ORTEK
ENVIRONMENTAL LABORATORY

QUALITY ASSURANCE
PROGRAM MANUAL

Revision # 1

Controlled Copy #

12

JUN 1993
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Approved By:

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General Manager

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REVISION 1

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CONTENTS, PAGE #S	DATE DISTRIBUTED

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NEESA HAZWRAP LQAP Element #	COE CDAP Topic #	EPA QAPjP Element #	QAPM Section #	TABLE OF CONTENTS SECTION TITLE	PAGE #	REVISION #
1	-	1	0	Title Page with Approvals	1	1
-	-	1	0	Acceptance Sheet	2	1
-	-	1	0	Distribution List	3	1
2	1	2	0	Table of Contents	8-14	1
			0	List of Tables	15	1
			0	List of Figures	16	1
			0	List of Appendices Available	17	1
-	-	-	1.0	INTRODUCTION	1	1
6,7,15	2	3	2.0	LABORATORY FACILITIES AND OPERATIONS	1	1
			2.1	**Equipment**	1	1
			2.2	**Material Procurement and Control**	1	1
			2.2.1	Purchasing	1	1
			2.2.2	QC Lot Checks	6	1
			2.2.3	Analytical Standards	6	1
			2.2.4	Material Storage and Disposal	8	1
			2.3	**Sample Management**	8	1
			2.3.1	Sample Containers/Preservatives	10	1
			2.3.2	Field Collection	17	1

NEESA HAZWRAP LQAP Element #	COE CDAP Topic #	EPA QAPjP Element #	QAPM Section #	TABLE OF CONTENTS SECTION TITLE	PAGE #	REVISION #
			2.3.3	Laboratory Storage	18	1
			2.3.4	Sample and Waste Disposal	18	1
			2.4	**Security**	19	1
3,4	4	4	3.0	LABORATORY ORGANIZATION AND RESPONSIBILITY	1	1
			3.1	**General Manager**	1	1
			3.2	**Laboratory Manager**	1	1
			3.3	**Marketing Manager**	3	1
			3.4	**Laboratory QA Officer**	3	1
			3.5	**Section Supervisors**	4	1
			3.6	**Analysts**	5	1
			3.7	**Support Staff**	5	1
			3.8	**Training**	6	1
-	3	5	4.0	QA OBJECTIVES	1	1
5	5	6	5.0	SAMPLING PROCEDURES	1	1
17	6	7	6.0	SAMPLE CUSTODY AND DOCUMENT CONTROL	1	1
			6.1	**Chain-of-Custody Documentation**	1	1
			6.1.1	Sample Receiving Procedures	1	1
			6.1.2	Sample Log-In Procedures	7	1
			6.1.3	Analytical Documentation	7	1
			6.2	**Document Control**	8	1

NEESA HAZWRAP LQAP Element #	COE CDAP Topic #	EPA QAPjP Element #	QAPM Section #	TABLE OF CONTENTS SECTION TITLE	PAGE #	REVISION #
10	7	8	7.0	CALIBRATION PROCEDURES AND FREQUENCY	1	1
			7.1	**Measurement Equipment and Supplies**	1	1
			7.2	**Instrument Calibration Procedures**	3	1
			7.2.1	GC/MS VOCs and BNAs	5	1
			7.2.2	GC VOCs	5	1
			7.2.3	Pesticides and PCBs - GC/EC	6	1
			7.2.4	Metals - ICAP	6	1
			7.2.5	AA Furnace and Cold Vapor Mercury Analyzer	6	1
			7.2.6	pH and Ion-Selective Electrodes	7	1
			7.2.7	Lachat Auto Analyzer	7	1
			7.3	**Calibration Records**	8	1
9,11	7	9	8.0	ANALYTICAL PROCEDURES	1	1
			8.1	**Detection Limits**	1	1
			8.2	**Variance From Analytical Methods/ORTEK SOPs**	4	1
14	7	10	9.0	DATA REDUCTION, VALIDATION & REPORTING	1	1
			9.1	**Data Reduction**	1	1
			9.1.1	Raw Data Generation	1	1
			9.1.2	Raw Data Verification	3	1

NEESA HAZWRAP LQAP Element #	COE CDAP Topic #	EPA QAPjP Element #	QAPM Section #	TABLE OF CONTENTS SECTION TITLE	PAGE #	REVISION #
			9.2	**Data Validation**	8	1
			9.2.1	Software	8	1
			9.2.2	Instrument Calibration	9	1
			9.2.3	Analytical Method Assessment	9	1
			9.3	**Data Reporting**	10	1
12	7	11	10.0	INTERNAL QC CHECKS AND FREQUENCY	1	1
			10.1	**Precision QC Samples**	1	1
			10.1.1	Lab Duplicate	1	1
			10.1.2	Blank Spike Duplicate (BSD)	1	1
			10.1.3	Matrix Spike Duplicate (MSD)	6	1
			10.2	**Accuracy QC Samples**	6	1
			10.2.1	Trip Blanks (TB)	6	1
			10.2.2	Field Blanks	6	1
			10.2.3	Rinsate Blanks	7	1
			10.2.4	Method Blanks (MB)	7	1
			10.2.5	Preservative or Solvent Blank (PS)	8	1
			10.2.6	Blank Spikes/Blank Spike Duplicate (BS/BSD)	8	1

NEESA HAZWRAP LQAP Element #	COE CDAP Topic #	EPA QAPjP Element #	QAPM Section #	TABLE OF CONTENTS SECTION TITLE	PAGE #	REVISION #
			10.2.7	Matrix Spike/Matrix Spike Duplicate (MS/MSD)	9	1
			10.2.8	Analytical (Post Digestion Spikes) (AS)	9	1
			10.2.9	Internal Standards (IS)	9	1
			10.2.10	Surrogate Spikes (SS)	10	1
			10.2.11	Laboratory Control Standards (LCS)	10	1
			10.3	**Representativeness QC Samples**	10	1
			10.3.1	Field Duplicate	11	1
			10.3.2	Collocated Sample	11	1
			10.4	**Completeness QC Samples**	11	1
			10.5	**Comparability QC Samples**	11	1
			10.5.1	Split Samples	11	1
			10.5.2	External Performance Evaluations	12	1
			10.6	**Control Charts**	12	1
16	7	12	11.0	PERFORMANCE AND SYSTEMS AUDITS AND FREQUENCY	1	1
			11.1	**Performance Audits**	1	1
			11.2	**Systems Audits**	2	1
			11.3	**Data Audits**	5	1
			11.4	**ORTEK Certifications**	5	1

NEESA HAZWRAP LQAP Element #	COE CDAP Topic #	EPA QAPjP Element #	QAPM Section #	TABLE OF CONTENTS SECTION TITLE	PAGE #	REVISION #
8	7	13	12.0	PREVENTIVE MAINTENANCE	1	1
19	7	14	13.0	SPECIFIC ROUTINE PROCEDURES TO ASSESS DATA PRECISION, ACCURACY AND COMPLETENESS	1	1
			13.1	Data Precision Calculation	1	1
			13.2	Data Accuracy Calculation	2	1
			13.3	Data Completeness Calculation	3	
			13.4	Data Set Assessment	4	1
13	7	15	14.0	CORRECTIVE ACTION	1	1
			14.1	Out-of-Control Conditions	1	1
			14.2	Corrective Action	1	1
			14.3	Responsibility for Corrective Action	2	1
			14.4	Corrective Action Requested by External Auditors	3	1
18	-	16	15.0	QUALITY ASSURANCE REPORTS TO MANAGEMENT	1	1
			15.1	Weekly Management Meetings	1	1
			15.2	Monthly Data Audit Reports	1	1
			15.3	Quarterly Internal Lab Audit Results	1	1
			15.4	Client Monthly Progress Reports/QA Reports	1	1
-	8	-	16.0	DATA DELIVERABLES	1	1
-	9	-	17.0	REFERENCES	1	1

NOTES:

NEESA:	Naval Energy & Environmental Support Activity
HAZWRAP:	U.S. Department of Energy Hazardous Waste Remedial Actions Program
COE:	U.S. Army Corps of Engineers
LQAP:	Laboratory Quality Assurance Plan
CDAP:	Chemical Data Acquisition Plan
QAPjP:	Quality Assurance Project Plan
QAPM:	Quality Assurance Program Manual

LIST OF TABLES	PAGE №	REVISION №
1. Major Analytical Instrumentation	2-3	1
2. Sample Containers/Preservatives and Holding Times	2-11	1
3. DQO Levels and Data Uses	4-2	1
4. Laboratory Requirements for DQOs	4-2	1
5. Equipment Periodic Calibration Requirements	7-2	1
6. Instrument Calibration Requirements	7-4	1
7. ORTEK Analytical Method Capabilities	8-2	1
8. Internal QC Checks & Frequency	10-2	1
9. Control Chart Parameters	10-13	1
10. PM	12-2	1
11. Data Deliverables	16-2	1

LIST OF FIGURES	PAGE №	REVISION №
1. Building Floor Plans	2-2	1
2. Preservative/Reagent or Solvent QC Checklist	2-7	1
3. Sample Collection and Shipment Form	2-9	1
4. Sample Bottle Request Form	2-11	1
5. Out-of-Control Form	2-16	1
6. ORTEK Management Structure	3-2	1
7. ORTEK Blind Performance Sample Evaluation Form	3-7	1
8. ORTEK Training Form	3-8	1
9. Sample Documentation Flow	6-2	1
10. Extract Chain-of-Custody Form	6-3	1
11. Chain-of-Custody/Analysis Request Form	6-4	1
12. ORTEK Sample Log-In Form	6-5	1
13. Sample Change Record	6-10	1
14. ORTEK Telephone Log	6-11	1
15. Laboratory Method SOP Format	8-5	1
16. Laboratory Data Processing Flow Chart	9-2	1
17. Wet Chemistry QC Checklist	9-4	1
18. Metals QC Checklist	9-5	1
19. GC QC Checklist	9-6	1
20. GC/MS QC Checklist	9-7	1
21. Data Validation Process Flow	9-11	1
22. Shewhart Control Chart	10-5	1
23. Surrogate Percent Recovery Control Chart	10-14	1
24. ORTEK Quality Control Report	16-5	1

LIST OF APPENDICES AVAILABLE
Personnel Resumes
Laboratory Operational Standard Operating Procedures (OP)
Wet Chemistry Standard Operating Procedures (WC)
Metals Standard Operating Procedures (M)
Organic Standard Operating Procedures (O)
Sample Preparation Standard Operating Procedures (P)

1.0 INTRODUCTION

Quality assurance (QA) is a planned system of activities that will provide a quality product. ORTEK's product is environmental data. The purpose of this QA manual is to provide each ORTEK laboratory technician/analyst with the requirements necessary to produce defensible environmental data of known quality.

