to 2X holding time), all positive results are estimated (J) and non-detects should be rejected (R).

3. If soil samples were not maintained at 4°C, positive results are estimated (J) and non-detects are estimated (UJ).

4. If aqueous samples were not adjusted to the proper pH, positive results are estimated (J) and non-detects are estimated (UJ).

3.0 SAMPLE PREPARATION

3.1 Objective

The objective of sample preparation procedures is to assure the proper preparation of samples for each analytical method and each individual analyte.

3.2 Criteria

1. Samples analyzed by ICP must be prepared by one of the following EPA SW-846 methods:

   a. Method 3005A for preparing groundwater and surface water samples for total recoverable or dissolved metals determination.

   b. Method 3010A for preparing aqueous samples and extracts for total metals determination.

   c. Method 3040A for preparing oils and hydrocarbon-based samples for dissolved metals determination.

   d. Method 3050A for preparing sediments, sludges, and soil samples for total metals determination.

2. Samples analyzed by GFAA for arsenic or lead analysis must be prepared by:

   a. Arsenic: Method 7060A (aqueous), Method 3050A (solid matrix).

   b. Lead: Method 3020A (aqueous), Method 3050A (solid matrix).

3. Samples analyzed by GFAA for selenium or thallium must be prepared by:

   a. Selenium: Method 7740A (aqueous), Method 3050A (solid matrix).

   b. Thallium: Method 3020A (aqueous), Method 3050A (solid matrix).

3.3 Evaluation Procedure

Examine the sample digestion logs to verify that the proper preparation method was used.

3.4 Action

1. If the correct method for sample preparation was not used for GFAA analysis of arsenic, lead, selenium, or thallium, all associated data is estimated (J or UJ).

2. If the correct method for sample preparation was not used for ICP analyses, all associated data is estimated (J or UJ).

4.0 CALIBRATION

4.1 Objective

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration is still valid.

4.2 Criteria

1. Initial Calibration

   a. Inductively Coupled Plasma (ICP) Analysis

      A blank and three standards must be used in establishing the analytical curve.

      After calibration and before beginning the analytical run, the highest calibration standard should be reanalyzed.

      If results are outside control limits, the instrument should be adjusted as recommended by the manufacturer and the highest calibration standard reanalyzed.

   b. Atomic Absorption (AA) Analysis

      Instruments must be calibrated daily with a blank and at least three standards.

   c. Mercury Analysis

      A blank and five standards must be used in establishing the analytical curve.

      NOTE: *Although SW-846 methods do not require the initial calibration correlation coefficient be greater than or equal to 0.995 (based on a least squares linear fit), the data reviewer should calculate the parameter to ascertain the accuracy of the calibration curve. If the initial calibration correlation coefficient is less than 0.995, professional judgment should be used to evaluate the data.*

2. Initial and Continuing Calibration Verification (ICV and CCV)

   An ICV standard must be analyzed after instrument calibration and before any analytical run. A CCV standard must be analyzed once every 10 samples and at the end of every analytical run. If the ICV or CCVs fall outside the control limits, terminate the run, recalibrate, and reanalyze the previous 10 samples.

The ICV and CCVs must fall within the control limits of 90-110 percent recovery (%R) of the true value.

4.3 Evaluation Procedure

1. Verify that the instrument was calibrated daily with a blank and the correct number of standards. Review the raw data to verify that the results for the ICV and CCV analyses were accurately transcribed onto the data summary report.

2. Recalculate one or more of the ICV and CCV %R using the following equation and verify that the recalculated value agrees with the %R reported on the data summary report.

$$\%R = \frac{\text{Value Found}}{\text{True Value}} \times 100$$

where:

Found = measured concentration (ug/L) of each analyte in the ICV or CCV solutions.
True = actual concentration (ug/L) of each analyte in the ICV or CCV source.

4.4 Action

1. If a blank and the minimum number of standards were not used for initial calibration, or if the instrument was not calibrated daily or each time the instrument was set up, qualify the data as rejected (R).

2. If the ICV or CCV %R falls outside the acceptance windows, use the following guidelines to validate the data:

   a. If the ICV or CCV %R is within the ranges of 75-89% or 111-125%, qualify positive results as estimated (J).

   b. If the ICV or CCV %R is within the range of 111-125%, non-detects are valid (V).

   c. If the ICV or CCV %R is within the range of 75-89%, qualify non-detects as estimated and undetected (UJ).

   d. If the ICV or CCV %R is less than 75%, qualify positive results and non-detects as rejected (R).

   e. If the ICV or CCV %R is greater than 125%, qualify positive results as rejected (R) and non-detects are valid (V).

3. If the initial calibration correlation coefficient is less than 0.995, professional judgment should be used to qualify the data.

5.0 BLANKS

5.1 Objective

Blank analysis results are assessed to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks applies to any blank associated with the samples.

5.2 Criteria

1. An Initial Calibration Blank (ICB) must be analyzed after the Initial Calibration Verification (ICV). A Continuing Calibration Blank (CCB) must be analyzed after every ten samples and at the end of the run.

2. A minimum of one preparation blank, prepared with the same reagents and volumes used for the samples, must be analyzed with each set of samples digested.

3. For any blank to be acceptable, the concentration in the blank must not be higher than the RL.

5.3 Evaluation Procedure

Review the raw data and verify that the results were accurately transcribed onto the data summary reports. Verify that an ICB was analyzed after the initial calibration and that a CCB was analyzed after every 10 samples and at the end of the run.

5.4 Action

1. If the absolute value of a blank is less than the RL, no action is taken.

2. If a blank value is greater than or equal to the RL:

   a. All sample results greater than the RL and less than five times the blank concentration are estimated and undetected (UJ).

   b. All sample results less than the RL are valid (V).

   c. All sample results greater than or equal to five times the blank concentration are valid (V).

NOTE: The blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples.

In instances where more than one blank associated with a given sample is greater than or equal to the RL, qualification should be performed using the associated blank with the highest concentration of contamination.

6.0 INTERFERENCE CHECK SAMPLE (ICS)

6.1 Objective

The ICP Interference Check Sample verifies the contract laboratory's interelement and background correction factors.

6.2 Criteria

The Interference Check Sample (ICS) must be run at the beginning and end of each analytical run or at a frequency of twice per 8 hours, whichever is more frequent. Results should be within ±20% of the true value.

6.3 Evaluation Procedure

1. Verify from the raw data (ICP printout) that the ICS was run at the beginning and end of each analytical run or at a frequency of twice per 8 hours.

2. Recalculate the ICS %Rs from the raw data using the following equation and verify that the recalculated value agrees with the values reported on the data summary forms.

$$\text{ICS } \%R = \frac{\text{ICS Found}}{\text{ICS True}} \times 100$$

where:

ICS Found = measured concentration (ug/L) of each analyte in the ICS solution.
ICS True = actual concentration (ug/L) of each analyte in the ICS solution.

6.4 Action

1. The following validation criteria for ICS solution AB must be followed:

   a. If the ICS recovery for an element is greater than 120% and the sample results are less than the RL, this data is considered valid (V).

   b. If the ICS recovery for an element is greater than 120% and the sample results are greater than the RL, qualify the data as estimated (J).

   c. If the ICS recovery for an element falls between 50 and 79% and the sample results are greater than the RL, qualify the affected data as estimated (J).

d. If sample results are less than the RL and the ICS recovery for that analyte falls within the range of 50 - 79%, the possibility of false negatives may exist. Qualify the data for these samples as estimated and undetected (UJ).

e. If the ICS recovery for an element is less than 50%, qualify all associated data as rejected (R).

7.0 MATRIX SPIKE ANALYSIS

7.1 Objective

The matrix spike sample analysis provides information about the effect of each sample matrix on the digestion and measurement methodologies.

7.2 Criteria

1. A pre-digestion matrix spike must be analyzed for every 20 samples or per analytical batch at a concentration between 10 and 100 times the IDL. The percent recovery (%R) should be within the limits of 80-120%.

2. Field blanks should not be used for matrix spike analysis.

7.3 Evaluation Procedure

1. Check the raw data and recalculate one or more of the %Rs found on the data summary forms and verify the values found in the raw data were transcribed correctly. Percent recoveries are calculated as follows:

$$\%R = \frac{(SSR-SR)}{SA} \times 100$$

where:

SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

2. Verify the spike added is at a concentration between 10 to 100 times the IDL and that the field blank was not used for matrix spike analysis.

7.4 Action

1. If the spike recovery is greater than 120%, all associated positive results are qualified as estimated (J) and non-detects are valid (V).

2. If the spike recovery falls within the range of 30-80%, all associated positive results are estimated (J) and non-detects are estimated and undetected (UJ).

3. If the spike recovery is less than or equal to 30%, positive results are estimated (J) and all non-detects are rejected (R).

4. If the field blank was used for matrix spike analysis, positive results are estimated (J) and non-detects are estimated and undetected (UJ).

5. If a matrix spike was not analyzed for each matrix or each analytical batch, positive results are estimated (J) and non-detects are estimated and undetected (UJ).

8.0 DUPLICATE SAMPLE AND MATRIX SPIKE DUPLICATE ANALYSIS

8.1 Objective

Duplicate analyses are indicators of laboratory precision based on each sample matrix.

8.2 Criteria

1. Either a laboratory duplicate or a matrix spike duplicate must be analyzed at a frequency of at least one per every 20 samples. The relative percent difference (RPD) should be within the control limits of 20%. The RPD for the matrix spike duplicate value must be within the control limits of 20% of the original matrix spike value.

2. Field blanks should not be used for duplicate sample analysis.

8.3 Evaluation Procedure

1. Review the data summary form and verify that results fall within the control limits of 20%.

2. Recalculate one or more RPDs and verify results were reported correctly. The RPD is calculated as follows:

$$RPD = \frac{|D_1 - D_2|}{(D_1 + D_2/2)} \times 100$$

where:

D_1 = original sample result or original matrix spike result
D_2 = duplicate sample result or duplicate matrix spike result

8.4 Action

1. If the RPD is not within 20%, all positive results are estimated (J) and all non-detects are estimated and undetected (UJ).

2. If the RPD for the duplicate matrix spike is not within the control limits of 20%, all positive results are estimated (J) and all non-detects are estimated (UJ).

3. If a field blank was used for duplicate analysis, all positive results are estimated (J) and all non-detects are estimated and undetected (UJ).

9.0 DUPLICATE CONTROL SAMPLE (DCS)

9.1 Objective

The duplicate control sample (DCS) serves as a monitor of the overall performance of the laboratory from sample preparation to sample analysis. It also monitors laboratory duplicate precision.

9.2 Criteria

1. Two DCSs must be prepared and analyzed for each matrix in each analytical batch of 20 samples or less.

2. The average of the DCS %Rs must fall within the control limits of 80-120%.

3. The DCS RPD must be less than 20%.

9.3 Evaluation Procedure

1. Verify that two DCSs were prepared and analyzed for each matrix at the required frequency.

2. Review the data summary form and verify that results fall within the control limits.

3. Check raw data and recalculate one or more percent recoveries. Percent recoveries are calculated as follows:

$$\text{DCS \%R} = \frac{\text{DCS Found}}{\text{DCS True}} \times 100$$

where:

DCS Found = measured concentration (ug/L for aqueous; mg/kg for solid) of each analyte in the DCS solution
DCS True = actual concentration (ug/L for aqueous; mg/kg for solid) of each analyte in the DCS solution

9.4 Action

1.

a. If the average %R of the DCSs are greater than 120%, all associated positive results are qualified as estimated (J) and non-detects are valid (V).

b. If the average DCS %R falls within the range of 50-79%, all associated positive results are qualified as estimated (J) and non-detects are estimated and undetected (UJ).

c. If the average DCS %R is less than 50%, qualify all associated data as rejected (R).

2. If DCSs were not analyzed at the required frequency, all positive results are estimated (J), and all non-detects are estimated and undetected (UJ).

3. If the DCS RPD is greater than 20%, qualify positive results as estimated (J) and non-detects are estimated (UJ).

10.0 FURNACE ATOMIC ABSORPTION QC

10.1 Objective

Analytical spikes establish the precision and accuracy of the individual analytical determinations and determine whether physical or chemical interferences may exist in each sample matrix type.

10.2 Criteria

1. The analytical spike recovery must fall within the control limits of 85-115 %R.

2. MSA analyses must be performed using the following criteria:

   a. The initial calibration curve must be linear over the concentration range of MSA standards analyzed. MSA correlation coefficient must be greater than 0.995.

   b. The slope of the MSA curve should be within 20% of the slope of the initial calibration.

10.3 Evaluation Procedure

1. Review the raw data to verify that all GFAA requirements were met.

2. If MSA was performed, recalculate the correlation coefficients to verify that they are greater than 0.995.

10.4 Action

1. If the furnace post digestion spike recovery is not within 85 - 115%, qualify positive results as estimated (J), and non-detects as estimated and undetected (UJ).

2. If the post-digestion spike recovery is < 40%, qualify positive results as estimated (J) and non-detects as rejected (R).

3. If MSA was required and not performed, qualify positive results as estimated (J), and non-detects as estimated and undetected (UJ).

4. If the MSA correlation coefficient is less than 0.995 for a sample, qualify positive results for the sample as estimated (J) and non-detects for the sample as estimated and undetected (UJ).

3. If MSA was required and not performed, qualify positive results as estimated (J), and non-detects as estimated and undetected (UJ).

4. If the MSA correlation coefficient is less than 0.995 for a sample, qualify positive results for the sample as estimated (J) and non-detects for the sample as estimated and undetected (UJ).

11.0 SAMPLE RESULT VERIFICATION

11.1 Objective

The objective is to ensure that the reported quantitative results are accurate.

11.2 Criteria

Verification of the reported values must be performed according to the procedures described in EPA SW-846.

11.3 Evaluation Procedure

Examine the raw data for any transcription or calculation errors and verify that the correct dilution factors were utilized to obtain the reported sample results. Verify that the sample prep weights and percent solids values were also properly incorporated into the determination of the reported values.

1. Examine the raw data for any anomalies (i.e., baseline shifts, negative absorbances, omissions, legibility, etc.).

2. Verify that no transcription or calculation errors occurred on any of the sample results.

3. Verify that instrument reporting limits are below the regulatory level for each analyte.

4. Verify that all ICP results fall within the linear ranges established for the instrument.

11.4 Action

If there are any discrepancies found, the laboratory may be contacted by the designated representative to obtain any additional information required to resolve they problems. If a discrepancy cannot be resolved, the reviewer may determine that qualification of the data is necessary.

12.0 OVERALL ASSESSMENT OF DATA

The overall data assessment qualifier represents the quality of the majority of the data. It can only be assigned once the data validation process is complete. At this point, each data point for each analytical result has been assigned a data qualifier based upon deficiencies in one or more of the QC categories (i.e., holding times, calibration, etc.). All results have been qualified. Determine which qualifier has been assigned to the majority of the analytical results and assign that qualifier as the overall data assessment qualifier.

APPENDIX I
ACRONYM LIST

ACRONYM LIST

AA	Atomic Absorption
CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
CLP	Contract Laboratory Program
COC	Chain-of-Custody
CVAA	Cold Vapor Atomic Absorption
GFAA	Graphite Furnace Atomic Absorption
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma
ICS	Interference Check Sample
ICV	Initial Calibration Verification
RL	Reporting Limit
DCS	Duplicate Control Sample
PB	Preparation Blank
MSA	Method of Standard Additions
RPD	Relative Percent Difference
VTSR	Verified Time of Sample Receipt

APPENDIX II

DATA QUALIFIER DEFINITIONS

DATA QUALIFIER DEFINITIONS

A	Data is acceptable, with qualifications.
J	The associated value is an estimated quantity.
R	Data is rejected.
U	The analyte was not detected at or above the instrument detection limit.
UJ	The analyte was not detected. The associated value is an estimate.
V	Data is valid.

