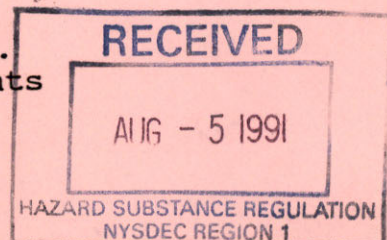


OCCIDENTAL CHEMICAL CORPORATION
HOOKER/RUCO SUPERFUND SITE
HICKSVILLE, NEW YORK

OPERABLE UNIT 2
REMEDIAL DESIGN WORK PLAN
INTERMEDIATE SITE DESIGN SUBMITTAL
VOLUME IA OF II

Prepared For
Occidental Chemical Corporation
July 1991

LEGGETTE, BRASHEARS & GRAHAM, INC.
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OCCIDENTAL CHEMICAL CORPORATION
PROJECT REA-91543-503
HOOKER/RUCO REMEDIATION PROJECT

SECTION 3: FIELD SAMPLING PLAN

(USEPA INDEX NUMBER - II CERCLA-10216)
SECTION 3: FIELD SAMPLING PLAN
REVISED JULY 25, 1991

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SECTION 3

3.0 FIELD SAMPLING PLAN

3.1 Scope and Purpose

The scope and purpose of the Field Sampling Plan (FSP) is to define procedures that will be used to collect samples from Operable Unit 2 of the Hooker/Ruco site during the Remedial Action (RA). The FSP has been prepared following specifications described in A Compendium of Superfund Field Operations Methods, Office of Emergency and Remedial Response, USEPA, EPA/540/P-87/001, Verification of PCB Spill Clean-up by Sampling and Analysis, Office of Toxic Substances, USEPA, EPA-560/5-85-026, and Field Manual for Grid Sampling of PCB Spill Sites to Verify Cleanup, Office of Toxic Substances, USEPA, EPA-560/5-86-017.

3.2 Project Description

A comprehensive description of the project's scope is presented in Section 2.0.

3.3 Project Organization and Responsibilities

3.3.1 Field Sampling Project Organization

Site Coordinator	Dr. Alan Weston (OCC)
Project Manager	Mr. Joseph Coveney (OCC)
QA/QC Officer	Mr. Patrick Garrity (OCC)
Remedial Leader	Mr. William T. West (LBG)
Onsite Geologists	Mr. Keith Yocis (LBG)
	Mr. Stephen Ritz (LBG)
Health and Safety Officer . .	Mr. Douglas Paschke (OCC)
Sampling Coordinator	Mr. William T. West

3.3.2 Definition of Responsibilities

Site Manager (SM): The SM is responsible for the successful execution of the work assignment. The SM shall implement and direct all sampling protocols required for the program. The SM shall determine the

sampling equipment and sample containers. The SM shall train and qualify field personnel in sampling procedures and field analytical procedures prior to sampling.

Onsite Geologist: All field personnel will report to the SM. The geologist will be required to be trained and qualified in all procedures of the FSP. The onsite geologists will perform all sample collection activities in accordance with the FSP and report any deviations in sample collection procedures to the SM.

Sample Coordinator: The sample coordinator is responsible for all sample handling and delivery of the collected samples to the analytical laboratory. The sample coordinator will ensure that samples are collected, labeled, preserved, stored and transported, as specified in accordance with the procedures or protocols. The sample coordinator will check that all sample documentation is correct and transmitted with the samples to the analytical laboratories. The sample coordinator will verify that all field analytical QC procedures are being followed.

3.4 Field Sampling Documentation

3.4.1 Field Books

Records of all sampling activities will be maintained and include field notebooks, scaled drawings, photographs and chain-of-custody forms. Field notebooks will be used to record pertinent observations, field measurements, and all sample collection procedures. Entries into the field books will be initialed for personnel identification and the date and time of all observations will be recorded. Entries will be made with waterproof ink and all field notebooks will be water resistant and weatherproof.

3.4.2 Field Measurements

All field measurements will be recorded in the field notebook. Sample locations will be clearly identified in the field notebook. The collection procedures, date and time of sample collection will be recorded in the field notebook. All field observations and climatic conditions during the sample event will also be recorded.

3.5 Air Monitoring - Real Time

Monitoring of the air quality in and around the work areas will be completed during remedial activities. Air monitoring will be conducted for total volatile organics and real-time particulates. Real-time monitoring indicates that readings are direct and immediate rather than requiring samples to be sent elsewhere from the site for analysis. Support activities, including backfilling, grading, soil transport and mobilization/demobilization will not require air monitoring.

3.5.1 Air Monitoring - Total Volatile Organics

Total volatile organic air monitoring will be completed using an HNU Model PI-101 PID or performance equivalent. PID instruments will be calibrated daily according to procedures presented in Appendix 3-1. Air monitoring for total volatile organics will be conducted prior to the start of remedial activities once each day, and once every two hours thereafter. Air monitoring for total volatile organics will be conducted from one upwind and three downwind locations at the perimeter of the active work areas. Specific monitoring locations will be determined based upon readings collected from an onsite wind gauge, and will be selected by the site manager. Air monitoring will be conducted at respirable height, approximately 4 to 6 feet above grade.

3.5.2 Air Monitoring - Real-Time Particulates

Air monitoring for real-time particulates will be completed using a GCA Miniram aerosol monitor or performance equivalent. The real-time monitoring unit will be calibrated daily according to procedures presented in Appendix 3-2. Real-time particulate levels will be monitored continuously at each active work area. Particulate levels will be recorded in the field notebook on an hourly basis, and more frequently during periods of wind gusts. Air monitoring for real-time particulates will be collected at specific upwind and downwind locations at respirable heights, approximately 4 to 6 feet above grade.

Remedial project work zones for this project are those specified in Section 5.9.1 of the HASP.

3.5.3 Air Monitoring - Confined Space

Air monitoring for total volatile organics, oxygen and flammability will be conducted during confined space activities. Work in confined space is anticipated to occur only in the deeper excavation in the Direct Spill Area. Confined space air monitoring will be used in conjunction with the Confined Space Entry Plan described in the HASP. All confined space air monitoring will be conducted continuously before and during the confined space activities. Total volatile organics will be monitored using a calibrated PID. Oxygen and flammability measurements will be collected using a MSA Model 260 portable combustible gas and oxygen alarm or performance equivalent. The combustible gas alarm will be set at a 25-percent Lower Explosion Limit (LEL) and the oxygen alarm will be set at 19.5 percent. The unit will be calibrated daily before use according to procedures presented in Appendix 3-3.

3.6 Air Sampling - Time-Weighted Average

Air sampling will be conducted for particulates and Aroclor 1248 on particulates during the remedial activities. Air sampling will be completed once prior to commencement of the field work and weekly during all excavation activities. Air sampling will not be completed during backfill, grading and reseeding operations, soil handling activities and mobilization/demobilization. The exact sampling locations will be selected daily by the SM and will be determined using an onsite wind gauge. Air sampling will be collected from one upwind and two downwind locations at the perimeter of the active work zones. Air sampling will be conducted at a respirable height (4 to 6 feet above grade) according to NIOSH methods. Onsite field measurements of windspeed, direction and relative humidity will be collected prior to sampling, and then every 4 hours thereafter. Field measurements will be augmented with meteorological data obtained from the Republic Airport at Farmingdale, New York. Information on meteorological data from Republic Airport will be collected daily prior to sampling by contacting the air tower at (516) 683-2916. All relevant air monitoring data will be recorded on the work sheets attached in Appendix 3-4, each time air sampling is completed.

3.6.1 Air Sampling - Particulates

Air monitoring for total particulates will be conducted utilizing a flow-controlled personal sampling pump and a tarred 37 mm, 5 um PVC filter, as described in NIOSH Method 0500. The personal sampling pump will be a DuPont ALPHA-1 air sampler or performance equivalent. The ALPHA-1 pumps are user programmable for start time, run time, tolerated low-flow time, and intermittent run time. User flow rates are selectable from 5 cc/min to 5,000 cc/min without requiring the use of critical orifices. The pumps will be

used at a flow rate of 1,500 to 2,000 cc/min, as specified in NIOSH Method 0500.

Collection filters utilized for particulate sampling will be SKC 37 mm, 5 um pore size, PVC filters, or performance equivalent, and will meet the requirements of NIOSH Method 0500.

Collection filters for the measurement of total particulates will be gravimetrically analyzed for total mass according to NIOSH Method 0500. The filters will be pre-numbered, desiccated and weighed on a microbalance capable of weighing to 0.01 mg. The filters will then be used for collection, desiccated again and re-weighed. The resulting change in weight will be calculated and the concentration in mg/m^3 will be reported.

Air sampling for particulates and specific pump calibration will be completed with procedures outlined in Appendix 3-4.

3.6.2 Air Sampling - Aroclor 1248 on Particulates

The analysis for Aroclor 1248 on particulates will be conducted utilizing a flow-controlled air sampling pump, as described in NIOSH Method 5503. The air sampling pump will be a DuPont ALPHA-1 air sampler or performance equivalent. The ALPHA-1 pumps are user programmable for start time, run time, tolerated low-flow time, and intermittent run time, as well as user flow rate selectable from 5 cc/min to 5,000 cc/min without requiring the use of critical orifices. The pumps will be used at flow rates between 500 and 1,000 cc/min for NIOSH Method 5503. They will be calibrated for flow rate utilizing a Teledyne-Hastings NBS traceable bubble meter or performance equivalent.

Hexane will be used to desorb chemicals from the glass fiber filters. The analysis will employ a gas chromatograph equipped with an electron-capture detector. The following

chemical will be analyzed for: Aroclor 1248. Concentrations above 0.01 mg/m³ will be reported.

Glass fiber filters utilized for Aroclor 1248 on particulates air sampling will be 13 mm glass fiber filters, or equivalent, and meet the requirements of NIOSH Method 5503.

QA/QC will consist of analysis of a field blank which will be an unopened filter taken into the field and returned to the laboratory for analysis. Desorption efficiency will also be determined by spiking Aroclor 1248 onto the filter media and desorbing according to NIOSH Method 5503.

Air sampling for Aroclor 1248 on particulates and specific equipment calibration will be completed with procedures outlined in Appendix 3-4.

3.7 Waste Classification Sampling

All soil and sediment waste generated during the remedial project and liquid waste from onsite decon procedures will be characterized in accordance with 40 CFR Part 261 and 6NYCCR Part 371, regulations concerning the identification and definition of hazardous waste. All waste classification samples will be subjected to analytical testing for ignitability, reactivity, corrosivity and the TCLP Test Method 1311 for toxicity. A summary of the testing procedures and regulatory levels are presented on table 1.

By definition, all solid waste containing 50 ppm or greater PCBs is listed hazardous waste. A portion of the excavated soils will contain concentrations of PCBs that, if tested, would be determined to be hazardous waste. Therefore, knowledge of the excavated soils will be used, rather than subject the soils to additional testing. Pursuant to 6NYCCR 371.4(e), the hazardous code for PCB wastes will be toxic (T). All excavated soils during the remedial project will have the following hazardous waste number assigned to the solid material:

B007: Other PCB wastes, including contaminated soil, solids, sludges, clothing, rags and dredged material.

3.7.1 Soil and Sediment Waste

As described in Section 2.0, soils that have been excavated will be stored onsite in containerized gondola rail cars. The waste material will be stored in the rail cars and separated by source location. One representative composite soil sample from the work areas containing 10 to 500 ppm of PCBs will be collected and submitted for analytical testing of the waste characterization parameters prior to the start of excavation. Soil sample for characterization and pre-qualifying the waste will be collected from the following sources:

- direct spill area;
- Sump 3;
- transported-related areas (Work Areas A through I); and
- stockpiled soils.

3.7.2 Liquid Waste

The remedial project will not involve the removal of either standing water or ground water. However, small quantities of liquid waste may be generated during the equipment or personnel decontamination procedures. During equipment or personnel decontamination, small volumes of water will be sprayed and contained. This liquid will be drummed and contained by source. Each specific waste stream (equipment decontamination, personnel decontamination) will be sampled separately, and one composite sample of each waste stream will be collected. The liquid samples will be tested for waste characterization parameters.

The liquid waste samples will be collected using the following procedure. The liquid waste drums will be opened and a clear bailer will be lowered to the base of each drum. After the bailer has been lowered to the base of the drum, it will be removed and visually inspected to determine if non-aqueous phase liquids (NAPLs) are present. If NAPLs are detected in any of the drums, two separate samples of the NAPLs will be collected. The NAPL sample collection will be completed using a bottom-loading bailer to prevent dilution of the NAPL samples. It should be noted, however, that NAPLs have never been encountered at the Hooker/Ruco site and are not expected to be found during the liquid waste sampling. If NAPLs are not encountered during the sample acquisition, one bailer from each drum of a distinct waste stream will be removed and placed in a 5-gallon nalgene container. The container will remain sealed between each sample acquisition. After all drums of a distinct waste stream have been sampled and composited, a sample of the composite water from each waste stream will be collected and submitted for testing. The drummed liquid waste will be resealed and remain on the Hooker/Ruco site until review of the laboratory data is completed.

3.7.3 Solid Waste

Solid waste consisting of decontamination rags used to swab equipment and non-porous surfaces and spent protective equipment will be containerized in sealable 55-gallon drums. Because the spent rags and protective equipment have been in direct contact with PCB articles/materials, knowledge of the waste will be used for disposal purposes. The solid waste will not be subjected to testing, but rather classified as hazardous waste based upon toxicity and be assigned hazardous waste number B007. The solid waste will be disposed of as hazardous waste with the soils containing PCBs between 10 and 500 ppm.

3.8 Verification Sampling

Work areas will be excavated to the desired target depth, as described in Section 2.0. Soil samples will then be collected from the excavated areas, beneath the stockpiled soils and the sides and base of Sump 3. The sample locations in each cleared area will be based upon a hexagonal grid configuration. Soil samples will be collected at each grid location and the verification samples will then be composited prior to analysis. All verification samples will be submitted to the receiving laboratory for analysis of PCBs by Method 8080. The post-excavation (verification) sample results will be used to assess the effectiveness of the remedial activities in achieving the performance standards.

The steps necessary to complete the verification sampling are presented below:

- Step 1 - Document the excavation/sample area.
- Step 2 - Determine the center and radius of the sample area.
- Step 3 - Determine the number of sample points.
- Step 4 - Construct the sample grid.
- Step 5 - Consider judgmental samples.
- Step 6 - Collect the verification samples.
- Step 7 - Composite the verification samples.

The following paragraphs provide narrative descriptions and describe the procedures to be employed to complete each step.

3.8.1 Step 1 - Document the Excavation/Sample Area

Each area to be sampled will be diagrammed. Both side and plan view scaled drawings of the sample area will be completed. All vertical surfaces, excavation dimensions and surface types will be identified on the diagrams. All excavation boundaries will be referenced by measured distance

to stationary objects and these reference points will be included on the drawings.

In addition to the scaled drawings, a minimum of one photograph of each sample area will be obtained. An appropriate scale, which can be used to reference sample area dimensions will be included in the photograph. A reference card containing the sample area's identification, site name and date will be included in the photographic record. The date, time and photograph number will be recorded in the field book.

3.8.2 Step 2 - Determining the Center and Radius of the Sample Grid

The shape and size of each sample area will be unique and will be different than other sample areas. The grid design that will be used to locate sample points will also be unique and tailored to each sample area. The sample grid will be determined for each area to be sampled using standard geometric techniques. The center and radius calculations will be determined from the one-dimensional plan view of the sample area completed in Step 1. The center and radius will be calculated according to procedures presented in Appendix 3-5. All dimensions will be computed and recorded to the nearest 10th of a foot.

3.8.3 Step 3 - Determine the Number of Sample Points

The sample area's size will govern the number of soil samples that will be collected. The sample area's size shall be determined and classified using the sample radius' length. Based upon the hexagonal grid configuration, sample grids will be of 7, 19 or 37 points in size. Figure 3-1 shows the proposed hexagonal sampling gride for verification sampling. Table 1 presents the sample area's size and the associated number of samples.

Sample radius (in feet)	Number of sample points
<10	7
>10 <30	19
>30	37

No more than 37 sample points will be plotted for a given sample area, excluding the judgmental sample locations described in Step 5.

3.8.4 Step 4 - Construction of the Sample Grid

Based upon the results of Step 3, the appropriate grid will be constructed in the sample area. The sample area's center will be determined and staked in the field following the procedures that were used in Step 2. The sample area's surface will be covered with plastic sheeting prior to completing measuring and grid construction activities. The sample grid will be constructed in the field according to procedures presented in Appendix 3-6. Actual sample locations will be staked with flagging and measured from a minimum of two stationary objects. Field measurements will be recorded in a coordinate table and also presented on a scaled drawing of the sample area.

3.8.5 Step 5 - Consider Special Case Sample Locations

After the grid sample design has been constructed, additional samples of suspect areas will be considered. The judgmental sample locations will be collected in addition to the sample grid when staining, visible oils or suspicious areas, including cracks and crevices, are located adjacent to, but beyond the sample area. As with all sample locations, the judgmental sample locations will be measured from at least two stationary objects and also presented on the scaled drawing of the sample area.

3.8.6 Step 6 - Verification Sample Collection

After the sample grid has been constructed on the site, a sample will be collected at each grid point. Soil samples for verification purposes will be collected according to procedures presented in Appendix 3-7. Samples, however, will not be collected from grid locations when any sample point meets one or more of the following conditions:

- A. The sample point is located in an adjacent work area that has been excavated and determined compliant.
- B. The sample point is located in an adjacent work area that has not been excavated.
- C. The sample point is located on a vertical surface between work areas that have not been excavated.
- D. The sample point, located on the scaled drawing, does not exist in the actual sample area.
- E. The sample point is located on a vertical surface of a site building or utility.

During sample collection activities, all pertinent data, including time, date, sample, personnel, sample methods, and other collection data, will be recorded in the field books.

3.8.7 Step 7 - Compositing Strategies

Samples collected from the verification grid locations will be composited prior to analysis. Procedures for the composite soil samples are presented in Appendix 3-8. Composite soil samples will be formed from 5 samples or less to prevent diluting a batched sample below the analytical quantification limits. Composite soil samples will be formed

from roughly equal number of soil samples from adjacent sample points. Figure 3-2 presents compositing strategies for the various verification sampling grids. Composite samples will be analyzed for Aroclor 1248 by Method 8080.

3.9 Results Evaluation - Compliance/Non-Compliance of Verification Samples

The objective of the verification sampling program is to determine if remedial actions have been effective in removing impacted soils having PCB concentrations above the performance standard of 10 ppm. The PCB concentration in a single soil sample, therefore, would be the target cleanup level of 10 ppm. Single soil samples analyzed and found to have PCB concentrations equal to or greater than 10 ppm will be considered to be above the performance standards, and PCB concentrations less than 10 ppm will be determined to be in accordance with performance standards.

Composite soil samples will be addressed similarly. PCB concentrations in the composite sample will be a result of the average concentration of all pooled samples. The composite sample concentration will be reviewed with respect to the statistical cutoff level of 10 ppm. If the results of the composite sample are at concentrations equal to or greater than 10 ppm, then at least one, if not more of the samples will have exceeded the compliance limit. However, if the resulting concentration is less than $10 \text{ ppm}/N$, where N equals the number of samples pooled to create the composite, not to exceed 5 samples, then all soil samples in the composite meet the remedial objectives. If the composite concentration results in some medium value, each sample used to form the composite will be analyzed individually to reach a decision.

If a reading in excess of 10 ppm is obtained by either the single sample analysis or the composite approach, the location of the sample or samples will be re-excavated to a limit of the radius of the smallest circle certain to be

sampled. The following table presents the procedure to be used to determine the smallest circle certain to be sampled:

Number of points	Radius of smallest circle certain to be sampled
7	0.5R
19	0.28R
37	0.19R

where R is the sample grid's radius, as determined by methods presented in Appendix 3-5.

The re-excavation will be completed in 1-foot lifts or at depth increments to be determined in the field. Re-sampling to confirm that the remedial objective has been achieved will be completed.

3.10 Confirmation Sampling

During the verification sampling process, sufficient quantities of soil will be collected from the individual sample locations to form duplicate composites. One of the composite samples will be analyzed rapidly for verification purposes. Once the verification sampling results show that the remedial action performance standards have been achieved, the duplicate composite soil sample will be submitted for analytical testing. One duplicate composite soil sample from each sample area will be submitted for confirmation analysis of Aroclor 1248 using CLP methodologies.