The precision, accuracy, completeness, representativeness and comparability of environmental data produced for ORTEK clients, must be consistently evaluated and documented in accordance with this manual. Personnel responsible for implementation, documentation and inspection of this QA Manual are accountable for quality from sample receipt through data reporting and sample disposal.

This QA manual is written in accordance with the following established guidelines as they apply to analytical laboratory measurements:

"Guidelines and Specifications for Preparing Quality Assurance Program Plans", EPA QAMS-004/80, EPA-600/8-83-024.

"Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans", EPA QAMS-005/80, EPA-600/4-83-004.

"Guidelines for the Preparation of Standard Operating Procedures (SOPs) for Field and Laboratory Measurements," US EPA Region V, March 16, 1989.

"Final Standard Quality Assurance Project Plan Content Document," US EPA Region V, June 1989.

"Chemical Data Quality Management for Hazardous Waste Remedial Activities," Department of the Army, US Army Corps of Engineers, ER-1110-1-263, 1 October 1990.

"Content Requirements for Quality Assurance Project Plan," US EPA Region V, January 1989.

"Requirements for Quality Control of Analytical Data," DOE/HWP-65/R1, July 1990.

"Sampling and Chemical Analysis Quality Assurance Requirements for the Navy Installation Restoration Program," NEESA 20.2-047B, June 1988.

This QA manual addresses the 16 essential elements of an EPA Quality Assurance Program Plan (QAPP), the 19 elements of a DOE, NEESA, and HAZWRAP Laboratory Quality Assurance Plan (LQAP) and the 9 topics of a COE Chemical Data Acquisition Plan (CDAP). The title page and table of contents were not considered separately numbered sections. As this ORTEK QA Manual is not intended to be project specific, the "project" is considered to be the laboratory. Accordingly, the project description and project organization and responsibility elements describe ORTEK's operations and QA structure only. Project Specific Quality Assurance Project Plans (QAPjPs) can be generated upon request.

This document is considered a "manual" as it is intended to be easily handled and referred to often. It is written for <u>use</u> and not <u>disuse</u> by ORTEK staff. The usual and worst fate of QA documents is to lie on a bookshelf collecting dust and to be trotted out during audits or to impress clients. This manual is not intended to impress, but to express the quality control requirements of the laboratory clearly and succinctly for all to follow.

2.0 LABORATORY FACILITIES AND OPERATIONS

The ORTEK laboratory consists of approximately 15,000 square feet and is located at 1609 Western Avenue, in Green Bay, Wisconsin. The facility floor plan is depicted in Figure 1. Adequate space exists for expansion of both analytical instrumentation and/or support functions.

2.1 Equipment

Major instrumentation available for analytical operations is listed in Table 1. Support documentation for the equipment listed includes the manufacturers operation manuals and bound logbooks that record service and maintenance. Section 7.0 and laboratory Standard Operating Procedures (SOPs) detail the instrument specific calibration requirements. Calibration results specific to the analyses are included in each client project file for GC, HPLC and GC/MS analyses.

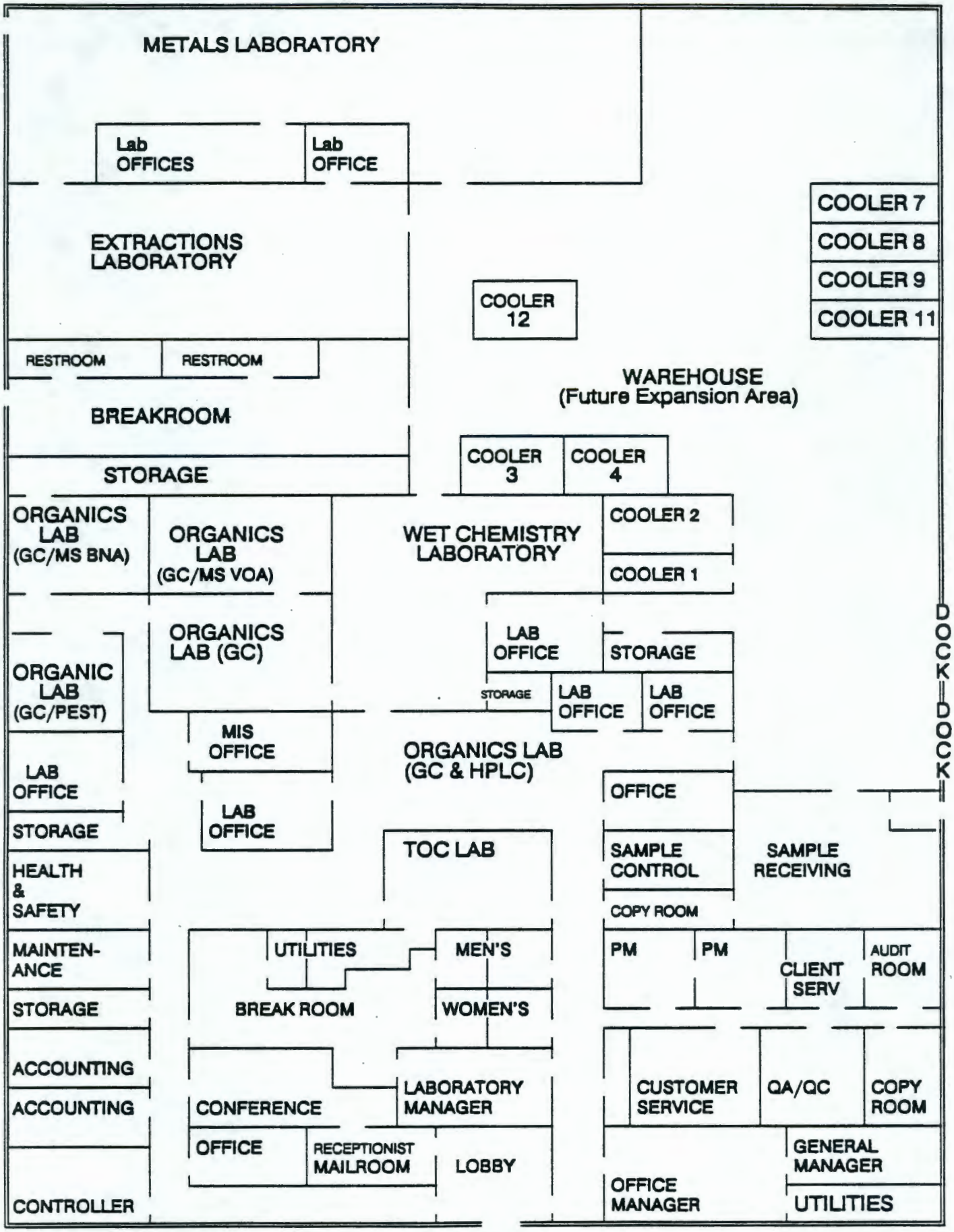
Lachat FIA, IR, TOC, TOX, AA and ICAP calibration results specific to the analyses are cross referenced by date to the samples, and are contained with raw data in batch run number logs kept in chronological order by each section supervisor.

2.2 Material Procurement and Control

2.2.1 Purchasing

The quality of materials ordered are defined by the reference analytical method included in the ORTEK Method SOPs. Each purchase order (PO) specifies the quality needed. Once the material is received the PO is used to cross check that they are of the appropriate quality. All materials are dated upon receipt and again upon opening. An inventory card system tracks the specific materials ordered.

ORTEK FLOOR PLAN



(Not to Scale)

TABLE 1 ***ORTEK INSTRUMENTATION***

INSTRUMENT ID		DATE OF PURCHASE	MANUFACTURER	MODEL NUMBER	ACCESSORIES
ORGANIC SECTION					
GC/MS	HP-D	1992	Hewlett-Packard	5970B	7673 Autosampler, RTE-A Data System
GC/MS	HP-A	1991	Hewlett-Packard	5970B	TEKMAR 2000/2016 Purge and Trap, RTE-A Data System
GC/MS	HP-C	1992	Hewlett-Packard	5970B	7673 Autosampler, RTE-A Data System
GC/MS	HP-B	1990	Hewlett-Packard	5970B	TEKMAR 2000/2016 Purge and Trap, RTE-A Data System
GC/MS	50BB	1989	Finnigan	Incos 50B	CTC A200 S Autosampler, Formaster® for CLP forms, Data General MV-1000
GC/MS	50CV	1989	Finnigan	Incos 50C	TEKMAR 2000/2016 Purge and Trap, Data General DG-10, Formaster® for CLP forms
GC	C	1990	Varian	3400	Photoionization Detector, Flame Ionization Detector, TEKMAR 2000 Purge and Trap, TEKMAR 2050 Autosampler
GC	I	1990	Varian	3400	Photoionization Detector, Flame Ionization Detector, Varian 8035 Autosampler
GC	E	1990	Varian	3400	Photoionization Detector, Flame Ionization Detector, O-I 4460A Concentrator, O-I Loop Sampling Module
GC	F	1990	Varian	3400	Flame Ionization/TSD Detector, HP 19395A Headspace Unit
GC	K	1992	Hewlett Packard	5890B	Dual ECD-Detectors 7673 Autosampler
GC	B	1990	Varian	3400	Hall Electrolytic Conductivity Detector, Photoionization Detector, TEKMAR 2000 Purge and Trap, TEKMAR 2016 Autosampler
GC	D	1991	Hewlett-Packard	5890	Hall Electrolytic Conductivity Detector, Photoionization Detector, TEKMAR 2000/2016 Purge and Trap Autosampler
GC	A	1991	Hewlett-Packard	5890	Hall Electrolytic Conductivity Detector, Photoionization Detector, OI 4560/MPM16
GC	G	1988	Perkin-Elmer	8420	Flame Ionization Detector, HP 19395A Headspace AS8300 Autosampler