TEXAS WORLD OPERATIONS, INC.
ORGANIC DATA VALIDATION GUIDELINES

EPA SW-846 METHOD 8150
CHLORINATED HERBICIDES ANALYSIS
(SEPTEMBER, 1986)

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October 1992

TEXAS WORLD OPERATIONS, INC.
ORGANIC DATA VALIDATION GUIDELINES

EPA SW-846 METHOD 8150
CHLORINATED HERBICIDES ANALYSIS

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APPENDICES

Appendix A Acronym List

Appendix B Data Qualifier Definitions

1.0 INTRODUCTION

This document is designed to offer guidance in laboratory data evaluation and validation of Chlorinated Herbicides in water, soil, and solid waste sample analyses. In some areas, specific guidelines and criteria are available. In other, less objective areas, only general guidance is offered due to the complexities and uniqueness of data relative to specific samples. The reviewer has a responsibility to inform the user concerning data quality and limitations, based on his/her technical judgment and evaluation, to assist the user in the proper use of the data.

Those areas where specific criteria are included are primarily areas in which definitive method performance requirements are established. These requirements are concerned with specifications that are not strictly sample dependent. They specify performance requirements normally under a laboratory's control. These specific areas include calibration standards, blanks, matrix spikes, and surrogate recoveries. Calculation and transcription errors must be rectified by laboratory resubmission of corrected data sheets if verification of the reported result is in doubt.

2.0 HOLDING TIMES

2.1 Objective

The objective is to ascertain the validity of results based on the holding time of the sample from the time of collection to the time of extraction and analysis.

2.2 Criteria

All samples and extracts must be iced or refrigerated at 4°C. Sodium thiosulfate should be added to water samples containing residual chlorine. (Although not used for qualification, these criteria are noteworthy.)

Water:
All samples must be extracted within seven days of collection and completely analyzed within 40 days of extraction.

Soil:
All samples must be extracted within fourteen days of collection and completely analyzed within 40 days of extraction.

2.3 Evaluation Procedure

Actual holding times are established by comparing the collection date on the Chain-of-Custody form with the dates of extraction and analysis found in the laboratory raw data. Holding times are determined as follows:

Extraction Holding Time (Days) = Extraction Start Date - Collection Date
Analysis Holding Time (Days) = Analysis Date - Extraction Start Date

2.4 Action

1. If the seven-day water extraction holding time, the fourteen-day soil extraction holding time, or the 40-day analysis holding time (after extraction) is exceeded, estimate (J) positive results and (UJ) non-detected results.

2. If the seven-day water extraction holding time, the fourteen-day soil extraction holding time, or the 40-day analysis holding time (after extraction) is grossly exceeded, estimate (J) positive results and reject (R) non-detected results. Grossly exceeded is defined as twice the acceptable holding time.

3.0 CALIBRATION

3.1 Objective

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents satisfactory performance of the instrument over specific time periods during sample analysis.

This will be done by a six-point quadratic curve.

3.2 Criteria

1. Initial Calibration

   a. Calibration standards, whether they are external or internal, must be prepared and analyzed at a minimum of six concentrations. One of the standards should be near but above the method detection limit which the laboratory has established. This applies to each GC column and instrument used for quantitation.

   b. The "RSE" of the calibration curve must be less than 15%.

2. Continuing Calibration

   a. The working calibration curve or calibration factor must be verified on each working day, every ten samples in the analysis sequence, and at the end of the analysis sequence by measuring a mid-level standard. The response for any analyte (compound) must be within ±20% of the initial calibration response or else the test must be repeated using a fresh calibration standard, or a new calibration curve or calibration factor must be generated. In addition, the retention time of any analyte should fall within the retention time window of that analyte from the initial calibration.

3.3 Evaluation Procedure

1. Initial Calibration

   a. Check the raw data and evaluate the quadratic curve.

   b. Verify that the "RSEs" are less than or equal to 15%.

   c. If errors are detected, pursue the explanation via more comprehensive recalculation.

2. Continuing Calibration

   a. Check the raw data and recalculate 10-20% of the %Ds.

   b. Verify that the %Ds are less than or equal to 20%.

   c. If errors are detected, pursue the explanation via more comprehensive recalculation.

3.4 Action

1. Initial Calibration

   a. Estimate (J) positive results for those compounds whose "RSEs" exceed 15% in the associated initial calibration. ("RSEs" remain undefined within the laboratory SOP. For the purpose of this action, "RSEs" are assumed to be an assessment of the curve's linearity.)

   b. Further information is required to assess the impact of "RSEs" which significantly exceed 15%.

2. Continuing Calibration

   a. Estimate (J) positive results for those compounds whose %Ds exceed 20% in the associated continuing calibration.

   b. Estimate (J) positive results and (UJ) non-detected results for those compounds whose %Ds exceed 50% in the associated continuing calibration.

   c. Estimate (J) positive results and reject (R) non-detected results for those compounds whose %Ds exceed 100% in the associated continuing calibration.

4.0 BLANKS

4.1 Objective

The assessment of method blank analysis results is to determine the existence and magnitude of contamination problems. If problems with any method blank exist, all data associated with the method blank must be carefully evaluated to determine whether or not there is an inherent variability in the data for the batch, or if the problem is an isolated occurrence not affecting other data. ("Associated" refers to those samples extracted and analyzed with a particular method blank.)

4.2 Criteria

A laboratory reagent blank (method blank) must be extracted and analyzed with each set of samples extracted (one every twenty samples) and when there is a change in reagents. No contaminants should be present in the blank(s).

4.3 Evaluation Procedure

1. Review the results of all associated blank(s) and raw data (chromatograms, mass spectra, quantitation reports, or data system printouts).

2. Verify that method blank analysis has been reported per matrix, per concentration, per GC system used to analyze samples, and per extraction batch.

4.4 Action

1. If a target compound is found in the method blank but not in the samples, no action is taken.

2. If a target compound is found in the method blank and is also found in the sample, apply the following:

    a. If the sample concentration is less than 5 times the blank concentration and less than the MDL: Report the MDL followed by (UJ).

    b. If the sample concentration is less than or equal to 5 times the blank concentration and greater than or equal to the MDL: Report the value followed by (UJ).

    c. If the sample concentration is greater than 5 times the blank concentration and greater than the MDL: Report the value unqualified.

3. If an associated method blank was not extracted and analyzed for the samples, estimate (J) positive results.

4. If an associated method blank was not extracted and analyzed for the samples, and there are positive results for the same target compound in all samples, reject (R) the positive results for that target compound in all samples.

5.0 SURROGATES

5.1 Objective

Laboratory performance on individual samples is established by means of spiking activities. The evaluation of the results of these surrogate spikes is not necessarily straightforward. The sample itself may produce effects due to such factors as interferences and high concentrations of analytes. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the review and validation of data based on specific sample results is frequently subjective and demands analytical experience and professional judgement. Accordingly, this section consists primarily of guidelines.

5.2 Criteria

1. Surrogate recoveries must fall within the control limits set by the laboratory. The laboratory will use the surrogate compound DCAA.

2. The surrogate must be added to the samples prior to the extraction process.

3. Samples in which surrogate recoveries are outside the control limits must be reanalyzed.

4. Samples reanalyzed due to failed surrogate recoveries which fail again must be reextracted and reanalyzed or the data must be "flagged as estimated concentration".

5.3 Evaluation Procedure

1. Check raw data (i.e., chromatograms, quant list, etc.) to verify the recoveries.

2. If recoveries are not within the control limits, check the raw data for possible interferences.

3. Check to verify that appropriate reanalyses or reextractions were performed.

5.4 Action

1. If the surrogate recovery is greater than the laboratory control limits, estimate (J) positive results. (Determine that high bias was not due to interference with the surrogate compound only.)

2. If the surrogate recovery is less than the laboratory control limits but greater than 10%, estimate (J) positive results and (UJ) non-detected results.

3. If the surrogate recovery is greater than zero but less than 10%, estimate (J) positive results and reject (R) non-detected results.

4. If zero pesticide surrogate recovery is reported, the reviewer should examine the sample chromatogram to determine if the surrogate may be present, but slightly outside its retention time window. If this is the case, in addition to assessing surrogate recovery for quantitative bias, the overriding consideration is to investigate the qualitative validity of the analysis. If the surrogate is not present, estimate (J) positive results and reject (R) non-detected results.

5. If appropriate reanalyses or reextractions were not performed, provide a comment in the validation report.

6.0 MATRIX SPIKE ANALYSIS

6.1 Objective

These data are generated to determine long-term precision and accuracy of the analytical method on various matrices. These data alone cannot be used to evaluate the precision and accuracy of individual samples.

6.2 Criteria

The laboratory is required to collect a portion of their samples to monitor spike recoveries. The frequency of spiked sample analyses must be one matrix spike (MS) per extraction batch (up to 20 samples). The MS must be spiked and analyzed using the same spiking solution as a laboratory control sample (which contains all target compounds). However, in most instances selected compounds will have been added (likely under contractual obligations).

6.3 Evaluation Procedure

1. Inspect results for the MS percent recoveries.
2. Verify transcriptions from raw data and verify calculations.
3. Verify that the MS recoveries fall within the control limits set by the laboratory.

6.4 Action

No action is taken on MS data alone to qualify an entire batch. However, using informed professional judgement the data reviewer may use the MS results in conjunction with other QC criteria and determine the need for some qualification of data.

7.0 ADDITIONAL QUALITY CONTROL

7.1 Objective

Laboratory control samples are required to establish acceptable accuracy and precision prior to any sample analyses and if any compound is outside its recovery range in the matrix spike analyses. (Data are not likely to be included as a deliverable.)

7.2 Criteria

Two laboratory control samples are prepared for every twenty samples. It is not clear if the laboratory control sample data will be included in the data package. If provided, the laboratory control sample recovery should be comparable to the data from Table 3 within the method or within the laboratory-generated limits. The relative percent difference (RPD) is limited to 20%. Overall, 80% of all QC values are required to be within QC limits.

7.3 Evaluation Procedure

Evaluate the accuracy of the data.

7.4 Action

Professional judgment should be used to evaluate the impact of outliers on the data.

8.0 COMPOUND IDENTIFICATION

8.1 Objective

The objective of the criteria for GC qualitative analysis is to minimize the number of erroneous identifications of compounds.

8.2 Criteria

1. The laboratory may establish retention time windows using actual retention time variation of standards over the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

2. Tentative identification of an analyte occurs when a peak falls within the daily retention time window. Normally, second column confirmation, GC/MS confirmation, or any other recognized confirmation technique is required. (Confirmation may not be necessary if the composition of the sample matrix is well-established by prior analyses.)

3. If peak detection and identification are prevented due to interferences, further cleanup is required.

8.3 Evaluation Procedure

1. Review Form I or the equivalent results summary, the associated raw data (chromatograms and data system printouts), and Form X or the equivalent summary. Confirm the reported positive results and verify the reported non-detected results using appropriate retention times and retention time windows.

2. Verify that positive identifications have dissimilar column analysis or GC/MS analysis.

3. Verify that cleanup techniques were employed for samples with interference.

8.4 Action

1. If confirmation data are not provided and a positive result is evident in the primary data, reject (R) the positive result.

2. If the criteria for positive identification (i.e. peak within its window on both columns or any evident shifts explained) are not met, and it is clear that an error in identification has occurred, change the result to non-detected (U).

3. If the criteria for identification are not met, and the misidentified peak(s) cause interference with potential detection of the target compound, then the value should be reported as an estimated quantitation limit (UJ).

4. If identification of any target compound is obscured by interference, professional judgement should be applied to estimate (UJ) the quantitation limits. (If no cleanup techniques were employed, provide a comment in the validation report.)

9.0 COMPOUND QUANTITATION

9.1 Objective

The objective is to ensure that the reported quantitation results and detection limits are accurate.

9.2 Criteria

1. Compound quantitation, as well as the adjustment of the method detection limit (MDL), must be calculated according to the method.

2. If the response for the peak exceeds the linear range of the system, the extract must be diluted and reanalyzed.

3. The internal standard 4,4'-dibromooctafluorobiphenyl may be used for quantitation if the %D of the internal standard areas between the calibration standard and the sample is less than 20%.

9.3 Evaluation Procedure

1. Examine the raw data to verify the correct calculation of all sample results. Quantitation lists, chromatograms, sample preparation logs, dilutions, and cleanups are compared to the reported sample results and quantitation limits.

2. Verify that responses for target compounds and standard peaks were measured consistently, i.e., all values were determined by either integrated areas or peak heights, not both.

3. Verify that if the response for any target compound exceeded the linear range of the system, the sample was diluted and reanalyzed.

4. Verify that the correct quantitation technique (internal or external standard) was used for quantitation.

   Provided that the quadratic equation can be verified to determine the initial ug/mL. The sample result can be calculated as follows:

$$\text{Concentration (ug/L or ug/Kg)} = \frac{\text{ug}}{\text{mL}} \quad \frac{V_t \times D}{(V_s \ \underline{\text{or}}\ W)}$$

   where:

   V_t = Volume of total extract, mL.
   D = Dilution factor.
   V_s = Volume of water extracted, L.
   W = Weight of soil extracted, Kg.

9.4 Action

1. If major discrepancies are found which prevent determination of the correct result, resubmittal will be necessary to resolve the problem.

2. If the response for any target compound exceeds the linear range of the system and the sample was not diluted and reanalyzed, the value should be qualified with an (E).

3. If the internal standard %D exceeded 30% and the internal standard quantitation technique was used, estimate (J) positive results.

10.0 SYSTEM PERFORMANCE

10.1 Objective

A thorough review of on-going data acquisition and examination of the raw data can yield indicators of instrument performance.

10.2 Criteria

Instrument performance indicators are as follows:

1. Abrupt, discrete baseline changes indicating electronic or detector changes.

2. Poor chromatographic performance characterized by high background levels, shifts in absolute retention times of standards, large extraneous peak interferences, baseline rise at elevated temperatures, loss of peak resolution, and peak tailing or peak splitting which could result in inaccurate quantitation.

10.3 Action

Based on the performance indicators discussed previously, the data reviewer must decide if the system has degraded to the point of affecting data quality or validity. If data quality may have been affected, data should be qualified based on the reviewer's technical judgment and subjective evaluation.

11.0 OVERALL DATA ASSESSMENT

The overall data assessment represents the quality of the majority of the data. It can only be assigned once the data validation process is complete. At this point, each data point for each analytical result has been assigned a data qualifier based upon deficiencies in one or more of the QC categories (i.e. holding times, calibration, etc.). All results have been qualified. Determine which qualifier has been assigned to the majority of the analytical results and assign that qualifier as the overall data assessment qualifier.

APPENDIX A
ACRONYM LIST

ACRONYM LIST

CF	Calibration Factor
%D	Percent Difference
MDL	Method Detection Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
RF	Response Factor
RPD	Relative Percent Difference
%RSD	Percent Relative Standard Deviation
SD	Standard Deviation

APPENDIX B

DATA QUALIFIER DEFINITIONS

DATA QUALIFIER DEFINITIONS

A Data are acceptable, with qualifications.

E The associated value exceeds calibration range. Dilute and reanalyze.

J The associated value is an estimated quantity.

R Data are rejected.

U The compound was analyzed for but not detected at or above the method detection limit.

UJ The compound was analyzed for but not detected at or above the method detection limit. The associated value is an estimated quantity.