3.11 Sample Equipment Cleaning

In order to minimize the potential for cross contamination during sample collection activities, disposable sample equipment will be used for verification sampling. Disposable equipment will include stainless-steel spoons/spatulas/trowels, Teflon sheets and disposable templates. However, should disposable soil sample equipment

not be available, sample equipment will be cleaned between sample collections. The sample equipment cleaning procedures are presented in Appendix 3-9.

3.12 Field and Equipment Blanks

Air sampling field blanks will be collected at a rate of one field blank per lot of air sampling filters. Air sampling blanks will only be assessed for Aroclor 1248. Air sampling field blanks will consist of an unopened filter taken into the field and returned to the laboratory for analysis. Desorption efficiency will also be determined for the field blank by spiking Aroclor 1248 onto the filter media and describing according to NIOSH Method 5503.

Field and equipment blanks will be collected during the investigation. One field blank will be collected for each type of sampling equipment used each day. Field and equipment blanks of the sample and excavation equipment will be collected by wipe sampling the surface. Wipe sampling will be completed by procedures presented in Appendix 3-10. Field and equipment blanks collected to assess sampling and remedial equipment quality will be analyzed for Aroclor 1248 by Method 8080. Equipment blanks will be collected from each type of excavation equipment used during the remediation; examples of equipment blanks include shovel blades used during hand excavation, backhoe or front-end loader buckets, and crane buckets.

3.13 Duplicates

Duplicate air samples will be collected at a rate of one duplicate per each sampling event. The air sampling duplicate will be collected from a downwind sampling location. Duplicate soil samples will be collected at a rate of 10 percent during the verification sampling process. Duplicate samples from the composite sampling procedures

will be collected at a rate of 1 per each sampled area. All duplicate soil samples will be blind coded.

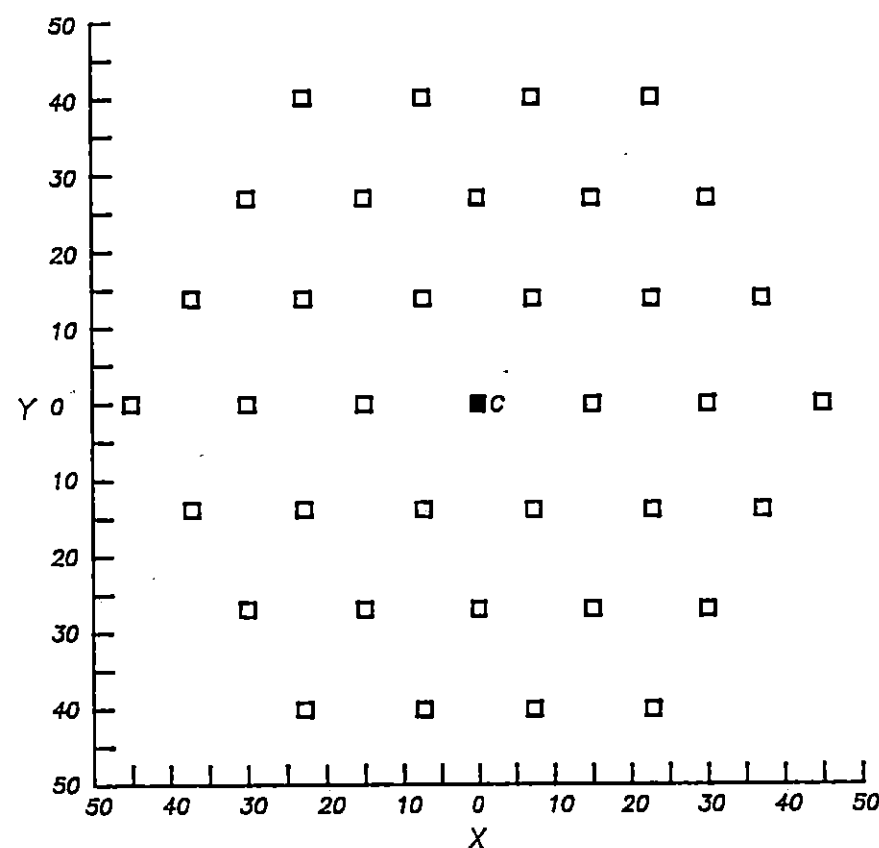
3.14 Containers

Three hundred (300) series containers will be obtained from I-Chem Research, Hayward, California, or performance equivalents. Appropriate sample containers for the various samples presented in Section 4.0 QAPP.

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July 25, 1991
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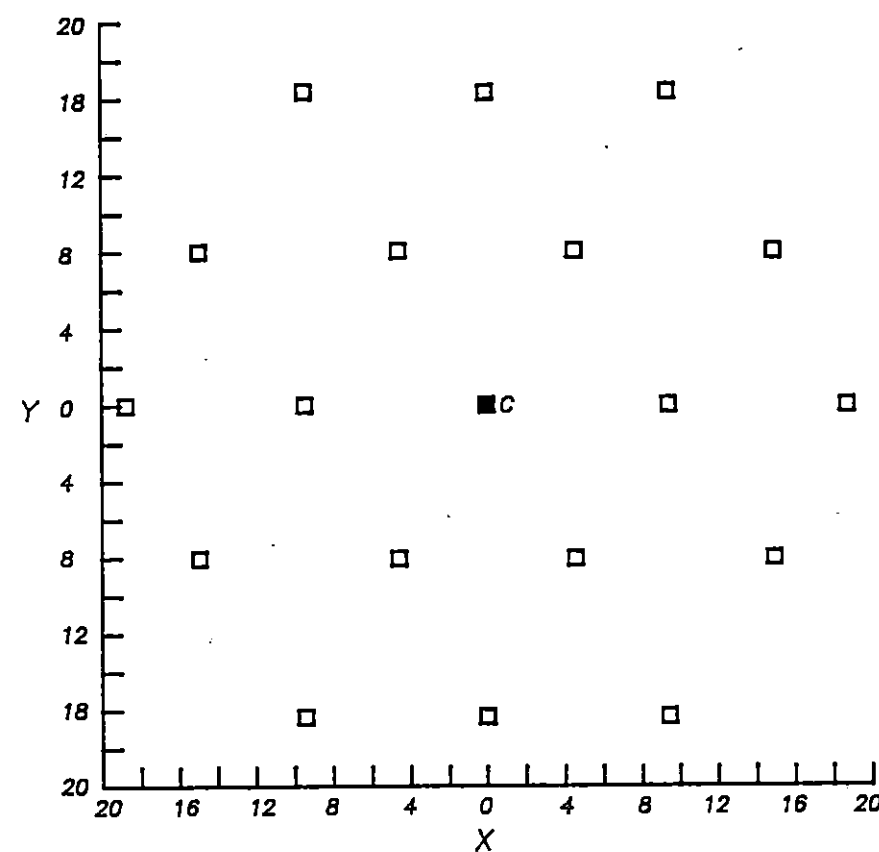
FIGURE 3-1

LOCATION OF SAMPLING POINTS IN A 37-POINT GRID



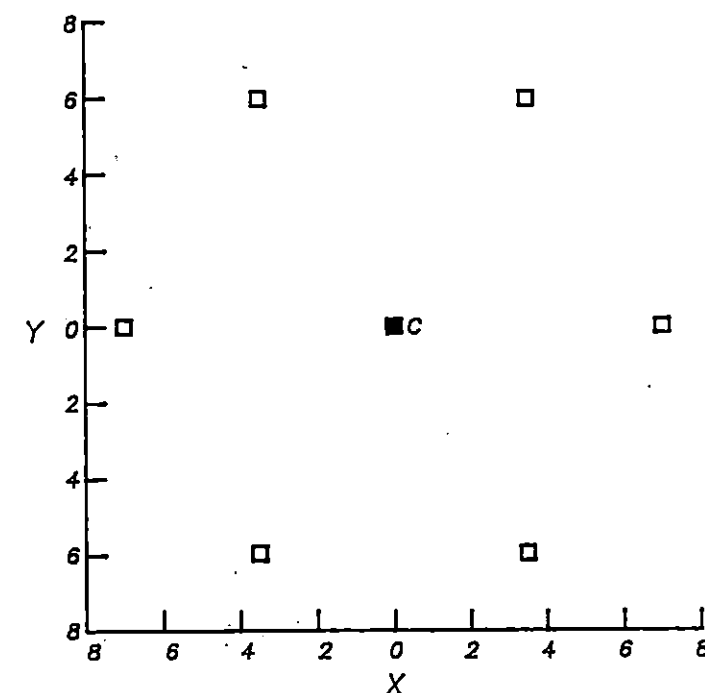
THE OUTER BODY OF THE SAMPLE AREA IS ASSUMED TO BE 50 FEET FROM THE CENTER (C) OF THE SPILL SITE.

LOCATION OF SAMPLING POINTS IN A 19-POINT GRID



THE OUTER BODY OF THE SAMPLE AREA IS ASSUMED TO BE 20 FEET FROM THE CENTER (C) OF THE SPILL SITE.

LOCATION OF SAMPLING POINTS IN A 7-POINT GRID



THE OUTER BODY OF THE SAMPLE AREA IS ASSUMED TO BE 8 FEET FROM THE CENTER (C) OF THE SPILL SITE.

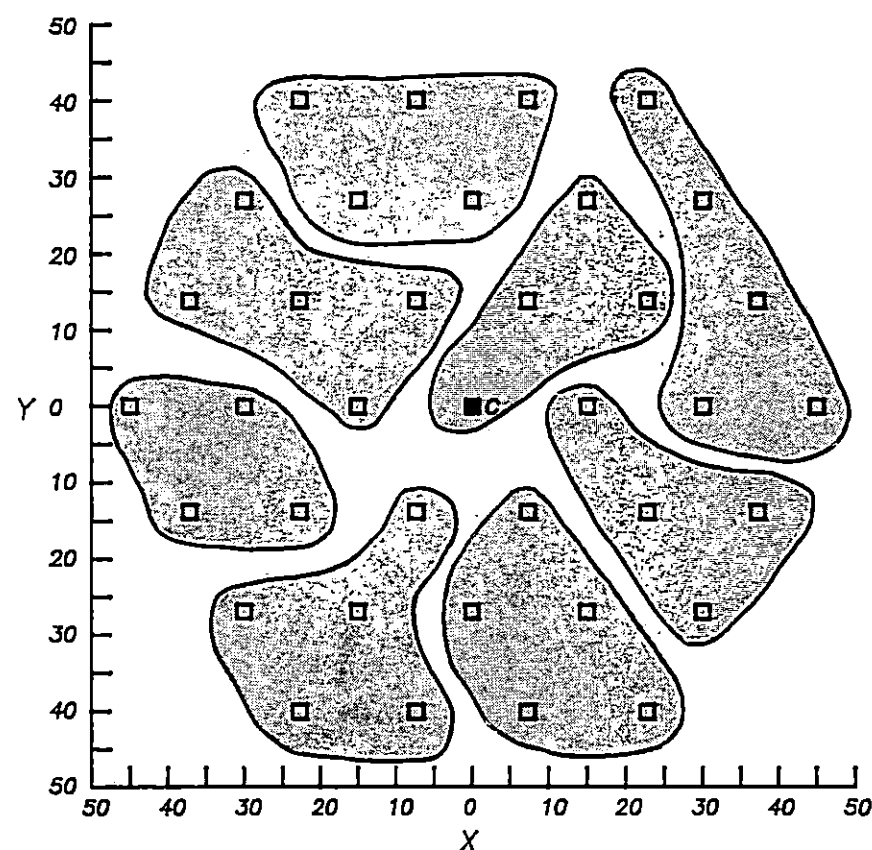
OCCIDENTAL CHEMICAL CORPORATION
HOOKER/RUCO SITE
HICKSVILLE, NEW YORK

HEXAGON SAMPLE GRIDS FOR
THE VERIFICATION SAMPLING

DATE	REVISED	PREPARED BY:	
		LECGETTE, BRASHEARS & GRAHAM, INC. Professional Ground-Water Consultants 72 Danbury Road Wilton, CT 06897 (203) 762-1207	
		DATE: 7/17/91	FIGURE: 3-1

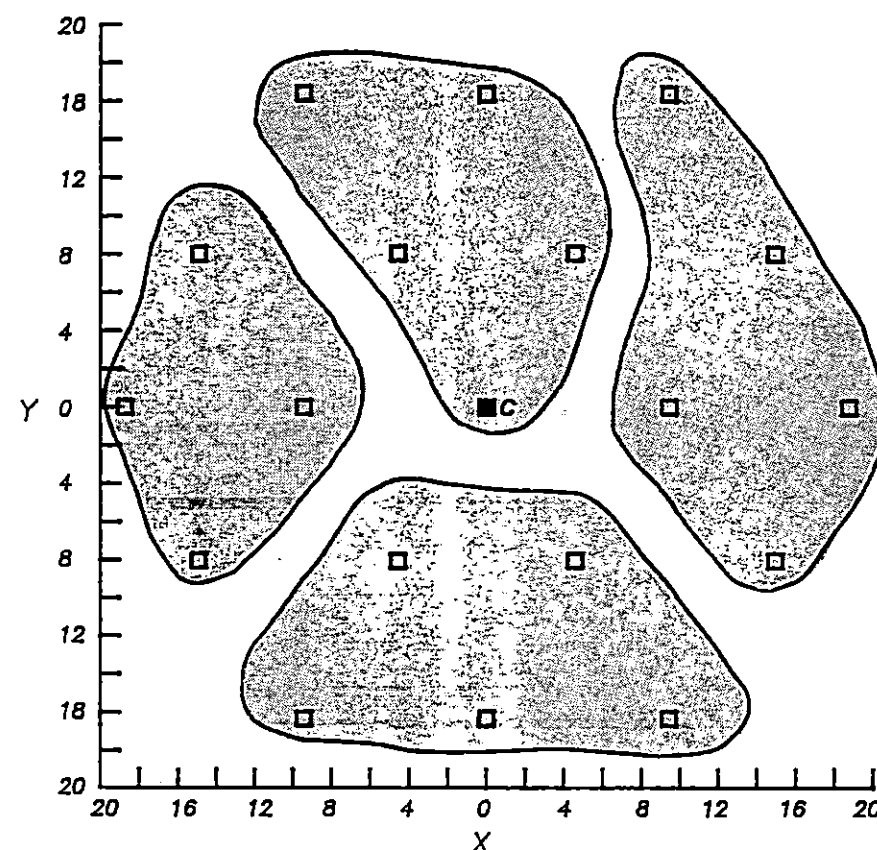
FIGURE 3-2

AN 8 GROUP COMPOSITING PLAN FOR 37 SAMPLE POINTS



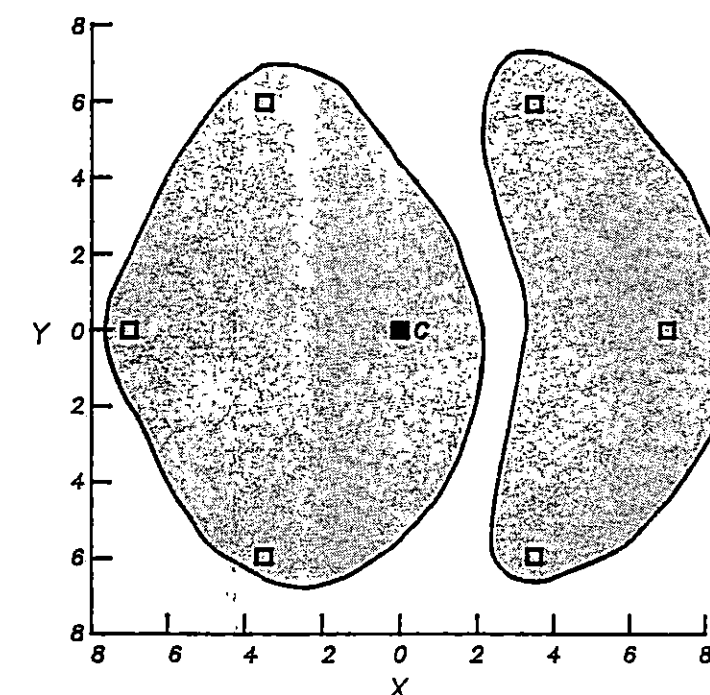
THE OUTER BODY OF THE SAMPLE AREA IS ASSUMED TO BE 50 FEET FROM THE CENTER (C) OF THE SPILL SITE.

A 4 GROUP COMPOSITING PLAN FOR 19 SAMPLE POINTS



THE OUTER BODY OF THE SAMPLE AREA IS ASSUMED TO BE 20 FEET FROM THE CENTER (C) OF THE SPILL SITE.

A 2 GROUP COMPOSITING PLAN FOR 7 SAMPLE POINTS



THE OUTER BODY OF THE SAMPLE AREA IS ASSUMED TO BE 8 FEET FROM THE CENTER (C) OF THE SPILL SITE.

OCCIDENTAL CHEMICAL CORPORATION
HOOKER/RUCO SITE
HICKSVILLE, NEW YORK

COMPOSITING PLANS FOR THE
VERIFICATION SAMPLING GRIDS

DATE	REVISED	PREPARED BY:	
		LEGGETTE, BRASHEARS & GRAHAM, INC. Professional Ground-Water Consultants 72 Danbury Road Wilton, CT 06897 (203) 762-1207	
		DATE: 7/17/91	FIGURE: 3-2

TABLE

TABLE 1

OCCIDENTAL CHEMICAL CORPORATION
HOOKER/RUCO SITE
HICKSVILLE, NEW YORK

Summary of Hazardous Waste Characterization
Parameters and Regulatory Levels for Liquid Waste

Parameter	Regulatory Levels
Ignitability	60°C
Corrosivity	pH ≤ 2 or ≥ 12.5
Reactivity	If the waste is unstable, explosive, generates toxic vapors or reacts violently with water

Toxicity (TCLP)	Hazardous Waste Regulatory Level (mg/l)	Part 703.6 NYS Class GA Effluent Discharge Standards (mg/l)	Safe Drinking Water Act ^{1/} (mg/l)	NYS Drinking Water Standards (mg/l)
Arsenic	5.0	0.05	0.05	0.05
Barium	100.0	2.0	1.0	1.0
Benzene	0.5	ND	0.005	0.005
Cadmium	1.0	0.02	0.005	0.01
Carbon tetrachloride	0.5	0.005	0.005	0.005
Chlordane	0.03	0.0001	0.002	--
Chlorobenzene	100.0	--	0.10	0.005
Chloroform	6.0	0.10	--	0.10 ^{2/}
Chromium	5.0	0.10	0.10	0.05
O, M, P Cresol	200.0 ^{3/}	--	--	--
Cresol	200.0 ^{3/}	--	--	--
2,4-D	10.0	0.0044	0.07	0.05
1,4-Dichlorobenzene	7.5	--	0.60	0.005
1,2-Dichloroethane	0.5	--	0.005	0.005
1,1-Dichloroethylene	0.7	--	0.007	0.005
2,4-Dinitrotoluene	0.13 ^{4/}	--	--	--
Endrin	0.02	ND	--	0.0002
Heptachlor	0.008	ND	0.0004 ^{5/}	--
Hexachlorobenzene	0.13 ^{4/}	0.00035	--	--
Hexachlorobutadiene	0.5	--	--	0.005
Hexachloroethane	3.0	--	--	--
Lead	5.0	0.5	0.05	0.05
Lindane	0.4	ND	0.0002	0.004

TABLE 1
(continued)

OCCIDENTIAL CHEMICAL CORPORATION
HOOKER/RUCO SITE
HICKSVILLE, NEW YORK

Summary of Hazardous Waste Characterization
Parameters and Regulatory Levels for Liquid Waste

Toxicity (TCLP)	Hazardous Waste Regulatory Level (mg/l)	Part 703.6 NYS Class GA Effluent Discharge Standards (mg/l)	Safe Drinking Water Act ^{1/} (mg/l)	NYS Drinking Water Standards (mg/l)
Mercury	0.2	0.004	0.002	0.002
Methoxychlor	10.0	0.035	0.04	0.05
Methyl ethyl ketone	200.0	--	--	--
Nitrobenzene	2.0	--	--	--
Pentachlorophenol	100.0	0.021	--	--
Pyridine	5.0 ^{4/}	--	--	--
Selenium	1.0	0.04	0.05	0.01
Silver	5.0	0.10	0.05	0.05
Tetrachloroethylene	0.7	--	0.005	0.005
Toxaphene	0.5	ND	0.003	0.005
Trichloroethylene	0.5	0.01	0.005	0.005
2,4,5-Trichlorophenol	400.0	--	--	--
2,4,6-Trichlorophenol	2.0	--	--	--
2,4,5-TP (silvex)	1.0	0.00026	0.05	0.01
Vinyl chloride	0.2	0.005	0.002	0.002

1/ Includes Phase II promulgated MCL's.