TABLE 1 ***ORTEK INSTRUMENTATION***

INSTRUMENT ID	DATE OF PURCHASE	MANUFACTURER	MODEL NUMBER	ACCESSORIES
ORGANIC SECTION (Continued)				
GC J	1988	Perkin-Elmer	8420	Electron Capture Detector, AS8300 Autosampler
GC H	1990	Hewlett-Packard	5890	Dual ECD Detectors, 7673 Autosampler
GC	1987	Perkin-Elmer	8500	Flame Ionization Detector, Electron Capture Detector, AS8300 Autosampler
HPLC	1988	Waters Associates	712 WISP	Autosampler, 590 Programmable Pump, UV-486 Detector, 470 Fluorescence Detector, 680 Gradient Controller
PREP SECTION				
Gel Permeation Chromatograph	1991	Analytical Bio-Chemistry Labs	1002B	OVD-1 ABC-5014, SCR-1 Chart Recorder
Gel Permeation Chromatograph	1992	Zymark	BF-1 Benchmate	Turbovap, SSI300 LC Pump, Foxy 200 fraction collector
Sonicator	1989	TEKMAR	TM600-2	
Sonicator	1988	Heat Systems	W-385	
Sonicator	1991	Heat Systems	XL-2020	Electronic Tuning
Sample Evaporator Bath	1991	Zymark Turbovap	ZW640-3	6 Unit
METALS SECTION				
ICAP 61E	1991	Thermo Jarrell Ash	61E	Simultaneous ICP Autosampler TJA300
ICAP P2	1988	Perkin-Elmer	Plasma II	Perkin-Elmer AS80 Autosampler
GFAA 5100Z	1991	Perkin-Elmer	Zeeman 5100	Zeeman Background Correction, Graphite Furnace, Perkin-Elmer AS60 Autosampler, EDL Power Supply
GFAA 5100Z2	1991	Perkin-Elmer	Zeeman 5100	Zeeman Background Correction, Graphite Furnace, Perkin-Elmer AS60 Autosampler, EDL Power Supply, Flame Port

TABLE 1 ***ORTEK INSTRUMENTATION***

INSTRUMENT ID	DATE OF PURCHASE	MANUFACTURER	MODEL NUMBER	ACCESSORIES
AA Spectrometer 3030B	1985	Perkin-Elmer	3030B	Graphite Furnace Cold Vapor Analysis, Perkin-Elmer AS60 Autosampler, EDL Power Supply, Flame Capabilities
AA Spectrometer 1100B	1987	Perkin-Elmer	1100B	Flame
Automated Mercury Analyzer PS200	1992	Leeman Labs	PS200	PS200 Autosampler
WET CHEM SECTION				
Autoanalyzer	1988	Lachat Quick Chem	II	Lachat Model 1100 Autosampler, Model 80 Circulating Water Bath
Fourier Transformation Infrared Spectrometer (FTIR)	1989	Perkin-Elmer	1610	PE Graphics Plotter
Total Hydrocarbon Analyzer	1992	Buck Scientific	HC-404	
Total Organic Carbon Analyzer	1992	O-I Corporation	700	Autosampling Module, Model 524 Purging & Sealing Unit
Total Organic Halide Analyzer	1992	Mitsubishi Kasei Corporation	TOX-10E	

2.2.2 QC Lot Checks

The quality of preservatives, acids and solvents used by ORTEK are documented in accordance with SOP OP-20 in order to provide assurance that analytical results reported are not biased. A preservative/reagent or solvent QC checklist (Figure 2) is completed for each lot received or prepared. Set aside lots are documented once upon receipt of the first shipment. All subsequent lots are verified by checking that the lot number matches the original vendor lot that was QC tested. The QC lot number assigned is used in all subsequent documentation such as extraction logbooks and runlogs.

2.2.3 Analytical Standards

The identity and purity of the neat standard must be documented by the vendor and supplied with the standards. All weights are traceable to NIST in accordance with SOP OP-28. This documentation is attached to the Standards Logbook, or kept in a Standards Binder for each section of the laboratory.

Expiration dates are written on each analytical standard prepared by ORTEK or purchased commercially. Metals working standards are less than 24 hours old at the beginning of the run, so expiration dates are not required. In addition, a preparation or opening date is recorded on the container. All bench records containing or referencing calibration results, reference the standards used in the file or runlog name by the Standards Logbook number and page where the documentation is recorded. Instrument log books for metals reference the standard number used. All lab working standards are clearly labeled in accordance with SOP OP-32.

When analytical standards are discarded, the entry in the Standards Logbook is crossed out with an X and the analyst discarding the standard enters his/hers initals, date discarded, and indicates how the standard was disposed of.

Only Class A volumetric glassware cleaned in accordance with SOP OP-08 is used to prepare standards. The analytical balance used to weigh standards is calibrated and leveled before use in accordance with SOP OP-5. Testing of pipettes is done in accordance with SOP OP-7.

FIGURE 2
PRESERVATIVE/REAGENT OR SOLVENT QC CHECK LIST

PRESERVATIVE		REAGENT OR SOLVENT	
HOW PREPARED:		HOW PREPARED:	
DATE PREPARED:		DATE PREPARED:	
VENDOR:		VENDOR:	
VENDOR LOT #:		VENDOR LOT #:	
DATE LOT RECEIVED		DATE LOT RECEIVED	
USES:		USES:	
QC LOT # ASSIGNED:		QC LOT# ASSIGNED:	

ANALYSIS	DATE ANALYZED:
LAB SAMPLE ID:	BATCH #:

HOW PREPARED FOR ANALYSIS: _____
_____
_____
_____ METHOD OF
ANALYSIS: _____

RESULTS	SUMMARY OF DETECTS:

_____
_____
_____
_____ APPROVED
BY: _____ DATE: _____

Original to: QC OFFICER

cc: Metals
Sample Receiving
Organics
Wet Chem

Data quality can be adversely affected by the storage of lab reagents and standards in inappropriate containers. No organic reagents or standards are stored in or prepared with any plasticware, in order to avoid phthalate contamination or analyte sorption. Metal standards are stored in high density polyethylene (HDPE) containers to reduce the probability of metals plating out on the container. Amber containers are used for photosensitive reagents.

2.2.4 Material Storage and Disposal

Manufacturer's recommendations regarding storage and disposal are followed. Standards and reagents are stored separately from samples in accordance with SOP OP-36. Incompatible reagents such as organic acids and flammable liquids are stored separately from oxidizing acids. Organic reagents are stored at or below 4°C. Stock standards in organic solvents are stored in freezers.

Refrigerator, freezer, and BOD incubator temperatures are monitored by an electronic system integrated with the building security alarms. Daily recording and verification of temperatures is done in accordance with SOP # OP-06. Thermometers are calibrated annually in accordance with SOP # OP-04.

Expired reagents, standards, and samples are segregated and contents/concentrations are compared against the City of Green Bay Metropolitan sewage discharge limits and TCLP limits for assessment of disposal options. Disposal is done according to ORTEK's Waste Management Plan (SOP OP-14).

2.3 Sample Management

Clients are encouraged to use sample containers and documentation supplied by ORTEK to aid in collecting a representative sample that is properly preserved and analyzed to meet their data needs. Figure 3 presents ORTEK's sample preservation instructional to clients. Although this manual defines the lab QA Program, field QA operations also impact sample integrity. Field QA is discussed below to alert ORTEK personnel to concerns and requirements for proper sample packaging, shipment, and collection.

FIGURE 3

SAMPLE COLLECTION & SHIPMENT REGULATIONS

Most analytical programs require
samples to be received
CHILLED
- but not frozen

Samples should therefore be
PACKED WITH ICE
to keep the temperature at
NO MORE THAN 4°C.

("blue ice" can be used, but ice
typically provides better cooling.)

***DATA FOR SAMPLES RECEIVED AT GREATER THAN 6°C
MAY BE REJECTED BY THE
WISCONSIN DNR
OR
OTHER REGULATORY AGENCIES!

ORTEK's "Temperature Blank" is a vial of water which
will be used to check cooler temperature on receipt.
The measured temperature is recorded on the Chain of Custody.

2.3.1 Sample Containers/Preservatives

ORTEK uses commercially available precleaned and virgin sample bottles. No sample bottles are reused. Two different levels of bottle quality are kept in inventory. One level consists of bottles of which a certain percentage are checked for purity in each lot by the vendor. A certificate of analysis is included with the bottles.

The other level of bottle quality available is the same as the precleaned and tested bottles, but without certificates of analysis. ORTEK also purchases presterilized bottles for coliform analyses. Any virgin bottles received are randomly checked for cleanliness after cleaning in accordance with SOP OP-03.

Preservatives are prepared by ORTEK and their quality documented on the Preservative/Reagent or Solvent QC Checklist (Figure 2) in accordance with SOP OP-20. Preservatives may be added to the sample container or contained separately in sealed ampules, depending on client need.

The client's request for bottles and shipping supplies is documented on a Sample Bottle Request Form (Figure 4). The lot number of trip blanks, bottles, and preservatives is recorded in order to trace possible sources of contaminated field/trip blanks. SOP OP-03 details the procedure for tracking sample bottles and preservatives sent to clients.

Sample containers, preservatives, and minimum volumes needed by ORTEK are listed in Table 2. EPA holding times are also listed to enable clients to time sampling and to permit efficient scheduling of analyses. Noncomformances discovered upon sample receipt are documented using the Sample Receiving Out-of-Control Form (Figure 5). Clients are notified and appropriate corrective action taken.

FIGURE 4

SAMPLE BOTTLE REQUEST FORM

Will Pick Up ☐ Yes ☐ No						Date:
Date:						Send Shipment to:
Time: ☐ A.M. ☐ P.M.						
Job No.:						
PLASTIC	TOTAL NUMBER	UNPRES	H_2SO_4	HNO_3	OTHER	Sterile (6 OZ.) ☐
1000 ml						Cooler(s) Needed ☐ Yes ☐ No
500 ml						Amount:
250 ml						Bottles Labeled ☐ Yes ☐ No
GLASS (CLEAR)						Special Instructions:
1000 ml						
500 ml						
GLASS (AMBER)						
1000 ml						Requested By:
500 ml						Order Filled by:
VIALS						Ship VIA
4 oz.						(Phone # & Contact needed for Fed. Ex.)
40 ml						
2 oz. Septum Top						
2 oz.						
Air Tube 100's						
Air Tube 32's						
Trip Blank						