V Data are valid.

WATER QUALITY PARAMETERS
DATA VALIDATION GUIDELINES

for

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APPENDICES

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Appendix C References

1.0 INTRODUCTION

This document is designed to offer guidance in laboratory data evaluation and validation. In some areas, specific guidelines and criteria are available. In other, more subjective areas, only general guidance is offered due to the complexities and uniqueness of data relative to specific samples.

Those areas where specific criteria are included are primarily areas in which definitive method performance requirements are established. These requirements are concerned with specifications that are not sample dependent; they specify performance requirements normally under a laboratory's control. These specific areas include blanks, calibration standards, calibration verification standards, laboratory control standards, and duplicates. Calculation and transcription errors must be rectified by resubmission of corrected data sheets.

The requirements to be checked during validation are listed below:

- Holding Times
- Calibration
  - Initial Calibration
  - Initial and Continuing Calibration Verification
- Blanks
- Laboratory Control Sample (LCS)
- Duplicate Sample Analysis
- Matrix Spike Sample Analysis
- Sample Result Verification
- Field Duplicates
- Overall Assessment of Data for a Case

2.0 HOLDING TIMES

2.1 Objective

The objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of analysis.

Note: The holding time is based on the date of collection rather than Verified Time of Sample receipt (VTSR) or date of preparation.

2.2 Criteria

Technical requirements for sample holding times have been established for water matrices. The following holding times and preservation requirements were established under 40 CFR 136 (Clean Water Act) or taken from Methods for Chemical Analysis of Water and Wastes.

Determination	Holding Time	Preservative
Acidity	14 days *	4°C
Fluoride	28 days	None
Chloride	28 days	4°C
Nitrate/Nitrite as N	28 days	4°C or 4°C (pH < 2(H_2SO_4))
Nitrite	48 hours	4°C
Sulfate	28 days	4°C
Alkalinity (Total)	14 days	4°C
Bicarbonate	14 days *	4°C
Carbonate	14 days *	4°C
Total Dissolved Solids	7 days	4°C
Total Suspended Solids	7 days	4°C
Phenolics (Total Recoverable)	28 days	4°C (pH < 2(H_2SO_4))
Phosphorus (Total)	28 days	4°C (filter)
pH	24 hours	4°C
Specific Conductivity	28 days	4°C
TOC	28 days (14 days soil)	4°C (pH < 2 HCl) (filter)
Ammonia	28 days	4°C (pH < 2(H_2SO_4))
Cyanide	14 days	4°C (pH > 12 NaOH) (filter) + 0.6 g ascorbic acid

* Actual requirements state as soon as practicably possible, with a maximum holding time of 14 days.

2.3 Evaluation Procedure

Actual holding times are established by comparing the sampling date on the Field Results Summary with the dates of analysis found in the laboratory raw data (Instrument Run Logs).

Analyte Holding Time (Days) = Analysis Date - Sampling Date

2.4 Action

1. If criteria for holding times and preservation are not met, qualify all results greater than the Instrument Detection Limit (IDL) or Method Detection Limit (MDL) as estimated (J) and results less than the IDL (or MDL) as estimated and undetected (UJ).

2. If holding times are grossly exceeded, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on sample results. The expected bias would be low and the reviewer may reject (R) results less than the IDLs, for appropriate analyses.

3. Soil holding time criteria are the same as those for water, except for TOC analysis. It is left to the professional judgement of the reviewer to take action if holding times are exceeded.

3.0 CALIBRATION

3.1 Objective

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification analyses demonstrate that the initial calibration is still valid.

3.2 Criteria

3.2.1 *Initial Calibration*

Instruments must be calibrated daily or before each analytical run.

- Colorimetric analysis
  - A blank and at least three standards must be used to establish the analytical curve.
  - The correlation coefficient for the standard curve must be greater than or equal to 0.995. (The correlation coefficient is a measure of the linear relationship of concentration vs. instrument absorbance readings.)
- Carbonate, Bicarbonate, Acidity, and Specific Conductivity determinations.
  - Follow the manufacturer's instructions for calibration.
- pH determination
  - Calibrate pH meters using at least two buffer solutions corrected to room temperature, if appropriate.
- Ion chromatography analysis
  - A blank and three standards must be used to establish the analytical curve for each analyte. Note: Column loading must not exceed 400 mg/L for any analyte.
  - An instrument calibration curve must be generated daily or once every 24 hours and each time the instrument is set up.
  - A four-point curve must be analyzed when eluent is changed.
  - The concentration range of the standards must not exceed the linear range of the system.

3.2.2 *Initial and Continuing Calibration Verification (ICV and CCV)*

An initial calibration verification (ICV) sample must be performed after instrument calibration before any analytical run to verify the calibration. The ICV must be from a different source than the continuing calibration verification (CCV) if an EPA standard is not used.

When the measurement exceeds control limits for the ICV, the analysis must be terminated, the instrument must be recalibrated, and the calibration must be verified.

Continuing calibration verification samples must be analyzed once every 20 samples and at the end of every analytical sequence in order to bracket all sample analysis, as applicable.

Analysis results must fall within the control limits of 85-115 percent recovery (%R) of the true value. If the CCV is outside control limits, the analysis must be terminated, the instrument must be recalibrated, the calibration must be verified, and the samples analyzed after the last CCV that was in control must be rerun.

3.3 Evaluation Procedure

1. Verify that the instrument was calibrated daily and each time the instrument was set up using the correct number of standards and blank.

2. Verify that the correlation coefficient for the instrument calibration is greater than or equal to 0.995.

3. Recalculate one or more of the ICV and CCV percent recovery (%R) per analyte, using the following equation and verify that the recalculated value agrees with the laboratory reported values. Due to possible rounding discrepancies, allow results to fall within 1% of the acceptance windows (e.g., 84-116%).

$$\%R = \frac{\text{Found}}{\text{True}} \times 100$$

Where,

Found = Measured concentration of each analyte (in mg/L) in the ICV or CCV solution.

True = Expected concentration of each analyte (in mg/L) in the ICV or CCV solution.

3.4 Action

1. If the minimum number of standards were not used for initial calibration, or the instrument was not calibrated daily and each time the instrument was set up, qualify the data as rejected (R).

2. If the correlation coefficient is less than 0.995, qualify results greater than IDL as estimated (J), and results less than IDL as estimated and undetected (UJ).

3. If the ICV or CCV %R falls outside the acceptance windows, use professional judgement to qualify all associated data. If possible, indicate the bias present.

   The following guidelines are recommended:

   - If the ICV or CCV %R falls outside the acceptance windows, but within the ranges of 50-84%, qualify results greater than IDL as estimated (J).
   - If the ICV or CCV %R is greater than 116%, results less than IDL are valid (V), and results greater than IDL are estimated (J).
   - If the ICV or CCV %R is 50-84%, qualify results less than IDL as estimated and undetected (UJ).
   - If the ICV or CCV %R is less than 50%, qualify all results less than IDL as rejected (R).

4.0 BLANKS

4.1 Objective

Blank analysis results are assessed to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks applies to those blanks associated with the sample analysis. If problems with the blanks exist, all data associated with the Batch must be carefully evaluated to determine if there is an inherent variability in the data for the Batch, or if the problem is an isolated occurrence not affecting other data.

4.2 Criteria

1. An Initial Calibration Blank (ICB) must be used to verify the base line immediately following calibration and prior to analytical sample analysis.

2. A continuing calibration blank (CCB) must be analyzed once every 20 samples and at the end of every analytical sequence in order to bracket all sample analyses.

3. A preparation blank (PB) should be analyzed with each method if a preparation procedure is required for the samples (i.e., cyanide).

4. If the ICB result exceeds the Contract Required Detection Limit (CRDL) for any analyte, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration verified.

5. Blanks must be deionized water.

6. If a blank concentration is above the CRDL, the lowest concentration of the analyte in the associated samples must be 5X the blank concentration. Otherwise, all samples associated with the blank, with the analyte's concentration <5X the blank concentration and above the CRDL, must be reanalyzed for that analyte.

7. If the concentration of the blank is below the negative CRDL, then all samples reported below 5X CRDL associated with the blank must be reanalyzed (ion chromatography, titrimetric, and gravimetric analyses).

4.3 Evaluation Procedures

No contaminants above the IDL should be present in the blank(s).

Review the results reported on the Blank Summary as well as the raw data (instrument printouts, strip charts, printer tapes, bench sheets, etc.) for all blanks and verify that the results were accurately reported.

4.4 Action

1. If the absolute value of a blank is less than the IDL, no action is taken.

2. If a blank value is greater than the IDL:

   - All sample results greater than the IDL and less than five times the blank concentration are estimated and undetected (UJ).

   - All sample results less than the IDL are valid (V).

   - All sample results greater than five times the blank concentration are valid (V).

3. If a blank value is less than zero, and the absolute value of the blank minus the sample results are:

   - less than the IDL, then the sample results are valid (V).

   - greater than or equal to the IDL, but less than two times the IDL, then the sample results are estimated and undetected (UJ).

   - greater than or equal to two times the IDL, then the sample results are rejected (R).

4. If a blank value is less than the negative IDL and the sample result is greater than the IDL but less than five times the absolute blank value, the results are estimated (J).

5. If the raw data sample results are less than the negative blank, the possibility of serious instrument problems or severe negative bias exists. The reviewer should carefully evaluate each sample point.

Note: The blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. In particular, soil sample results may not be on the same basis (units, dilution) as the calibration blank data. The reviewer may find it easier to work from the raw data when applying 5X criteria to soil sample data/calibration blank data.

In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant. Sample results may not be corrected by subtracting any blank values. The only exception is for color blank subtraction for turbidimetric analyses.

5.0 LABORATORY CONTROL SAMPLE (LCS)

5.1 Objective

The laboratory control sample serves as a monitor of the overall performance of all steps in the analysis, including sample preparation.

5.2 Criteria

1. A LCS sample must be analyzed every 20 samples or one for each Sample Delivery Group (SDG), whichever is more frequent.

2. All aqueous LCS results must fall within the control limits of 80-120%R where applicable.

3. All solid LCS results must fall within the control limits of 80-120%R, where applicable.

4. EPA LCS solutions shall be used. If EPA solutions are not available, LCSs may be prepared using stock standards in reagent grade water at a different concentration and source than a calibration verification sample, and within the calibrated range for the instrument. For soils, if EPA solid LCS is unavailable, other EPA QA check samples or other certified materials may be used.

5. The laboratory must verify, with raw data, the reported recoveries. Calculate percent recoveries by comparing the reported value against the true value. All aqueous and soil LCS results must be within the control limits of 80-120% recovery.

6. All LCS results which fall outside control limits require that the analysis be terminated, the problem corrected, and the samples associated with that LCS reanalyzed.

7. A LCS must be prepared and analyzed with each sample batch.

8. LCS analysis does not apply for acidity.

5.3 Evaluation Procedure

1. Verify that results fall within the control limits.

2. Check raw data (instrument printout, strip charts, bench sheets, etc.) to verify the reported recoveries. Recalculate one or more of the recoveries (%R) using the following equation:

$$\text{LCS } \%R = \frac{\text{LCS Found}}{\text{LCS True}} \times 100$$

5.4 Action

1. Aqueous LCS

   - If the LCS recovery for any analyte falls within the range of 50-79% or greater than 120%, qualify results greater than IDL as estimated (J).
   - If the LCS recovery is greater than 120% and results are less than IDL, the data is valid (V).
   - If the LCS recovery is 50-79% and the results are less than IDL, qualify the data as estimated and undetected (UJ).
   - If the LCS recovery is less than 50%, qualify the data for these samples as rejected (R).

2. Solid LCS

   - If the solid LCS recovery falls outside the control limits, qualify all sample results greater than IDL as estimated (J).
   - If the LCS results are higher than the control limits and the sample results are less than IDL, the data is valid (V).
   - If the LCS results are lower than the control limits, qualify all sample results less than IDL as estimated and undetected (UJ).

6.0 DUPLICATE SAMPLE ANALYSIS

6.1 Objective

Duplicate analyses are indicators of laboratory precision based on each sample matrix.

6.2 Criteria

1. Samples identified as field blanks cannot be used for duplicate sample analysis.

2. A control limit of ±20% (35% for soil) for the Relative Percent Difference (RPD) shall be used for sample values greater than 5X the Contract Required Detection Limit (CRDL).

3. A control limit of ± CRDL (±2X CRDL for soil) shall be used for sample values less than 5X CRDL, including the case when only one of the duplicate sample values is less than 5X CRDL.

4. A duplicate sample should be analyzed with every SDG or for every 20 samples, whichever is more frequent.

5. The RPD is calculated by the difference between the sample value and duplicate sample value and dividing by the average of both readings.

6. A matrix spike duplicate does not constitute a duplicate sample analysis.

6.3 Evaluation Procedure

1. Verify that results fall within the control limits.

2. Check the raw data and recalculate one or more RPD using the following equation to verify that results have been reported correctly.

$$RPD = \frac{|S-D|}{(S+D)/2} \times 100$$

Where,

S = Original Sample Value
D = Duplicate Sample Value

3. Verify that the field blank was not used for duplicate analysis.

4. Verify that a duplicate was analyzed every 20 samples or SDG.

6.4 Action

1. If duplicate analysis results for a particular analyte fall outside the appropriate control windows, qualify the results for that analyte in all associated samples of the same matrix as estimated (J).

2. If the field blank was used for duplicate analysis, all other QC data must be carefully checked and professional judgement exercised when evaluating the data. Blank duplicate analysis does not provide a measure of analytical precision above method detection limits. All other QC data must be evaluated to determine if qualification is necessary based upon precision criteria.

7.0 MATRIX SPIKE SAMPLE ANALYSIS

7.1 Objective

The matrix spike sample analysis provides information about the effect of each sample matrix on the measurement methodology.

7.2 Criteria

1. Samples identified as field blanks cannot be used for spiked sample analysis.

2. Spike recovery (%R) must be within the limits of 75-125% where applicable. However, spike recovery limits do not apply when sample concentration exceeds the spike concentration by a factor of 4 or more.

3. A spike sample should be run with every SDG or every 20 samples, whichever is more frequent.

4. A spike concentration is chosen for each analyte in the ion chromatographic analyses and is measured in the respective working standard ranges of the calibration curves.

5. Matrix spike analysis does not apply for bicarbonate, carbonate, acidity, pH, and conductance.

7.3 Evaluation Procedure

1. Verify that results fall within the specified limits.

2. Check raw data and recalculate one or more %R using the following equation to verify that results were correctly reported.

$$\%R = \frac{(SSR-SR)}{SA} \times 100$$

Where,

SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Concentration Added

3. Verify that the field blank was not used for spike analysis.

4. Verify that a spike sample was analyzed every 20 samples or for each SDG.

5. Verify that the laboratory provided results for the spiked sample result, the original sample analysis, and the concentration of spike solution added.

7.4 Action

1. If the spike recovery is greater than 125% or less than 74%, and the sample results are greater than IDL, qualify the data for these samples as estimated (J).

2. If the spike recovery is greater than 125% and the sample results are less than IDL, the data is valid (V).

3. If the spike recovery falls within the range of 30-74% and the sample results are less than IDL, qualify the data for these samples as estimated and undetected (UJ).

4. If the spike recovery results fall less than 30% and the sample results are less than IDL, qualify the data for these samples as rejected (R).

5. If the field blank was used for matrix spike analysis, all other QC data must be carefully checked and professional judgement exercised when evaluating the data.

8.0 SAMPLE RESULT VERIFICATION

8.1 Objective

The objective is to ensure that the reported quantitation results are accurate.