2/ The limit established for total Trihalomethanes is 0.1 mg/l.

3/ If the O, M and P Cresol concentrations cannot be differentiated, then the total Cresol concentration is used. The regulatory level for total Cresol is 200 mg/l.

4/ Quantitation limit is greater than the regulatory level. The quantitation limit, therefore, becomes the regulatory level.

5/ Heptachlor epoxide maximum contaminant level is 0.0002 mg/l.

mg/l - Milligrams per liter.

ND - Not Detected.

occ.tbl/91-14

APPENDIX 3-1

HUN MODEL PI-101 PHOTOIONIZATION ANALYZER

SPECIFICATIONS AND CALIBRATION

EQUIPMENT SPECIFICATIONS
HNU MODEL PI-101 PHOTOIONIZATION ANALYZER

Application: Detection of trace volatile organic vapors
in ambient air

Method Detection: Photoionization

Ranges: 0-20, 0-200 and 0-2000 ppm (Benzene referred)

Sensitivity: 0.1 ppm

Repeatability: $\pm 1\%$ of full scale

Operational
Temperature: greater than 32°F

Response Time: Less than 3 seconds to 90% of full scale

Dimensions: Probe - 2-1/2" dia. x 11-1/4" long
Unit - 8-1/4" w. x 6-1/2" h. x 5-3/16" d.

Power: Internal rechargeable battery - 12 VDC

Maximum Continuous
Operation: 10 hours on fully charged battery

Outputs: Analog meter; 0-20, 0-200, and 0-2000 ppm
Signal output for recorder - 0-5 VDC

Alarms: Audible and visual alarm to be set at 5 ppm.

STANDARD OPERATING PROCEDURE

HNU MODEL HW-101 PHOTOIONIZATION DETECTOR

Meter Use

1. Unclamp the cover from the main readout assembly and connect the probe cable to the 12 pin keyed connector on the readout assembly panel.
2. Screw the filter nozzle securely into the probe end cap.
3. Check the battery operation. Turn the function switch to the BATT position. If the battery is fully charged, the needle should move to the right and go into the green zone of the scale. If the needle is below the green zone or if the low battery indicator comes on, the batteries must be recharged.
4. Check the zero adjustment. Turn the function switch to the STANDBY position. The needle should align with the zero position on the scale. If this does not occur, then adjust the needle until a zero reading is achieved using the zero adjustment.
5. Select an appropriate operating range using the function switch. It is recommended that the user start with a 0 to 2,000 position and switch to a more sensitive range as required. Once the appropriate operating range has been selected, the instrument is now operational and ready for use.

Calibration Procedure

1. Attach the regulator to the calibration cylinder which has a mixture of 100 ppm isobutylene in pure air. Attach the analyzer directly to the output of the regulator using a short piece (butt connected) of flexible tubing.

2. Open the regulator and allow the calibrant gas to flow directly from the cylinder to the analyzer.

3. Unlock the span control knob on the main readout assembly by turning the locking mechanism counter-clockwise. Adjust the span control knob to read the required setting shown on the calibrant cylinder. (Note, the span knob should be set at 9.8 and the needle should read 57 ppm when the function switch is positioned on the 0 to 200 scale, using a 10.2eV lamp and 100 ppm isobutylene calibrant gas.) After setting the span knob to the correct setting, relock the knob by turning the locking mechanism clockwise.

4. After adjusting the span knob, set the function switch back to STANDBY position and recheck the zero setting. If the zero setting requires adjustment, complete the adjustment and recalibrate the span setting using the calibrant gas.

5. If the span setting is less than 9.0, after zero readjustments, or calibration cannot be achieved, then the lamp bulb must be cleaned.

Lamp Cleaning

1. The function switch must be in the off position prior to disassembling the instrument.

2. Disassemble the probe following directions outlined in Paragraph 6-2.1 of the operation manual.

3. Clean the lamp bulb with a mild detergent, rinse with deionized water and wipe dry with lens paper.

4. If rigorous cleaning of the lamp bulb is required, clean the lamp with special HNU cleaning compound supplied by the manufacturer.

5. Reassemble the probe and recheck the calibration of the analyzer.

lms
May 9, 1991
sop/hsp

APPENDIX 3-2

**GCA MODEL MINIRAM REAL-TIME AEROSOL SENSOR
SPECIFICATIONS AND CALIBRATION**

EQUIPMENT SPECIFICATIONS
GCA MODEL MINIRAM REAL-TIME AEROSOL SENSOR

Application:	Detection of airborne particulates in ambient air
Method Detection:	Near-forward scattered electromagnetic radiation
Ranges:	0.01 to 10 mg/m ³ and 0.1 to 100 mg/m ³
Sensitivity:	0.1% of full scale
Repeatability:	<u>+0.2%</u> of full scale
Operational Temperature:	10°F to 120°F
Response Time:	10 seconds to full scale
Dimensions:	4" x 4" x 2" (approximate)
Power:	7.5 VDC rechargeable battery pack
Maximum Continuous Operation:	12 hours on fully charged battery
Alarm:	Contact closure, internally adjustable
Outputs:	Digital meter real time or time averaged

OPERATION/CALIBRATION: GCA MINIRAM REAL TIME AEROSOL MONITOR**1.0 PURPOSE**

Describes the operation and calibration techniques for the MINIRAM Aerosol Sampler.

2.0 DEFINITIONS

The MINIRAM real time aerosol monitor is an aerosol sensor designed for personal aerosol monitoring. The MINIRAM provides real time and time averaged concentration readouts of airborne particulates. The digital signal can be read from the front panel. The MINIRAM unit of measure is mg/m³ and operates in two ranges: 0.01 to 10 mg/m³ and 0.1 to 100 mg/m³.

3.0 RESPONSIBILITIES

3.1 The Site Technician will be responsible for the operation and calibration of the MINIRAM.

3.2 The Site Technician will check the following:

- Zero
- Precision

3.3 The Safety Officer will be responsible for insuring that the work is performed and that the required data is collected.

4.0 OPERATION

4.1 The normal operation procedure is as follows:

- 1) Turn the unit on and program the internal microprocessor for sample time, averaging time, alarm level, and range per instruction manual.
- 2) Allow the unit to warm up for 5 minutes before calibration or sampling.

4.2 Calibration Check

- 1) The MINIRAM is factory calibrated against a filter-gravimetric reference using standard test dust, and therefore the manufacturer does not recommend a field span calibration.

- 2) If a question is raised as to the accuracy of a field MINIRAM and/or the precision check is out of tolerance, a filter/gravimetric sampler will be used to verify and/or adjust the MINIRAM. This will be done in accordance with the procedure recommended by the manufacturer in the instrument instruction manual.

4.3 Zero Adjust Procedure (Daily)

- 1) Assure that the unit is in the sample mode and the display selector is in real time.
- 2) Attach the zero check/pump module to the unit.
- 3) Sample air and adjust if necessary.
- 4) Return to normal operation.

4.4 Precision Checks

A precision check will be conducted on each field MINIRAM weekly during Active Operations at the Working Site. The precision check is conducted by comparing the reading of the field MINIRAM to that of the standard MINIRAM when their sensors are held close together. The readings of the two units are recorded in the log book as "Precision Check".

5.0 REPAIR AND MAINTENANCE

If the instrument fails to function as per manufacturer's specifications, it will be replaced with a spare. The defective unit will be sent to the factory for repair. Appropriate spare equipment will be available either at the Project Site or at a location near the Durez plant.

APPENDIX 3-3

**MSA MODEL 260 COMBUSTIBLE GAS AND OXYGEN ALARM
SPECIFICATIONS AND CALIBRATION**

A8-10
Equipment Specifications

MSA Model 260 Combustible Gas and Oxygen Alarm

Application:	Monitor areas for combustible gases, and/or oxygen deficiency
Method Detection:	Catalytic combustion and REDOX reactions
Range:	0-100% lower explosive limit (LEL) 0-25% oxygen
Sensitivity:	1% of full scale
Repeatability:	$\pm 1\%$ of full scale for LEL $\pm 5\%$ for oxygen
Operational Temperature:	14°F to 140°F
Response Time:	90% in less than 20 seconds
Dimensions:	10" w. x 7" H. x 3 3/4" d.
Power:	Internal 2.4 volt rechargeable battery pack
Maximum Continuous Operation:	8 to 10 hours on fully charged battery
Alarms:	Audio and visual at 50% LEL and less than 19.5% oxygen
Output:	Analog meter

OPERATION/CALIBRATION: COMBUSTIBLE GAS AND OXYGEN ALARM

1.0 Applicability

The combustible gas and oxygen alarm is designed to monitor areas for combustible gases, and/or oxygen deficiency. The instrument is portable, but its audible/visible alarm permits the instrument to be used effectively as a semi-continuous monitor in work areas. The alarm will be operated on AC power when available.

2.0 Apparatus

- 2.1 Combustible gas and oxygen alarm: MSA Model 260 or equivalent.
- 2.2 Zero air: charcoal filtered ambient air or equivalent
- 2.3 Calibration gas: 0.6% methane in air (27% LEL) (blind tolerance $\pm 5\%$, certification tolerance $\pm 2\%$).
- 2.4 Calibration Kit: MSA, Model R and RP or equivalent

3.0 Responsibilities

- 3.1 The Site Technician will be responsible for the calibration, operation and maintenance of the Combustible Gas and Oxygen Alarm.
- 3.2 The Safety Officer will be responsible for ensuring that the work is performed and that the required data is collected.

4.0 Operation

The operation of the combustible gas and oxygen alarm is essentially identical to that of the MSA Model 100, except that the oxygen meter needs to be calibrated to 20.8% in fresh or ambient air.

5.0 Repairs and Maintenance

If the calibration checks are not within the limits specified in the manufacturer's instruction manual, the instrument should be replaced with a spare, if available, and returned to the factory for diagnostic treatment and correction. Spare equipment will be available either at the Project Site or at a location near the Durez plant.

APPENDIX 3-4
AIR MONITORING PROCEDURES
SPECIFICATIONS AND CALIBRATION

CALIBRATION OF THE DUPONT ALPHA-1

- c. Adjustment for the desired flow rate will be made using the low-flow valve. (The high-flow valve may have to be opened slightly if the desired flow rate cannot be attained using the low-flow valve by itself.)
- o High-Flow Range (1000-5000 cc/min)
 - a. Close the bypass valve completely by gently turning it clockwise until it seats.
 - b. Adjustment for the desired flow rate will be made by using the high-flow valve. The low-flow valve may be used as a "fine tuning" control.
- 4.5 In order to simulate the pressure drop that will be encountered during sampling, attach a sample line along with the type of sample collection media that will be utilized for sampling, to the inlet of the pump.
- 4.6 Measure the flow rate through the sampling system with the respective rotameter and adjust the flow control valve to read the approximate desired flow rate. Allow 10 to 15 seconds after adjustment for the flow control mechanism to respond.
- 4.7 Remove the rotameter. Wet down the inside of the bubble tube with soap solution by allowing several bubbles to pass up the full length of the bubble tube. Attach the bubble tube to the pump sample line and collection media system.
- 4.8 Determine the observed volume flow rate. Use a stopwatch to measure the time it takes for one bubble to rise between any two graduated marks. The observed volume is the value difference from one mark to another. This volume divided by the elapsed time will equal the observed flow rate. (The bubble must be allowed to travel at least one liter while being timed with the stopwatch.)

$$\text{Flow Rate} = \text{Volume/Time}$$
- 4.9 If the flow rate needs adjustment, repeat the procedure in 4.6 through 4.8 until the desired flow rate is obtained.
- 4.10 After all adjustments, if any, are completed, run five individual tests with the bubble meter, average the flows, and record the volume and elapsed time on the calibration data sheet.

AIR SAMPLING WORKSHEET

OCCIDENTAL CHEMICAL CORPORATION
INDUSTRIAL HYGIENE

FACILITY _____

SAMPLING DATE _____

PERSON PERFORMING SAMPLING _____

Employee (Name, Social Security Number) _____

Job Title _____

Pump Checks and Adjustments _____

PPE (Type) _____

Job Description, Operation, Work Location(s), Ventilation and Controls _____

Temperature: _____

Relative Humidity: _____

Wind Speed, Direction: _____

PUMP NUMBER: _____

SAMPLING DATA

Lab Sample Number						
Sample Submission Number						
Sample Type						
Sample Media						
Filter/Tube Number						
Time ON/OFF						
Total Time (In Minutes)						
Flow Rate <input type="checkbox"/> l/min <input type="checkbox"/> cc/min.						
Volume (In Liters)						
Net Sample Weight (In mg)						
Analyze Samples For:	Indicate Which Samples to Include in TWA, Ceiling, STEL, etc. Calculations					
Interferences and Comments to Lab	Supporting Samples		Chain of Custody		Initials	DATE
	a. Blanks: _____		a. Seals Intact?		Y	N
	b. BULKs: _____		b. Rec'd in Lab			
			c. Rec'd by Anal			
			d. Anal Compl'd			
			e. Calc Checked			
			f. Supt OK'd			

P R E	Pump Mfg & SN	Flow Rate Calculations				
	Voltage Checked					
	Yes No					
	Location / T & Alt.					
		Flow Rate	Method	<input type="checkbox"/> Bubble <input type="checkbox"/> PR	Initials	Date / Time

[illegible]

WATER LOSS CALCULATIONS					
Filter No.					
Final Weight (mg)					
Initial Weight (mg)					
Weight Gained (mg)					
Blank Adjustment					
Net Sample Weight (mg)					

CALCULATIONS AND NOTES:

This image shows a single sheet of white paper with horizontal black ruling lines. The lines are evenly spaced and run across the width of the page. There are approximately 20 lines visible. The paper appears to be a standard notebook or ledger page.

SAMPLING TOTAL PARTICULATES USING GRAVIMETRIC FILTER ANALYSIS

1.0 General Applicability

- 1.1 Ambient air concentrations of total particulates will be determined by drawing ambient air through a pre-weighed PVC filter, and then re-weighing the filter to determine the total weight gained. The sample collection procedure involves determining the volume of air sampled through measurement of flow rate and sample duration.**
- 1.2 The collection filter consists of a 37 mm diameter polyvinyl chloride (PVC) filter with a pore size of 5 μ m, a cellulose support pad, and a polystyrene filter holder. These filters will meet the requirements set forth in NIOSH Method 0500.**
- 1.3 The sampling and analysis procedure used in this program will follow NIOSH Method 0500.**

NUISANCE DUST, TOTAL

ISSUED: 2/15/84

PROPERTIES: quartz less than 1% [1]

ACGIH: 10 mg/m³, total dust less than 1% quartz

SYNONYMS: boron oxide (CAS #1303-86-2) and nuisance dusts [1] including alumina (CAS #1344-28-1), calcium carbonate (CAS #1317-65-3), cellulose (paper fiber; CAS #9004-34-6), glycerin mist (CAS #56-81-5), limestone (CAS #1317-65-3), etc.

APPLICABILITY: The working range is 3 to 20 mg/m³ for a 100-L air sample. This method is nonspecific and determines the total dust concentration to which a worker is exposed. It may be applied, e.g., to gravimetric determination of fibrous glass [4] in addition to the other ACGIH nuisance dusts [1].

INTERFERENCES: Organic and volatile particulate matter may be removed by dry ashing [4].

OTHER METHODS: This method is similar to the criteria document method for fibrous glass [4] and Method 5000 for carbon black. This method replaces Method S349 [5]. Impingers and direct-reading instruments may be used to collect total dust samples, but these have limitations for personal sampling.

EQUIPMENT:

1. Environmental chamber at constant temperature and humidity (e.g., $20\text{ }^{\circ}\text{C} \pm 0.3\text{ }^{\circ}\text{C}$ and $50\% \pm 5\% \text{ RH}$).
2. Sampler: 37-mm PVC, 2- to 5- μm pore size membrane or equivalent hydrophobic filter and cellulose supporting pad in 37-mm cassette filter holder.
3. Personal sampling pump, 1.5 to 2 L/min, with flexible connecting tubing.
4. Microbalance, capable of weighing to 0.01 mg.
5. Vacuum desiccator.
6. Static neutralizer: e.g., Po-210; replace nine months after the production date.

SPECIAL PRECAUTIONS: None.

PREPARATION OF FILTERS BEFORE SAMPLING:

1. Dry filters and backup pads under vacuum in the vacuum desiccator for at least 15 min.
2. Release the vacuum, remove the desiccator cover and equilibrate the filters in the environmental chamber for at least 1 hr.
3. Number the backup pads with a ballpoint pen and place them, numbered side down, in filter cassette bottom sections.
4. Weigh the filters in the environmental chamber. Record the filter tare weight, W_1 (mg).
 - a. Zero the balance before each weighing.
 - b. Handle the filter with forceps (nylon forceps if further analyses will be done).
 - c. Pass the filter over an antistatic radiation source. Repeat this step if filter does not release easily from the forceps or if filter attracts balance pan. Static electricity can cause erroneous weight readings.
5. Place the weighed filters on top of the backup pads in the filter cassette bottom sections and allow to stand an additional 8 to 16 hrs in the environmental chamber.
6. Reweigh the filters. If this tare weight differs by more than 0.01 mg from the first tare weight obtained in step 4 above, discard the filter.

NOTE: Insert a rod through the outlet hole of the filter cassette bottom section to raise the backup pad and filter so that the filter can be grasped with forceps.
7. Assemble the filter in the filter cassettes and close firmly so that leakage around the filter will not occur. Place a plug in each opening of the filter cassette. Place a cellulose shrink band around the filter cassette, allow to dry and mark with the same number as the backup pad.

SAMPLING:

8. Calibrate each personal sampling pump with a representative sampler in line.
9. Sample at 1.5 to 2 L/min. Do not exceed a total filter loading of approximately 2 mg total dust.

SAMPLE PREPARATION:

10. Wipe dust from the external surface of the filter cassette with a moist paper towel to minimize contamination. Discard the paper towel.
11. Remove the top and bottom plugs from the filter cassette. Place the filter cassettes in a vacuum desiccator under vacuum for at least 15 min, followed by equilibration for at least 1 hr in the environmental chamber.
12. Remove the cassette band, pry open the cassette and remove the filter. Handle the filters very gently by the edge to avoid loss of dust.

NOTE: If the filter sticks to the underside of the cassette top, very gently lift away by using the dull side of a scalpel blade. This must be done carefully or the filter will tear.

CALIBRATION AND QUALITY CONTROL:

13. Zero the microbalance before all weighings. Use the same microbalance for weighing filters before and after sample collection. Maintain and calibrate the balance with National Bureau of Standards Class M weights.
14. Take two to four replicate samples for every batch of field samples for quality assurance on the sampling procedures. The set of replicate samples should be exposed to the same dust environment, either in a laboratory dust chamber [6] or in the field. The quality control samples must be taken with the same equipment, procedures and personnel used in the routine field samples. The relative standard deviation calculated from these replicates should be recorded on control charts and action taken when the precision is out of control.

MEASUREMENT:

15. Weigh each filter, including field blanks. Record this post-sampling weight, W_2 (mg), beside its corresponding tare weight. Record anything remarkable about a filter (e.g., overload, leakage, wet, torn, etc.).

CALCULATIONS:

16. Calculate the concentration of total nuisance dust, C (mg/m³), in the air volume sampled, V (L):

$$C = \frac{(W_2 - W_1) + B}{V} \cdot 10^3, \text{ mg/m}^3$$

where: W_1 = tare weight of filter before sampling (mg)

W_2 = post-sampling weight of sample-containing filter (mg)

B = mean change in field blank filter weights between tare and post-sampling (mg)
(+ or -).

EVALUATION OF METHOD:

Lab testing with blank filters and generated atmospheres of carbon black was done at 8 to 28 mg/m³ [2,6]. Precision and accuracy data are given on page 0500-1.

REFERENCES:

- [1] TLVs - Threshold Limit Values for 1983-84, Appendix D, ACGIH, Cincinnati, OH (1983).
- [2] This Manual, Method 5000.
- [3] Unpublished data from Non-textile Cotton Study, NIOSH/ORDS/EIB.
- [4] NIOSH Criteria for a Recommended Standard ... Occupational Exposure to Fibrous Glass, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-152, 119-142 (1977).
- [5] NIOSH Manual of Analytical Methods, 2nd ed., V. 3, S349, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C (1977).
- [6] Documentation of the NIOSH Validation Tests, S262 and S349, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977).