TABLE 2 ORTEK SAMPLE CONTAINERS/PRESERVATIVES AND HOLDING TIMES

ANALYTE	MATRIX	EPA METHODS	HOLDING TIME		CONTAINER			PRESERVATION							MINIMUM SAMPLE VOLUME
			TO EXTRACT	TO ANALYSIS	#	SIZE	CONSTRUCTION	4°C	HCl	HNO_3	H_2SO_4	NaOH	NO HEADSPACE	OTHER	
Alkalinity	water	310.1	--	14 days	1	250 ml	HDPE	✓							100 ml
Ammonia	water	350.2		28 days	1	500 ml	HDPE	✓			✓				100 ml
BOD	water	405.1		48 hour	1	500 ml	HDPE	✓							500 ml
COD	water	410.4		28 days	1	500 ml	HDPE	✓			✓				100 ml
Chloride	water	325.1	--	28 days	1	500 ml	HDPE	✓							100 ml
Conductivity	water	120.1,9050	--	28 days	1	500 ml	HDPE	✓							200 ml
Coliform, safe/unsafe	water	909C	--	30 hrs. max 24 hrs.	1	200 ml	HDPE	✓						Sterile NA_2SO_3	100 ml
Coliform, total	water	colilert	--	30 hrs max 24 hrs.	1	200 ml	HDPE	✓						Sterile NA_2SO_3	100 ml
Cyanide	water	335.2	--	14 days	1	L	HDPE	✓				✓		pH > 12	1000 ml
Cyanide	soil	9010	--	14 days	1	8 oz	glass	✓							50 g
Flashpoint	waste	1010	--	14 days	1	4 oz	glass	✓							10 g
Flashpoint	water			28 days	1	500 ml	HDPE								100 ml
	soil														100 gm
Fluoride	water	340.2	--	28 days	1	500 ml	HDPE	none							100 ml
Free liquids	waste	9095		28 days	1	L	HDPE	✓							500 ml
Hardness	water	130.2	--	180 days	1	500 ml	HDPE			✓				pH < 2	100 ml
TKN	water	351.2	--	28 days	1	L	HDPE	✓			✓				500 ml
Nitrate	water	353.2	--	48 hours	1	250 ml	HDPE	✓							100 ml
Nitrite	water	353.2	--	48 hours	1	250 ml	HDPE	✓							100 ml
$NO_2 + NO_3$	water	353.2	--	28 days	1	250 ml	HDPE	✓			✓			pH < 2	100 ml
Oil & grease	water	9070,413.1	--	28 days	1	L	HDPE	✓			✓			pH < 2	1L

TABLE 2 ORTEK SAMPLE CONTAINERS/PRESERVATIVES AND HOLDING TIMES

ANALYTE	MATRIX	EPA METHODS	HOLDING TIME		CONTAINER			PRESERVATION							MINIMUM SAMPLE VOLUME
			TO EXTRACT	TO ANALYSIS	#	SIZE	CONSTRUCTION	4°C	HCl	HNO_3	H_2SO_4	NaOH	NO HEADSPACE	OTHER	
Oil & grease	soil	9071	--	28 days	1	8 oz	glass	✓							50 g
pH	water	150.1	--	24 hours	1	500 ml	HDPE	✓							100 ml
pH	soil	9045	--	24 hours	1	4 oz	glass	✓							50 g
Phenol, total	water	420.2,9066	--	28 days	1	L	glass	✓			✓			pH < 2	1000 ml
Phosphorus, ortho	water	365.4	--	48 hours	1	500 ml	HDPE	✓						Filter	100 ml
Phosphorus, total	water	365.4	--	28 days	1	500 ml	HDPE	✓			✓			pH < 2	100 ml
Sulfate	water	375.2,9036	--	28 days	1	500 ml	HDPE	✓							200 ml
Sulfide	water	376.1,9030	--	7 days	1	500 ml	HDPE	✓				✓		pH > 9 Zn Ac	200 ml
TDS	water	160.1	--	7 days	1	500 ml	HDPE	✓							200 ml
TS	water	160.3	--	7 days	1	500 ml	HDPE	✓							200 ml
TSS	water	160.2	--	7 days	1	500 ml	HDPE	✓							200 ml
TVSS	water	160.4	--	7 days	1	500 ml	HDPE	✓							200 ml
TOC	water	415.1,9060	--	28 days	1	250 ml	HDPE	✓			✓				25 ml
TOX	water	9020	--	7 days	1	16 oz	glass	✓			✓				250 ml
TRPH - WIDNR	water	DNR	--	14 days	1	quart	amber glass	✓	✓						1000 ml
	soil	DNR	--	14 days	1	4 oz	glass jar	✓							10 g
METALS															
Hexavalent Chromium	water	7196	--	24 hours	1	L	HDPE	✓							500 ml
Hexavalent Chromium	soil	7196 Mod.	24 hours	--	1	8 oz	glass jar	✓							20 g
Mercury*	water	7470,245.1	--	28 days	1	L	HDPE			✓					500 ml

TABLE 2 ORTEK SAMPLE CONTAINERS/PRESERVATIVES AND HOLDING TIMES

ANALYTE	MATRIX	EPA METHODS	HOLDING TIME		CONTAINER			PRESERVATION							MINIMUM SAMPLE VOLUME
			TO EXTRACT	TO ANALYSIS	#	SIZE	CONSTRUCTION	4°C	HCl	HNO_3	H_2SO_4	NaOH	NO HEADSPACE	OTHER	
Mercury*	soil	245.5, 7471	--	28 days	1	8 oz	glass jar	✓							10 g
TCLP Metals	waste	1311	14 days	180 days	1	8 oz	glass jar	✓							20 g
Metals	water	ICAP GFAA	--	180 days	1	L	HDPE			✓					1000 ml
Metals	soil	ICAP GFAA	--	180 days	1	8 oz	glass jar	✓							20 g
Mercury	water	245.1, 7470	26 days	28 days											
Mercury	soil	245.5, 7471	26 days	28 days											
ORGANICS															
Volatiles	water	601/602, 8010/8020, 8021, 624, 8240, 8260	--	14 days	2	40 ml	VOA Vials	✓	✓				✓		5 ml
Volatiles	soil	8010, 8020, 8260, 8021, 8240	--	14 days	2	4 oz	glass septa vials	✓					✓		5 g
Semivolatiles	water	625,8270	7 days	40 days	1	L	amber glass	✓							1000 ml
Semivolatiles	soil	8270	14 days	40 days	1	120 ml	glass jar	✓							50 g
PCB/Pesticides/ Herbicides	water	608,8080 8150	7 days	40 days	1	L	amber glass	✓							1000 ml
PCB/Pesticides/ Herbicides	soil	8080, 8150	14 days	40 days	1	8 oz	glass jar	✓							50 g
GRO	water	DNR	--	14 days	2	40 ml	VOA vials	✓					✓		
	soil	DNR	--	14 days	1	60 ml	Septum jar	✓						Tared, methanol	
DRO	water	DNR	--	14 days	1	liter	amber glass	✓						Tared	1000 ml
	soil	DNR	3½ days	14 days	1	60 ml	septum jar	✓						$MeCl_2$	25-40 gm
TCLP Organics	waste	1311			1	80 oz	amber glass jar	✓							100 g

TABLE 2 ***ORTEK SAMPLE CONTAINERS/PRESERVATIVES AND HOLDING TIMES***

ANALYTE	MATRIX	EPA METHODS	HOLDING TIME		CONTAINER			PRESERVATION							MINIMUM SAMPLE VOLUME
			TO EXTRACT	TO ANALYSIS	#	SIZE	CONSTRUCTION	4°C	HCl	HNO_3	H_2SO_4	NaOH	NO HEADSPACE	OTHER	
VOA			0	14 days											
SVOA, PEST, HERB			7 days	40 days											

FIGURE 5

ORTEK SAMPLE RECEIVING OUT OF CONTROL FORM

COMPANY NAME:	CONTACT PERSON:
PROJECT NAME:	REPORTED BY:
BATCH NUMBER:	SAMPLE NUMBER(s):
DATE: TIME:	PHONE NUMBER:
☐ HOLD TIME ☐ IMPROPER PRESERVATIVE ☐ PAPERWORK DISCREPANCY ☐ IMPROPER CONTAINER	☐ SAMPLE TEMPERATURE ____°C ☐ METHODOLOGY ☐ INSUFFICIENT VOLUME ☐ OTHER

DESCRIPTION:

PERSON RESPONSIBLE FOR RESOLUTION:

CLIENT RESPONSE:

SIGNATURE:	TIME:	DATE:

cc: Working File
Mary Jo Nash
Tori Cook
Earl Schmoll
Barb Rutten
John Rather
Chris Groh
Becki Detaege
Jan Leaf
Alan Orr
Mary Unger

2.3.2 Field Collection

Prior to collecting and shipping samples, clients are urged to contact ORTEK to ensure adequate time for delivery or courier pickup of sample bottles, trip blanks and preservatives. The capacity of the lab to handle any quick turn analyses should also be verified. The potential hazard level of the samples and specific analytical requirements and any special QC or reporting needs should also be discussed and documented. Field instrument readings such as HNU, PID or field observations should be indicated on the paperwork accompanying the samples to aid lab personnel in avoiding saturation of instrument detectors. Arrangements for Saturday delivery to the lab, or after hours courier drop off, should be made prior to sample shipment.

Those samples requiring preservation at 4°C should be iced prior to packaging and shipment in order for the temperature to be within 4 ± 2°C upon receipt. "Blue ice" packs are usually not sufficient to keep the temperature at 4 ± 2°C so cube ice or block ice is recommended. Figure 3 presents the form ORTEK sends with sample bottles to alert clients to these requirements. Courier services, commercial shippers and delivery in person by the client, all necessitate that the sample be iced as soon as the sample has been taken to ensure that all reasonable effort was made to properly chill the sample.

Field personnel should initiate either an ORTEK or client specific Chain-of-Custody Form, and indicate the analyses required. Sample containers should be labeled to indicate the sample location, date, time, sampler and preservatives used. Any trip blanks necessary should be listed on the Chain-of-Custody Form. Containers as listed in Table 2 are recommended to ensure that the sample is compatible with the intended analysis. ORTEK Sample Receiving personnel will examine the condition, preservation, container and documentation. Any discrepancies will be noted on an Out-of-Control Form (Figure 5). The client will be notified before the sample is accepted for analysis and logged into the laboratory sample tracking system. Details of the ORTEK log-in and sample receipt process is contained in SOP OP-02.

2.3.3 Laboratory Storage

Samples are stored in restricted access refrigerators or on shelves which are controlled in accordance with SOP OP-12A. This sample storage location number is entered on the Chain-of-Custody and the sample label. Soil samples are stored separately from water samples and suspected medium or high level organic samples are segregated from the other organic samples. Holding blanks are, when requested by clients, are prepared for volatile organic analysis and stored with the samples in accordance with SOP OP-18.

The refrigerated storage temperature is monitored by a digital system linked to the building security alarms. The system monitors the temperature every 15 seconds and an audible alarm is sounded if the temperature exceeds the acceptable upper range of for more than an hour. After hours, the temperature alarm sounds at the security agency and laboratory management personnel are called. The temperature probes were initially factory calibrated against a reference and are checked daily against an independent calibrated thermometer. Temperatures are recorded according to SOP OP-06 and thermometer calibration is done annually against an NIST reference thermometer in accordance with
SOP OP-04.