8.2 Criteria

Analyte quantitation must be calculated according to the appropriate Statement of Work (SOW).

8.3 Evaluation Procedure

The raw data should be examined to verify the correct calculation of sample results reported by the laboratory. Bench sheets, instrument printouts, strip charts, etc. should be compared to the reported sample results.

1. Examine the raw data for any anomalies (i.e., baseline shifts, negative absorbances, omissions, legibility, etc.).

2. Verify that there are no transcription or reduction errors (e.g., dilutions, percent moisture, sample weights) for one or more samples.

3. Verify that results fall within the calibrated range of the instrument.

4. Interferences may occur in ion chromatography from substances which have similar retention times or overlap the analyte of interest. Sample dilution and/or spiking is used to solve most problems with interferences. These anomalies should be noted in the case narrative section of the data package.

5. Verify that percent solids results are greater than 30% to be considered a soil sample.

8.4 Action

1. If there are any discrepancies found in the raw data, the laboratory may be contacted by the data reviewer to obtain additional information that could resolve any differences. If a discrepancy cannot be resolved, the reviewer may determine if qualification of the data is warranted based upon the circumstances of the discrepancy.

2. If the samples are soil samples and percent solids are < 30%, estimate (J) all positive values and reject (R) all non-detects.

3. If the samples are soil samples and percent solids are < 10%, reject (R) all analytical results associated with that sample.

9.0 FIELD DUPLICATES

9.1 Objective

Field duplicate samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than lab duplicates which measure only lab performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field samples.

9.2 Criteria

There are no review criteria for field duplicate analyses comparability at this time.

9.3 Evaluation Procedures

Samples which are field duplicates should be identified using sample field sheets. The reviewer should compare the results reported for each sample and calculate the Relative Percent Difference (RPD), if appropriate.

The RPD can be calculated by using:

$$RPD = \frac{|S - D|}{(S+D)/2} \times 100$$

Where,

S = Sample Result
D = Duplicate Sample Result

9.4 Action

Any evaluation of the field duplicates should include the reviewer's comments.

10.0 OVERALL ASSESSMENT OF DATA FOR A CASE

It is appropriate for the data reviewer to make professional judgments and express concerns and comments on the validity of the overall data for a Case. This is particularly appropriate when there are several QC criteria out of specification.

Appendix A

Acronym List

ACRONYM LIST

CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
CRDL	Contract Required Detection Limit
FB	Field Blank
ICB	Initial Calibration Blank
ICV	Initial Calibration Verification
IDL	Instrument Detection Limit
LCS	Laboratory Control Sample
MB	Method Blank
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
SOW	Statement of Work
TB	Trip Blank
VTSR	Verified Time of Sample Receipt

Appendix B

Data Qualifier Definitions

DATA QUALIFIER DEFINITIONS

A Data is Acceptable, with Qualifications.

J The associated value is an estimated quantity.

R Data is rejected.

U The analyte was analyzed for, but not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.

UJ The material was analyzed for, but was not detected. The associated value is an estimate.

V Data is valid.

Appendix C

References

REFERENCES

APHA, Standard Methods for the Examination of Water and Wastewater, 17th Edition, 1989.

Methods for Determination of Inorganic Substances in Water and Fluvial Sediments, Techniques of Water-Resources Investigations of the United States Geological Survey, Book 5, Chapter A1, GPO 1979.

USEPA, Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses, July, 1988.

USEPA, Methods for Chemical Analysis of Water and Wastes, March, 1984.

USEPA SW-846, Test Methods for Evaluating Solid Waste, Third Edition, 1986.

SOP Number: LM-RMA-3001
Title: GC/MS Analysis of Volatile Organics

This addendum is specific to the Chemical Waste Management, Vickery, Ground Water Monitoring Plan QAPjP. Procedural changes or clarification statements presented herein are not applicable to Enseco SOPs for other projects, and are made here in response to an EPA letter dated March 17, 1992 to CWM containing comments (with additional attachment) regarding the CWM-Vickery GWMP QAPjP.

Section 1.2: See QAPjP Appendix A-10 for the project specific "reporting limits".

Section 7.3 to Section 7.6: Referenced SOPs are included in QAPjP Appendix B-13A.

Section 8.3: Referenced SOP LM-RMA-3022 is included ia QAPjP Appendix B-13A.

Section 8.4: All samples are homogenized prior to analysis. Aqueous samples only will be analyzed for this project, percent moisture determination is not applicable.

Section 8.7.1.2: Ion Abundance Criteria for BFB is found in Table C-1 of the SOP Appendix C, which is present near the end of QAPjP Appendix B-13.

The wording of the section's second and third paragraphs remain as stated, and are intended to be instructive to the analyst.

Section 8.7.2: For the purpose of this project, the next-to-last paragraph of the section is deleted. Specific compounds being analyzed for are listed in the project parameter listings in Section 7 of the GWMP.

Section 8.9: Dilutions of samples are performed within accepted EPA guidelines in SW-846 and the CLP methods. All samples are screened prior to analysis. The screening results are used to optimize the sample analysis to achieve the lowest possible reporting limits. Dilutions are only performed relative to compounds that would interfere with the analysis. Thus, a high concentration of inorganic ions such as sodium would not result in a dilution being performed for volatile organics.

Section 8.9.3.9 to 8.9.3.10:	Acceptance control limits for surrogate spikes and matrix spike recovery will be set after experience working with the matrix is obtained. Limits will be specified in the raw data.
Section 11.2:	Project specific reporting limits were listed in QAPjP Appendix A-10.
SOP Appendix A:	Target compounds are provided in this appendix. The relative retention times for individual compounds per instrument conditions is indicated in the screening calibration data found within the latter portions of QAPjP Appendix B-13B.

GC/MS ANALYSIS OF VOLATILE ORGANICS

SOP No.:	Revision No.:	Effective Date:
LM-RMA-3001 VOA by GC/MS	3.0	4/24/90

Supersedes: 2.0

1. SCOPE AND APPLICATION

1.1 Analytes: Quantitative analysis is performed for the compounds listed in Appendix A. Qualitative analysis by mass spectral identification can be performed on other purgeable, chromatographable compounds.

1.2 Reporting Limits: Test Specific

1.3 Applicable matrices: Water, soil, solids, sludge and waste.

1.4 Dynamic range: Analytes can typically be quantitated between 5 ppb and 200 ppb for water samples and soils analyzed by the low level direct purge method. Analytes in soils analyzed by the methanol extraction method can be quantitated between 500 and 20000 ppb.

1.5 Analysis time: Approximate analytical time is 40 minutes per GC/MS run. The time required for data reduction will be dependent upon the complexity of the sample.

Prepared by:	Date: 4/24/90
Management Approval:	Date: 4/24/90
QA Officer Approval:	Date: 4-24-90

1.6 The applicability of this Standard Operating Procedure to particular matrices and/or analytical requests not specifically stated herein is determined by the GC/MS department manager and communicated to the client manager. The GC/MS department manager has the authority to make modifications to the SOP for certain matrices and/or analyses if his technical judgement deems it necessary and appropriate.

2. METHOD SUMMARY

2.1 Aqueous samples are analyzed by quantitative and/or qualitative purge and trap-GC/MS using an internal standard method.

2.2 Solid samples are extracted with methanol and the extract is analyzed by purge and trap-GC/MS for quantitative and/or qualitative identification using the same internal standard method.

2.3 Alternatively, lower detection limits can be achieved for analytes in soil samples by using the direct purge method.

3. COMMENTS

3.1 All deviations made from this SOP **MUST** be approved by the supervisor and well documented.

3.2 All reusable glassware should be washed with hot, soapy water and rinsed thoroughly. This is followed by a rinse with HPLC grade (or better) Methanol. Finally, the glassware is baked in a 105° oven.

3.3 Care should be taken not to introduce contamination into the samples. Particular attention should be paid to common laboratory solvents, such as acetone and methylene chloride.

3.4 Care should be taken when handling samples for volatiles analysis to avoid loss of analytes.

3.5 Do not overstock traps. Tenax has a lifetime of only a few months. Traps will also degrade with use due to the breakdown of tenax from rapid temperature changes.

4. SAFETY ISSUES

4.1 The toxicity or carcinogenicity of each chemical used in this procedure has not been precisely defined, however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals should be reduced to the lowest possible.

4.2 A Material Safety Data Sheet (MSDS) is available for all laboratory standard and reagent chemicals. The appropriate MSDS must be read before handling the chemical(s).

4.3 All laboratory personnel should be thoroughly familiar with the laboratory Safety Manual and Hazard Communication Standard before undertaking any laboratory work.

4.4 Samples should be considered as hazardous. Appropriate protective clothing, such as gloves, lab coat and safety glasses should be worn when handling samples.

5. SAMPLE COLLECTION, PRESERVATION, CONTAINERS AND HOLDING TIMES

5.1 Standard 40 mL glass screw-cap VOA vials with Teflon-faced silicone septa may be used for both liquid and solid matrices. Solid samples may also be collected in wide-mouth glass jars with Teflon-lined caps. Samples should be introduced into the containers with minimum agitation to avoid loss of volatile components. For liquid samples, each VOA vial should be filled without introduction of bubbles. Fill until there is a meniscus over the lip of the vial. The lid with septum (Teflon side toward the sample) should be tightened onto the vial. After tightening the lid, the vial should be inverted and

tapped to check for air bubbles. If there are any air bubbles present the sample must be retaken. Sample containers for solid samples should be filled as completely as possible with minimum air space.

5.2 Water samples may be preserved with HCl to extend the holding time of samples to be analyzed for aromatic compounds. Aromatic compounds are particularly susceptible to biodegradation at normal pH. The pH of the sample should be adjusted to less than 2 with HCl in the field at the time of sampling.

5.3 All samples must be iced or refrigerated at 4°C from the time of collection until analysis.

5.4 All aqueous samples must be analyzed within 7 days of collection. If aqueous samples are preserved with HCl, the holding time is extended to fourteen days from date of collection. Medium level soil/waste samples must be extracted and analyzed within 14 days of collection. Soil samples which are analyzed by the low level method must be analyzed within 14 days from the sampling date.

6. APPARATUS

6.1 Gas-tight syringes: 10 uL, 25 uL, 50 uL, 100 uL, 250 uL, 500 uL, and 1 mL.

6.2 Syringe valve: two-way with Luer ends.

6.3 5 mL gas tight syringe, luer-lock type.

6.4 1 mL disposable pipet.

6.5 Top-loading balance capable of weighing 0.1 g.

6.6 Glass culture tubes with screw cap and teflon liner.

6.7 Volumetric flasks: 10 mL - 100 mL.

6.8 Screw top vials with teflon-lined septa for storage of methanol extracts and extract dilutions.

6.9 Disposable pasteur pipets.

6.10 Stainless steel spatula.

6.11 Gas Chromatograph for screening with all required accessories such as syringes, analytical columns, gases, flame ionization detector, recording device and optional autosampler or optional headspace analyzer. A data system is recommended for data acquisition, integration and output.

6.12 Heated bath or heater capable of maintaining the purging chamber to within +/- 1°C over the temperature range of ambient to 100°C.

6.13 Purge-and-trap device including the sample purger, the trap and the desorber. (Tekmar LSC-2 or equivalent) Device may include automatic sampler (Tekmar ALS or equivalent.)

6.14 Purging Chamber

6.14.1 Two piece glass purge cell with ground glass joint and airtight o-ring connection, single piece glass purge tube, and/or glass-fritted sparger or equivalent.

6.15 Traps Four phase: Supelco Volatile Purge trap, or equivalent.

6.15.1
1. 1 cm 3% SP-2100 on 60/80 Chromosorb WAW
2. 15 cm Tenax TA
3. 8 cm 35/60 Silica Gel Gd-15
4. 1.5 cm Carbosieve added to base

6.16 Chromatographic Columns

6.16.1 Screening column: JW P/N 125-1334 DB624. Mega-Bore 30m X .53mm I.D., or equivalent

6.16.2 Analytical Column:

A. JW P/N 125-1334. DB624 Mega-Bore 30m X .53mm I.D., or equivalent.

6.17 Gas Chromatograph/Mass Spectrometer

6.17.1 Gas chromatograph: An analytical system that includes a temperature-programmable gas chromatograph including all required accessories such as syringes, columns, and gases.

6.17.2 Mass spectrometer: Capable of scanning from 35 to 300 amu every 1 second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode.

6.17.3 GC/MS interface: An interface that gives acceptable calibration points at 50 ng or less per injection for each of the analytes and achieves all acceptable performance criteria. An all glass interface is recommended.

6.17.4 Data system: A computer system must be interfaced to the mass spectrometer which will allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the analytical run. The software should be capable of producing extracted ion profiles and integrating these abundances. The EPA/NIH Mass Spectral Library should also be available on the system.

7. REAGENTS AND STANDARDS

7.1 Reagent water - Carbon filtered house distilled water, continuously sparged with an inert gas prior to use for volatiles analyses. Interferents should not be observed at or above the reporting limit of the parameters of interest.

7.2 GC/MS Calibration solution: 50 ng/uL bromofluorobenzene in methanol.

7.3 Internal standard and surrogate spiking solutions.
MS-VOA-IS (See SOP No. LS-RMA-010)
MS-VOA-SCS (See SOP No. LS-RMA-011)

7.4 Laboratory Control Sample standard spiking solution.
MS-VOA-LCS (see SOP No. LS-RMA-012)

7.5 GC screening standard. (See RMAL SOP "Volatiles Screening Analysis by GC/FID - SOP No. LM-RMA-3022.)

7.6 Calibration standard solutions:

A. MS-VOA-CS1 (See SOP No. LS-RMA-001)
B. MS-VOA-CS2 (See SOP No. LS-RMA-009)
C. MS-VOA-AP9-CSA (See SOP No. LS-RMA-015)
D. MS-VOA-API-CSA (See SOP No. LS-RMA-013)
E. MS-VOA-B&V-CSA (See SOP No. LS-RMA-039)

7.7 Standards containing other purgeable compounds can be prepared on special request by clients.

7.8 Methanol: purge-and-trap grade or equivalent. Store apart from other solvents.

8. PROCEDURE

8.1 Scheduling Samples for Analysis

8.1.1. Samples will be scheduled for analysis by experienced analysts and/or supervisors according to the following priorities:

a) Meet holding times
b) Meet promised due date

8.2 Methanol extraction of soil samples for analysis by the medium-level method. (minimum reporting limit: 500 ug/kg)

8.2.1 Carefully mix the contents of the sample container. Do not discard any supernatant liquids. Weigh 5 g of sample to the nearest 0.1 g. Note and record the actual weight.

8.2.2 Determine the percent moisture as in section 8.4 (if required).

8.2.3 Add 10 mL purge & trap methanol, (25 ug/mL). Cap and shake for 2 minutes.

8.2.4 Allow the soil to settle or centrifuge and transfer a portion of the extract to a labelled autosampler vial, leaving a minimum of headspace. Store these extracts at 4°C prior to analysis.

8.2.5 Prepare methanol blank for each day of extraction using same procedure as medium level.

8.2.6 Screen the methanol extracts as described in section 8.3

8.3 SCREENING. For complete information on the screening procedures for GC/MS Volatile compounds, refer to RMAL SOP No. LM-RMA-3022, Volatiles Screening Analysis by GC/FID.

8.4 Percent Moisture Determination (optional)

8.4.1 Weigh 5-10 g of sample into a tared weighing dish. Record the samples wet sight. Allow to dry overnight at 105°C. Re-weigh the sample. Record the samples dry weight. (Correct for weight of weighing dish) Calculate percent moisture using equation 10.2.7.