METHOD WRITTEN BY: Kathy Moring, Jerry Clare, and Frank Hearl, P.E., NIOSH/ORDS.

SAMPLING AND ANALYSIS: Polychlorobiphenyls Using NOISH Method 5503

FORMULA: mixture: $C_{12}H_{10-x}Cl_x$
[where $x = 1$ to 10]

M.W.: ca. 258 (42% Cl ; $C_{12}H_7Cl_2$);
ca. 326 (54% Cl ; $C_{12}H_5Cl_5$)

POLYCHLOROBIPHENYLS

METHOD: 5503

ISSUED: 2/15/84

REVISION #1: 8/15/87

OSHA: 1 mg/m³ (42% Cl);
0.5 mg/m³ (54% Cl)
NIOSH: 0.001 mg/m³ [1,2]
ACGIH: 1 mg/m³ (42% Cl); STEL 2 mg/m³
0.5 mg/m³ (54% Cl); STEL 1 mg/m³
(skin)

PROPERTIES: 42% Cl: BP 325 to 366 °C; MP -19 °C;
d 1.38 g/mL @ 25 °C;
VP 0.01 Pa (8×10^{-5} mm Hg;
1 mg/m³) @ 20 °C [3]
54% Cl: BP 365 to 390 °C; MP 10 °C;
d 1.54 g/mL @ 25 °C;
VP 0.0004 Pa (3×10^{-6} mm Hg;
0.05 mg/m³) @ 20 °C [3,4]

SYNONYMS: PCB; CAS #1336-36-3; 1,1'-biphenyl chloro (CAS #27323-18-8); chlorodiphenyl, 42% Cl (Aroclor 1242; CAS #53469-21-9), and 54% Cl (Aroclor 1254; CAS #11097-69-1)

SAMPLING	MEASUREMENT
SAMPLER: FILTER + SOLID SORBENT (13-mm glass fiber + Florisil, 100 mg/50 mg)	! TECHNIQUE: GAS CHROMATOGRAPHY, ECD (⁶³ Ni) ! ! ANALYTE: polychlorobiphenyls !
FLOW RATE: 0.05 to 0.2 L/min or less	! DESORPTION: filter + front section, 5 mL hexane; ! back section, 2 mL hexane !
VOL-MIN: 1 L @ 0.5 mg/m ³ -MAX: 50 L	! INJECTION VOLUME: 4 µL with 1-µL backflush !
SHIPMENT: transfer filters to glass vials after sampling	! TEMPERATURE-INJECTION: 250 - 300 °C ! -DETECTOR: 300 - 325 °C ! -COLUMN: 180 °C !
SAMPLE STABILITY: unknown for filters; 2 months for Florisil tubes [5]	! CARRIER GAS: N ₂ , 40 mL/min !
BLANKS: 10% of samples	! COLUMN: glass, 1.8 m x 2 mm ID, 1.5% OV-17/1.95% ! QF-1 on 80/100 mesh Chromosorb WHP !
ACCURACY	! CALIBRATION: standard PCB mixture in hexane !
RANGE STUDIED: not studied	! RANGE: 0.4 to 4 µg per sample [6] !
BIAS: none identified	! ESTIMATED LOQ: 0.03 µg per sample [6] !
OVERALL PRECISION (s _p): not evaluated	! PRECISION (s _p): 0.044 [5] !
APPLICABILITY: The working range is 0.01 to 10 mg/m ³ for a 40-L air sample [5]. With modifications, surface wipe samples may be analyzed [7,8].	
INTERFERENCES: Chlorinated pesticides, such as DDT and DDE, may interfere with quantitation of PCB. Sulfur-containing compounds in petroleum products also interfere [9].	
OTHER METHODS: This method revises Methods S120 [10], 5503 (dated 2/15/84), and P&CAM 244 [5]. Methods S121 [11] and P&CAM 253 [12] for PCB have not been revised.	

REAGENTS:

1. Hexane, pesticide quality.
2. Florisil, 30/48 mesh sieved from 30/60 mesh. After sieving, dry at 105 °C for 45 min. Mix the cooled Florisil with 3% (w/w) distilled water.
3. Nitrogen, purified.
4. Stock standard solution of the PCB in methanol or isooctane (commercially available).*

*See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: 13-mm glass fiber filter without binders in a Swinnex cassette (Cat. No. SX 0001300, Millipore Corp.) followed by a glass tube, 7 cm long, 6 mm OD, 4 mm ID containing two sections of 30/48 mesh deactivated Florisil. The front section is preceded by glass wool and contains 100 mg and the backup section contains 50 mg; urethane foam between sections and behind the backup section. Join the cassette and Florisil tube with PVC tubing, 3/8" L x 9/32" OD x 5/32" ID, on the outlet of the cassette and with another piece of PVC tubing, 3/4" L x 5/16" OD x 3/16" ID, complete the union.
2. Personal sampling pump, 0.05 to 0.2 L/min, with flexible connecting tubing.
3. Tweezers.
4. Vials, glass, 4- and 7-mL, with aluminum or PTFE-lined caps.
5. Gas chromatograph, electron capture detection (⁶³Ni), integrator and column (page 5503-1).
6. Volumetric flasks, 10-mL and other convenient sizes for preparing standards.
7. Syringe, 10-μL.

SPECIAL PRECAUTIONS: Avoid prolonged or repeated contact of skin with PCB and prolonged or repeated breathing of the vapor [1,2,13].

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Break the ends of the Florisil tube immediately before sampling. Connect Florisil tube to Swinnex cassette and attach sampler to personal sampling pump with flexible tubing.
3. Sample at an accurately known flow rate between 0.05 and 0.2 L/min for a total sample size of 1 to 50 L.

NOTE: At low PCB concentrations, the sampler was found to be efficient when operated at flow rates up to 1 L/min, for 24 hours [8]. Under these conditions, the limit of detection was 0.02 μg/m³.

4. Transfer the glass fiber filters to 7-mL vials. Cap the Florisil tubes with plastic (not rubber) caps and pack securely for shipment.

SAMPLE PREPARATION:

5. Place the glass wool and 100-mg Florisil bed in the same 7-mL vial in which the filter was stored. Add 5.0 mL hexane.

NOTE: For surface wipe samples, extract each gauze pad with 25 mL hexane [7].

6. In a 4 mL vial, place the 50-mg Florisil bed including the two urethane plugs. Add 2.0 mL hexane.
7. Allow to stand 20 min with occasional agitation.

CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least five working standards over the range 10 to 500 ng PCB/mL.
 - a. Add known amounts of stock standard solution to hexane in 10-mL volumetric flasks and dilute to the mark.
 - b. Analyze together with samples and blanks (steps 11 and 12).
 - c. Prepare calibration graph (sum of areas of selected peaks vs. ng PCB/mL).
9. Determine desorption efficiency (DE) at least once for each lot of glass fiber filters and Florisil used for sampling in the calibration range (step 8). Prepare three tubes at each of five levels plus three media blanks.
 - a. Remove and discard back sorbent section of a media blank Florisil tube.
 - b. Inject known amounts of stock standard solution directly onto front sorbent section and onto a media blank filter with a microliter syringe.
 - c. Cap the tube. Allow to stand overnight.
 - d. Desorb (steps 5 through 7) and analyze together with working standards (steps 11 and 12).
 - e. Prepare a graph of DE vs. µg PCB recovered.
10. Analyze three quality control blind spikes and three analyst spikes to ensure that the calibration graph and DE graph are in control.

MEASUREMENT:

11. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 5503-1. Inject sample aliquot manually using solvent flush technique or with autosampler.

NOTE 1: Where individual identification of PCB is needed, a procedure using a capillary column may be used [14].

NOTE 2: If peak area is above the linear range of the working standards, dilute with hexane, reanalyze and apply the appropriate dilution factor in calculations.

12. Sum the areas for five or more selected peaks.

CALCULATIONS:

13. Determine the mass, ng (corrected for DE) of PCB found on the glass fiber filter (W) and in the Florisil front (W_f) and back (W_b) sorbent sections, and in the average media blank filter (B) and front (B_f) and back (B_b) sorbent sections.

NOTE: If $W_b > W_f/10$, report breakthrough and possible sample loss.

14. Calculate concentration, C , of PCB in the air volume sampled, V (L):

$$C = \frac{(W + W_f + W_b - B - B_f - B_b) \cdot 10^{-9}}{V}, \text{ ng/m}^3.$$

EVALUATION OF METHOD:

This method uses 13-mm glass fiber filters which have not been evaluated for collecting PCB. In Method S120, however, Aroclor 1242 was completely recovered from 37-mm glass fiber filters using 15 mL isooctane [12,15,16]. With 5 mL of hexane, Aroclor 1016 was also completely recovered from 100-mg Florisil beds after one-day storage [5]. Thus, with no adsorption effect likely on glass fiber filters for PCB, 5 mL hexane should be adequate to completely extract PCB from combined filters and front sorbent sections. Sample stability on glass fiber filters has not been investigated. Breakthrough volume was >48 L for the Florisil tube at 75% RH in an atmosphere containing 10 mg/m³ Aroclor 1016 [5].

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- [16] NIOSH Research Report-Development and Validation of Methods for Sampling and Analysis of Workplace Toxic Substances, U.S. Department of Health and Human Services, Publ. (NIOSH) 80-133 (1980).

METHOD REVISED BY: James E. Arnold, NIOSH/DPSE; S120 originally validated under NIOSH Contract 210-76-0123.

Table 1. Composition of some Aroclors [3].

Major Components	Aroclor 1016	Aroclor 1242	Aroclor 1254
Biphenyl	0.1%	<0.1%	<0.1%
Monochlorobiphenyls	1	1	<0.1
Dichlorobiphenyls	20	16	0.5
Trichlorobiphenyls	57	49	1
Tetrachlorobiphenyls	21	25	21
Pentachlorobiphenyls	1	8	48
Hexachlorobiphenyls	<0.1	1	23
Heptachlorobiphenyls	none detected	<0.1	6
Octachlorobiphenyls	none detected	none detected	none detected

EQUIPMENT SPECIFICATIONS
DU PONT ALPHA-1 AIR SAMPLER

Application:	Low-flow personal sampling pump
Pump Type:	Dual opposed diaphragm
Flow Rate Range:	5-5000 cc/min constant flow
Preserve Drop Capability:	25" W.C. maximum
Control Capability:	Microprocessor controlled for: <ul style="list-style-type: none">• run time• start time• tolerated restricted flow time• flow rate readout• average air temp. readout
Constant Flow Control:	$\pm 5\%$ of set flowrate
Power:	Internal rechargeable battery pack
Dimensions:	2-1/4" x 4" x 5"

EQUIPMENT SPECIFICATIONS
FILTERS FOR NIOSH 0500

Application: Collection of total respirable particulates in air
Supplier: SKC
Dimensions: 37 mm diameter
Pore size: 5.0 μ m
Material: Polyvinyl Chloride
Catalog Number: 225-8

EQUIPMENT SPECIFICATIONS
TEFLON FILTERS FOR NIOSH METHOD 5503

Application:	Collection of particle associated semivolatile organic compounds
Supplier:	SKC
Dimensions:	13mm diameter
Pore Size:	1.0 um
Material:	Polytetra fluoroethylene XAD-2 CARTRIDGE FOR NIOSH METHOD 5503
Application:	Collection of semivolatile organic compounds
Supplier:	SKC
Dimensions:	8mm diameter x 100mm/long
Sections:	2
Sorbent mg:	50/100
Cartridge Ends:	Scaled glass

CALIBRATION OF THE DUPONT ALPHA-1

1.0 Applicability

This procedure describes the steps necessary for calibration of the DuPont, Alpha-1 Sampling Pump.

2.0 Responsibilities

The site technician or field operator will be responsible for calibrating the DuPont Alpha-1 at the frequency required in the Project Work Plan.

3.0 Supporting Materials

- DuPont Calibrator Case (or other standard bubble tube calibrator with stop watch)
- Two indicating rotameters - 0-500 cc/min and 0-5000 cc/min
- Mercury thermometer
- Aneroid Barometer (or other means of obtaining local, barometric air pressure)
- Calibration Data Sheet

4.0 Procedure

- 4.1 Determine the flow rate and the type of sampling media that will be used for sampling.
- 4.2 Record the date, time, project #, sample media type, pump serial number, ambient temperature and barometric pressure on the calibration data sheet.
- 4.3 Turn "on" the Alpha-1 by pressing the On/Off and Hold/Run push buttons.
- 4.4 Set the flow rate for the Alpha-1 to the desired flow range as follows:
 - Low-Flow Range (5-1000 cc/min)
 - a. Open the bypass valve fully by turning it three turns counterclockwise from the fully closed position.
 - b. Close the high-flow valve completely by gently turning it clockwise until it seats.

CALIBRATION OF THE DUPONT ALPHA-1

4.11 Determine the standard flow rate as follows:

4.11.1 Average the run times for the five tests completed in 4.10 above and record on the calibration data sheet.

4.11.2 Determine the flow rate at standard conditions, (760 mm Hg, 25°C, and 0% water vapor), using the equation below:

$$\text{Standard Flow Rate} = \frac{\text{Vol (ml)}}{\text{Time (min)}} \times \frac{(P - P_v)}{760} \times \frac{298.16}{(273 + T)}$$

where

P = atmospheric pressure, mm Hg
 P_v = vapor pressure of water, mm Hg (see Table 4.1)
 T = temperature of gas, °C

4.11.3 Record the initial flow rate at standard conditions on the data sheet.

4.12 Disconnect the sample line and sample collection media from the pump.

4.13 The pump is now calibrated and ready for field use. (Note: All pumps should remain running until sampling is completed. Turning the pumps off may affect the calibrated flow rate.)

4.14 Following the sampling period, and before turning the pumps "off" a final flow calibration check will be completed. This will be done by repeating steps 4.7, 4.8, 4.10, 4.11 and 4.12 and, without making any adjustments, record necessary information on the calibration data sheet.

4.15 The average standard flow rate is determined by averaging the pre-test standard flow rate with the post-test standard flow rate.

5.0 Acceptance Criteria

5.1 All blank spaces on the calibration data sheet will be filled out or marked "NA".

CALIBRATION OF THE DUPONT ALPHA-1

5.2 The pre-test standard flow rate must agree with the post-test standard flow rate to within $\pm 10\%$. If it does not, re-run the post-calibration tests and re-check temperature readings, pressure readings, and all calculations. If repeated validations of the post calibration test result in flow differentials of $>10\%$ then the average standard flow rate should be treated as suspect. If the corresponding sample is selected for actual analyses then the resulting values should be treated as approximate concentrations.

6.0 Document Submission

6.1 All calibration data sheets will be submitted to the program manager or field coordinator within 3 days.

CALIBRATION OF THE DUPONT ALPHA-1

Table 4-1

SATURATION VAPOR PRESSURE OVER WATER ($^{\circ}\text{C}$, mm Hg)^a
 Values for Fractional Degree Between 50 and
 89 Were Obtained by Interpolation

T _{mm}	0.0	0.2	0.4	0.6	0.8	T _{mm}	0.0	0.2	0.4	0.6	0.8
-16	1.436	1.414	1.390	1.363	1.343	42	61.50	62.14	62.80	63.46	64.12
-15	1.360	1.344	1.327	1.311	1.293	43	64.80	65.48	66.16	66.84	67.52
-14	1.281	1.264	1.247	1.231	1.213	44	68.28	68.97	69.66	70.34	71.02
-13	1.204	1.186	1.168	1.151	1.133	45	71.84	72.53	73.22	73.90	74.58
-12	1.128	1.109	1.090	1.072	1.053	46	75.48	76.18	76.87	77.55	78.23
-11	1.052	1.032	1.013	0.994	0.975	47	79.20	79.90	80.59	81.27	81.94
-10	0.976	0.955	0.935	0.915	0.895	48	83.00	83.70	84.38	85.05	85.71
-9	0.900	0.878	0.857	0.836	0.815	49	86.88	87.58	88.25	88.91	89.56
-8	0.824	0.801	0.779	0.757	0.734	50	90.84	91.54	92.21	92.86	93.50
-7	0.748	0.724	0.701	0.678	0.654	51	94.88	95.58	96.24	96.88	97.51
-6	0.672	0.647	0.623	0.598	0.573	52	99.00	99.70	100.36	100.99	101.60
-5	0.596	0.570	0.544	0.518	0.491	53	103.20	103.90	104.55	105.17	105.77
-4	0.520	0.493	0.466	0.439	0.411	54	107.48	108.18	108.82	109.43	110.02
-3	0.444	0.416	0.388	0.360	0.331	55	111.84	112.54	113.17	113.77	114.34
-2	0.368	0.339	0.310	0.281	0.251	56	116.28	116.98	117.60	118.19	118.75
-1	0.292	0.262	0.232	0.202	0.171	57	120.80	121.50	122.11	122.69	123.24
0	0.216	0.185	0.154	0.123	0.091	58	125.40	126.10	126.70	127.27	127.81
1	0.140	0.108	0.076	0.044	0.012	59	130.08	130.78	131.37	131.93	132.46
2	0.064	0.032	0.000	-0.032	-0.064	60	134.84	135.54	136.12	136.67	137.19
3	0.000	-0.032	-0.064	-0.096	-0.128	61	139.68	140.38	140.95	141.49	142.00
4	-0.064	-0.096	-0.128	-0.160	-0.192	62	144.60	145.30	145.86	146.39	146.89
5	-0.128	-0.160	-0.192	-0.224	-0.256	63	149.60	150.30	150.85	151.37	151.86
6	-0.192	-0.224	-0.256	-0.288	-0.320	64	154.68	155.38	155.92	156.43	156.91
7	-0.256	-0.288	-0.320	-0.352	-0.384	65	159.84	160.54	161.07	161.57	162.04
8	-0.320	-0.352	-0.384	-0.416	-0.448	66	165.08	165.78	166.30	166.79	167.25
9	-0.384	-0.416	-0.448	-0.480	-0.512	67	170.40	171.10	171.61	172.09	172.54
10	-0.448	-0.480	-0.512	-0.544	-0.576	68	175.80	176.50	176.99	177.45	177.88
11	-0.512	-0.544	-0.576	-0.608	-0.640	69	181.28	181.98	182.46	182.91	183.33
12	-0.576	-0.608	-0.640	-0.672	-0.704	70	186.84	187.54	188.01	188.45	188.86
13	-0.640	-0.672	-0.704	-0.736	-0.768	71	192.48	193.18	193.64	194.07	194.47
14	-0.704	-0.736	-0.768	-0.799	-0.830	72	198.20	198.90	199.35	199.77	200.16
15	-0.768	-0.799	-0.830	-0.860	-0.890	73	204.00	204.70	205.14	205.55	205.93
16	-0.830	-0.860	-0.890	-0.920	-0.950	74	209.88	210.58	210.99	211.37	211.72
17	-0.890	-0.920	-0.950	-0.980	-1.010	75	215.84	216.54	216.93	217.29	217.62
18	-0.950	-0.980	-1.010	-1.040	-1.070	76	221.88	222.58	222.95	223.29	223.60
19	-0.980	-1.010	-1.040	-1.070	-1.100	77	228.00	228.70	229.05	229.37	229.66
20	-1.010	-1.040	-1.070	-1.100	-1.130	78	234.20	234.90	235.23	235.53	235.80
21	-1.040	-1.070	-1.100	-1.130	-1.160	79	240.48	241.18	241.50	241.78	242.03
22	-1.070	-1.100	-1.130	-1.160	-1.190	80	246.84	247.54	247.85	248.12	248.36
23	-1.100	-1.130	-1.160	-1.190	-1.220	81	253.28	253.98	254.27	254.53	254.76
24	-1.130	-1.160	-1.190	-1.220	-1.250	82	259.80	260.50	260.77	261.01	261.22
25	-1.160	-1.190	-1.220	-1.250	-1.280	83	266.40	267.10	267.35	267.57	267.76
26	-1.190	-1.220	-1.250	-1.280	-1.310	84	273.08	273.78	274.01	274.20	274.37
27	-1.220	-1.250	-1.280	-1.310	-1.340	85	279.84	280.54	280.76	280.94	281.09
28	-1.250	-1.280	-1.310	-1.340	-1.370	86	286.68	287.38	287.58	287.74	287.88
29	-1.280	-1.310	-1.340	-1.370	-1.400	87	293.60	294.30	294.49	294.64	294.76
30	-1.310	-1.340	-1.370	-1.400	-1.430	88	300.60	301.30	301.47	301.61	301.72
31	-1.340	-1.370	-1.400	-1.430	-1.460	89	307.68	308.38	308.53	308.65	308.75
32	-1.370	-1.400	-1.430	-1.460	-1.490	90	314.84	315.54	315.67	315.77	315.85
33	-1.400	-1.430	-1.460	-1.490	-1.520	91	322.08	322.78	322.89	322.97	323.03
34	-1.430	-1.460	-1.490	-1.520	-1.550	92	329.40	330.10	330.19	330.26	330.31
35	-1.460	-1.490	-1.520	-1.550	-1.580	93	336.80	337.50	337.57	337.62	337.65
36	-1.490	-1.520	-1.550	-1.580	-1.610	94	344.28	344.98	345.03	345.06	345.08
37	-1.520	-1.550	-1.580	-1.610	-1.640	95	351.84	352.54	352.58	352.60	352.61
38	-1.550	-1.580	-1.610	-1.640	-1.670	96	359.48	360.18	360.21	360.22	360.22
39	-1.580	-1.610	-1.640	-1.670	-1.700	97	367.20	367.90	367.91	367.91	367.91
40	-1.610	-1.640	-1.670	-1.700	-1.730	98	375.00	375.70	375.69	375.67	375.64
41	-1.640	-1.670	-1.700	-1.730	-1.760	99	382.88	383.58	383.55	383.51	383.46
42	-1.670	-1.700	-1.730	-1.760	-1.790	100	390.84	391.54	391.49	391.43	391.36

^aHandbook of Chemistry and Physics, 45th Edition, Chemical
 Rubber Publishing Company, 1965.