After analysis, samples are moved to a refrigerated holding area. Metals samples are stored at room temperature after digestion and are grouped by the blank number analyzed with them. All samples are routinely held 30 days after reports are released, unless other arrangements for longer storage have been made. Any samples with analytical results indicating that they are hazardous waste are flagged in accordance with SOP OP-14.

2.3.4 Sample and Waste Disposal

Samples that are classified as hazardous are returned to the client unless prior arrangements have been made. Solvents are recycled when appropriate, through an EPA approved facility. Sample residual waste, hazardous and nonhazardous waste and other lab waste is handled in accordance with SOP OP-14.

2.4 Security

ORTEK maintains a controlled access facility to ensure client confidentiality, proper sample custody and document control in accordance with SOP OP-13.

Visitors are required to sign in with the receptionist and are issued visitor badges. All visitors are accompanied by an ORTEK employee while in the laboratory and data operations areas.

Managers and analysts are assigned unique code numbers to activate and deactivate the security alarm system and a printout of alarm activity is provided to the Laboratory Manager by the security firm. Security breaches occurring during non-working hours release an alarm to the security agency who alerts the local police and the Laboratory Manager.

Computer access to client records, financial data and analytical data is restricted in accordance with SOP OP-15. Backup of the computer system and secure storage of the files is also detailed in this SOP.

Dependent on the QC review and data approval responsibilities and authority, each analyst or employee is restricted to the areas of the Laboratory Information Management System (LIMS) they can access or revise. No data or client files are removed from the facility and all working data files are controlled by a check out card system in accordance with SOP OP-11. QA reports to clients that include other client samples are censored so that sample results are not traceable to other clients.

3.0 LABORATORY ORGANIZATION AND RESPONSIBILITY

The Laboratory Manager has overall responsibility for the technical quality, cost control, laboratory personnel management, and adherence to project schedules. The Laboratory Quality Assurance Officer is responsible for monitoring the quality of laboratory work and taking appropriate actions to ensure that quality standards are being met. The QA Officer reports directly to the General Manager and is independent of laboratory cost or profit responsibilities, schedules and personnel supervision other than QA Assistants. Responsibilities for fulfilling day to day quality control requirements is delegated to each Laboratory Section, and the performance monitored by the QA Officer. The Section Supervisors (GC/MS/EC, GC/HPLC, Sample Preparation, Sample Receiving, Metals, Wet Chemistry, Project Management) are responsible for technical quality of work within their respective sections, including adherence to prescribed procedures for calibration, preventive maintenance, data validation, training, out-of-control forms and corrective actions. The Section Supervisors are responsible for meeting project commitment dates, (including preparation and analysis of samples within holding times), and reporting data and QA information as required by the client. Each analyst is responsible for implementing and documenting this Laboratory QA Program in daily activities which includes the preparation of out-of-control forms, taking corrective actions, and responding to QC review requests.

The specific responsibilities and authority of ORTEK personnel for QA/QC activities are described below. Resumes of personnel are available upon request. An organizational chart indicating reporting and communication lines is contained in Figure 6.

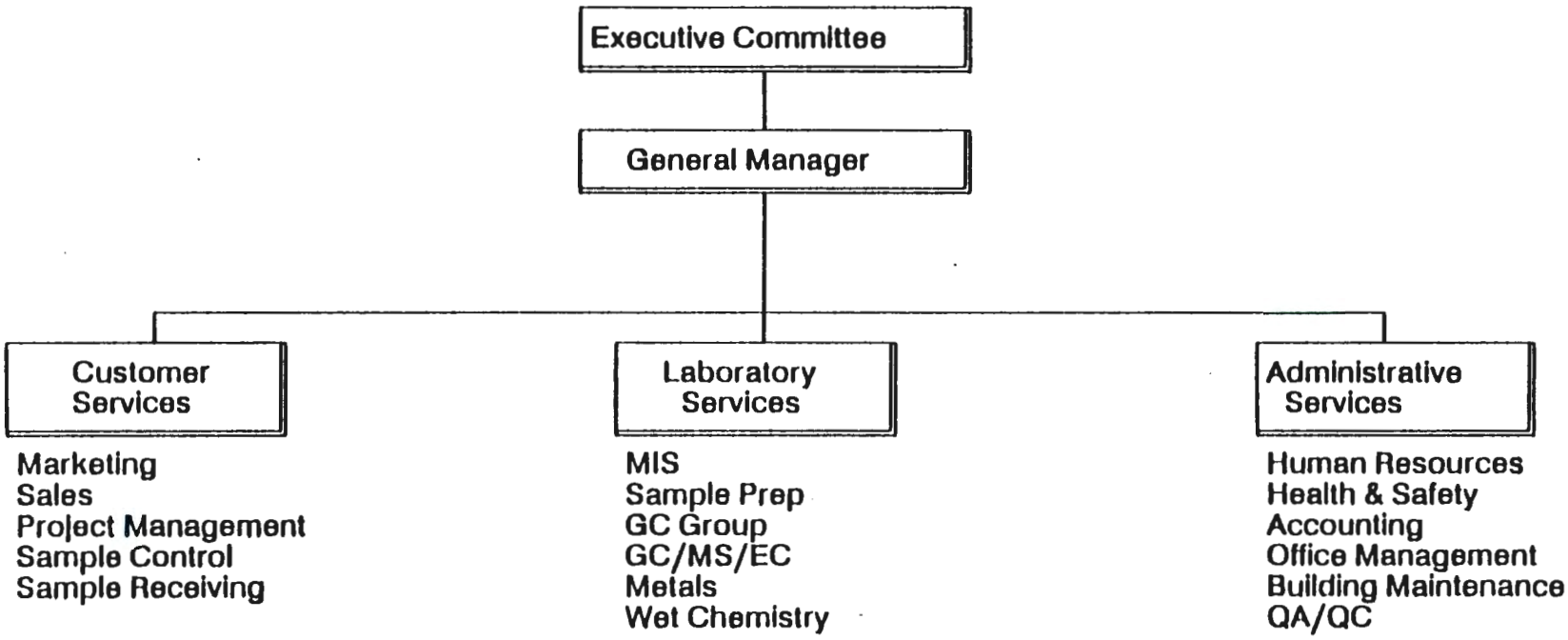
3.1 General Manager

The General Manager is responsible for long range planning and establishing the operating policies of the laboratory. He provides the resources and commitment to the Laboratory QA Officer to implement this QA Program Manual. He provides for strategic planning and communicates with the Executive Committee and Board of Managers on profit, scheduling and resource issues. Quality related laboratory performance issues are relayed to him by the QA Officer.

3.2 Laboratory Manager

The Laboratory Manager is responsible for hiring, assignment of personnel, and the purchase of new equipment. He reports to the General Manager and is responsible for the management of laboratory resources to accomplish designated goals and turnaround times.

ORTEK ORGANIZATIONAL CHART
April 29, 1993



He assesses productivity of each section of the laboratory and determines with each Section Supervisor how to optimize efficiency without compromising quality.

Additional Laboratory Manager's duties and responsibilities include the following:

- Direct the laboratory's analytical programs and physical operations.
- Coordinate and prioritize projects and associated workloads.
- Execute laboratory administrative functions.
- Ensure that analytical methods comply with client needs, this QA Manual and regulatory requirements.

3.3 Marketing Manager

The Marketing Manager is responsible for coordinating the project management and marketing efforts of the Laboratory. This involves coordinating with the Laboratory Manager in ensuring that the analytical needs of the client are met and with the QA Officer in ensuring that the client's Data Quality Objectives (DQO's) are defined and met. The Marketing Manager reports to the General Manager.

For assigned projects, his staff coordinates field supplies, tracks sampling and sample analysis status, maintains contact with the client's project manager, and reviews data for completeness. The Marketing Manager keeps the Lab Manager appraised of project schedule, analysis and QA status.

The Marketing Manager is also responsible for obtaining feedback from clients on quality and timeliness of service. The Marketing Manager prepares price quotes, responds to RFQ's and develops SOQs and other sales oriented literature.

3.4 Laboratory QA Officer

The Laboratory QA Officer has the authority to stop production of data in the laboratory, when review of the QC data or analytical procedures indicates that data quality is compromised or is not sufficient to meet client requirements. The QA Officer reports directly to the General Manager and communicates QC deficiencies and corrective actions to the Lab Manager and affected Section Supervisors. Other primary duties and responsibilities of the position are:

- Update QA Program Manual at least annually and maintain distribution list.

- Responsible for writing, maintaining and distributing laboratory operational SOPs.

- Conduct laboratory and data audits to assess effectiveness of QA Program with a monthly report in writing to Laboratory Manager and General Manager.

- Responsible for completion of Monthly Progress Reports (NEESA, HAZWRAP projects) and other client QA reports when requested.

- Maintain laboratory certification/approval records, review performance evaluation sample data, and provide responses to certifying agencies as required.

- Respond to client data challenges and provide feedback to management on outcome of challenges.

- Provide technical assistance to Section Supervisors by defining new method validation requirements and instrument detection limit verification criteria. Calculate method accuracy and precision control chart limits.

3.5 Section Supervisors

The laboratory is divided into five technical sections, each headed by a Section Supervisor. Each Section Supervisor has hands-on experience in all of the tests in the section and serves as the lead analyst and technical resource to the staff. Each reports to the Laboratory Manager. Each Section Supervisor's duties and responsibilities are:

- Organize, schedule and prioritize the section analyses with consideration for sample-holding times and client due dates.

- Check that required number and type of QC checks are performed. Identify and correct nonconformances.

- Assign analysts for data processing and data validation activities.

- Review and approve section analytical data and sign data reports.

- Evaluate instrument performance and supervise instrument calibration and preventive maintenance programs.

- Report out-of-control situations to the Laboratory QA Officer.

- Implement and verify the effectiveness of corrective actions.

3.6 Analysts

Laboratory analysts are responsible for determining and documenting the quality of the data they generate. They are responsible for equipment maintenance, calibration and documentation in accordance with ORTEK SOPs. They are to include the appropriate number and type of QC samples as defined by this QA manual, analytical method, and specific client request. They are to identify out-of-control situations and document them to their Section Supervisor and QA Officer for resolution. They are to conduct corrective actions promptly and document their effectiveness.

3.7 Support Staff

Delivery of a quality product to clients is not limited to lab analysis. Ancillary to analytical operations are the functions of sample receipt, client services, MIS, accounting, and health & safety.