8.5 Instrument Set-Up for the GC/MS

8.5.1 Chromatographic Conditions

8.5.1.2 Mega-bore Capillary Column (See Section 6.17.3)

8.5.1.3 Temperature Program for Capillary Column:
Initial Temperature: -10°C
Temperature Ramp:
Program 1: 20°/min 1.5 minutes to 20°
Program 2: 5°/min 16 minutes to 100°
Program 3: 20°/min 3 minutes to 160°
Final Time: 7 minutes

These parameters are subject to change and are given as guidelines.

8.5.2 Mass Spectrometer parameters:

8.5.2.1 Mass range: 35-300 AMU

8.5.2.2 Scanning rate: 1 second/scan

8.5.3 Purge and Trap Parameters

8.5.3.1 Trap: As described in Section 6.16

8.5.3.2 Purge Flow: 30-50 mL/min (measured at vent in purge mode).

8.5.3.3 Purge pressure: 20 lb/in^2 (measured on the gauge)

8.5.3.4 Purge Time: 12 minutes
Desorb Time: 2-3 minutes
Bake Time: 8-10 minutes

8.6 Installation and conditioning of trap in the purge-and-trap device.

8.6.1 All traps should be installed with accompanying brass nut down and inlet marker to the top. A brass nut and ferrule should be used at the bottom connections. A teflon ferrule should be used at the top. The top nut should only be finger tightened.

8.6.2 Purge flow is adjusted by measuring the flow at the vent in purge mode.

The faster the purge flow the more efficient the purging process. The limit to increasing the flow is governed by the ability of the trap to adsorb and contain the purged analytes.

If the total purge volume is too large (flow rate too high) then the lightest compounds such as the gases will break through the end of the trap and their respective RFs will drop correspondingly. If the purge flow is slow the heavier and the more polar compounds will not purge efficiently. Thus, to improve the response of the CS2 compounds, the purge flow should be decreased and to improve bromoform response the purge flow should be increased.

In general, the purge flow should be between 30 and 50 mL/min.

If optimization of overall RFs by changing the purge flow cannot be attained, a new trap may need to be installed.

8.6.3 The trap is conditioned by placing an empty sparge cell on the purge & trap device and placing the unit into purge mode. After a few minutes, step into "bake" mode for 10-15 minutes.

8.7 Instrument Calibration

8.7.1 BFB

8.7.1.1 Each GC/MS MUST be hardware tuned to meet SW-846 3rd. Edition ion abundance criteria for BFB. The instrument tune MUST be verified at the start of every operation. When the tuning criteria are met, standards and samples may be run for a period of 12 hours depending on contract or method requirements. Ion abundance criteria are listed in SW-846 3rd Edition.

8.7.1.2 50 ng of BFB is directly injected on to GC column and peak data are acquired.

The spectrum of BFB is evaluated. Ion abundance criteria given in Table C-1 must be met. Choosing a scan that is one scan to the left or right of the peak apex and reasonable background subtraction or enhancement is acceptable.

Manipulations which will distort the spectrum, such as excessive background subtraction or multiple scan additions, are not acceptable.

8.7.1.3 If the ion abundance criteria are not met, the instrument is retuned and BFB reinjected.

8.7.1.4 If the ion abundance criteria are met, a hardcopy of the spectrum, mass abundance table, and ion abundance summary is generated and stored on file.

8.7.2 Initial calibration. An initial calibration must be performed before analysis of samples can begin. This consists of a five point calibration curve for the analytes of interest. Low-level soil analysis requires an initial calibration in which the standards in water are heated to 40° C during the purge step. Standard solutions are prepared in reagent water at concentrations of 20 ug/L, 50 ug/L, 100 ug/L, 150 ug/L and 200 ug/L, and purged, desorbed and analyzed on the GC/MS/DS. All 5-points must be injected within 12 hours of BFB injection which has met abundance criteria.

Certain compounds have been selected to monitor instrument performance. These compounds are identified as System Performance Check Compounds (SPCC) and Calibration Check Compounds (CCC).

The average response factor for each System Performance Check Compounds (SPCC) MUST be greater than or equal to 0.300, except for bromoform, which must be greater than or equal to 0.250.

The percent relative standard deviation (%RSD) for each Calibration Check Compound (CCC) MUST be less than 30%. The %RSD for all compounds should also be reviewed. Any values greater than 40% should be evaluated before analyses are continued.

If samples are NOT being analyzed for these specific compounds, the criteria for these compounds need not be met. The fact that these compounds are not in the analyte set should be documented.

See current CLP-SOW for a summary of CCC and SPCC criteria.

8.7.2.1 The five standard solutions are typically analyzed in the following order: 50 ppb, 20 ppb, 100 ppb, 150 ppb and 200 ppb.

Standards are prepared using a 5 mL calibrated gas-tight syringe which is rinsed with purged reagent water and then filled to a volume of 5 mL.

Internal standards, surrogates and analytes are then added.

8.7.2.2 Standards are analyzed using the ALS. Standards are introduced into a purge chamber on the ALS followed by purging & desorption into the GC/MS/DS as previously described.

8.7.2.3 The area counts of the internal standards in the 50 ppb standard are evaluated before calibration is continued. The first internal standard EICP (Bromochloromethane) should be between 15,000-50,000 counts depending on the instrument. This will minimize saturation in the higher level standards. If the peak area is outside these limits, the analyst should adjust the electron multiplier and re-inject the 50 ppb standard.

8.7.2.4 The SPCC and CCC limits MUST be verified and met. If any criteria is not met, the analyst and supervisor will choose appropriate corrective action. Sample analysis cannot take place until CCC and SPCC criteria are met for the initial calibration.

8.7.2.5 The quantitation list is reviewed to ensure that the internal standards are correctly identified and that the analytes are properly quantitated. Special attention is payed to the following compounds to ensure correct peak assignment:

dichlorodifluoromethane
chloroethane
vinyl chloride
acetone
cis-1,3-dichloropropene
trans-1,3-dichloropropene
2-butanone
2-hexanone
4-methyl-2-pentanone
xylene isomers
cis-1,2-Dichloroethene
trans-1,2-Dichloroethane
dichlorobenzene isomers
cis-1,2-Dichloroethene
trans-1,2-Dichloroethene

8.7.2.6 If the standard evaluation criteria have been met, update with the response factors and retention times of the 50 ppb standard just analyzed. Standards and samples are processed.

8.7.2.7 The 20 ppb, 100 ppb, 150 ppb and 200 ppb standard are then analyzed and evaluated in the same manner as the 50 ppb standard, using programs on the data system designed for multipoint calibration data.

8.7.2.8 A table which lists each standards Response factors, the average Response factors and % RSD for each compound is generated. The % RSD for CCC and Response factors or SPCC are then evaluated. Compounds having % RSD greater than 30% are checked to verify accuracy of quantitation of each standard. Response factors which differ greatly from the compound average are also investigated.

8.7.2.9 If all CCC and SPCC criteria are met, the data are stored in an Initial Calibration file. This file is then referred to during continuing calibration.

8.7.3 Continuing calibration

The 50 ppb standard is used as the continuing calibration standard. It MUST be analyzed for each twelve hour period, immediately following a successful BFB analysis. The BFB injection time starts the twelve hour clock.

The response factor of each CCC compound should be compared to the initial calibration using multipoint calibration software. The percent difference for each CCC compound should be less than or equal to 25%. Analyses may continue if any one CCC or any one SPCC is not within limits but only if contractually allowable or approved by a supervisor. Response factors for all analytes should be reviewed. Any significant deviations from the initial calibration should be evaluated and quantities checked before analyses are continued. (e.g. deviations of greater than 30%)

8.7.3.1 The continuing calibration standard is run as described in section 8.7.2.1. The concentration of each of the surrogate compounds and the internal standard compounds is 50 ppb.

8.7.3.2 Process the data file as described in Section 8.7.2.2. (See Section 8.10.1).

8.7.3.3 The quantitation list is reviewed to ensure that the internal standards are correctly identified and that the analytes are properly quantitated. Check the compounds listed in 8.7.2.5 for proper integration and peak assignment.

8.7.3.4 A program on the data system is used to compare the continuing calibration standard to the initial calibration. The CCC and SPCC compounds are evaluated as outlined in Section 8.7.3. The RF and percent deviations from the initial calibration of all the other compounds are reviewed. (e.g. compounds with deviations > 30% are checked for proper peak assignment and integration.)

8.7.3.5 If the standard evaluation criteria are NOT met, corrective action must be taken. Corrective action may include replacement of the trap or the analytical column or replacement of calibration standards which may have degraded. Document corrective action taken. Repurge the continuing calibration standard and/or perform a new initial calibration. (See Section 8.7.2.)

8.7.3.6 If these criteria are not met and the decision is made to continue analysis, the rationale must be documented, approved by a supervisor, and kept on file with the continuing calibration record.

8.7.3.7 If the standard evaluation criteria have been met or the decision has been made to continue, update the data system with the Response factors and retention times of the continuing calibration standard just analyzed.

8.8 File Naming Conventions

The file names of BFB, standards, QC, and samples acquisitions are based on the instrument letter designation, and a continuing numeral integer progression from 0001 to 9999. When file number 9999 is reached, go to 0001. File names are recorded in the instrument log book on a daily basis as acquisitions are conducted.

8.9 Sample Analysis

8.9.1 Scheduling

8.9.1.1 Analyses are scheduled by the supervisor or a senior analyst generated from the Laboratory Information Management System (LIMS). Backlogs are available in the laboratory.

8.9.1.2 The analyst will determine the type of analysis requested. Modifications to the normal analytical protocol such as low detection limits, special compounds or special criteria will be indicated on the backlog or found in the LIMS database.

8.9.1.3 Sample ID's are tracked based on RMA project and sample numbers assigned by Sample Receiving. These ID's should be used when entering the "Sample ID" accounting parameter in data acquisition programs.

8.9.2 Analysis Sequence

8.9.2.1 A method blank (surrogate control sample) must always be analyzed after a calibration standard. See section 9.3 for method blank acceptance criteria. Duplicate control samples (DCS) are analyzed as required by SOP No. M-EQA-002. SCS and DCS sample results must be verified before samples can be analyzed. See Section 9.0 for QC criteria for SCS and DCS samples.

8.9.2.2 If the QC criteria are met, the sample analysis can begin. Consult the Enseco QAPP for specific requirements. Matrix spike and matrix spike duplicates and any other Project Specific QC when requested by the client, are analyzed in conjunction with the associated sample.

8.9.2.3 Samples must be injected within 12 hours of the BFB injection, unless a method or contract specifies otherwise.

8.9.3 Analysis of Water Samples

8.9.3.1 All samples must be allowed to warm to ambient temperature before analysis.

8.9.3.2 All purging cells are washed with soap and hot water, rinsed with water, then methanol and baked dry.

8.9.3.3 Screen data is first reviewed to determine if any samples require dilutions. When screens indicate samples are clean, or have low levels of volatile compounds, 5 mL will be used for analysis. Sample volume will be measured using a 5 mL gas-tight syringe. If screens indicate high levels of volatile compounds are present, sample volume will be adjusted so that the compound in highest concentration will quantitate at approximately 100 ppb. The final volume of sample is adjusted with reagent water so that 5 mL is always purged. Dilutions of up to 1:1000 (5 ul of sample or more) can be made directly in the 5 mL syringe. If necessary, serial dilutions of 1:10 are made so that 5 ul of sample will be the minimum volume injected into the 5 mL syringe.

8.9.3.4 The sample is carefully poured into the syringe barrel nearly filling the barrel. Vent any residual air while adjusting the samples volume to 5.0 mL. A second syringe may be filled at this time (if capped off), if there is only one vial for analysis. This will maintain the integrity of the sample. A sample may be stored, refrigerated, in a syringe for 24 hours.

8.9.3.5 Add 10 uL of the internal standard + surrogate (VOA IS/SCS) mix to the sample using 25 uL gas-tight syringe.

8.9.3.6 The syringe valve assembly is attached to the valve on the purging chamber. The valve on the chamber is opened and the sample is injected into the purging chamber.

8.9.3.7 The valve on the purging chamber is closed and the syringe is removed. The sample is then purged and analyzed using the same instrument parameters as those used to analyze the calibration standards.

8.9.3.8 Sample number and sample concentrations are recorded in the instrument log book.

8.9.3.9 Surrogate control samples and duplicate control samples are prepared using 5.0 mL of purged reagent water. Add 10 uL of internal standard and surrogate solution (VOA IS/SCS) to the water in the syringe. For DCS samples, add 10 uL of DCS spike solution in addition to the surrogates and internal standards. Place each aliquot into adjacent purge vessels. SCS must meet blank criteria described in Section 9.3.

8.9.3.10 Matrix spikes are prepared in the same manner. The additional aliquot(s) of the sample to be spiked is (are) placed in 5 mL gas-tight syringes. If the sample requires dilution, dilute the matrix spike aliquot as well. Add 10 uL of the internal standard + surrogate (VOA IS/SCS) mix and 10 uL of the matrix (VOA-DCS) spike solution to each aliquot prepared. Analyze the sample and matrix spike(s) consecutively, if possible.

8.9.4 Analysis of Methanol Extracts for the Medium Soil Method

8.9.4.1 The samples are extracted and screened as described in Section 8.2 and 8.3.3. A medium level blank consisting of 5 mL reagent water, 10 mL IS/SCS mix and 100 ul of methanol is run with each set of medium level samples.

8.9.4.2 A 5 mL gas-tight syringe is filled with purged reagent water. Adjust the volume to 5 mL. If screening data indicates sample requires between 5 uL and 100 uL of extract, then the appropriate volume of the methanol extract is added to the reagent water. If less than 5 uL of extract is indicated, a 1:10 dilution in Methanol is required. The extract is further diluted as necessary so that at least 5 uL of diluted extract is added to the reagent water purged. The maximum amount of Methanol extract that should be purged is 100 mL. It is acceptable to add the extract directly to the syringe, or directly to the reagent water after it has been added to the purge cell. Use Hamilton microliter syringes to deliver the 5 uL to 100 uL aliquot.

8.9.4.3 Add 10 uL of VOA IS/SCS mix to the syringe and place the sample in the purging chamber as described above.

8.9.4.4 Matrix spike samples are prepared at the instrument. The same volume of extract used in the sample analysis is used in the matrix spike. The matrix is prepared as described in sample analysis using 5 mL reagent water, 10 uL VOA IS/SCS mix, and 10 uL DCS mix.

8.9.5 Low Level Soil Analysis

8.9.5.1 QC SCS and DCS samples will be analyzed as described in Section 8.9.3.9, with one exception, soil DCS samples are to include 5 grams of Ottawa sand.

8.9.5.2 The screen data is required to determine the weight of samples to be purged (between 0.5g and 5.0g).

8.9.5.3 Mix the contents of the sample container. Do not discard any supernatant liquids. The amount of sample to be purged as determined by the screen is weighed into a tared purge vial. Note and record the actual weight to the nearest 0.1 g.

8.9.5.4 Prepare a 5.0 mL aliquot of reagent water containing 10 uL of VOA IS/SCS solution in a 5 mL syringe. Add the water to the vial, purge vial + sample by injecting the water through the valve on the purge and trap device as the purge cell is lifted into position. This will mix the soil and water and ensure the helium purge tube will not be blocked. Low level soils (and their corresponding calibrations) are purged at a temperature of 40°C.