DUPONT ALPHA-1 CALIBRATION DATA SHEET

PROJECT #: _____

PRE-TEST

POST-TEST

LOCATION: _____

TIME: _____

DATE: _____

BAROMETRIC PRESS. (mm Hg): _____

SAMPLE MEDIA TYPE: _____

VAPOR PRESS. (mm Hg): _____

BUBBLE TUBE S/N: _____

TEMP. (DEG C): _____

[illegible]

DO PRE-TEST AND POST-TEST STANDARD FLOW RATES FOR EACH PUMP AGREE WITHIN + OR - 10% ? Y N

ARE ALL CALIBRATIONS AND DATA CLEARLY DOCUMENTED ? Y N

DATE: _____ TIME: _____ TECHNICIAN: _____

$$\text{STANDARD FLOW RATE} = \frac{\text{VOL (ml)}}{\text{TIME (min)}} \times \frac{(P - P_v)}{760} \times \frac{298.16}{(273 + t)}$$

APPENDIX 3-5
GRID DESIGN PROCEDURES

GRID DESIGN PROCEDURES

Equipment

disposable gloves	string
plastic sheeting, various sizes	wire flagging markers
fiberglass tape with tenth of foot increments	indelible pen
	field book/calculator

Procedure (see attached figure)

1. Secure plastic sheeting over the entire sampling area and secure the ground cover.

2. Using a fiberglass measuring tape, measure the longest sample area's dimension. The longest dimension will be recorded in the field book, as length L_1 . Dimension measurements will be complete to the nearest foot.

3. Divide the length of L_1 in half to determine the mid-point (P) of L_1 . Record the distance between the end point of L_1 and P in the field book.

4. Using the fiberglass measuring tape, measure the length of the sample area's dimensions along a line through mid-point P, perpendicular to dimension L_1 . Record this dimension as L_2 .

5. Divide the length of L_2 in half to determine the sample area's center (C). Record the distance between the end point L_2 and center C in the field book.

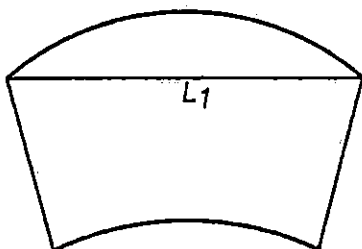
6. Measure the distance between the sample area's center (C) and the end point of L_1 . The distance is recorded in the field book as the sample radius (R).

NOTE: In determining the length of any dimension, both horizontal and vertical surfaces will be measured and the combined length will be used to determine the axial dimension. Vertical surfaces between adjacent work areas and vertical surfaces as a result of shoring materials will, however, not be measured. The sample area dimensions will be determined by measuring only the length of the resulting horizontal surface.

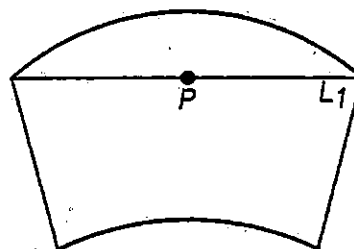
occ.fsp/91-29

FIGURE

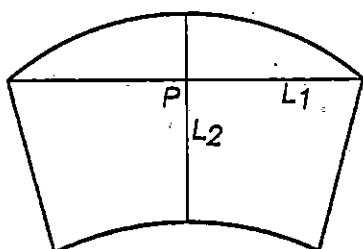
DRAW LONGEST DIMENSION,
 L_1 , ON SITE DIAGRAM



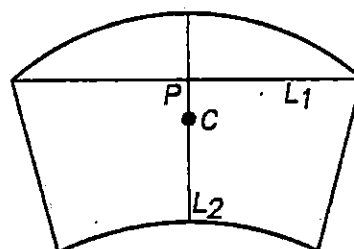
FIND MIDPOINT, P, OF L_1



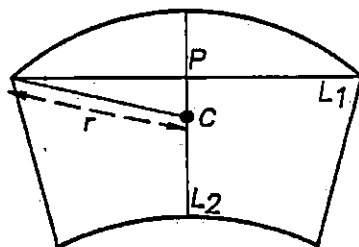
DRAW LINE, L_2 , THROUGH P
PERPENDICULAR TO L_1



THE MIDPOINT, C, OF L_2 IS THE
CENTER OF THE SAMPLING CIRCLE



THE DISTANCE FROM C TO THE END
OF L_1 IS THE SAMPLING RADIUS, r



OCCIDENTAL CHEMICAL CORPORATION
HOOKER/RUCO SITE
HICKSVILLE, NEW YORK

PROCEDURE TO DETERMINE THE CENTER AND RADIUS
OF THE VERIFICATION SAMPLE AREA

DATE	REVISED	PREPARED BY:
		LECCETTE, BRASHNARS & GRAHAM, INC.
		Professional Ground-Water Consultants
		72 Danbury Road
		Wilton, CT 06897
		(203) 762-1207
		DATE: 7/17/81
		FIGURE: 3-5

APPENDIX 3-6
SAMPLE GRID CONSTRUCTION PROCEDURE

GRID LAYOUT PROCEDURES

Equipment

disposable gloves	nylon string
plastic sheeting, various sizes	wire flagging markers
fiberglass tape, with tenth of foot increments	indelible pen
	field book/calculator

Procedure (see attached figure)

1. Using a fiberglass measuring tape, measure all excavation dimensions and record the following measurements, L_1 , P, L_2 , C and R according to Appendix 3-5.
2. Using nylon string, construct the largest dimension L_1 , in the excavation by installing stainless-steel spads at each end point and extending the string between the spads.
3. Mark the nylon string defining length L_1 at its mid-point (P) with a polyethylene marking pen.
4. Using a 90 degree miter-form, construct length L_2 using nylon string. Length L_2 will be perpendicular to length L_1 and bisects L_1 at mid-point (P). L_2 will be constructed by installing stainless-steel spads at each end point and extending the nylon string between the two points.
5. Mark nylon string, length L_2 at its center point (C) with a polyethylene marking pen.
6. Using nylon string, measure the sample radius (R) by extending a string from mid-point C to the end point of L_1 , to the nearest tenth of a foot.

7. Determine the distance between sample points (S), and the distance between adjacent rows (U), by means of the following calculations where R is the sample radius defined in Step 6:

**Geometric Parameters for Hexagonal Grid
Designs for Sampling Radius R**

Number of samples	Distance (S) between adjacent samples	Distance (U) between suc- cessive rows
7	0.87R	0.75R
19	0.48R	0.42R
37	0.30R	0.26R

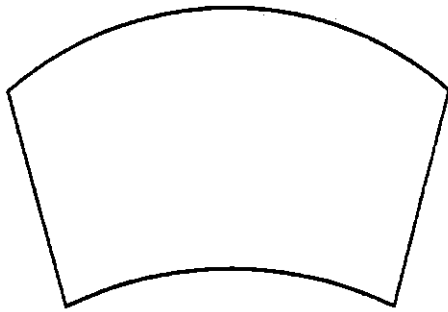
8. Point C will be the first sampling location in the excavation. Additional sampling locations will be staked-out, (S) feet apart along the circle's diameter. Actual sample points will be determined by extending a nylon string of length (S) and marking the end point with wire-stake flagging.

9. At the end of each sample row, a parallel sample row will be constructed. The successive rows will be parallel to the sample area's diameter and separated by distance (U). The sample row will be constructed by measuring the distance (S), from the two most previous sampling locations. Nylon string of length (S) will be extended from the two reference sample points and each string will be fully extended. The apex between the two strings will be the starting point for the successive rows. (See attached figure, at Sample Locations 4, 5 and 6 for reference.)

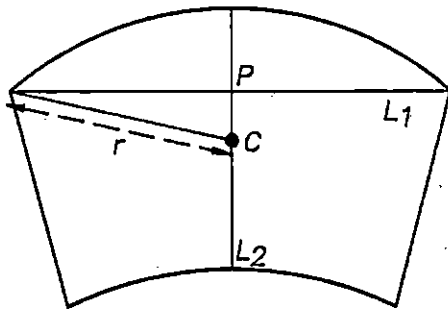
10. Using a wire-stake flagging, mark each sample location. Each flag will be identified with the site name, excavation work area and sample location number.

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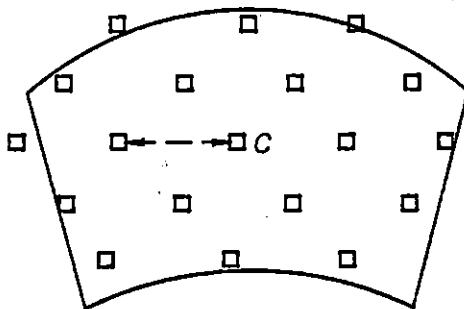
FIGURE



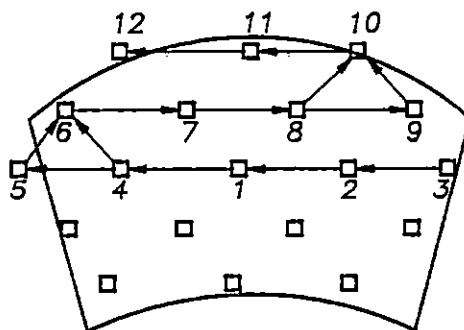
ORIGINAL CLEANUP AREA



LOCATING THE CENTER OF THE SAMPLING CIRCLE



CENTERING THE HEXAGONAL GRID



STAKING OUT THE GRID POINTS

OCCIDENTAL CHEMICAL CORPORATION
HOOKER/RUCO SITE
HICKSVILLE, NEW YORK

PROCEDURE TO CONSTRUCT THE SAMPLE GRID IN THE FIELD

DATE	REVISED	PREPARED BY:
		LEGGETT, BRASHEARS & GRAHAM, INC.
		Professional Ground-Water Consultants
		72 Danbury Road
		Wilton, CT 06897
		(203) 762-1207
		DATE: 7/17/91
		FIGURE: 3-6

VERIFICATION SOIL SAMPLE COLLECTION PROCEDURES

APPENDIX 3-7

VERIFICATION SOIL SAMPLE COLLECTION PROCEDURES

Equipment

disposable gloves	plastic sheeting, various sizes
laboratory glassware, 40 ml and 8 oz. glass jars	teflon sheets
stainless-steel spatula, scoop or trowels	disposable teflon templates, 6 x 6" ²
ice chests, ice packs	stainless steel scissors

Procedure

1. Prior to soil sampling, a 6" x 6" template will be placed over the sample location and the outline of the template will be marked on the plastic sheeting overlying the sample area.

2. The template outline on the plastic sheeting will be trimmed away exposing the soil sample area.

3. Soil will be scraped to a depth of approximately 1/2 inch using a stainless-steel trowel, scoop or spatula. Approximately 200 grams of soil will be scraped from each sample location.

4. The soil sample will be placed on a disposable teflon sheet and homogenized.

5. A portion of the sample will be transferred into a 40-ml (milliliter) glass jar (approximately 50 grams) and the remaining soil will be placed in an appropriate 8-ounce glass jar. The 40-ml soil sample will be used during the compositing procedures. The 8-ounce glass jar will be held, should individual soil analysis be required.

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APPENDIX 3-8
COMPOSITE SAMPLE PROCEDURES

COMPOSITE SAMPLING PROCEDURES

Equipment

soil sample containers, both 40 ml and 8 oz.

disposal gloves

stainless-steel spatula

teflon sheets

cooler, ice packs, tape

Procedure

1. The individual soil samples requiring compositing will be removed from storage and assembled into the appropriate composite groups. No more than five (5) soil samples can be composited together.

2. Open each 40-ml soil sample that will be composited and combine the contents of each sample jar on a disposable polypropylene sheet. Continue adding soil samples to the sheeting until all soil samples requiring compositing have been pooled together.

3. Thoroughly homogenize the pooled soil samples using a stainless-steel spoon, spatula or trowel.

4. Transfer the homogenized soil sample to an appropriate 8-ounce glass container, and seal the soil sample container.

5. Should a split or duplicate composite soil sample be required for confirmation analysis, fill appropriate analytical containers. During split or duplicate sample formation, alternately fill the analytical jars so that all jars are uniformly filled.

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APPENDIX 3-9

SOIL SAMPLING EQUIPMENT CLEANING PROCEDURES

SOIL SAMPLING CLEANING EQUIPMENT PROCEDURES

Equipment

disposable gloves	plastic sheeting
wire brush	containers of detergent
containers of tap water	containers of pesticide
buckets, various sizes	grade hexane/methanol
containers of deionized water	aluminum foil

Procedure

1. With a dedicated wire brush, remove any solid material remaining in or on the sampling equipment.
2. Wash with detergent and tap water.
3. Rinse with tap water.
4. Rinse with methanol.
5. Rinse with hexane.
6. Rinse with deionized water.
7. Air dry.
8. Reassemble sampling equipment with gloved hands.
9. Wrap in aluminum foil, shiny side out.

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APPENDIX 3-10
WIPE SAMPLE COLLECTION PROCEDURES

WIPE SAMPLE COLLECTION PROCEDURES

Equipment

disposal gloves	laboratory glassware
container of reagent grade solvent (isooctane/hexane)	box of 11 cm filter paper (Whatmans 40 ashless or Whatmans 50 smear tabs)
stainless-steel forceps	
10 x 10 cm ² template	ice chests, ice packs, tape

Procedure

1. Wearing dedicated disposable gloves, remove one filter paper from the box. A new set of gloves should always be worn when collecting a new sample.

2. Moisten the filter paper with collection solvent (either isooctane or hexane). The filter should be wet, but not dripping.

3. Delineate the sample area by marking with a disposable template.

4. Using either stainless-steel forceps or disposable gloves, thoroughly rub the sample area with the filter paper.

5. Without allowing the filter paper to contact any surface other than the sample area, fold the filter paper with the exposed side in and place the filter paper into the appropriate laboratory glassware.

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OCCIDENTAL CHEMICAL CORPORATION
PROJECT REA-91543-503
HOOKER/RUCO REMEDIATION PROJECT

SECTION 4: QUALITY ASSURANCE PROJECT PLAN

(USEPA INDEX NUMBER - II CERCLA-10216)
SECTION 4: QUALITY ASSURANCE PROJECT PLAN
REVISED JULY 25, 1991

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APPENDIX 4-3: Short Form

TABLE
(at end of report

Table

1 Sample Analysis Program

FIGURE
(at end of report)

Figure

1 Chain-of-Custody Form

4.0 QUALITY ASSURANCE PROJECT PLAN (QAPP)

4.1 Introduction

The QAPP defines all Quality Assurance/Quality Control (QA/QC) procedures which will be used during the analysis of samples from Operable Unit 2 of the Hooker/Ruco Site during the remedial action. The QAPP has been prepared following specifications and definitions described in "Test Methods for Evaluating Solid Wastes" (SW-846), November 1986, Region II CERCLA Quality Assurance Manual (October 1989), Guidance for Preparation of Combined Work/Quality Assurance Project Plans for Environmental Monitoring, Office of Water Regulations and Standards, USEPA, May 1984.

4.2. Project Description

A comprehensive description of the project is contained in Section 2.0 of the RDWP.

4.3 Project Organization and Responsibility

4.3.1 Project Organization, Remedial Design Implementation

Site Coordinator	Dr. Alan Weston (OCC)
Project Manager	Joseph Coveney (OCC)
Project Engineer	William Beckman (LBG)
Remedial Leader	William T. West (LBG)
Health and Safety Officer.	Douglas Paschke (OCC)
QA/QC Officer	P. Garrity (OCC)
External Laboratory Coordinator (ELC)	Determined by individual laboratory
Sampling Coordinator	William T. West (LBG)

4.4 Definintion of Responsiblities

a. The QA/QC Officer (QAO) is responsible for the following:

- selecting and reviewing all sampling and analytical protocols required for measuring and monitoring;
- selecting analytical laboratories;
- directing the activities of the external analytical laboratory used for the project;
- reviewing all QA/QC results;

- has overall responsibility for management of the analytical program and the validity of all data;
 - reviewing and advising on all aspects of QA/QC;
 - making QC evaluations to assist in reviewing QA/QC procedures, and, if problems are detected, making recommendations to the ELC to rectify the problem;
 - evaluating and recommending corrections to sample custody procedures;
 - informing the Project Manager that appropriate QA/QC procedures have been established and are being implemented by the proper personnel; and
 - evaluating and recommending corrections in sampling and/or analytical techniques.
- b. The ELC is responsible for the following:
- the laboratory's activities;
 - training and qualifying personnel in specified laboratory QC and analytical procedures, prior to receiving samples;
 - informing the QAO if any review of data quality appears to warrant repeat analysis of some or all samples;
 - receiving samples from the field and verifying that incoming samples correspond to the packing list or chain-of-custody sheet;
 - maintaining records of all incoming samples, tracking those samples through subsequent processing, analysis and ultimately, appropriate disposal of those samples at the conclusion of the project;
 - preparing QC samples for analysis prior to and during the program;

- preparing QC and sample data for review by the QAO;
- review of raw data with laboratory chemists against calibration and QC records;
- approval of finished data; and
- preparing QC and sample data for transmission to the QAO.

c. Sampling Coordinator

The sampling coordinator is responsible for the following:

- coordinating field activities and delivery of samples to the analytical laboratory;
- determining appropriate sampling equipment and sample containers to minimize contamination;
- training and qualifying field personnel in sampling procedures and field analytical procedures prior to sampling;
- ensuring that samples are collected, labeled, preserved, stored, transported, and, when necessary, filtered as specified in the procedures or protocols;
- checking that all sample documentation is correct and transmitted with the samples to the analytical laboratory and the APM;
- verifying that field analytical QC procedures are being followed as specified in the QA/QC protocol and prepares QC for review by the APM and QAO; and
- participating in field analytical/sampling quality audits with the APM and QAO.

4.5 Quality Assurance Objectives

The overall QA objective is to develop and implement procedures for field sampling, chain-of-custody, laboratory analyses and reporting that will provide accurate data. Specific procedures to be used for chain-of-custody, calibration, laboratory analysis, reporting, quality control, audits, preventive maintenance and corrective actions are presented in other sections of this QAPP. Procedures relative to sampling are presented in Section 3.0 of the Work Plan and Section 4.6 of the QAPP.

Data quality objectives (DQOs) have been established in accordance with the USEPA guidance document entitled "Data Quality Objectives for the RI/FS Process", dated March 1987 (Ref. 1), to ensure that the database developed during the Waste Excavation and Disposal Program meets the objectives and quality necessary for its intended use, namely determination of proper treatment/disposal requirements and characterization of soils remaining in the base of the excavation.

The purpose of this section is to define the goals for the level of QA effort. Objectives for accuracy, precision, sensitivity, completeness, representativeness and comparability of measurement data from the analytical laboratory will be identified. In addition, QA objectives for field measurements will be defined.

4.6 Sampling Procedures

A comprehensive description of the field sampling procedures is contained in the FSP. Samples will be collected to achieve four objectives: assessing work site conditions, classifying the exhumed waste, verifying that the cleanup objectives have been met, and confirming that the project is complete using CLP methodologies. Analytical results of the waste classification samples from the excavated soil will be used for transport and disposal purposes. Verification samples will be collected to determine residual PCB soil

concentrations in the direct spill and transport related areas. Additional split samples will be submitted for confirmation analysis by the CLP. A copy of the sample analysis program is presented in table 1.