Sample receiving personnel ensure that submitted samples are properly documented, preserved and entered into the Laboratory Management Information System. Sample receiving personnel clarify vague or questionable analysis requests with the client. Conversations with the client are documented and problems resolved <u>before</u> samples are logged in for analysis. MIS personnel ensure that the lab tracking system and instrument interfaces are operational with minimum downtime. Proper sample labeling, computer log-in and analyses are then verified by Project Management and the client contacted to clarify discrepancies. Client Services prepares and assembles client data packages in accordance with SOP OP-10 to include the invoice, chain-of-custody forms and analytical result sheets. Accounting personnel verify correct invoice totals and per sample charges against price quotes and/or the Analytical Services Manual list prices. Compliance with federal health & safety regulations is assessed by the Health & Safety Officer, to provide analysts a safe environment in which to conduct quality analyses.

3.8 Training

New employees are required to read this laboratory QA Manual and sign that they have accepted its requirements. On a method-by-method basis each analyst first reads the applicable SOP, performs the method, and analyzes independently prepared QC samples at least twice with acceptable results before they can be considered "certified" to produce sample data. Documentation of Certification is recorded on Figure 7 and kept by the QA Officer. SOP # OP-09A describes the details of ORTEK's technical training system.

On an ongoing basis, results of client/government agency performance evaluation samples and blind internal performance evaluation samples are recorded for each analyst to provide feedback on their performance. The QA Officer maintains these records. Figure 8 is used to record these sample results.

New employees also review laboratory safety rules before any lab work can start. Annually, videotapes from a commercial vendor are viewed and classroom refreshers on hazard communication and the Chemical Hygiene Plan are given. Attendance is mandatory and tests are given. A score of at least 70% must be attained on the safety exams or the analyst must repeat the training. A Safety Committee consisting of at least one analyst from each section of the lab meets regularly to discuss employee concerns in their areas and make written recommendations to management. The Chemical Hygiene Plan is supplied to all analysts and it is stressed that safety is everyone's responsibility.

FIGURE 7
ORTEK BLIND PERFORMANCE SAMPLE EVALUATION

ANALYTE:			ANALYST:		
METHOD:					
MATRIX:					
BLIND SAMPLE #:			SOURCE:		
DATE ANALYZED:					
ANALYST VALUE	UNITS	TRUE VALUE	UNITS	ACCEPTANCE LIMITS	LIMIT BASIS

Comments/Corrective Action:

FIGURE 8
ORTEK TRAINING FORM

Trainee:

Method:

Trainer:

Date of Certification: ___/___/___

This is to certify that the trainee can perform the above method without supervision. The trainee has successfully completed the following steps in training:

1. Has been shown all steps of the test. ☐
2. Has performed the test under supervision. ☐
3. Can independently complete all necessary calculations. ☐
4. Has successfully analyzed EPA performance samples on two separate occasions. ☐

DATE	REFERENCE TRUE VALUE	ACCEPTANCE LIMIT	ANALYST RESULT

Signatures:

Trainee: _____

Trainer: _____

QC Officer: _____

4.0 QA OBJECTIVES

The primary QA objective of the laboratory is to develop, implement and document the specific QC criteria that provide for legal, defensible data. Regardless of the client or end use of the data, all analytical results are to be traceable to properly stored and secured samples analyzed by the appropriate method on correctly calibrated instruments. All data are of known precision and accuracy as determined by the results of the internal QC checks.

The requirements for acceptable internal QC check results and frequency are often established by the client by use of Data Quality Objectives (DQOs).

DQOs are qualitative and quantitative statements that specify the quality of the data necessary to support the client's or regulatory agency's use of the data. DQOs are based on the end use of the data to be collected, the type of decision to be made, the allowable uncertainty in the decision and the risk associated with a "wrong" decision. Clients are encouraged to define their data needs and uses, applicable regulatory limits of concern, critical samples and specific methodology requirements to ORTEK as soon as possible in order to ensure use of the proper DQO.

DQO's have been segregated into levels by the US EPA, Navy and DOE in order to support the different types of decisions made based on the analytical data. Five levels of data quality were originally defined by the US EPA under the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA), more commonly referred to as Superfund. DOE/HAZWRAP adopted the same levels but labeled them as Levels A-E. The Navy selected three of the DOE/EPA levels C-E. Table 3 presents all of these DQO levels and a brief description of their intended data uses. Table 4 lists the laboratory QC requirements ORTEK follows for each level.

TABLE 3 DQO LEVELS AND DATA USES

EPA LEVEL	DOE HAZWRAP LEVEL	NAVY LEVEL	DATA USE(S)	TYPE OF ANALYSIS	LIMITATIONS
I	A	-	Screening, process monitoring, field health and safety monitoring.	Real time in-field measurements.	Nonspecific, high detection limits, background levels interfere.
II	B	-	Initial site characterization, process monitoring.	On-site lab, same day turnaround.	Concentration usually reported as range, compound identity uncertain.
III	C	C	Risk assessment, site characterization on nonSuperfund sites. Hazardous waste/RCRA analyses, remediation monitoring.	EPA approved methods in off-site or on-site lab.	Tentative organic compound identity compound unless MS or 2nd column GC analyses used.
IV	D	D	Superfund sites PRP determination risk assessment site characterization remediation monitoring	Quantitative, legally defensible data package using CLP methods (ICAP, GFAA, GC/EC, GC/MS).	TAL & TCL list compounds only available. Independent data validation of packages requires time and dollars.
V	E	E	Risk assessment PRP determination	Unusual matrices or analyses requiring method development (i.e., tissues, explosives)	Costly, limited number of labs available, long lead time for method validation required.

PRP = Potentially Responsible Party

TABLE 4 ORTEK LABORATORY REQUIREMENTS FOR DQOs

EPA LEVEL	NAVY DOE HAZWRAP LEVEL	CALIBRATION	LAB BLANK	LCS OR BLANK SPIKE	LAB DUPLICATE	MATRIX SPIKE	MATRIX SPIKE DUPLICATE	DATA PACKAGE
III	C	5 point initial calibration check every 12 hours GC 2nd column confirmation	1 per every 12 hours	1/20	1/20 (metals and wet chemistry only)	1/20	1/20 (organics only)	Batch size variable. CLP Forms only. Blank spike control charts.
IV	D	CLP requirements	CLP	1/20	NR (organics) 1/20 metals BS, BSD, MS, MSD *	CLP 1/20	CLP 1/20 (organics only)	Batch size 20. Full CLP documentation. Blank spike control charts
V	E	3 point initial calibration and check every 12 hours	1 per batch	1 per batch	NR.	NR	NR	Batch size variable. Sample result sheets, blank results, Blank spike control chart.

Legend: CLP = Contract Laboratory Program
NR = Not Required

* BS and BSD only if insufficient amount of sample per sample delivery group.

The purpose of complying with these specified DQO levels is to define the precision, accuracy, representativeness, comparability and completeness achieved for the sample analysis. In general, data generated by ORTEK should:

- Be accurate in comparison to true or reference values within an accepted tolerance limit.
- Be precise to within a specified degree of variability between replicate measurements.
- Be representative of the source sampled.
- Be comparable to analytical results obtained by other laboratories following the same DQO level and method.
- Be complete in terms of the amount of valid data obtained versus all analyses requested.

These general QA objectives are fulfilled by the ORTEK QA program that defines the specific QC samples to be analyzed and their acceptable limits. The limits are based on historical data collected and method validation studies conducted in-house. When not enough data have been collected by ORTEK to set acceptance limits, advisory limits are set using EPA data. These limits and the frequency of QC sample analyses are specified in Section 10.0 and SOP OP-21 contains procedures for creating control charts.

5.0 SAMPLING PROCEDURES

As ORTEK does not provide for sample collection, this section is not applicable. Clients are advised to use the certified sample containers supplied by ORTEK to collect their samples and contact ORTEK prior to collection to verify laboratory capacity and minimum sample volume requirements.

6.0 SAMPLE CUSTODY AND DOCUMENT CONTROL

A sample is legal evidence collected by the client representative of the site. In order for ORTEK to produce legally defensible data representative of a sample, the custody and documentation of the sample must be traceable and secure. This section discusses the lab operations necessary to ensure sample and document integrity. Figure 9 presents the flow of sample and analysis documentation in the laboratory. The sections below describe the specific documents generated and controlled.

6.1 Chain-of-Custody Documentation

The trail of the sample's journey, from collection to disposal, is documented by an unbroken written record that accounts for the secure location of the sample at all times. This unbroken written record is termed the chain-of-custody. A sample is considered in custody if it is:

- In a person's hands-on possession.
- In a person's view.
- Locked or sealed so tampering can not be done.
- In a secured area, restricted to authorized personnel only.

At ORTEK, the entire laboratory is considered a secured area restricted to analysts only, and the chain-of-custody is considered unbroken until the sample is disposed of by the sample custodian. Intralab transfer of custody occurs when samples or sample extracts are transferred from one analyst to another and is recorded on an extract chain of custody form (Figure 10).

The chain-of-custody is documented in Figure 11. These forms are sequentially numbered. The form number is entered in the sample log-in Form (Figure 12) to enable cross referencing of ORTEK sample numbers, client ORTEK batch numbers, sample storage location and custody form. Any client specific QA report or DQO level needed should also be entered in the Special Instructions Comments section to alert analysts and the QA Officer.

6.1.1 Sample Receiving Procedures

The specific steps of sample reception are detailed in SOP OP-2. Briefly, this procedure consists of the following steps:

DOCUMENTATION FLOW

FIGURE 9

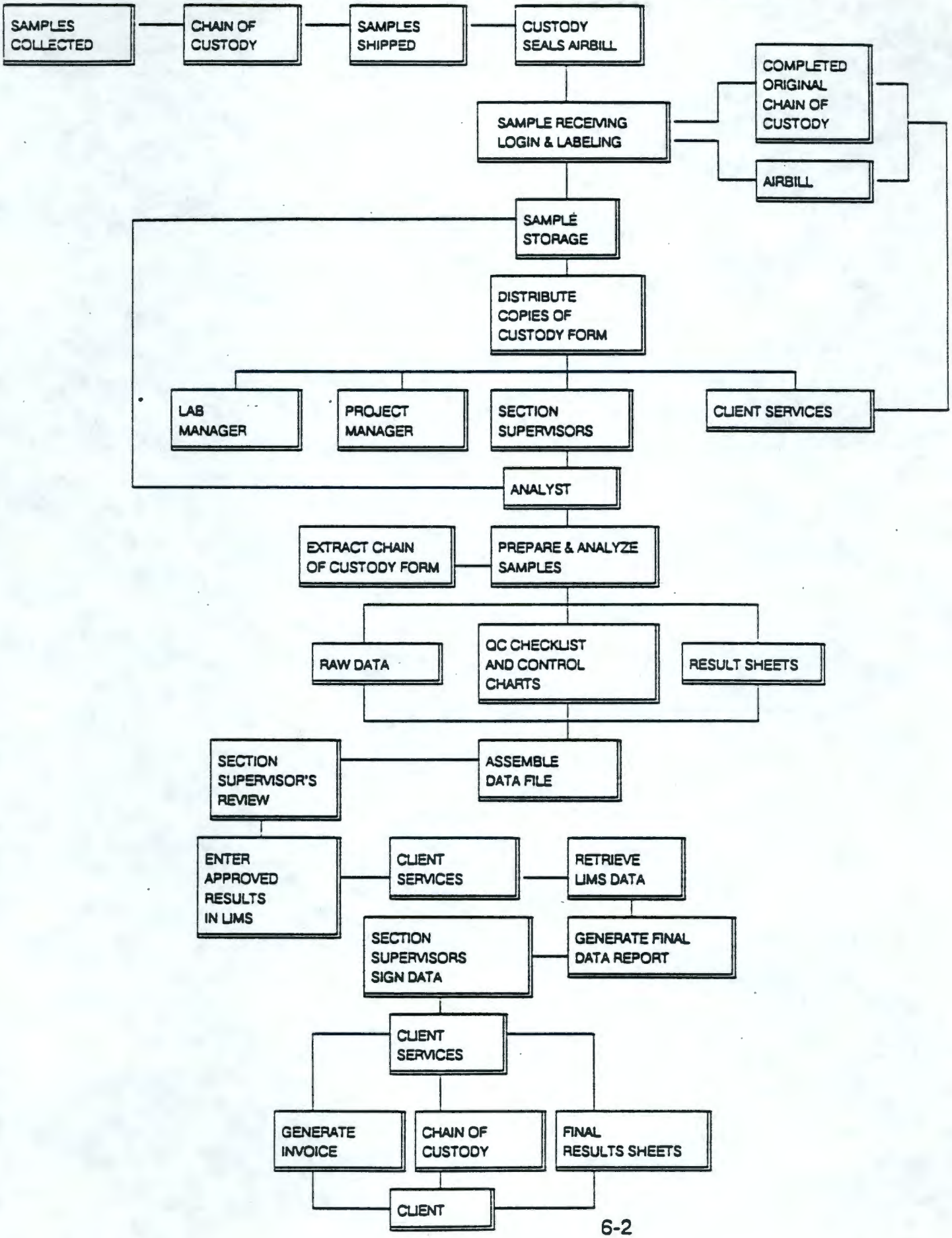


FIGURE 10

ORTEK EXTRACT CHAIN OF CUSTODY FORM

Company Name: _____

Project No./Client _____

Extracted By: _____

Bottle Size/Preservative

No.: 126

Date	Time	Sample I.D./Description	No. of Bottles	Total	ANALYSIS REQUESTED	Remarks	ORTEK Batch No. / Extraction Date / ID Number

INCLUDE EXTRACTION LOG AND ALL OTHER APPROPRIATE PAPERWORK WITH THIS EXTRACT CHAIN OF CUSTODY.

CUSTODY TRANSFERS - INTERNAL TO ORTEK

Relinquished by:	Date:	Time:	Received by:	Date:	Time:
1.					
2.					
3.					
4.					
5.					
6.					
7.					
8.					
9.					

Date Received: _____

Date Due: _____ URUSH

Results To: _____

Attention: _____

COMMENTS/SPECIAL INSTRUCTIONS:

FIGURE 11

ORTEK CHAIN OF CUSTODY/ANALYSIS REQUEST FORM

Company Name: _____

Project No./Client _____

Sampling Location: _____

Sampler _____

Bottle Size/Preservative

No.: 1578

Date	Time	Sample I.D./Description	No. of Bottles	Total	*Sample Type	ANALYSIS REQUESTED	Remarks	ORTEK Batch No. / Lab Use Only / ID Number

COMMENTS/SPECIAL INSTRUCTIONS:

☐ If Pb > 5ppm do TCLP

*Sample Type
S - Soil
SE - Sediment
SO - Solid
SW - Surface Water
DW - Drinking Water
WW - Wastewater
GW - Groundwater
H - Hazardous Liquid
A - Air
O - Oil
X - Other _____

Date Received: _____
Date Due: _____ ☐ RUSH
Quotation #: _____ (approved by lab)
Purchase Order #: _____

Results To: _____

Billing Address: _____

Attention: _____

Phone: _____ FAX _____

To Be Completed by Client

Seal Intact Upon Receipt by Sampling Co.: ☐ Yes ☐ No

Packed By: _____

Sealed For Shipping By: _____ Seal # _____

CUSTODY TRANSFERS

Relinquished by:	Date:	Time:	Received by:	Date:	Time:
1.					
2.					

Received for Laboratory: _____

Shipping Details - To Be Completed By ORTEK

Seal Intact Upon Receipt by Laboratory ☐ Yes ☐ No

Method of Shipment: _____

Contents Temperature _____ °C Refrig. # _____

ORTEK
2495 West Mason Street
Green Bay, WI 54307-2433
(414) 490-2222

FIGURE 12

ORTEK SAMPLE LOG-IN/COMPLETE FORM

DATES _____ TO _____

SAMPLE #'S	CLIENT	DATE RECEIVED	SAMPLE STATUS	LOCATION	BATCH #	COC #	DUE DATE	DATE MAILED	INVOICE #

1. The condition of the shipping containers and sample bottles are inspected and documented upon receipt and the temperature measured and recorded (if appropriate). If samples are indicated as hazardous, this procedure is done in the hood and the sample custodian wears gloves and a respirator in case of sample leakage.

2. The condition of the custody seals (intact/not intact) is recorded on the Chain of Custody form (Figure 11) and the client notified of any damage.

3. The presence or absence of the following documents accompanying the sample shipment are verified and a Sample Receiving Out-of-Control Form (Figure 5) completed to document discrepancies:

   - Airbills or airbill stickers.
   - Custody seals.
   - Custody form.
   - Sample tags/labels.

4. The sample custodian signs and dates all forms (e.g., custody records, packing lists, and airbills) accompanying the samples at the time of sample receipt.

5. The sample custodian contacts the client to resolve discrepancies and problems such as absent documents, conflicting information, unclear analytical requests, broken custody seals, and unsatisfactory sample condition (e.g., improper preservation, leaking sample bottle) as listed on the Sample Receiving Out-of-Control Form.

6. The sample custodian records the resolution of discrepancies and problems on the Sample Receiving Out-of-Control Form and forwards the form with the original Chain-of-Custody Form to the Project Manager. The Project Manager faxes the completed custody form back to the client for verification.

6.1.2 Sample Log-In Procedures

The specific steps of sample log-in into the Laboratory Information Management System (LIMS) are detailed in SOP OP-2. Briefly, this procedure consists of the following steps:

1. The sequential ORTEK laboratory sample number and client sample number are entered into LIMS by the sample custodian. The requested turnaround time, due date, holding time, client name, address, date collected and matrix are also entered for each sequential ORTEK laboratory sample number. The appropriate refrigerator storage location code is also entered.

2. The sample labels containing the information are then printed by LIMS and placed on each sample bottle.

3. Samples are placed in the refrigerator location printed on the label. The sample custodian alerts the appropriate Section Supervisor of samples with short holding times or quick turn requests. Analysts must return all containers to the refrigerator location marked on the label.

4. The chain-of-custody and all supporting documentation received with the samples, or generated by the sample custodian (i.e., Out-of-Control Forms) are copied for distribution to the Laboratory Manager, Section Supervisors, Project Manager and Client Services. All original documentation is sent to Client Services for inclusion in the client project file.

6.1.3 Analytical Documentation

Any entry into laboratory notebooks or forms must include the date, and the signature or initials of the person making the entry. SOP OP-24 details the use of laboratory notebooks. Initials used by each analyst and their signature are recorded on the acceptance page of this QA Manual and the original is kept by the QA Officer. Sample extraction, preparation logs, and standards preparation logs are reviewed at least weekly by the Section Supervisors. Section Supervisors countersign and date the pages reviewed. This signature indicates that they have checked the information for compliance with the applicable SOPs.

Error corrections are done in accordance with SOP OP-23. All laboratory documentation are made in ink, preferably permanent ink. The use of pencil is not allowed. All corrections to documentation are made by crossing out the error with a single line and placing the correction above it. The error line is initialled and dated by the person making the correction. No error correction fluid or "white out" is permissible.

Bound logbooks with sequentially numbered pages are used for recording all laboratory data. All preprinted laboratory forms contain the date(s) they apply to and the name ORTEK. Copies of all applicable pages are included with the data for the project file, or the logbook and page number where the documentation can be found is traceable to the final reported results either by batch number, sample number or both.

6.2 Document Control

The goal of ORTEK's document control system is to be able to supply to any client all documents relating to the analysis of his or her samples. These documents include but are not limited to; chain-of-custody forms, LIMS Sample Change records (Figure 13), sample bottle lots used, sample tags (if removable), airbills, price quotes, bench sheets, logbooks, telephone conversation records (Figure 14), out-of-control forms, QA and/or progress reports, corrective action forms, accompanying QC sample results, calibration records, worksheets with calculations, instrument printouts, and final result sheets. The criteria for an acceptable document control system is that the data and records are secure, retrievable and complete.

The following document control procedures are followed, to assure that these laboratory records are able to be assembled and stored, for efficient retrieval upon request. Details of document storage, tracking and disposition are contained in SOPs OP-11 and OP-25.

- All laboratory records are stored in a secured area so they are not accessible to laboratory visitors or instrument service personnel.

- Locked file cabinets are utilized to store completed records, and a check out card system is used for removal of working project files or archived files.

- All original laboratory forms and data will be included in the project file, when all data from the project is compiled.

- All preprinted laboratory forms and logbook pages must contain the signature of the person conducting the work and the date the work was performed.

- The pages in bound and unbound logbooks must be sequentially numbered. No pages will be removed. Bound logbooks themselves will also be assigned a unique number.

- Proper error correction procedures must be followed. No information shall be obliterated or rendered unreadable. The unused portions of documents shall be crossed out with a "Z" and the person's initials and date entered.

- All documents, notebooks and forms are to be completed in ink.

- Completed logbooks are kept in a secured area and controlled by a checkout system. Completed project files are stored on-site for the last year. Off-site secured storage for older records will be utilized for clients specifying storage for more than three years.

- Unless specified otherwise, project files are kept for 3 years after completion of the project.

- Any changes to LIMS must be documented on a Sample Change Record (Figure 13), and be done by the Sample Custodian or MIS Analyst.

- All telephone conversations with clients must be documented on an ORTEK Telephone Conversation Record (Figure 14), and the original filed with the project file. All faxes, fax cover sheets, and transmission reports must also be filed in the project file.