8.9.5.5 Repeat steps 8.9.5.3 and 8.9.5.4 for each sample.

8.9.5.6 Surrogate control samples and duplicate control samples are prepared as stated in section 8.9.3.9 with one exception. Each DCS sample also contains 5.0 grams of a solid, contaminant-free matrix, such as Ottowa Sand. No solid matrix is used. Add 10 uL of VOA IS/SCS solution to the water in the syringe. For DCS samples, also add 10 uL of matrix spike (VOA DCS) solution. Place each aliquot into a purge vial and affix to the unit as described above.

8.9.5.7 Matrix spikes are prepared using the same procedure as the samples. Weigh the same amount of sample for each aliquot required. In addition to the 10 uL of VOA IS/SCS added to the reagent water, 10 uL of DCS solution is added. Matrix spike samples should be analyzed along with the corresponding sample. Affix each aliquot to the unit as described above.

8.9.6 Data Acquisition

8.9.6.1 Samples are analyzed using the same instrument parameters as the calibration standards. Recommended conditions are listed in section 8.5. Blank, SCS and DCS data must be verified before sample analysis.

8.9.6.2 Sample data is acquired either as a single run, or as in an automated series of sample runs.

If a limited list of compounds is being used, the run time (number of scans desired) may be shortened, as appropriate to the compounds being analyzed. The actual number or scans collected may vary slightly from one instrument to another.

8.9.6.3 File names, the date, analyst initials, sample identification and appropriate dilution are recorded in the instrument log book.

8.10 Data Processing and Reduction

8.10.1 Following sample data acquisition, each sample data file is processed using the same software used to search for, identify and quantitate target compounds as was used for calibration data.

8.10.2 The data package generated contains many of the following printouts:

8.10.2.1 **Log Page:** Included on this page are the header information, GC conditions, scan parameters, and instrument tuning parameters that were used at the time of data acquisition.

8.10.2.2 **Chromatogram:** Included on this page is the header information and the reconstructed ion chromatogram displaying the range of scans used during data acquisition.

8.10.2.3 **Diagnostic Report:** Included in this page is a Table of Library entries and chromatographic and Library search information used in the computerized target compound evaluation.

8.10.2.4 **Quantitation Report:** Included in this section is the list of target compounds, internal standards and surrogate standards used for processing the data file, and compound library entry numbers. This section contains: **1)** Chromatographic information such as retention times (scan numbers) and relative retention times and, **2)** Quantitation information such as peak areas, updated library response factors, calculated response factors and quantitated amounts for all library entries.

8.10.2.5 **Quantitation Summary:** Included on this page is a list of internal standards, surrogates and those target compounds which were found in the AutoQuan target compound search routine. Listed along with each compound is the mass, scan, indication of internal reference standard, peak area, response factor and quantitation amounts.

8.10.2.6 **Spectra:** A mass spectra of the peak or each target compound is printed.

8.10.2.7 **Summary Cover Page:** Sample summary which lists surrogate recoveries + all target compounds. These target compounds which were found to have quantitation amounts, have dilution adjustments.

8.10.3 Internal Standard area counts must be monitored on all sample and QC runs. Areas or Percents are recorded in the log book. The areas should not vary by more than a factor of two (-50% to +100%). If the areas do vary by more than a factor of two, the analyst should first check to see if the peak has been correctly integrated. If the area is still outside the limits, the purge & trap/GC/MS system should be checked and corrections made if necessary. The sample will be reanalyzed if problems were detected and corrected. If, upon verification that the instrument is operating properly, the areas are still outside of the limits, submit both analyses with the data package and document the actions taken. If no problems were detected with the instrument, involve a supervisor to determine if sample reanalysis is necessary.

8.10.4 Surrogate recovery values are calculated and compared to recovery limits in Table D-2. Samples with surrogate recoveries outside QC limits should be checked for proper quantitation. Secondary ions may be used if there are interferences on primary quantitation ions. CLP contracted samples having surrogates outside the limits are reanalyzed. If surrogates are outside limits for the second analysis, both analyses are submitted with data package. Non-CLP samples, with one or more surrogates outside limits, are documented and evaluated on a case-by-case basis. Need for reanalysis will be determined by the analyst and supervisor.

8.10.5 Verify that the sample was analyzed at the proper dilution. If any sample contains target compounds that are greater than the highest point in the initial calibration plus 10% (over 220 ug/L on the QUAN report), the sample should be diluted by the appropriate factor and reanalyzed. Exceptions to this include acetone and 2-butanone for non-CLP work which have demonstrated linearity to 1200 ug/L and 500 ug/L respectively. Any sample that has been diluted should contain levels of compounds (either target or non-target compounds) that are high enough to justify the dilution. In the absence of target compounds, the peak height of non-target compounds should be approximately greater than or equal to the peak height of the nearest internal standard. If not, the sample should be reanalyzed at the correct dilution.

8.10.6 Spectra Verification

The data processing routines generate spectra for all target compounds > 1 ug/L for analyst interpretation. Each positive hit picked by the data system, must be examined by the analyst to either confirm or negate what the processing routine has chosen.

Prior to quantitative identification of target compounds in a sample "standard" mass spectra of known identity should be obtained on the instrument(s) used for sample analysis. Hardcopies of mass spectra from a calibration standard (e.g. 50 ug/mL continuing calibration standard) run on the instrument under similar tuning conditions would serve this purpose.

8.10.6.1 Each mass spectrum generated for a sample data file should be compared to the standard spectrum. EPA criteria for quantitative identification by comparison of mass spectra should be followed. The EPA criteria for comparing spectra are:

1) All ions present in the standard spectrum at a relative intensity greater than 10% of the base peak MUST be present in the sample spectrum.

2) The relative intensities of the ions specified in (1) must agree within plus or minus 20% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard specrtum, the corresponding sample ion abundance must be between 30% and 70%.)

3) Ions greater than 10% in the sample spectrum, but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. If the analyst can easily identify a co-eluting compound (e.g. an alkane or a surrogate spike compound) then the sample spectrum should be labelled with the contaminant.

8.10.6.2 The analyst should circle the name of target compound on the spectrum hard copy of spectra that meet the criteria listed in Section 8.10.6.1. All spectra that do not meet the EPA criteria should be labelled with the word "No" and the corresponding entries on the quantitation

summary should be crossed out (line out the amounts calculated for those compounds which are not "hits") by the analyst making the judgement.

8.10.6.3 If a compound cannot be verified by all of the criteria listed in 8.10.6.1, but in the technical judgement of the analyst the identification is correct, the compound should be reported.

8.10.7 Summary Sheets

A number of summary sheets are computer generated at the instrument following data file processing and pulling (hardcopying) target compound spectra. These summaries include:

8.10.7.1 Target Compound Summary (Also referred to as "Sum Sheets"): This one or two page summary tabulates pertinent information about a sample analysis including header information (e.g. sample number, dilution used for analysis, sample matrix), data analyzed, analyst's initials, run factor (refer to calculations in Section 10.1.1, final reporting units, surrogate % recoveries, target compounds, amounts >1.0 ug/L, scan number, amount listed on quantitation summary and concentration in sample (expressed in final reporting units, usually ug/L for waters and ug/kg for soils) are tabulated.

8.10.7.2 DCS Summary (Refer to section 9.0 for discussion of DCS QC samples): This one page summary tabulates pertinent information about the DCS samples representing a specific QC lot including QC Lot number, matrix (soil or water), date analyzed, analyst's initials, DCS data file

numbers, list of samples belonging to the QC lot and the QC limits. The program calculates the amount and average percent recovery for each spike compound and the RPD (Relative Percent Difference) between the two DCS recoveries for each spike component. Percent recoveries and RPD's that fall outside the acceptable QC limits are flagged with an asterisk (*) and require further action by the analyst (see Section 9.0).

When generating this summary sheet the analyst can input a complete list of samples belonging to the QC lot for QC tracking purposes. This information can be found in the QC summary notebook kept at each instrument.

8.10.7.3 SCS Summary (See Section 9.0 for discussion of QC samples): This one page summary tabulates pertinent information about the surrogate control sample representing a specific ongoing calibration. Included are QC lot number, matrix (soil or water), date analyzed, analyst's initials, data file number, list of samples belonging to the lot, names of surrogates and QC limits for surrogate % recoveries, and calculated amount and % recovery for each surrogate. Percent recoveries that fall outside the acceptable QC limits are flagged with an asterisk (*) and require further action by the analyst see Section 9.0). When generating this summary sheet, the analyst inputs the complete list of samples associated with the SCS sample for QC tracking and data reporting (Blank Subtraction) purposes. This information can be found in the QC summary note book kept at each instrument.

8.10.8 Library Searches (Optional)

Some data packages will include mass spectral library searches on non-target compounds (excluding internal standards, surrogates & spikes) as outlined in contract requirements or requested by the client. If library searches (also referred to as "TIC's", "TID's" or Tentatively Identified Compounds) are necessary to the data package, the 10 or 15 largest unidentified peaks that are greater than 10% of the peak (RIC) area of the nearest internal standard will be searched against the NBS library for possible matches.

8.10.8.1. The generation of TIC data is partially automated through the use of several RMA programs which perform the following functions:

1) Select non-target, unidentified peaks from the chromatogram for tentative identification,

2) Quantitate unknown peaks using RIC area of closest uninterfered standard as reference peak using a response factor of 1 and run factor adjustment. (See Section 10.2.8 for run factor calculations)

3) Perform and generate hardcopy mass spectral library searches on the unknown peaks (searching against the NBS library of mass spectra),

4) Select preliminary "Best Match" spectra from the library searches and tabulate them with quantitation information for all unknowns,

5) Allow editing of computer's final selections for compound names,

6) Allow manual quantitation of unusually shaped peaks,

7) Generate final summary with amounts calculated to reflect concentration in sample (i.e. run factor applied).

8.10.8.3 After reviewing each library search spectrum, the analyst should indicate the compound name selected by labelling the spectrum (or circling the correct name if it appears on the library search spectrum as one of the computer's selections). If the compound is to be reported as "Unknown" (See Section 8.11.6.5) the spectrum should be labelled as such.

8.10.8.4 When all of the library comparisons have been printed out, a summary sheet listing each peak with the best library fit will print out or be filled out by the analyst. The operator should go through each comparison, determine the probability that the match is correct, and assign a confidence level based on that probability. The confidence levels are:

3 = High, verfied by an authentic standard
2 = Moderate
1 = Low

In addition, two qualifiers,
I = Isomer
C = Chemical Class
can be assigned to the identification.

8.10.8.5 The EPA criteria to be used for making these decisions for tentative identifications are as follows:

1) Relative intensities of major ions present in the library spectrum at a relative intensity greater than 10% of the base peak should be present in the sample spectrum.

2) The relative intensities of the ions specified in (1) must agree within +/- 20% between the library and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%.)

3) Molecular ions present in the library spectrum should be present in the sample spectrum.

4) Ions present in the sample spectrum, but not present in the standard spectrum should be considered and accounted for by the analyst making the comparison. If the analyst can easily identify a co-eluting compound (e.g. an alkane or a surrogate spike compound) then the sample spectrum should be labelled with the contaminant.

5) Ions present in the library spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting compounds.

8.10.8.6 If in the technical judgement of the analyst, no valid tentative identification can be made, the compound should be reported as unknown. The analyst should consult with a more experienced analyst or a supervisor if there are questions concerning compound identification.

8.10.9 Label the RIC to identify the internal standards surrogate compounds, target compounds, and TIC if requested. If the sample is particularly dirty and the peaks are buried in other peaks, estimate the location of the peak.

For industrial, non-CLP work the abbreviations IS-internal standard, SS-surrogate standard, SP or SC-spike compound, TC-target compound and TIC-tentatively identified compound are sufficient. In CLP work the internal standards and surrogates are usually stamped with the compound names; the abbreviations SC, TC and TIC are normally sufficient for contract work for spike compounds, target compounds and tentatively identified compounds provided a legend is clearly indicated on each chromatogram page.

8.10.9.1 Each data package submitted by the analyst for secondary review must include an anomaly sheet summarizing any problems or unusual circumstances incurred during sample analysis. If no problems were encountered during analysis, the analyst must still submit an anomaly sheet to that effect. The supervisor must sign the anomally sheet before the package progresses to the data review group.

8.10.9.2 QC Summary Sheets (SCS & DCS)

Each data package submitted for secondary review must include copies of DCS and-SCS summary sheets for all QC related to the sample set in the data package.

8.10.10.3 Instrument and sample data are organized by the analyst at each instrument. Instrument tuning records and BFB data are filed at the instrument. STD, SCS, and DCS Raw data are filed separately by date at the instrument. If the above data is required as part of the client deliverable package, copies are made and added to the data package assembly. The data package assembly will

include hardcopies of printouts described in Section 8.10.11 for each sample. TIC library comparison spectra and summaries are included when required.

8.10.11 Summary of Data Package Requirements

When initial data review has been completed by the analyst the data package should be assembled to include the following:

Anomaly Sheet
DCS Summary Sheet
SCS Summary Sheet
Blank Target Compound Summary Sheet
Calibration Data
Out of Control Forms (if necessary) see Section 9.0
Sample Raw Data
Sample Target Compound Summaries (attached to sample raw data)

TIC data and summary (when requested) in some cases contract requirements or client requests will necessitate including tuning and calibration information (i.e. BFB), daily standard raw data and initial and continuing calibration response factor tables.

8.10.12 Upon completion of the data package assembly and review, the data package is passed on to the data review specialist for Level 2 review.

9. QA/QC Requirements (See SOP no.: M-EQA-002)

9.1 DCS. Duplicate laboratory control samples are analyzed for every 20 samples. DCS components are spiked into organic-free water. A solid matrix (Ottawa sand) is incorporated as a representative solid matrix. Aqueous and Solid DCS Q.C. lots are maintained separately.

9.2 SCS. A single control sample is analyzed with every analytical lot. This sample serves as the method blank. An analytical lot for volatile organics is defined as samples analyzed within a twelve hour period.

9.3 Blank. A Method Blank is run with each daily calibration. For samples run under Enseco QC guidelines the SCS will serve as the Method Blank. Medium level soils will also require a medium level blank. All target compounds should be below reporting limits. Consult with a supervisor if any target compound is present at or above rep. limit. Surrogate recoveries must fall between limits set for SCS acceptance. Reanalyze blank or SCS if surrogate recoveries are outside of limits. If recoveries still out, troubleshoot system and recalibrate if necessary.

9.4 Surrogates Spiked into Samples. Surrogate compounds are required to be spiked into all samples and QC samples for this method.

9.5 Matrix specific QC. Matrix spike and matrix spike duplicates are performed at the request of the client. Matrix spike compounds will be the same as those used for the DCS. These compounds will be spiked into aliquots of the sample specified by the client at the same concentration level as the DCS.

9.6 Data acceptability is based upon the results of the Duplicate Control Samples unless otherwise negotiated with the client. QC data must fall within established control limits in order for the laboratory to be considered "in control" when samples from that QC lot were analyzed. An out-of-control form must be filled out if any of the DCS or SCS criteria is not met.

9.6.1 At least 80% of the DCS average recovery data, and at least 80% of the DCS precision data and at least 80% of the SCS recovery data must be within established control limits in order for the laboratory to be considered "in control". Blank values must be acceptable. For this method the maximum number of values permitted outside control limits are:

	average recovery	precision
DCS:	1	1
SCS:	0	

9.6.2 Control Limits: Control limits can be found in the current CLP-SOW. Data will be accumulated and limits statistically adjusted over time.

9.6.3 Holding blanks will be run at a frequency determined by the Enseco QC Department. A holding blank consists of a VOA vial properly filled with purged reagent water. These will be analyzed at a frequency that reflects the maximum allowable holding time of a sample.