4.7 Sample Custody Procedures

It will be the responsibility of the Sampling Coordinator to maintain and document sample handling. This will be completed using a chain-of-custody form. The ELC will provide documentation that the samples have been properly disposed of after completing the analyses.

The designated laboratory will provide sample chain-of-custody as prescribed in the USEPA CLP Statement of Work for Organic Analyses, 2/88 and for Inorganic Analysis, 12/87. At minimum, the record will contain the following types of information:

- sample number;
- signature of collector;
- date and time of collection;
- sample matrix (e.g., soil, air, wipes);
- identification of sample location;
- number of containers;
- parameters requested for analysis;
- signature of person(s) involved in the chain of possession; and
- inclusive dates of possession.

A copy of the chain-of-custody form is included as figure 4-1. To prevent misidentification of samples, legible labels will be affixed to each sample container. The labels will be sufficiently waterproof and durable to remain legible even when wet and will contain the following information:

- sample identification number;
- name of collector;

- date and time of collection;
- place of collection; and
- parameter(s) requested (if space permits).

In cases where samples may leave the site project coordinator's immediate control, such as shipment to laboratory by a common carrier, a seal will be provided on the shipping container or individual sample bottles to ensure that the samples have not been disturbed during transportation.

4.8 Sample Storage Procedures and Holding Times

Sample preservation techniques, and holding times, are presented in Appendix 4-1. Sample size, preservation, storage and holding times are addressed in "Volumetric Techniques", Quality Assurance Manual, Appendix 4-2 and in table 1 of Appendix 4-3.

4.9 Sample Preparation Methods

Sample preparation methods for Aroclor 1248 on particulate and particulate analyses are addressed in NIOSH Method 5503 and 0500 (Appendix 4-3). Sample preparation methods for waste classification and verification samples will be completed according to procedures presented in "Test Methods for Evaluating Solid Waste (SW-846)", November 1986. Confirmation soil sample preparation is addressed in USEPA-CLP Statement of Work (SOW) for "Organic Analyses, Multi-Media, Multi-Concentration", 2/88.

4.10 Analytical Procedures

4.10.1 Air Samples

Air samples will be analyzed for particulates and Aroclor 1248 on particulates according to NIOSH Methods 0500 and 5503. Actual method references are presented in Appendix 4-3.

4.10.2 Waste Classification Samples

The Waste Classification soil samples will be analyzed for ignitability, reactivity, corrosivity, and toxicity. Testing parameters and regulatory levels applicable to the disposal of the waste material will be completed pursuant to 40CFR 261.24.

4.10.3 Verification Samples

Verification samples will be analyzed for Aroclor 1248 according to "Test Methods for Evaluating Solid Waste, November, 1986" by EPA Method 8080.

4.10.4 Confirmation Samples

USEPA-CLP SOW for "Organic Analyses, Multi-Media, Multi-Concentration, February, 1988", will be used in the analyses of confirmation soil samples for Aroclor 1248.

4.11 Calibration Procedures and Frequency

All calibration procedures and their frequency shall be followed as described in NIOSH Methods 5503 and 0500, "Test Methods For Evaluating Solid Waste (SW-846)" and CLP SOW Organics, 2/88. All standards used for quantitation must be traceable to an EPA EMSL standard whenever possible, and if not, to a verified standard. This is a compound whose purity has been determined by at least two different analytical procedures. Linearity of detector response for each parameter must be demonstrated by generation of a linearity curve containing five concentrations of that parameter. All sample calculations must be made from responses which fall within this linearity range. During the course of the analysis, standards must be interspersed at frequent intervals to check the calibration. The preparation of all standards, including purity verification, dilutions, linearities, etc., must be recorded in a bound notebook with each page or work unit signed and dated by the analyst.

4.12 Data Reduction, Validation and Reporting

All raw data will be examined, evaluated and then reduced to final results by the ELC. The final results will be expressed in units of measurement that permit comparison with data generated from similar projects and analyses performed pursuant to the remediation at the referenced site.

All raw data shall be reviewed and validated against calibration and QC records to ensure that data are reliable, and that the data are in compliance with the QA/QC objectives. Any data determined to be invalid shall not be used in the final reporting, however, will be made available to EPA upon request. However, the fact that data have been invalidated and the reasons for the invalidation will be reported to the following people in the following order:

- QAO;
- Site Coordination; and
- EPA.

The report format will include at least the following:

- sample ID number or code;
- place of collection;
- date sampled; and
- date analyzed.

4.12.1 Turnaround Time

All samples will be sent to the laboratory promptly. The normal expected turnaround period for the various analyses are summarized below:

Air samples:	21 days
Waste classification:	30 days
Waste classification: (rush analysis)	3 days
Aroclor 1248 (verification):	1 day
Aroclor 1248 (confirmation):	40 days

4.13 Internal Quality Control Checks

The QC checks described below will be used to assess the quality of both the sampling procedures and of the sample analyses used for this project.

1. Method Blank(s): Method blanks are to be prepared in the laboratory and analyzed to assess possible laboratory contamination.
2. Laboratory Control Samples (Method Spikes): Method spikes (blank spikes) will be prepared and analyzed. Reagent grade water is spiked with one or more selected compounds prior to extraction. The recovery of the compound(s) is used as a measure of the accuracy of the sample preparation and analysis procedures. At least ten percent of the total number of samples analyzed will also be method spike samples.
3. Calibration Check Sample(s): During the course of analysis, every twentieth sample shall be a calibration check standard. This standard shall be prepared from a "second source", that is, a supplier(s) different from the primary calibration standard. The purpose of this calibration check is to ensure the validity of the calibration standard.
4. Replicate Sample(s): These samples are analyzed in order to establish control and assess the precision of analysis and/or of sampling. At least ten percent of the total number of samples to be analyzed will be replicated.
5. Matrix-Spiked Sample(s): Matrix-spiked samples are from site(s) sampled in duplicate. This sample is spiked with one or more selected compounds prior to extraction. The recovery of the compound(s) is used as a measure of the accuracy of the sample preparation and analysis procedures. At least ten

percent of the total number of samples analyzed will also be spiked samples.

6. Control Charts: Precision and accuracy will be monitored by use of control charts. Accuracy will be expressed in terms of percent recovery. A minimum of twenty data points are needed to construct the percent recovery control chart. The details of control charting are beyond the scope of this document, but at a minimum will include the following:

- the average (mean) recovery of twenty analyses (\bar{X});
- the standard deviation of the mean (SD);
- an upper and lower warning limit, which is the mean plus or minus two standard deviation units ($\bar{X} \pm 2 \times \text{SD}$); and
- an upper and lower control limit, which is the mean plus or minus three standard deviation units ($\bar{X} \pm 3 \times \text{SD}$).

Percent recoveries will then be plotted on the control chart to determine whether or not they are acceptable.

7. Surrogate Compounds: Surrogate compounds will be used to determine extraction efficiency and analytical accuracy as described in USEPA CLP Statement of Work for Inorganic, 12/87 and Organic Analyses, 2/88.
8. Reagent Quality Control Checks: Reagent and solvent blanks are prepared in the laboratory and analyzed to determine background of reagents and solvents used in the routine analysis.

4.14 Specific Routine Procedures Used to Assess Data Precision, Accuracy and Completeness

Assessment of precision and accuracy of analytical data is accomplished via review of duplicate analyses (precision) and spike recovery (accuracy) in sample matrices. Precision is generally expressed as the coefficient of variation (CV). Accuracy is expressed as percent recovery. Precision will be assessed for each matrix since distribution of parameters may be non-homogeneous, especially in non-water matrices. Precision in samples will be reviewed with knowledge of the matrix and level of analyte present. Corrective action and documentation of substandard precision is a laboratory responsibility. Accuracy will reflect the impact of matrix interferences. Each method which provides QC requirements and acceptance criteria also specifies the method of generating the data to be reviewed. It is also the laboratory's responsibility to attempt to identify the source of substandard recoveries and either take corrective action or document the cause. Calculation are presented below:

$$\%R = \frac{\text{observed value}}{\text{theoretical value}} \times 100$$

$$CV = (s/x) \times 100$$

where,

%R = percent recovery;
CV = coefficient of variation;
s = sample standard deviation; and
x = mean value of data set.

Completeness is generally assessed as a percentage of data intended to be generated.

4.15 Quality Assurance Reports

On a predetermined schedule, the QAO will meet with the APM to review QC data summaries, documentation and other aspects of the analytical performance. The assessment of the QA/QC data shall be reported to the Project Manager. This report will highlight any areas that appear to require corrective action, and will also present proposed plans to rectify the apparent problems. Included in this report shall be any results of earlier corrective action that had been initiated.

QA audits are performed to assure and document that QC measures are being utilized to provide data of acceptable quality and that subsequent calculation, interpretation and other project outputs are checked and validated.

System and performance audits will be conducted by the QAO. The APM and the QAO will conduct project audits of calculations, interpretations and reports which are based on the measurement system outputs. In addition, personnel from the State or its authorized representatives may obtain access to performance audits.

1. Performance Audits: These audits are intended primarily for analytical and data generation system. This audit will be accomplished by the use of performance evaluation samples. These samples will be randomly submitted by either the APM or QAO during the period when surveys and studies are being carried out for the duration of the program. In addition, audit samples may be submitted by the State.
2. System Audit: A systems audit will be conducted on all components of measurement systems to determine proper selection and utilization. The systems audit includes evaluation of both field and

laboratory procedures. Systems audits will be made at regular intervals at each laboratory used and whenever a new analysis is initiated. The results of the systems audit will be reported in an appropriate QA report.

3. Organization and Personnel: The QA Plan organization is reviewed for compliance with the proposed organization and for clarity of assigned responsibility. Personnel assigned to the project will be evaluated to determine that assigned responsibility, skill and training of the personnel are properly matched. The APM maintains firsthand knowledge of his team's capabilities and will discuss the organization's efficiency with the QAO. Assigned personnel may be interviewed by the QAO during an audit.
4. Facilities and Equipment: The audit will address whether field tools and analytical instruments are selected and used to meet requirements specified by the QA Plan objectives. Equipment and facilities provided for personnel health and safety will also be evaluated. Calibration and documentation procedures for instruments used in the field will receive special attention.
5. Analytical Methodology: A review of analytical methodology in regard to the data requirements for the QA Plan will be performed. An onsite observation of analyst technique, data reduction and record keeping may be performed if determined necessary. A review of precision and accuracy data will be performed for each batch of samples.

6. Sampling and Sample Handling Procedure: An audit of scheduled samples versus samples collected versus samples received for analysis will be performed. Field documentation will be reviewed. If deemed necessary, field or laboratory visits will be made to assure that designated control procedures are practiced during sampling activities.
7. Data Handling: During a systems audit, the QAO will review data handling procedures with the APM. Accuracy, consistency and documentation will be discussed.
8. QA Plan Audit: QA Plan audits encompass the aspects of both the systems audit and the performance audit. The QA Plan audit typically occurs once but may occur more often if required. Timing is keyed to the systems involved an the QA Plan objectives.
9. QA Plan Audit Report: A written QA Plan Audit Report will be prepared for each QA Plan Audit and will include:
 - an assessment of QA Plan team status in each of the major QA Plan areas;
 - clear statements of areas requiring improvement or problems to be corrected. Recommendation and assistance will be provided regarding proposed corrective actions or system improvements. If no action is required, the report will state that the QA Plan audit was satisfactorily completed;
 - a timetable for any corrective action required; and

- a follow-up to assure that recommendations have been implemented.

The format for the QA Plan Audit is found below. The QA Plan Audit Report will be distributed to the APM and the Project Coordinator.

QUALITY ASSURANCE PLAN AUDIT REPORT FORM

(Topics for inclusion in report)

Organization and Personnel

Facilities Utilized

Analytical Methodologies

Sampling and Sample Handling

Quality Control Measures Utilized

Data Handling

Quality Assurance Deficiencies

Recommended Corrective Actions and Schedule

4.16 Preventative Maintenance

It will be the responsibility of the ELC to follow the instrument manufacturer's suggested instrument maintenance program for all instruments and equipment which are to be utilized by the external laboratory. The ELC should be prepared documentation of proper institution all maintenance programs.

The PID will be inspected daily to determine if the ionization lamp is free of dust and the wand in free of obstructions. The sample inputs of both the explosivity and oxygen meter will be checked daily for obstructions.

4.17 Corrective Action

Corrective Actions will be deemed necessary and/or appropriate by the APM, QAO or the ECL. Corrective actions may include and are not limited to the following:

- additional training and/or reassignment of personnel;
- replacement of solvents and/or reagents that yield unacceptable blank values;
- reclamation of insurgents with fresh standards; and/or
- replacement of the analytical equipment.

The analytical laboratory utilized will be equipped with multiple instruments required to perform each procedure to minimize the chance of excessive down time due to equipment failure.

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TABLE

TABLE 1

**OCCIDENTAL CHEMICAL CORPORATION
HOOKER/RUCO SITE
HICKSVILLE, NEW YORK**

Sample Analysis Program

Sample Activity	Parameter	Method reference	Matrix
Baseline Sampling	Aroclor 1248	NIOSH 5503	Particulates
	Particulates	NIOSH 0500	Air
Waste Disposal Sampling	Ignitability	EPA 1010/1020	Soil/water
	Corrosivity	EPA 9040/9041	Soil/water
	Reactivity	EPA 9010/9030	Soil/water
	Toxicity	EPA 1311	Soil/water
Verification Sampling	Aroclor 1248	EPA 8080	Soil
Confirmation Sampling	Aroclor 1248	EPA SOW 2/88	Soil

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FIGURE

CONSULTING HYDROGEOLOGISTS

72 DANBURY RD. WILTON CT. 06897 (203) 762-1207

CHAIN OF CUSTODY

APPENDIX 4-1

**SAMPLE STORAGE PROCEDURES
AND HOLDING TIMES**

TABLE 1

OCCIDENTAL CHEMICAL CORPORATION
HOOKER/RUCO SITE
HICKSVILLE, NEW YORK

Parameters

Parameters	Method reference	Matrix	Sample presentation	Holding time	Sample container
Aroclor 1248	NIOSH 5503 ^{1/}	Air	Sealed cartridge	2 months	glass fiber
Particulates	NIOSH 0500 ^{1/}	Air	Sealed cartridge	--	PVC filter
Ignitability	SW-846 1010 ^{2/}	Soil	4°C	--	8 oz. glass
Corrosivity	SW-846 9040/9041	Soil	4°C	--	8 oz. glass
Reactivity					
Cyanide bearing	SW-846 9010	Soil	4°C	--	8 oz. glass
Sulfide bearing	SW-846 9030	Soil	4°C	--	8 oz. glass
Toxicity					
Non-volatile extraction	SW-846 1311	Soil	4°C	56 days ^{3/} 360 days ^{4/}	8 oz. glass
Zero-headspace extraction	SW-846 1311	Soil	4°C	28 days ^{5/} 54 days ^{6/}	8 oz. glass
Aroclor 1248	SW-846 8080	Soil	4°C	Extract in 10 days Analyze in 40 days	8 oz. glass

TABLE 1
(continued)

OCCIDENTAL CHEMICAL CORPORATION
HOOKER/RUCO SITE
HICKSVILLE, NEW YORK

Parameters

Parameters	Method reference	Matrix	Sample presentation	Holding time	Sample container
Aroclor 1248	SW-846 8080	Soil	4°C	Extract in 10 days Analyze in 40 days	8 oz. glass
Aroclor 1248	EPA SOW 2/88 ^{7/}	Soil	4°C	Extract in 10 days Analyze in 40 days	8 oz. glass

- 1/ NIOSH Manual of Analytical Methods, Third Edition, Revision 1.0, United States Department of Health, Education and Welfare, Published (NIOSH) 77-157-A (1987).
- 2/ Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Office of Solid Waste and Emergency Response, November 1986.
- 3/ Mercury requires extraction within 28 days and analysis within 28 days; total elapsed holding time is 56 days.
- 4/ Metals, excluding mercury require extraction within 180 days and analysis within 180 days, total elapsed holding time is 360 days.
- 5/ Volatiles require extraction within 14 days and analysis within 14 days; total elapsed holding time is 28 days.
- 6/ Semi-volatiles require extraction within 7 days, prepare extraction within 7 days and analysis within 40 days; total elapsed holding time is 54 days.
- 7/ United States Environmental Protection Agency, "Contract Laboratory Protocols, Statement of Work for Organic Analyses, Multi-Media, Multi-Concentration", February 1988.

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APPENDIX 4-2

VOLUMETRIC TECHNIQUES
QUALITY ASSURANCE MANUAL

VOLUMETRIC TECHNIQUES, Ltd.

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QUALITY
ASSURANCE
MANUAL

CONSULTING CHEMISTS • COMPLETE LABORATORY TESTING

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1. SCOPE AND SIGNIFICANCE

1.1 Purpose of This Manual

The purpose of this Manual is ensure the following:

- o Quality and consistency of analytical results
- o Conformance with all regulatory requirements
- o Highest quality of professional services to our clients

To achieve these goals, this Manual directs implementation of a thorough Quality Control program headed by a Quality Assurance (QA) Director, addressing all aspects of operation; describes responsibilities and duties of all personnel; and addresses all aspects of this Laboratory's operation.

1.2 Implementation of Goals and Objectives

It is our intention to ensure that all goals and objectives of our Quality Program are met. Quality policies and procedures are integrated into our daily work, and are constantly reviewed by Laboratory Management and by the Quality Assurance (QA) Director. These include:

- o Integrity of analytical results
- o Completion of Quality Control (QC) samples
- o Proper documentation of analytical data
- o Good laboratory technique that ensures a contamination-free environment
- o Clear job descriptions delineating responsibilities of each employee involved in all steps of laboratory procedures, data analysis and report generation
- o Use of latest analytical technology including review of current literature to capture recent applicable developments
- o Review of reports to clients

The goals and objectives of our program are described in this Manual. All employees must learn the extent and limitations of their responsibilities as described in this Manual and adhere to them in order for this Laboratory to meet its commitments to clients.

1.3 Program Review

The Quality Program will be reviewed at least annually by the QA Director. It will also be reviewed any time a problem arises that indicates a possible program flaw. In such an instance, the QA Director will discuss the problem with Laboratory Management and Analysts to ensure needed input from all levels within the Laboratory.

DELEGATION OF RESPONSIBILITY

The following section describes the responsibilities of all laboratory personnel, broken down into the following categories: technical, administrative, and QA. Training requirements are described in the Training section of this Manual.

2.1 Laboratory Director

Technical

The Laboratory Director makes all final technical decisions for the Laboratory including:

- o Selecting and developing analytical methods
- o Requirements for laboratory equipment and supplies
- o Adjudicating analytical problems
- o Training requirements for analysts

The Laboratory Director ensures that all laboratory operations are technically sound.

Administrative

The Laboratory Director is responsible for over-all administration of laboratory operations. He ensures that policies are developed and understood by all personnel. He ensures that work-scheduling procedures adequately address client needs. He also ensures appropriate responses to client complaints are taken as described in Section 9 - Responding to Client Complaints which includes reviewing the complaint and writing a responding letter. He also approves all employee reviews and promotions. He provides the Managing Director with information regarding purchase of equipment and supplies and expenses for out-of-house training.

QA

The Laboratory Director ensures that proper QA standards are established and administered. He is ultimately responsible for seeing that the QA Program is conscientiously implemented. He reviews the QA Program with the QA Director to ensure completeness and effectiveness. He supports the QA Director in carrying out the program by use of authority.

2.2 QA Director

Technical:

The QA Director establishes and oversees the entire QA Program as described in Section 10 -- Performance Monitoring of this Manual. He develops statistical protocols for data reduction. He defines requirements for resubmitting QC samples. He sets standards for analytical performance.

Administrative

The QA Director is responsible for maintaining the QA Manual and all Standardized Operating Procedures (SOPs). He develops protocols for documenting and tracking QC samples. He reviews and approves all QC records. He sets standards for laboratory practices. He confers with the Laboratory Director on all QA policies. He supports the Laboratory Supervisor in the daily management of the QC Program.

QA

The QA Director's purpose is specifically to establish and implement the QA Program as described in the preceding paragraphs.

2.3 Laboratory Supervisor (Department Head)

Each department or group within this Laboratory is headed by a Laboratory Supervisor. He reports to the Laboratory Director except for issue of QA where he reports to the QA Director.