9.6.4 When trip blanks are generated by the laboratory to be shipped to a sampling site, a separate blank will be prepared on a weekly basis. This blank is analyzed for volatile organics and the data evaluated for contaminants, to ensure that contaminant-free trip blanks are sent into the field.

10. Calculations

10.1 Calculations required to reduce data

10.1.1 Concentration factor for sample (CF):

CF = NOMINAL SAMPLE SIZE/ACTUAL SAMPLE AMOUNT USED

The nominal sample size is 5 mL for water and 5 g for soil.

10.1.2 VOA SURROGATE CONCENTRATION FACTOR (SCF):

SCF = (Vol purged X 100) / amt spiked (total ng)

10.2 Calculations required to verify automated data reduction

10.2.1 Response factors

$$RF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

A_x = Area of the characteristic ion for the compound to be measured.

A_{is} = Area of the characteristic ion for the specific internal standard.

C_{is} = Concentration of the internal standard (ng/uL).

C_x = Concentration of the compound to be measured (ng/uL).

10.2.2 Average response factor

$$RF_{ave}=[(RF_{20})(2.5)+RF_{50}+(RF_{100}/2)+(RF_{150}/3)+(RF_{200}/4)]/5$$

Where RF_x = Response factor of a compound in each of the calibration solutions.

10.2.3 Percent Relative Standard Deviation

$$\%RSD = \frac{SD}{RF_{ave}} \times 100$$

Where:

RSD = Relative Standard Deviation

SD = Standard Deviation of initial response factors (per compound)

Where: $$\overline{SD} = \sum_{i=1}^{N} \sqrt{\frac{(x_i - x)^2}{N-1}}$$

RF_{ave} = mean of initial response factors (per compound)

10.2.4 Percent Deviation from average response factor

$$\% \text{ Difference} = \frac{RF_I - RF_C}{RF_I} \times 100$$

Where:

RF_I = average response factor from initial calibration.

RF_C = response factor from current calibration check standard.

10.2.5 Percent Recovery

$$\text{Matrix Spike Percent Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where:

SSR = Spike Sample Results
SR = Sample Result
SA = Spike Added from spiking mix

10.2.6 Relative percent difference

$$RPD = \frac{D_1 - D_2}{(D_1 + D_2)/2} \times 100$$

Where:

RPD = Relative Percent Difference
D_1 = First Sample Value
D_2 = Second Sample Value (duplicate)

10.2.7 Percent moisture

$$\frac{\text{wet (sample+dish)} - \text{dry (sample+dish)}}{\text{wet (sample+dish)}} \times 100 = \%\ \text{moisture}$$

10.2.8 Run factors: CF are multiplied by the actual T.C. concentration found in the quan list to get the concentration found in sample.

10.2.8.1 Aqueous:

$$CF = \frac{.005\ l}{XL}$$ where X = Volume of sample used.

10.2.8.2 Soil (Low Level):

$$CF = \frac{.005\ kg}{Xkg}$$ X = Weight of sample used.

Projects which require % moisture corrections

$$CF = \frac{0.005\ kg}{Xkg}\ \left(\frac{1-\%\ moisture}{100}\right)$$

10.2.8.3 Medium Level:

$$CF = \frac{.005\ l\ (10\ ml)}{(Xkg)(YmL)}$$ X=amount of sample used in extraction

Y=volume of MEOH from extr act used

Projects which require % moisture

$$CF = \frac{005\ l\ (10\ ml)}{Xkg(YmL)\left(1-\frac{\%moisture}{100}\right)}$$

11. Reporting requirements

11.1 Units: Aqueous samples will be reported in ug/L
Solid and sludge samples will be reported in ug/Kg or mg/kg on a wet weight basis, unless requested otherwise by the client.

11.2 Reporting limits: Test Specific

Reporting limits will be adjusted according to sample dilution.

11.3 Significant figures: All data <10 ug/L will be reported with one significant figure. All other data will be reported with two significant figures.

11.4 LDMS data entry

12. References

12.1 Method Source: Method 8240, SW-846 Third Edition, USEPA, Office of Solid Waste and Emergency Response, Washington, DC 20460, September 1986.

12.2 Deviations from Source Methods

12.2.1 Deviations from the specified criteria for continuing calibration are allowed, provided the rationale is documented. Additionally, non-routine compounds may be analyzed for using a single, midpoint calibration.

12.2.2 An internal QA/QC program has been adopted by Enseco, Inc. This is described in Enseco's "Quality Assurance Program Plan for Environmental Chemical Monitoring." Specific elements of the QC program are described in SOP NO. M-EQA-002: "Internal QC Checks--Laboratory Performance QC." The laboratory control samples generated in this program are used to monitor method performance. Matrix spikes are performed at the request of the client.

12.2.3 The medium level method for soil referred to in this SOP is equivalent to the High-level method described in method 8240. The only exception is that this SOP uses a 5 g sample compared to the 4 g sample recommended in Method 8240.

INTERNAL STANDARDS AND SURROGATES

- BROMOCHLOROMETHANE
- 1,4-DIFLUOROBENZENE
- CHLOROBENZENE-D5
- 1,2-DICHLOROETHANE-D4
- TOLUENE-D8
- BROMOFLUOROBENZENE

CHLOROMETHANE
BROMOMETHANE
DICHLORODIFLUOROMETHANE
VINYL CHLORIDE
CHLOROETHANE
METHYLENE CHLORIDE
ACETONE
CARBON DISULFIDE
TRICHLOROFLUOROMETHANE
1,1-DICHLOROETHENE
1,1-DICHLOROETHANE
TRANS-1,2-DICHLOROETHENE
CHLOROFORM
TRICHLORO-TRIFLUORO-ETHANE
1,2-DICHLOROETHANE
1,4-DIOXANE
2-BUTANONE
1,1,1-TRICHLOROETHANE
CARBON TETRACHLORIDE
VINYL ACETATE
BROMODICHLOROMETHANE
1,2-DICHLOROPROPANE
CIS-1,3-DICHLOROPROPENE
TRICHLOROETHENE
DIBROMOCHLOROMETHANE
1,1,2-TRICHLOROETHANE
BENZENE
TRANS-1,3-DICHLOROPROPENE
1,2-DIBROMOETHANE
2-CHLOROETHYL VINYL ETHER
BROMOFORM
4-METHYL-2-PENTANONE
2-HEXANONE
TETRACHLOROETHENE
1,1,2,2-TETRACHLOROETHANE
TOLUENE
CHLOROBENZENE
ETHYLBENZENE
STYRENE
M-XYLENE
O+P XYLENES
M-DICHLOROBENZENE
O-DICHLOROBENZENE
P-DICHLOROBENZENE

INTERNAL STANDARDS
- BROMOCHLOROMETHANE
- 1,4-DIFLUOROBENZENE
- CHLOROBENZENE-D5

DICHLORODIFLUOROMETHANE
ACETONITRILE
IODOMETHANE
ACROLEIN
ACRYLONITRILE
TRICHLOROFLUOROMETHANE
ETHYLCYANIDE
3-CHLOROPROPENE
DIBROMOMETHANE
METHACRYLONITRILE
ISOBUTYL ALCOHOL
1,4-DIOXANE
2-CHLORO-1,3-BUTADIENE
1,2-DIBROMOETHANE
1,1,1,2-TETRACHLOROETHANE
1,2,3-TRICHLOROPROPANE
T-1,4-DICHLORO-2-BUTENE
1,2-DIBROMO-3-CHLOROPROPANE

GC/MS ANALYSIS OF VOLATILE ORGANICS

SOP No.: LM-RMA-3001 VOA by GC/MS	Revision No.: 3.0	Effective Date: 4/24/90

Appendix B Tables of Solutions Used in this Method - To Include Analyte List, Concentration of Solution, and Amount to Use to Spike Samples or Inject

Table B-1 VOA-IS/SCS
Table B-2 VOA-DCS
Table B-3 Calibration Standard Solutions
Table B-4 Preparation of Standard for 5-Point Calibration

Table B-1 VOA IS/SCS

Compounds	Standard Concentration ug/mL	Volume Added
Bromochloromethane	50	5 ul in 5 mL
1,4-Difluorobenzene	50	
Chlorobenzene-D5	50	
Tuluene-D8	50	
Bromofluorobenzene	50	
1,2-Dichloroethane-	50	

Table B-2 VOA-DCS

Compounds	Units ug/mL	Volume Added
1,1-Dichloroethene	50	5 ul in 5 mL
Tichloroethene	50	
Chlorobenzene	50	
Toluene	50	
Benzene	50	

Table B-3 Calibration Standard Solutions

A. CS1

Compounds	Units ug/mL	Volume Added
Chloromethane	50	5 ul in 5 mL
Bromomethane	50	
Vinyl Chloride	50	
Chloroethane	50	
Dichlorodifluoromethane	50	
Trichlorodrifluoromethane	50	

Table B-3 Calibration Standard Solutions

B. CS2

Compounds	Units ug/mL	Volume Added
Methylene Chloride	50	5 ul in 5 mL
Acetone	50	
Carbon Disulfide	50	
Trichlorofluoromethane	50	
1,1-Dichloroethene	50	
1,1-Dichloroethane	50	
Trans-1,2-Dichloroethene	50	
Chloroform	50	
1,2-Dichloroethane	50	
2-Butanone	50	
1,1,1-Trichloroethane	50	
Carbontetrachloride	50	
Vinyl Acetate	50	
Bromodichloromethane	50	
1,2-Dichloropropane	50	
Cis-1,3-Dichloropropene	70 *	
Trichloroethene	50	
Dibromochloromethane	50	
1,1,2-Trichloroethane	50	
Benzene	50	
Trans-1,3-Dichloropropene	29 *	
Bromoform	50	
4-Methyl-2-Pentanone	50	
2-Hexanone	50	
Tetrachloroethene	50	
1,1,2,2-Tetrachloroethane	50	
Toluene	50	
Chlorobenzene	50	
Ethylbenzene	50	
Styrene	50	
O-Xylene	50	
M+P Xylenes	50	
M-Dichlorobenzene	50	
O-Dichlorobenzene	50	
P-Dichlorobenzene	50	

* = Cis and Trans Isomers may vary over time requiring adjustments of Library Update amounts.

Table B-3 Calibration Standard Solutions

C. API

Compounds	Units ug/mL	Volume Added
1,4-Dioxane	1000	5 ul in 5 mL
1,2-Dibromoethane	100	
Isobutanol	100	
1,1,1,2-Tetrachloroethane	50	

Table B-3 Calibration Standard Solutions

D. Appendix IX

Compounds	Units ug/mL	Volume Added
Acetonitrile	50	5 ul in 5 mL
Iodomethane	100	
Acrolein	50	
Acrylonitrile	50	
Ethylcyanide	50	
3-Chloropropene	50	
Dibromomethane	50	
Metharcrylonitrile	50	
Isobutyl Alcohol	100	
2-Chloro-1,3-Butadiene	50	
1,1,1,2-Tetrachloroethan	50	
1,2,3-Trichloropropane	50	
T-1,4-Dichloro-2-Butene	50	
1,2-Dibromo-3-Chloroprop	100	

Table B-4 Preparation of Standard for 5-Point Calibration Curve

A. Water and Low-Level Soil

The amount of standard solutions added to the reagent water for each standard level will be identical for the water and low-level soil. The low-level soil standards will be heated to 40°C when purged.

Standard Level	Vol of Purge Water	Vol of IS + Surrogate	Vol of Std	Vol of Gases
50 ppb	5 mL	10 uL	5 uL	5 uL
20 ppb	5 mL	10 uL	2 uL	2 uL
100 ppb	5 mL	10 uL	10 uL	10 uL
150 ppb	5 mL	10 uL	15 uL	15 uL
200 ppb	5 mL	10 uL	20 uL	20 uL

B. Medium-Level Soil

The standards for the medium level soil are prepared as above for water. The same curve is used for water and medium level soil. The volume of standard is the same. The standard levels are identical when the appropriate sample equivalent is taken into account. That is, for a medium-level soil analysis 0.05 g or 50 mg of soil is purged while for water 5 mL or .005 L is purged.

Appendix C

Table C-1

BFB KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	15.0 - 40.0 percent of the base peak
75	30.0 - 60.0 percent of the base peak
95	base peak, 100 percent relative abundance
96	5.0 - 9.0 percent of the base peak
173	less than 1.00 percent of the base peak
174	greater than 50.0 percent of the base peak
175	5.0 - 9.0 percent of mass 174
176	greater than 95.0 percent but less than 101.0 percent of mass 174
177	5.0 - 9.0 percent of mass 176

Table C-2

CALIBRATION CHECK COMPOUNDS

Initial Calibration: The percent relative standard deviation for the each compound listed below should be <30%.

Continuing Calibration: The percent deviation from the average response factor determined in the initial calibration should be <30% for each compound listed below.

1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl Chloride

SYSTEM PERFORMANCE CHECK COMPOUNDS

Initial Calibration: The average response factor for each compound listed below should be >0.300, 0.250 for Bromoform.

Continuing Calibration: The response factor for each compound listed below should be >0.300, 0.250 for Bromoform.

Chloromethane
1,1-Dichloroethane
Bromoform
1,1,2,2-Tetrachloroethane
Chlorobenzene

STANDARD
OPERATING
PROCEDURE

Subject or Title: MS-VOA-CS1		Page 1 of 2
SOP No.: LS-RMA-0001	Revision No.: 4.1	Effective Date: 05/26/92
Supersedes: Revision 4.1		

1. TYPE OF STANDARD: VOA HSL Calibration Standard

   1.2 SOLVENT/MATRIX: Methanol

2. PREPARATION NOTES: To a 5.0 mL screw top volumetric flask add 1.0 mL each of Purg. A, Purg. B, Purg. E stock (LS-RMA-0002) and carbon disulfide, and 1.0 mL MeOH. The final volume will be 4.0 mL. Transfer into two 2 mL microreaction vessels and one 100 uL conical for future reference. Use Hamilton syringes.

3. SHELF LIFE: 1 Month

   3.1 Prepare every 2 weeks.

4. SAFETY ISSUES: Refer to the MSDS's

5. COMMENTS: See Lab Procedures SOPS (SOP NO. LP-RMA-0018 thru LP-RMA-0023) for specific lab practices i.e. dilutions, care of glassware, SMI pipets, etc.

Prepared by: Susan Davis/Heather Esbenson	Date: 05/26/92
Management Approval: Paul Winkler	Date: 5/27/92
QA Officer Approval: Cindy Patterson	Date: 5/26/92

SOP No.:	Revision No.:	Effective Date:
LS-RMA-0001	4.1	05/26/92

6. CONSTITUENTS/CONCENTRATION:

CHEMICAL/STOCK NAME (Conc., open date, etc.)	FINAL CONC.
Purgeable A (MeOH 200 ug/mL Supelco):	
Methylene Chloride	50 ug/mL
Trichlorofluoromethane	50 ug/mL
1,1-Dichloroethylene	50 ug/mL
1,1-Dichloroethane	50 ug/mL
Chloroform	50 ug/mL
Carbon Tetrachloride	50 ug/mL
1,2-Dichloropropane	50 ug/mL
Trichloroethylene	50 ug/mL
1,1,2-Trichloroethane	50 ug/mL
Dibromochloromethane	50 ug/mL
2-Chloroethylvinylether	50 ug/mL
Tetrachloroethylene	50 ug/mL
Chlorobenzene	50 ug/mL
Purgeable B (MeOH 200 ug/mL Supelco):	
trans-1,2-Dichloroethylene	50 ug/mL
1,2-Dichloroethane	50 ug/mL
1,1,1-Trichloroethane	50 ug/mL
Bromodichloromethane	50 ug/mL
trans-1,3-Dichloropropylene	50 ug/mL
cis-1,3-Dichloropropylene	50 ug/mL
Benzene	50 ug/mL
Bromoform	50 ug/mL
1,1,2,2-Tetrachloroethane	50 ug/mL
Toluene	50 ug/mL
Ethylbenzene	50 ug/mL
Purgeable E (MeOH 200 ug/mL):	
2-Butanone	50 ug/mL
4-Methyl-2-pentanone	50 ug/mL
2-Hexanone	50 ug/mL
Vinyl Acetate	50 ug/mL
Styrene	50 ug/mL
m-Xylene	50 ug/mL
o-Xylene	50 ug/mL
o-Dichlorobenzene	50 ug/mL
p-Dichlorobenzene	50 ug/mL
m-Dichlorobenzene	50 ug/mL
Acetone	50 ug/mL
cis-1,2-dichloroethene	50 ug/mL
Separate Stock: (200 ug/mL)	
Carbon Disulfide	50 ug/mL

STANDARD
OPERATING
PROCEDURE

Subject or Title: MS6-VOA-CS2 and GC-601/602-CS2		Page 1 of 2
SOP No.: LS-RMA-0009	Revision No.: 5.0	Effective Date: 12/10/90
Supersedes: Rev. 4.0		

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1. TYPE OF STANDARD: VOA Calibration standard.

2. SOLVENT/MATRIX: Methanol.

3. PREPARATION NOTES:

   3.1 Standards made for the extraction laboratory (DCSs, SCSs, MS/MSDs) and the instrumentation laboratory (calibration standards, check standards) must each be prepared from different sources. The other source may be another chemical supplier, the EPA, or the National Institute of Standards and Technologies (NIST). Refer to SOP: LP-RMA-0019.

Prepared by: Bob Sprengeler	Date: 03/13/90
Management Approval: M. Hoffman	Date: 1/21/91
QA Officer Approval: Jan Rodenbarger	Date: 1/21/91

3.2 To a 5 mL micro reaction vessel using a 1.0 mL Hamilton gas tight syringe add 1.0 mL of trichlorotrifluoroethane (Freon 113) at 200 ug/mL, add 1.0 mL of dichlorodifluoromethane (Freon 12) at 200 ug/mL, add 1.0 mL of dichlorofluoromethane (Freon 21) at 200 ug/mL and Purgeable C at 200 ug/mL to the vessel. Slowly invert vessels three times to mix. Transfer the 4.0 mL of solution to four 1 mL micro reaction vessel using the Hamilton gas tight syringe. Also fill a 100 uL conical for GC-department for future reference and verification purposes.

4. SHELF LIFE: 2 Weeks.

5. SAFETY ISSUES: Refer to the MSDS's.

6. CONSTITUENTS/CONCENTRATION:

CHEMICAL/STOCK NAME (Conc.)	FINAL CONC.
Purgeable C (200 ug/mL, methanol)	
Chloromethane	50.0 ug/mL
Bromomethane	50.0 ug/mL
Vinyl Chloride	50.0 ug/mL
Chloroethane	50.0 ug/mL
Dichlorodifluoromethane (200 ug/mL, methanol)	50.0 ug/mL
Trichlorotrifluoroethane (200 ug/mL, methanol)	50.0 ug/mL
Dichlorofluoromethane (200 ug/mL, methanol)	50.0 ug/mL

7. NOTES:

STANDARD
OPERATING
PROCEDURE

Subject or Title: MS-VOA-IS		Page 1 of 2
SOP No.: LS-RMA-0010	Revision No.: 4.0	Effective Date: 04/30/91
Supersedes: Revision 3.0		

1. TYPE OF STANDARD: VOA internal standard

   1.2 SOLVENT/MATRIX: Methanol

2. PREPARATION NOTES: To a 10.0 mL screw cap volumetric flask add 2 mL MEOH. Then add 1.0 mL Restek VOA LM Internal Standard Mix @ 2500 ug/mL (or equivalent). Use gastight Hamilton syringe. Dilute to volume with MEOH. Transfer into 2 - 5 mL microreaction vessels plus a 100 uL conical for future reference.

3. SHELF LIFE: 1 Month

   3.1 Prepare every 3 weeks.

4. SAFETY ISSUES: Refer to the MSDS's

5. COMMENTS: See Lab Procedures SOPS (SOP NO. LP-RMA-0018 thru LP-RMA-0023) for specific lab practices i.e. dilutions, care of glassware, SMI pipets, etc.

Prepared by: Susan Davis	Date: 04/30/91
Management Approval: Deborah J Cayl	Date: 6-4-91
QA Officer Approval: Jane Ryan	Date: [illegible]

6. CONSTITUENTS/CONCENTRATION:

CHEMICAL/STOCK NAME (Conc., open date, etc.)	FINAL CONC.
VOA LM Internal Standard Mix (2500 ug/mL Restek)	
Bromochloromethane	250 ug/mL
1,4-Difluorobenzene	250 ug/mL
Chlorobenzene-d5	250 ug/mL

STANDARD
OPERATING
PROCEDURE

Subject or Title: MS-VOA-SCS		Page 1 of 2
SOP No.: LS-RMA-0011	Revision No.: 3.0	Effective Date: 03/15/90
Supersedes: Rev. 2.0		

1. TYPE OF STANDARD: Sample control spike stock.

   1.2 SOLVENT/MATRIX: Methanol

2. PREPARATION NOTES: To a 25 mL vol. flask add 10 mL MeOH. Then add 1250 uL of each of the three 5000 ug/mL stocks. Dilute to volume with MeOH. Transfer into a 35 mL screw cap glass bottle with a Miniert Syringe valve. Use a Hamilton syringe.

3. SHELF LIFE: 1 Month

   3.1 Prepare Every 3 weeks

4. SAFETY ISSUES: Refer to the MSDS's

5. COMMENTS: See Lab Procedures SOPS (SOP NO. LP-RMA-0018 thru LP-RMA-0023) for specific lab practices i.e. dilutions, care of glassware, SMI pipets, etc.

Prepared by: Bob Sprengeler	Date: 03/13/90
Management Approval: Deborah J. Coyle	Date: 4/6/90
QA Officer Approval: Tina Sign	Date: 4/3/90

SOP No.:	Revision No.:	Effective Date:
LS-RMA-0011	3.0	03/15/90

6. CONSTITUENTS/CONCENTRATION:

CHEMICAL/STOCK NAME (Conc., open date, etc.)	FINAL CONC.
Toluene-d8 (MeOH 5000 ug/mL)	250 ug/mL
4-Bromofluorobenzene (5000 ug/mL MeOH)	250 ug/mL
1,2-Dichloroethane-d4 (5000 ug/mL MeOH)	250 ug/mL

STANDARD
OPERATING
PROCEDURE

Subject or Title: MS-VOA-DCS		Page _1_ of _2_
SOP No.: LS-RMA-0012	Revision No.: 3.0	Effective Date: 04/03/90
Supersedes: Rev. 2.0		

1. TYPE OF STANDARD: Laboratory control spike

   1.2 SOLVENT/MATRIX: Methanol

2. PREPARATION NOTES: To a 10.0 mL screw cap volumetric flask add 2 mL MeOH. Then add 500 uL of each stock at 5000 ug/mL. Dilute to volume with MeOH. Transfer to 2 - 5 mL microreaction vessels. Use Hamilton syringes.

3. SHELF LIFE: 1 Month

   3.1 Prepare Every 4 weeks

4. SAFETY ISSUES: Refer to the MSDS's

5. COMMENTS: See Lab Procedures SOPS (SOP NO. LP-RMA-0018 thru LP-RMA-0023) for specific lab practices i.e. dilutions, care of glassware, SMI pipets, etc.

Prepared by: Bob Sprengeler	Date: 04/03/90
Management Approval: Deborah J. Cay[illegible]	Date: 5/23/91
QA Officer Approval: [illegible]	Date: [illegible]

SOP No.:	Revision No.:	Effective Date:
LS-RMA-0012	3.0	04/03/90

6. CONSTITUENTS/CONCENTRATION:

CHEMICAL/STOCK NAME (Conc., open date, etc.)	FINAL CONC.
1,1-Dichloroethene (vinylidene chloride)(5000 ug/mL MeOH)	250 ug/mL
Trichloroethene (5000 ug/mL MeOH)	250 ug/mL
Chlorobenzene (5000 ug/mL MeOH)	250 ug/mL
Toluene (5000 ug/mL MeOH)	250 ug/mL
Benzene (5000 ug/mL MeOH)	250 ug/mL

STANDARD
OPERATING
PROCEDURE

Subject or Title: MS-VOA-AP9-CSA		Page 1 of 2
SOP No.: LS-RMA-0015	Revision No.: 5.0	Effective Date: 05/26/92
Supersedes: Revision 4.0		

1. TYPE OF STANDARD: Calibration standard for Appendix 9 volatiles.

   1.2 SOLVENT/MATRIX: Methanol

2. PREPARATION NOTES: To a 5.0 mL screw top volumetric flask add approximately 2 mL MeOH. Add 500 uL ea of Isobutyl Alcohol and Acetonitrile stocks, 250 uL of Acrolein stock, 200 uL of Acrylonitrile stock, 100 uL ea of Iodomethane, DBCP, Ethyl Methacrylate, and Methyl Methacrylate stocks, 25 uL of the 3-Chloropropene stock. Use Hamilton syringes. Dilute to volume with MeOH. Transfer to a 5 mL amber microreaction vessel plus a 100 uL conical for future reference.

3. SHELF LIFE: 1 Month

   3.1 Prepare every 3 weeks

4. SAFETY ISSUES: Refer to the MSDS's

5. COMMENTS: See Lab Procedures SOPS (SOP NO. LP-RMA-0018 thru LP-RMA-0023) for specific lab practices i.e. dilutions, care of glassware, SMI pipets, etc.

Prepared by: Heather Esbenson	Date: 05/26/92
Management Approval: Paul W. Miller	Date: 5/27/92
QA Officer Approval: Cindy Patterson	Date: 5/26/92

SOP No.:	Revision No.:	Effective Date:
LS-RMA-0015	5.0	05/26/92

6. CONSTITUENTS/CONCENTRATION:

CHEMICAL/STOCK NAME (Conc., open date, etc.)	FINAL CONC.
Acetonitrile (5000 ug/mL MeOH)	500 ug/mL
Ethyl Cyanide (5000 ug/mL MeOH) (Proprionitrile)	50 ug/mL
3-Chloropropene (5000 ug/mL MeOH) (Allylchloride)	25 ug/mL
DBCP (5000 ug/mL MeOH)	100 ug/mL
Methacrylonitrile (5000 ug/mL MeOH)	50 ug/mL
Isobutyl alcohol (5000 ug/mL MeOH)	500 ug/mL
2-Chloro-1,3-Butadiene (5000 ug/mL MeOH)	50 ug/mL
1,1,1,2-Tetrachloroethane (5000 ug/mL)	50 ug/mL
Methyl Methacrylate (5000 ug/mL)	100 ug/ml

STANDARD
OPERATING
PROCEDURE

Subject or Title: MS-VOA-524.2-DCS-CSA		Page _1_ of _2_
SOP No.: LS-RMA-0039	Revision No.: ORIGINAL	Effective Date: 04/26/91
Supersedes:		

1. TYPE OF STANDARD: Calibration standard spike stock for 524.2 method

   1.2 SOLVENT/MATRIX: Methanol

2. PREPARATION NOTES: To a 5.0 mL screw top volumetric flask, add about 2 mLs MeOH, then add 250 uL of Supelco custom mix ENS-524.2 (or equivalent) using a Hamilton syringe. Bring to volume with MeOH. Transfer to a 5 mL microreaction vessel.

3. SHELF LIFE: 2 months

   3.1 Prepare every 6 weeks.

4. SAFETY ISSUES: Refer to the MSDS's

5. COMMENTS: See Lab Procedures SOPS (SOP NO. LP-RMA-0018 thru LP-RMA-0023) for specific lab practices, i.e. dilutions, care of glassware, SMI pipets, etc.

Prepared by:Susan Davis	Date: 04/26/91
Management Approval: Deborah J. Cay	Date: 5/23/91
QA Officer Approval: Jim [illegible]	Date: [illegible]

SOP No.:	Revision No.:	Effective Date:
LS-RMA-0039	Original	04/26/91

6. CONSTITUENTS/CONCENTRATION:

CHEMICAL/STOCK NAME (Conc., open date, etc.)	FINAL CONC.
ENS-524.2 (5,000 ug/mL in MeOH, Supelco custom mix)	
Benzene	250 ug/mL
Toluene	250 ug/mL
Chlorobenzene	250 ug/mL
Ethyl benzene	250 ug/mL
1,3-dichlorobenzene	250 ug/mL
1,1-dichloroethane	250 ug/mL
Bromodichloromethane	250 ug/mL
Chloroform	250 ug/mL
Trichloroethylene	250 ug/mL
Hexachlorobutadiene	250 ug/mL

STANDARD
OPERATING
PROCEDURE

Subject or Title: MS VOA API/B&V Addition-CSA		Page 1 of 2
SOP No.: LS-RMA-0045	Revision No.: 1.0	Effective Date: 04/30/91
Supersedes: Original		

1. TYPE OF STANDARD: Calibration standard stock

   1.2 SOLVENT/MATRIX: Methanol

2. PREPARATION NOTES: To a 5.0 mL screw cap volumetric flask add 2 mL of MeOH. Add 1.0 mL of 1,4-Dioxane, 500 uL of Ethanol, 200 uL Acrylonitrile, 250 uL Acrolein, 100 uL ea of Iodomethane, 1,2-Dibromoethane, and Ethyl Methacrylate, and 50 uL each of remaining stocks. Dilute to volume with MeOH then transfer to 2 - 2 mL microreaction vessels plus a 100 uL conical for future reference. Use Hamilton syringes.

3. SHELF LIFE: 1 month

   3.1 Prepare every 3 weeks

4. SAFETY ISSUES: Refer to the MSDS's

5. COMMENTS: See Lab Procedures SOPS (SOP NO. LP-RMA-0018 thru LP-RMA-0023) for specific lab practices i.e. dilutions, care of glassware, SMI pipets, etc.

Prepared by: Susan Davis	Date: 04/30/91
Management Approval: Deborah J. Cayl	Date: 5/23/91
QA Officer Approval: [illegible]	Date: 6/4/91

SOP No.:	Revision No.:	Effective Date:
LS-RMA-0045	1.0	04/30/91

6. CONSTITUENTS/CONCENTRATION:

CHEMICAL/STOCK NAME (Conc., open date, etc.)	FINAL CONC.
1,4-Dioxane (5000 ug/mL MeOH)	1000 ug/mL
1,2-Dibromoethane (5000 ug/mL MeOH)	100 ug/mL
Hexane (5000 ug/mL MeOH)	50 ug/mL
Tetrahydrofuran (5000 ug/mL MeOH)	50 ug/mL
Acrylonitrile (5000 ug/mL)	200 ug/mL
Dibromomethane (5000 ug/mL)	50 ug/mL
Ethyl Methacrylate (5000 ug/mL)	100 ug/mL
1,2,3-Trichloropropane (5000 ug/mL)	50 ug/mL
t-1,4-Dichloro-2-Butene (5000 ug/mL)	50 ug/mL
Indomethane (5000 ug/mL)	100 ug/mL
Ethanol (5000 ug/mL)	500 ug/mL
Acrolein (5000 ug/mL)	250 ug/mL
MTBE (5000 ug/mL)	50 ug/mL