Technical

The Laboratory Supervisor is responsible for ensuring technically correct analysis on a daily basis in his department. He addresses all technical issues that arise from routine analyses, and confers with the Laboratory Director on issues of a less routine nature.

Administrative

The Laboratory Supervisor assigns and manages daily work, and approves all final reports. He ensures that daily analytical data is properly recorded and reviews such data. He also reviews employee performance, and makes recommendations based on these reviews. He also ensures that routine maintenance schedules are followed for all equipment as described in Section 4.2 of this Manual.

QA

The Laboratory Supervisor is responsible for implementing daily QC analyses, and provides such data to the QC Director. He ensures that good laboratory practices, as described in this manual, are followed for all analyses performed under his jurisdiction.

2.4 Analyst (Technician)

All Analysts report directly to their Laboratory Supervisors.

Technical

The Analyst is responsible for performing calibrations of equipment, assigned analyses, and recording of all analytical data according to established procedures. The Analyst must use good analytical technique. He must provide analytical results suitable for issuing a client report.

Administrative

The Analyst manages all work assigned to him. He completes all paperwork in accordance with established Laboratory procedures and reviews all paperwork for correctness and completeness. He ensures that work progresses in a timely and productive manner.

QA

The Analyst performs analysis on QC samples. He records his analytical results and maintains them for review purposes by his Laboratory Supervisor and the QA Director.

2.5 Sample Receiving Clerk

The Sample Receiving Clerk reports to the Laboratory Supervisor.

Technical

The Sample Receiving Clerk reviews paperwork for all incoming samples to ensure completeness and correctness. He logs in samples, and initials all entries. He inspects samples to ensure sample integrity is retained, and that packaging is not compromised. (He makes sure all samples placed in proper storage area to await analysis?).

Administrative

The Sample Receiving Clerk delivers incoming samples to the Laboratory. He informs the Supervisor or Analyst of any special priorities regarding the samples. He also informs them if there are any concerns noted regarding sample integrity. The Sample Receiving Clerk should also be aware of sample origin as it impacts regulatory requirements.

QA

The Sample Receiving Clerk follows all sample-tracking protocols in handling samples. He ensures that proper numbering is used, and is written directly on sample containers.

SAMPLE TRACKING

Rigorous sample tracking is fundamental to an QA Program. The most thorough and complete analysis is useless if performed on the wrong sample.

Our sample-tracking program is designed, to the extent that it is possible, to meet all litigation requirements. It is also designed to have redundancy safeguards wherever possible.

3.1 Chain of Custody

A sample will probably not meet litigation requirements without a Chain of Custody that begins at the sample collection point. ~~If~~ Since the client, and not ourselves, collects samples for analysis, we cannot be responsible for issuing a Chain of Custody at the time of sampling.

The Sample Receiving Clerk, or anyone else receiving samples, will properly sign all Chain of Custody documents accompanying incoming samples, and assigns job or batch and sample ID numbers. If called for, a copy will be returned to the client at this point, serving only as the sample receipt. The original remains with the samples, and is signed by any party handling them. When analysis is complete, and the sample will not be analyzed further, the Chain of Custody is returned to the client, and a copy retained in the Laboratory's permanent Master File with the report.

If a sample is received without a Chain of Custody (or an incomplete Chain of Custody), the Sample Receiving Clerk will prepare one at log-in, if requested. Such a form will only guarantee custody within this Laboratory, and the custody form shall so note on the document sent to the client.

In the absence of a custody form, the logging and data-recording requirements are structured to serve as a documented custody record within the Laboratory.

3.2 Logging

Logging of samples is normally done by the Sample Receiving Clerk, but may be done by any other employee familiar with the process. The following data must be recorded in the sample tracking logbook:

- o Date received
- o Job number or sample batch number (sequential)
- o Sample ID number(s)
- o Number of samples received
- o Sample number range (sequential)
- (o Method of arrival - by mail, delivered in person by client, collected by in-house technician)
- o Client name or ID
- o Brief sample description
- o Sample collection location and method
- o Priority
- o Required analyses (Appropriate SOP indicated)
- o Sample Receiving Clerk initials or Name of employee other than Clerk receiving sample
- o Any additional information (storage area if more than one in Laboratory, department performing analysis if more than one)

The Sample Receiving Clerk inspects the samples for integrity verifies that all samples in the Chain of Custody are present, and logs them in. If he detects any damage, he reports it to the Laboratory Supervisor.

The Sample Receiving Clerk records the batch and ID number on each sample and places it in the "Incoming Sample" storage area.

3.3 Sample Acceptance Criteria

Prior to accepting samples the Sample Receiving Clerk inspects them to determine if they conform to laboratory acceptance criteria. If they do not, the Laboratory Supervisor will determine whether the damage to integrity is sufficient to cause rejection. Rejections of samples are to be followed up by immediate notification of the client with an explanation and return of the questionable sample.

Samples are judged unacceptable under the following circumstances:

- o Improper labeling
- o Improper packaging
- o Sample too small (or too large)
- o Impossible deadlines
- o Obvious faulty sampling technique

Clients are to ship samples in clearly labeled, non-breakable airtight containers and to package such samples so as to minimize damage or change in condition of the samples. Samples shipped by air should be placed in containers which allow them to

be maintained upright to minimize jostling and damage.

3.4 Routing

The Laboratory Supervisor assigns all work. Analysts are either assigned specific batches or take the sample with the next ID number. All samples are to be analyzed in sequential order unless assigned otherwise by the Supervisor. The Laboratory Supervisor assigns a specific sample or batch to an Analyst by notifying both the Sample Receiving Clerk and the Analyst. In either case, when accepting a sample, the Analyst records his name in the log book and the date and time he removes the sample from the "Incoming Sample" storage area.

3.5 Analysis and Data Gathering

All analyses must be carried out in accordance with the SOP(s) indicated in the log book. All SOPs used in this Laboratory will be found in the Appendices of this Manual.

Each SOP has a specific preprinted Data Recording Worksheet. Analysts are to obtain the appropriate worksheet from the Sample Receiving Clerk before beginning an analysis. All data must be recorded on the sheet.

3.6 Data Reporting

Once an analysis of a sample is completed, the Analyst records the date and time in the log book and makes a photocopy of the Data Recording Worksheet. He gives the original to the Sample Recording Clerk and the copy to the Laboratory Supervisor.

The Laboratory Supervisor reviews the copy and notifies the Sample Recording Clerk of approval or rejection. The Laboratory Supervisor maintains the copy in his own file. If approved, the Sample Recording Clerk assigns a typist to complete a Final Client Report using the data recorded on the Data Recording Worksheet. Each SOP has its own specific Client Report. Typists are to use a sample report as a model to determine proper procedures for completing the Client Report.

The Sample Recording Clerk also completes the Chain of Custody documents, recording who performed the analysis, and date and time analysis was initiated and completed. The completed Client Report and

Chain of Custody are sent to the client and copies of those documents as well as the original Data Recording Worksheet are filed in the permanent Laboratory Master File.

If the Data Recording Worksheet is judged unacceptable, the Laboratory Supervisor can either require the Analyst to repeat those sections of the SOP judged questionable or assign another technician to them. In either case, a copy of the Data Recording Worksheet goes to the Laboratory Supervisor and the original to the Sample Recording Sheet. If results are acceptable, the preceding steps are then followed, including recording the time and date in the log book. All analysts who participated in the analysis of a sample must be recorded on all documents.

3.2 Disposal of Sample

Once the analysis is complete and the Data Recording Worksheet approved, the analyst disposes of the sample in the appropriate Disposal Box indicated in the SOP and records sample batch and ID numbers, and date and time in the storage log book. All Disposal Boxes are to be stored in a safe manner for the period of time indicated for that category of waste, in accordance with regulatory requirements. When a Disposal Box is full, the date of the most recent sample enclosed is marked on it. A new Disposal Box replaces the old one which is then to be stored until time of disposal when it is disposed in accordance with regulatory requirements.

4.

EQUIPMENT AND CALIBRATION

4.0 Usage

A usage logbook is to be maintained for each instrument that requires calibration. At each use of an instrument the following information is to be recorded in it:

- o Date and time of usage
- o Initials of analyst
- o Sample ID and batch # of sample ^{in which} analysis was performed ~~on~~

4.1 Calibration

Calibrations of instruments are necessary to maintain our high standards of results and to provide our clients with legally defensible data on which to base decisions. Therefore all laboratory personnel are required to comply with calibration procedures of this Laboratory.

Instruments are to be calibrated on a regular basis in accordance with frequency of use. The calibration schedule of each instrument will be established by the QA Manager. A Calibration SOP for each instrument can be found in the Appendices of this Manual.

All Calibration SOPs must be in accordance with regulatory agency requirements and will include complete descriptions of calibration, required calibration curves and calibration schedule.

A Calibration Logbook will be maintained for each instrument. A calibration curve will be maintained for each instrument whose SOP calls for it. All logbooks and curves must include the following:

- o Calibration schedule
- o Date and time of each calibration
- o Initials of analyst who performs calibration
- o Number of times instrument used since last calibration
- o Pertinent comments

All analysts are responsible for compliance with calibration requirements and must check the usage logbook for the instrument about to be used before carrying out an analysis. If the logbook indicates a calibration is due, that analyst must do it, even if he must interrupt an analysis of a batch of samples.

4.2 Maintenance

Maintenance schedules for equipment will be established by the Laboratory Director. The Laboratory Director shall also determine whether each instrument is maintained and repaired in-house or by an outside agency. Servicing will also be performed when indicated by dramatic changes in calibration.

A maintenance logbook will be maintained for all equipment which will contain a maintenance checklist, schedule and log sheet for each instrument. The schedule will be in the form of a table with maintenance elements listed by row and frequency by column:

Frequency:	1 mo.	2 mo.	3 mo.	4 mo.
Maintenance				
Elements				

Each instrument maintenance log sheet shall contain the following information:

- o Date and time
- o Initials of who performed servicing (include if in-house or outside agency)
- o Scheduled or unscheduled check
- o Maintenance element examined and if any repairs/replacement of component; were made
- o Pertinent comments
- o Due date for next servicing

The Laboratory Supervisor is responsible for ensuring these routine maintenance schedules are enforced.

4.3 Manuals

The QA Director is responsible for maintaining and reviewing all instrument manuals pertaining to calibration and maintenance. Any noteworthy items are to be included in the monthly QA report.

The QA Director additionally distributes the manufacturer's manual on each instrument to the head of each department in which the particular instrument is located. It is the policy of this Laboratory to be informed of and current with all

new releases of information on all utilized equipment. Therefore the QA Director is also responsible for receiving and distributing all updates on manufacturer's manuals to the appropriate department heads.

5 QUALITY OF MATERIALS

The high quality of materials used in this Laboratory shall be assured through specific purchasing and verification procedures and/or proper preparation techniques.

5.1 Reagents

The reagents used in this Laboratory are to be classified into three levels of use priority:

1. Industrial Grade
2. Analytical Grade
3. Special High Priority Grade

Selection of the appropriate grade of reagent(s) is designated in the reagent section of each analysis SOP and in addition may be specified by the Laboratory Supervisor in unusual circumstances.

Reagents shall be purchased in accordance with the analysis needs of this Laboratory as determined by the Laboratory Director. Reagent preparation is described in Section 6 -- Laboratory Practices of this Manual.

Verification will consist of confirming that the priority grade is recorded on the reagent label unless analysis difficulties indicate a possible problem or regulatory agency requirements specify otherwise. In the latter case, the appropriate analytical SOP will indicate the proper verification procedure.

5.2 Apparatus

All apparatus shall be maintained in good condition and in working order. Any items found to be defective will be taken out of use and repaired or, if necessary, replaced.

Analysts will use apparatus for the intended purpose and not needlessly expose items to corrosive materials and/or inappropriate conditions such as allowing acid to boil over onto hot plates. Apparatus and equipment shall not be used for food preparation.

Verification will consist of one of the following procedures:

- o Standard manufacturers' labels
- o Letter from the manufacturer certifying that the purchased item is of the selected type and class and is manufactured to specifications
- o In accordance with regulatory agency requirements
- o In accordance with specific procedures developed by the QA Director

The QA Director will determine which of these procedures is to be followed for each piece of apparatus.

5.3 Consumable Supplies

Consumable supplies are to be purchased on the basis of analysis needs as determined by the Laboratory Director. Analysts are to use the appropriate items as specified in analytic SOPs. SOPs will indicate the specific grades and classes of consumable supply items to be used. Analysts are not to re-use expendable materials intended for single-use purposes such as filter paper.

Verification procedures will be one of the following:

- o Each shipment of consumable supplies will be tested at the rate of 1% in accordance with procedures established by the QA Director at the time of arrival.
- o Standard manufacturers' labels
- o Letter from the manufacturer certifying that the purchased item is of the selected type and class and is manufactured to specifications
- o In accordance with regulatory agency requirements

The QA Director will select the verification procedure for each type of consumable supplies.

LABORATORY PRACTICES

This Section describes reagent control, contamination management and use of controlled procedures for this Laboratory. Proper observance of these procedures is necessary to guarantee the safety of Laboratory staff members.

6.1 Reagent Control

Reagents will be prepared either according to the analytic SOPs specifying their usage (if a reagent is not commonly used in this Laboratory) or in a separate compendium of reagent SOPs (if a common reagent). All such documents are located in the Appendices of this Manual.

All reagents will be stored in either their original containers or, if prepared in this Laboratory, in specified containers used for this purpose. All reagent container labels will bear the following information:

- o Active component
- o Matrix description
- o Concentration
- o Date prepared
- o Expiration date
- o Technician who prepared it (if in-house)
- o Manufacturer/supplier (if prepared outside)

All reagent containers are to be stored in a non-combustible, properly ventilated room in a flammable materials cabinet. Handling of volatile reagents is performed under a ventilated hood with rubber gloves for the safety of Laboratory personnel.

Small quantities of commonly used reagents are kept at work stations to facilitate their use during the work day. Those specific materials will be determined by the Laboratory Supervisor.

6.2 Contamination Management

Contamination both of samples and of the environment (including reagents used in analysis) must be avoided to provide the highest quality, legally defensible data to our clients. In order to achieve this goal, Laboratory staff must adhere to various preventative measures and use the regular testing procedures for contamination detection as established by the QA Director.

6.2.1 Contamination Control

Contamination control is focused both on sources and targets of contamination. Sources would include:

- o Samples
- o Laboratory debris

Targets would include:

- o Samples
- o Equipment, such as tools
- o Supplies, such as slides and mounting media
- o Work areas

Contamination control programatically consists of 2 parts:

- o Avoidance
- o Verification

To avoid contamination of the previously listed targets, the following procedures must be followed:

- o Cleanliness (housekeeping)
- o Controlling work areas
- o Isolating pathways

To achieve cleanliness, Laboratory personnel will comply with the following steps:

1. Clean all tools before and after preparing each sample.
2. Clean and wrap tool sets at the end of the work day.
3. Dispose of wipers after use. Do not let them pile up during the work day.
4. Wipe all work surfaces before and after sample preparation. Surfaces include bench tops, slide trays, stereo microscope stage, and slide preparation surface.

Work areas should be controlled as follows:

1. Bulk samples are opened and examined using the stereo microscope only in the hood.
2. Slides are prepared only in the hood.

2. Slide preparation or sample handling shall occur only in the assigned areas. Only prepared slides in a slide tray or other clean surface shall be kept in that area.

3. Small numbers of active samples are kept near the hood. The sample containers are kept closed at all times. Inactive samples are stored in a suitable, out-of-the-way, area.

Laboratory personnel will comply with the following procedures to isolate contamination pathways:

1. Target containers - for example, samples, mounting media, slides, cover glasses - are opened one at a time. Each is closed before another is opened. Two target containers are not to be opened simultaneously.

2. Prepared slides are stored in a protected manner. Covered slide trays are ideal.

3. Mounting media never touches a sample. Place media on a clean slide before the sample is placed there.

Analysts are to work only on clean surfaces.

6.2.2. Verification of Control

In addition to the previously delineated steps, contamination control must be verified. An example of this procedure for slides is as follows:

1. At the start and end of each day a blank slide, consisting of mounting media and a cover glass, is prepared and examined.

2. If the start-slide shows contamination, the area and tools are cleaned, and another slide is prepared. If the second slide shows contamination, another bottle of mounting media is checked. If the second bottle is clean, the first bottle is considered contaminated. If the second bottle also shows contamination, a complete investigation is conducted to determine the contamination source.

4. If the end-slide shows contamination, the same steps are taken. In addition, all samples done that day by that analyst that contain less than a predetermined percentage (generally 10%) of the analyte of interest are checked.

5. At least once a week prepare a slide from a homogenous, non-fibrous, reagent grade material that is permanently stored in the microscope hood. If contamination is noted, take the steps described above.

The weekly blanks are recorded in a separate log book. Daily blanks are recorded only if contamination is found. Neither of these blanks are to be used for statistical quality control.

6.3 Controlled Procedures

Only controlled procedures, i.e., controlled SOPs as described in Section 7 -- Documentation Preparation and Control, shall be used in analyses in this Laboratory. No unauthorized SOPs are to be used.

DOCUMENTATION PREPARATION AND CONTROL

In order to prepare and distribute documents in an organized fashion, the following procedures for initiation, preparation, review, approval and issuance of controlled copies will be followed. This program is a coordinated effort involving both technical review and custodial control. Analysts are to use only controlled, i.e., approved, documents for all calibrations, analyses and other activities performed in this Laboratory. Documents include:

- o Analytic SOPs
- o Reagent preparation SOPs
- o Calibration SOPs
- o Contamination management SOPs
- o Quality Assurance Manual

7.1 Initiation and Preparation

Initiation of a new document or a request for revision of an existing document can arise from a variety of sources within this Laboratory. Such sources include any employee involved in laboratory operation such as secretaries, analysts, Laboratory Director, QA Director, Laboratory Supervisor, Analysts and the Sample Receiving Clerk. Assignment of preparation or revision of a document will vary depending on its purpose. However, the person most familiar with the material covered in the document generally will be the person so assigned and that decision will be made by the Laboratory Director.

7.2 Review and Approval

Once the individual assigned to the preparation and or revision task has completed a first draft of the new document, it will be submitted for review according to the normal chain of command in this Laboratory. In other words every staff member above that individual will see the document and make any necessary comments. No matter who prepares a document the Laboratory Director and QA Director must be included. A typical document affecting a task performed by an analyst would be prepared by the analyst and submitted for approval to the Laboratory Supervisor of that particular department, QA Director and Laboratory Director.

Once the document has gone through the chain of command, the preparer institutes all approved changes and submits it again for what is (normally) the last review. The Laboratory Director gives final approval to all documents and procedures.

7.3. Distribution of Controlled Copies

In this Laboratory, only controlled copies of procedural documents will be issued in order to ensure the following:

- o that all laboratory activities are carried out in a uniform fashion
- o that distribution of SOPs and other documents is organized
- o to facilitate updating these documents.

The QA Director is responsible for distribution and control of approved documents.

Each copy of a document is assigned a specific number. The original document along with a Document Distribution Form for every copy will be maintained in the Master Laboratory file. The Distribution form will list the document title; copy number; version; name, title, and signature of the individual receiving that copy; and the date the copy is received. Every time a revised document is issued, the outdated version will be exchanged to assure implementation of revisions. The new information will be recorded on the appropriate Distribution Form.

REPORTING RESULTS

8.1 Client Report Requirements

As described in Section 3 -- Sample Tracking of this Manual, all data performed in this Laboratory shall be recorded on preprinted Data Recording Worksheets. Each SOP has its own specific Worksheet.

Final Client Reports are prepared by typists from Data Recording Worksheets that have been approved by the Laboratory Supervisor. Each final report will have the following information:

- o Laboratory identification and address
- o Name and address of client
- o Department(s) performing analyses and date
- o Sample IDs and descriptions
- o Sampling procedure
- o Identification and description of test procedures performed
- o Any deviations or additions to test specifications
- o Statement of measurement uncertainty
- o Statement of authenticity of results signed by Laboratory Director
- o Statement that report cannot be reproduced except in full with the approval of this Laboratory
- o Statement that this report relates only to the items tested

8.2 Approval

All Final Client Reports are to be reviewed and approved by the Laboratory Director prior to being sent to the client. They are also subject to approval by the QA Director. Quality Control statistics shall be reviewed on a regular basis as determined by the QA Director in accordance with regulatory agency requirements and delineated in the appropriate SOPs. As long as those statistics are deemed acceptable, Client Reports will continue to be processed.

8.3 Records Retention

The following records shall be maintained for three years in the Laboratory Master Files:

- o Copy of Chain of Custody Documents
- o Client Report
- o Original Data Recording Worksheets
- o Location of all other records relating to the preparation of the Client Report

Client Reports are to be filed by client name and by Job number.

Each department within this Laboratory shall retain the following records in their permanent files for three years:

- o Copy of Data Recording Worksheets
- o Calibration, usage, and verification data
- o Contamination monitoring data
- o Equipment and maintenance
- o Performance monitoring

All records must be maintained in sufficient condition so as to meet regulatory agency requirements and to withstand regular inspections by those same agencies.

PROCEDURES FOR DEALING WITH CLIENT COMPLAINTS

If a client makes a complaint about a test result, the sample in question will be reanalyzed by a second Analyst. If the second result agrees with the original (is within the original's uncertainty range), the Laboratory Director shall send a letter stating that a quality control check has confirmed the original analysis.

If the second result does not agree with the original, a third Analyst shall perform a test to determine the correct analysis. If this third result rejects the original analysis, the Laboratory Director shall send a letter to the client stating that a quality control check has found the original analysis in error. The letter should either accompany an amended Final Client Report or state that such a report will be sent shortly. In either case, the amended report will contain the corrected result that was confirmed by two Analysts.

10 TECHNICAL QUALIFICATIONS

Analysts are required to meet certain training and performance criteria as mandated by federal and state regulations and by this Laboratory's hierarchy before being permitted to analyze samples and perform procedures on a routine basis. The following sections describe the formal and informal training programs as well as the opportunities provided for on-the-job review. Procedures for new technicians to obtain authorization to perform analyses as well as review and approval of those analyses and future quality characterization are also described.

10.1 Training

Training for all analyses performed in this Laboratory according to the SOPs found in the appendices of this Manual shall consist of formal and informal training as well as opportunities for on-the-job review.

10.1.1 Formal Training

Formal training will consist of a 2-year Associates Degree or higher in Chemistry or Chemical Technology at an accredited institution. Alternatively, formal training requirements may be met by "equivalent professional experience" as defined by federal law.

In addition, Analysts will be required to complete training seminars and mini-courses on use of instruments used in this Laboratory offered by manufacturers of those instruments.

10.1.2 Informal Training

Informal training for a new Analyst with minimal experience will take place on the job and consist of the following steps:

1. The trainee will be asked to study the appropriate SOPs for sample and reagent preparation, analysis and instrument calibration.
2. The trainee will observe an experienced Analyst perform the reagent preparation, sample preparation and analysis, and instrument use and calibration procedures.
3. Ample opportunity will be provided for

the trainee to continue to practice analyses by using samples previously analyzed by ~~by~~ an experienced technician.

4. When formal and informal training has been completed, including sufficient opportunity for the trainee to practice all required procedures, his or her performance will be evaluated prior to being given authorization to perform analyses as described in Section 10.2 of this Manual.

For more experienced new Analysts, the Laboratory Supervisor will determine which of the previous steps may be bypassed with the exception of items 1 and 4.

10.1.3 On-The-Job-Review

Formal reviews of each Analyst's job performance will occur two times each year counting from the initial date of employment.

In addition, all aspects of each Analyst's work will be subject to ongoing on-the-job review by his/her Laboratory Supervisor who will provide the appropriate feedback as needed.

Examination of all Quality Control sample analyses performed by Analysts will provide information for on-the-job review. All analysts' performances will be monitored as described in Section 11 of this Manual. If the Quality Control statistics indicate out-of-control points on the control chart, or if an Analyst has an increase in number of rejections or deviations from expected quality control results over a previous period of time, one of the main corrective actions to be taken will be to provide an opportunity for on-the-job review.

Analysts are always encouraged to consult with each other on any analysis difficulties. This type of confirming analysis is a less structured but equally significant aspect of review procedures and quality control. To perform a confirming analysis, one Analyst notes the results of another and checks to see if he agrees with the results. The first step taken as a corrective action if these results do not

agree, or for inconsistencies found with other Quality Control samples, is for the two Analysts to examine the sample together, discuss it, and come to an agreement on the results. Such consultations provide another input for on-the-job review.

10.2 Authorization to Perform Analysis

The procedure to obtain authorization to perform analysis consists of an evaluation process, review and approval and ongoing quality characterization.

10.2.1 Examination Process

After the Analyst Training Program has been completed, each new Analyst will be evaluated before being given authorization to perform analyses.

A Quality Card on each Analyst will be maintained and stored in the personnel section of the Laboratory Master files. The card will list the name of the SOP for every analysis performed in this Laboratory. As the new Analyst attains proficiency in performance of a particular analysis, as determined by the Laboratory Supervisor, that Laboratory Supervisor will note, date and sign the Quality Card in the appropriate spot. In addition, records of the Analyst's test performance (whether it results in authorization or not) will be maintained in the files.

The examination to determine proficiency for an SOP will have two sections: a re-analysis of samples already analyzed by an experienced Analyst and analysis of quality control samples. The Supervisor will use the results of both areas to determine competence.

The new Analyst's result must agree with that of the experienced Analyst within the realm of margin of error. Performance must fall within the action limits on the control chart for overall lab performance. Quality control results must be on par with the rest of Laboratory personnel.

If the new Analyst fails to obtain authorization at this time, he will be asked to consult with an experienced Analyst on test discrepancies. Any further training

measures deemed necessary will be implemented, and the Analyst will be asked to continue to practice by reanalyzing other technicians' samples. When the new Analyst is ready for a retest, he will repeat the process.

10.2.2 Review and Approval

When the Laboratory Supervisor determines that the new Analyst meets all criteria as previously described, the results of the tests will be submitted to the Laboratory Director for review and approval prior to the Analyst receiving authorization to perform the analysis.

10.2.3 Quality Characterization

Once a new Analyst receives authorization to perform analyses, the quality of his performance will be monitored as described in Section 11 of this Manual. Such performance will be part of both informal on-the-job review and formal semi-annual review.

The above procedures also apply to experienced personnel who wish to attain proficiency in new areas for the purpose of career advancement.

11.0 PERFORMANCE MONITORING

The overall Quality Control program as established and managed by the QA Director ensures that this Laboratory is fulfilling our commitment to our clients, that our data is legally defensible and that all personnel perform their responsibilities properly.

The Quality Control program includes intra-lab sample testing, participation in inter-lab programs and statistical analysis.

11.1 Intra-lab Sample Testing

Each analysis performed in this Laboratory has its own Quality Control requirements which have been determined by the QA Director and are delineated in that SOP. These requirements are in full compliance with the NYS DOH ELAP program and are detailed in the ELAP manual. These requirements include at least one if not all of the following:

o Replicates

A portion of a sample is prepared and reanalyzed by the original analyst. The Analyst will then submit his result to his Supervisor who reviews it and in turn submits it to the QA Director for statistical evaluation.

o Duplicates

A portion of a sample is prepared and reanalyzed by a second Analyst. The Analyst will then submit his result to his Supervisor who reviews it and in turn submits it to the QA Director for statistical evaluation.

o Reference

Known standards and blanks are relabeled, prepared and analyzed as if they were samples.

o Spike

A known quantity of the analyte of interest is added to a sample which is then relabeled, prepared and analyzed.

o Surrogate

A known quantity of a substance other than the analyte of interest is added to a sample which is then relabeled, prepared and analyzed. This procedure is usually reserved for SOPs involving chromatography.

The QA Director will determine how QC testing is implemented, either on a frequency basis, e.g., after the analyses of every ten samples, or on a percent of workload basis where QC testing occurs on a regular basis with, for example, 10% of the previous time period's workload tested. This is also delineated in the SOPs and is dependent on the analyte in question. The QA Director in addition will inspect the results of all QC testing on a regular basis and provide the necessary support and directives to the Laboratory Director to ensure the QC program is properly executed.

In all cases, the NYS DOH ELAP requirements are taken as a minimum. Other QA/QC protocols may be required, depending on the sample type, e.g., food samples require FDA protocols.

Additionally, the laboratory will perform custom QC for a client upon request. Such custom QC protocols will be worked out with the client, and the client will be furnished with all QC data, including control charts, for those samples. The cost of such work will be decided by the Laboratory Director, and must have his approval. Custom QC protocols, at a minimum, must meet NYS DOH ELAP requirements.

The Laboratory Supervisor (Department Head) of each department is responsible for implementing the day-to-day QC testing and ensuring the correct type of testing at the appropriate frequencies occur. The Laboratory Supervisor is also responsible for ensuring complete records of QC testing are maintained. Data collection is described in Section 11.4.1.

11.2 Inter-lab Program

This laboratory will participate in both proficiency and round robin testing programs with outside organizations.

11.2.1 Proficiency Testing

This Laboratory participates in the mandatory proficiency testing administered by the NYS DOH ELAP program and in the voluntary testing administered by the American Industrial Hygiene Association (AIHA) and the Environmental Protection Agency (EPA). The QA Director determines which of the latter two organizations' programs each department takes part in. This decision is based on the particular analysis areas of each group.

In all programs each organization sends four samples and one blank at regularly scheduled intervals which will be determined by the QA Director. These samples are analyzed by at least five Analysts and results are averaged to obtain the final result reported to the testing agencies. The organizations then evaluate the results by determining if they are within the acceptable three standard deviations range based on the results of all participating laboratories. The number of standard deviations each result is from the mean is also reported.

11.2.2 Round Robin Testing

This Laboratory is currently part of a round robin testing program which includes two other area labs. All samples for each type of analysis performed in this Laboratory are exchanged on a semi-annual basis. Results are compared.

The QA Director recommends the particular outside labs, maintains contact with the equivalent officials at those organizations and coordinates the process for this Laboratory.

11.3 Data Validation

At regular intervals which will be determined by the QA Director, all QC data will be scanned for questionable values. Criteria for judging a result questionable will include deviation from prior data from the same sample, from another sample within the same job batch, or from a sample collected at the same site within one month of the time of collection. Any questionable results will be rechecked with other Quality Control samples.

11.4 Statistical Analysis

Copies of all data produced in intra-lab and round robin testing will be handed in by the Laboratory Supervisors to the QA Director. The QA Director then carries out the appropriate statistical analysis. Data collection and statistical analysis are described in the following.

11.4.1 Data Collection

Quality Control data are recorded in bound notebooks with separate sections for replicate, duplicate, reference, spike and surrogate analysis. Information included in these sections is to include:

- o Date of original analysis
- o Sample ID
- o Relabeled ID
- o Original and where appropriate duplicating Analysts
- o Original result and second result

Each department will maintain its own notebooks for each SOP and hand them in to the QA Director on a regular basis as delineated in the SOP.

11.4.2 Data Evaluation

The QA Director will plot the data to create control charts, graphs and spreadsheets as called for by regulatory agencies and delineated in each SOP. Control charts will contain warnings and action limits for reference samples.

In addition, the QA Director will prepare a monthly report highlighting the following:

- o Summary of all QC activities
- o Results of investigations of any QC out-lier results
- o Overall laboratory performance
- o Results of any internal or external audits

PERFORMANCE CRITERIA

Both standards of performance and corrective action to be taken when those standards are not met are discussed in this section

12.1 Performance Criteria and Standards

Performance criteria will be determined two ways.

1. Results from intra-lab and round robin testing will be plotted to see if they fall within warning and action limits.
2. The administering agencies for proficiency testing will determine performance criteria.

Either or both methods may be used and this is delineated in each SOP. Performance criteria results will be maintained for both individual Analysts and for the entire Laboratory where applicable.

12.2 Corrective Action

Corrective action will be taken when results fall outside performance levels, i.e., outside warning and action limits. The QA Director is responsible for flagging such occurrences and reporting them to the Laboratory Supervisor who then takes the appropriate action to determine the the reason for the unacceptable result.

If an Analyst is found to be at fault, the occurrence is documented and placed in the personnel file of that Analyst. If the Analyst is found to be performing below par, individual informal training will be provided. A record of the training will be placed in that Analyst's personnel file.

In round robin testing, this Laboratory's results must be within two standard deviations of the mean of the other two participating laboratories' results. If they are not, and a review of other Quality Control Statistics from this Laboratory indicates the problem is not internal, a joint meeting with the other two labs will be held to discuss the disparity. As a final, drastic step, termination of the association with the outside lab¹¹¹ deemed at fault and participation with another lab will be considered.

APPENDIX 4-3

SHORT FORM

WORK/QA PLAN SHORT FORM

**RD/RA at Occidental Chemical Corporation
Hooker/Ruco Site**

Occidental Chemical Corporation

Project Officer's Signature _____

Project Officer's Name: Patrick J. Garrity

Project Quality Assurance Officer's Signature _____

Project Quality Assurance Officer's Name: William Leroux

1. **Project:** Hooker/Ruco Remedial Action
2. **Project Requested by:** USEPA
3. **Date of Request:** July 12, 1991
4. **Date of Project Initiation:** N/A
5. **Project Officer:** (QA Program Manager) Patrick Garrity
6. **Quality Assurance Officer:** William Leroux
7. **Project Description:** Detailed description is presented in Section 2.0 RDWP.

- A. **Objective and Scope Statement:** To collect analytical data from surface and subsurface soils, performed pursuant to Section 106(A) of CERCLA, 42 U.S.C. §9606(A).
- B. **Data Usage:** To define the presence, magnitude and extent of residual Aroclor 1248 during the remedial action within the facility's boundaries.
- C. **Monitoring Network Design and Rationale:** Air Sampling (upwind and downwind locations), waste characteristic sampling, Aroclor 1248 verification and confirmation samples. Sample results will determine compliance with performance standards.
- D. **Monitoring Parameters and their Frequency of Collection:** See E. for parameters; will be a single even sampling.

E. Parameter Table

Parameter	Number of samples	Sample matrix	Analytical method reference*	Sample preservation	Holding time	Container
See attached table 1						

* if other than EPA, must be attached.

F. QA Sample Parameter Table

	Parameter	Number of samples	Sample matrix	Analytical method reference*	Sample preservation	Holding time	Containers
Field blank	all as above	10%	all as above				
Field split	all as above	10%	all as above				
Laboratory duplicate	all as above	10%	all as above				

* if other than EPA, must be attached.

8. Project Fiscal Information (Optional):

A. Survey Costs: NA

Salaries: _____

Supplies: _____

Equipment: _____

Mileage: _____

B. Laboratory Services: _____

C. Administrative Overhead: _____

D. Consultant Services: _____

Total Project Costs: _____

9. Schedule of Tasks and Products: Reference RDWP for schedule.

- 10. Project Organization and Responsibility:** The following is a list of key project personnel and their corresponding responsibilities:

Leggette, Brashears & Graham - sampling operations

Leggette, Brashears & Graham - sampling QC

P. Garrity, OCC - laboratory analysis

W. Leroux, OCC - laboratory QC

P. Garrity, OCC - data processing activities

P. Garrity, OCC - data processing QC

P. Garrity, OCC - data quality review

W. Leroux, OCC - performance auditing

W. Leroux, OCC - systems auditing

W. Leroux, OCC - overall QA

A. Weston, OCC - overall project coordination

(Note: an organizational chart should be supplied with this plan) See figure 1.

- 11. Data Quality Requirements and Assessments:**

Parameter	Sample matrix	Detection limit	Quantitation limit	Estimated accuracy	Accuracy protocol	Estimated precision	Precision protocol
See attached table 2							

- 12. Documentation, Data Reduction and Reporting:**

- A. Objective and Scope Statement:** Data sheets, field logs, photographs and chain of custody will be kept by all applicable personnel until the project is closed.

- B. Data Usage:** The designation laboratory will calculate and report the data to the OCC Site Coordinator, who will transmit the data to the EPA.
- 13. Data Validation:** The OCC Quality Assurance Officer (QAO) will evaluate the data based on surrogate recoveries, detection limits, instrument standards and evaluation of chromatogram. The QAO will determine precision and accuracy and utilize the QA criteria set fourth in the methodology of the analysis to validate the data.
- 14. Performance and Systems Audits:** Audits will be conducted in accordance with Section 4.0 of RDWP.
- 15. Corrective Action:** Corrective Actions will be conducted in accordance with Section 4.0 RDWP.
- 16. Reports:** The final output of the project will be a sampling and analysis report. The report shall include the following: a) map of sample locations, b) sample I.D. numbers, c) sample analysis information, d) sample analysis results, e) QA/QC data, f) QA/QC review and g) assessment of the data.

cmp
July 24, 1991
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TABLES

TABLE 1

OCCIDENTAL CHEMICAL CORPORATION
HOOKER/RUGO SITE
HICKSVILLE, NEW YORK

Parameters

Parameters	Method reference	Matrix	Sample presentation	Holding time	Sample container
Aroclor 1248	NIOSH 5503 ^{1/}	Air	Sealed cartridge	2 months	glass fiber
Particulates	NIOSH 0500 ^{1/}	Air	Sealed cartridge	--	PVC filter
Ignitability	SW-846 1010 ^{2/}	Soil	4°C	--	8 oz. glass
Corrosivity	SW-846 9040/9041	Soil	4°C	--	8 oz. glass
Reactivity					
Cyanide bearing	SW-846 9010	Soil	4°C	--	8 oz. glass
Sulfide bearing	SW-846 9030	Soil	4°C	--	8 oz. glass
Toxicity					
Non-volatile extraction	SW-846 1311	Soil	4°C	56 days ^{3/} 360 days ^{4/}	8 oz. glass
Zero-headspace extraction	SW-846 1311	Soil	4°C	28 days ^{5/} 54 days ^{6/}	8 oz. glass
Aroclor 1248	SW-846 8080	Soil	4°C	Extract in 10 days Analyze in 40 days	8 oz. glass

TABLE 1
(continued)

OCCIDENTAL CHEMICAL CORPORATION
HOOKER/RUCO SITE
HICKSVILLE, NEW YORK

Parameters

Parameters	Method reference	Matrix	Sample presentation	Holding time	Sample container
Aroclor 1248	SW-846 8080	Soil	4°C	Extract in 10 days Analyze in 40 days	8 oz. glass
Aroclor 1248	EPA SOW 2/88 ^{1/}	Soil	4°C	Extract in 10 days Analyze in 40 days	8 oz. glass

- 1/ NIOSH Manual of Analytical Methods, Third Edition, Revision 1.0, United States Department of Health, Education and Welfare, Published (NIOSH) 77-157-A (1987).
- 2/ Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Office of Solid Waste and Emergency Response, November 1986.
- 3/ Mercury requires extraction within 28 days and analysis within 28 days; total elapsed holding time is 56 days.
- 4/ Metals, excluding mercury require extraction within 180 days and analysis within 180 days, total elapsed holding time is 360 days.
- 5/ Volatiles require extraction within 14 days and analysis within 14 days; total elapsed holding time is 28 days.
- 6/ Semi-volatiles require extraction within 7 days, prepare extraction within 7 days and analysis within 40 days; total elapsed holding time is 54 days.
- 7/ United States Environmental Protection Agency, "Contract Laboratory Protocols, Statement of Work for Organic Analyses, Multi-Media, Multi-Concentration", February 1988.

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LEGGETTE, BRASHEARS & GRAHAM, INC.

TABLE 2A

OCCIDENTAL CHEMICAL CORPORATION
HOOKER/RUCO SITE
HICKSVILLE, NEW YORK

Remedial Data Quality
Requirements and Assessments

Parameter	Method reference	Matrix	Precision/accuracy objectives (percent recovery and RSD)	Percent completed
Aroclor 1248	NIOSH 5503	Particulates	Per method	95
Particulates	NIOSH 0500	Air	Per method	95
Ignitability	EPA 1010/1020	Soil/water	Per method	95
Corrosivity	EPA 9040/9041	Soil/water	Per method	95
Reactivity	EPA 9010/9030	Soil/water	Per method	95
Toxicity	EPA 1311	Soil/water	Per method	95
Aroclor 1248	EPA 8080	Soil	Per method	95
Aroclor 1248	EPA SOW 2/88	Soil	Per method	95

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TABLE 2B

OCCIDENTAL CHEMICAL CORPORATION
HOOKER/RUCO SITE
HICKSVILLE, NEW YORK

Distribution of Analysis

Parameter	Laboratory
Aroclor 1248	Volumetric Techniques
Particulates	Volumetric Techniques
Ignitability	RECRA
Corrosivity	RECRA
Reactivity	RECRA
Toxicity	RECRA
Aroclor 1248	Volumetric Techniques
Aroclor 1248	Weston Analytical

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FIGURE

QA PROJECT ORGANIZATION HOOKER/RUCO SITE

REMEDIAL PROJECT TEAM