- General laboratory records including detection limit studies, method development data, SOPs, training and personnel files, health and safety records, audit reports, control charts and performance evaluation results will be kept as long as ORTEK is in existence.

FIGURE 13

BATCH #	SAMPLE ID #
SAMPLE CHANGE RECORD	
Please mark appropriate box for LIMS change. Also initial appropriate box if you have completed the change or check appropriate box for person who needs to make the change.	
PLEASE MAKE CHANGE/DATE:	CHANGE COMPLETED/DATE:
SAMPLE CONTROLLER	SAMPLE CONTROLLER
SAMPLE REC.	SAMPLE REC.
SAMPLE PREPARATION	SAMPLE PREPARATION
WET CHEM	WET CHEM
METALS	METALS
ORGANICS	ORGANICS
CHANGE FROM:	CHANGE TO:
LAB #	LAB #
BATCH #	BATCH #
LOGIN DATE/TIME	LOGIN DATE/TIME
DUE DATE	DUE DATE
COLLECTION DATE/TIME	COLLECTION DATE/TIME
COLLECTION LOCATION	COLLECTION LOCATION
PRES-METHOD	PRES-METHOD
SAMPLE MATRIX	SAMPLE MATRIX
CLIENT/CONTACT	CLIENT/CONTACT
ADDRESS/PHONE	ADDRESS/PHONE
ADD TEST	
DELETE TEST	
REASON FOR CHANGE	
REQUESTED BY:	

COPIES TO:	COC FILE	WORKING FILE	ORG. EXTRACTION	ORGANICS	METALS	WET CHEM	SAMPLE CONTROLLER

FIGURE 14

ORTEK

TELEPHONE CONVERSATION RECORD

WHO:

YOUR NAME: _____ DATE: ___/___/___ TIME: _____

CALLED ☐ RECEIVED CALL ☐ RETURNED CALL ☐

CLIENT NAME: _____ COMPANY: _____

PHONE Nº: ( ) _____ FAX Nº: ( ) _____

WHAT:

PROJECT: _____ BATCH Nº: _____

ANALYSES: _____

SUMMARY OF CONVERSATION: _____

WHEN:

ACTION REQUIRED: _____

ACTION BY: _____ DATE: ___/___/___

COMMENTS: _____

CC: Working file, QC Department, Extractions, Sample Receiving, Metals/Wet Chem, Organics, Lab Director, Project Manager, Client Services, Administration

7.0 CALIBRATION PROCEDURES AND FREQUENCY

The purpose of calibration is to verify that the analytical instrument/equipment can provide data of known and acceptable precision and accuracy. Instrument calibration is performed by the analyst in accordance with method and instrument SOPs. Equipment calibration is periodically performed at prescribed intervals for balances, pipettes and thermometers which are relatively stable in performance. The Section Supervisors are responsible for equipment calibration and for supplying the documentation to the QA officer.

Laboratory analysts record and document all instrumental calibration runs in designated Laboratory Instrument or Run Logbooks. These logbooks identify instrument operating parameters, settings, and performance data associated with each instrumental calibration run.

This section describes the general calibration practices by the analyte and instrument group. Instrument specific calibration procedures are detailed in ORTEK Method SOPs.

7.1 Measurement Equipment and Supplies

ORTEK complies with good laboratory practices in the use of measuring equipment, glassware, laboratory pure water and chemical reagents. All laboratory glassware, balances, thermometers, and subsequent volume, mass, and temperature measurements are directly traceable to primary standards. Chemical reagents are purchased of the quality specified in the SOP and/or reference method. Table 5 lists the equipment calibration frequency and limits. Laboratory volumetric glassware conforms to NIST Class A standards and is used in accordance with SOP OP-33. The SOP for cleaning and storing glassware (OP-8) is posted at wash stations.

Laboratory balances are annually serviced and calibrated by an independent vendor manufacturer's service contract. Additional balance performance evaluations are conducted daily (before first use) by comparison against NIST Class P certified weights using SOP OP-5. Unacceptable performance requires service adjustments. Both balance service and daily calibrations are recorded and documented in designated Laboratory Balance Calibration Logbooks.

Laboratory thermometers are calibrated against a NIST certified thermometer annually using SOP OP-4 and recorded in the designated Laboratory Thermometer Calibration Logbook.

TABLE 5 ***EQUIPMENT PERIODIC CALIBRATION REQUIREMENTS***

INSTRUMENT	CALIBRATION FREQUENCY	ACCEPTANCE LIMITS	CORRECTIVE ACTIONS
Analytical Balances	Daily: Sensitivity (with a Class "P" weight) Annually: Class "S" weights, Check vs P Every three years: Class "S" weights check	See SOP OP-5	Adjust, Sensitivity Service Balance Replace Weights
Ovens	Temperature readout device checked against indicating pyrometer semi-annually	±10%	Service oven
Thermometers	Calibrate annually in constant temperature bath at ice point against NIST reference thermometer	±0.5°C or ±0.2°C	Tag and remove from service, replace
Pipetters	Volume check quarterly	High volume (>100 μL): ≤ 1.0% relative error and RSD Low volume (<100 μL): ≤ 2.0% relative error and RSD	Service or replacement
NIST Thermometer	Recertified every three years		Replace

Laboratory drying ovens, incubators, and refrigerators contain these calibrated thermometers. Temperature readings are recorded daily on the Laboratory Temperature Logsheet. Unacceptable deviation from the acceptable temperature range requires immediate corrective action as described in SOP OP-6.

Laboratory pure water is generated by a commercial on-line water purification system consisting of mixed resin deionizing and carbon filtration cartridges. Cartridges are routinely replaced and serviced by the manufacturer or as indicated by an on-line resistivity indicator recorded in accordance with SOP OP-29.

The Laboratory uses various types and purities of chemical reagents, solvents, and gases depending upon their intended use. Laboratory stock and working standards are derived from commercially available primary standards and solvents whenever possible. SOPs OP-28, 34 and 35 detail reagent, compressed gases, solvent and analytical standard practices.

7.2 Instrument Calibration Procedures

All instruments subject to calibration are uniquely numbered/identified so that calibration records can be traced to a specific instrument. EPA and manufacturer's specific calibration protocols are followed. Source of analytical standards used, standard preparation and documentation of the instrument calibration must comply with the requirements in Section 2.0 and follow the Method SOPs. Minimum operations necessary to satisfy most EPA criteria for calibration are contained in Table 6. Specific procedures are briefly described below by instrument:

7.2.1 GC/MS VOCs and BNAs

- Every 12 hours the instrument is tuned and must meet EPA established abundance criteria for DFTPP or BFB to assure that instrument response meets EPA specifications.

- Generation of five point calibration curves, as the method requires, for all method compounds at least quarterly, or more frequently if needed. Recalibration is done when continuing calibration criteria is not met as specified in the EPA method.

- Verification of volatile system cleanliness by the analysis of at least one daily reagent blank.

TABLE 6 ***INSTRUMENT CALIBRATION REQUIREMENTS***

INSTRUMENT	CALIBRATION STANDARDS USED	CALIBRATION ACCEPTANCE LIMITS	CORRECTIVE ACTION	STANDARD SOURCE
FTIR	Wavelength calibration and curve, minimum three levels and blank	± 5 cm true value	1) make new standards 2) service	Variable
AA (GF,CV)	Every analysis, minimum of 3 concentration standards and one blank. Verification (ICV): Second source standard immediately following calibration near the midpoint but not at the continuing (CCV) concentration. Continuing (CCV) midpoint concentration standard, every ten samples.	Calibration: $r^2 \geq 0.995$ ICV (AA): ± 10% true value ICV (CV): ± 20% true value CCV (AA): ± 10% true value CCV (CV): ± 20% true value AA Blanks (ICB & CCB) 2X or 3X standard deviation of mean blank value (SW846, 200.7). < CRDL (CLP) CV Blanks (ICB and CCB) <0.2 ug/L	AA: Clean furnace, replace tube, platform, and contact rings as needed. Modify temperature programs. Recalibrate, make new standards. CV: Check fluid and vapor flows, Check optical cell, Check drying tube, Reanalyze standard(s) Recalibrate, make new standards	Variable, typically Baker and Fisher
ICAP	Every analysis, typically one concentration standard and one blank. Exception: Cu on Plasma II (3 point curve) Verification (ICV): Second source standard immediately following calibration (CLP) or high standard analysis (SW846) at or near the midpoint of the curve. Exception: Ag at 1000 ug/L. Continuing (CCV): Midpoint Concentration standard, every 10 samples.	High standard analysis (calibration standards) ± 5% true value (SW846) ICV: ± 10% true value CCV: ± 10% true value Blanks (ICB and CCB) 2X or 3X standard deviation of mean blank value (SW846, 200.7). < CRDL (CLP)	Check ICAP for proper operation, recalibrate, make new standards.	Spex or Inorganic Ventures
LACHAT UV-Visible Spectrophotometer	Initial: 3 levels and blank Daily: Check standard	$r^2 > 0.995$ ± 10% true value	1) make new standards 2) recalibrate	Variable
GC	Initial: 5 concentrations and blank Continuing: Varies	RF < 20% RSD or $r^2 > 0.995$, blank < detection limit (except acetone and methylene chloride)	1) make new standards 2) recalibrate	Supelco Ultra
GC/MS	Tuning (DFTPP or BFB) Initial: 5 concentrations and blank Continuing: midrange conc.	EPA criteria RRF, RSD criteria for SPCC, CCC compounds for SPCC, CCC compounds	1) Retune, service 2) Recalibrate 3) Make new standards, recalibrate	Supelco Ultra
pH/Conductivity Meter	Daily: pH buffers 3 standard concentrations, conductivity standard	± 0.05 pH unit ± 10% true conductivity	Clean or replace electrode, service	Variable

- Maintain sample response within linear range of instrument by dilution.

- Addition of internal standards to each sample that must meet area count criteria of -50% to +100%.

7.2.2 GC VOCs

- Generation of five point calibration curves for all analyzed compounds at least quarterly, or prior to any sample analysis as stated in the analytical method. Recalibration is done when continuing calibration criteria is not met and the compound of interest is present in the sample.

- The initial calibration curve must have a Relative Standard Deviation (RSD) of ≤20% for Method 8010/8020, and ≤10% for Method 601/602 with continuing calibrations of ≤15%, and ≤10% respectively. (RSDs are calculated based on guidance found in SW846, Method 8000, Section 7.4.4.2.). Alternately, the linear regression performed must have a correlation coefficient greater than 0.995.

- Maintain sample response with linear range of instrument by dilution.

- Monitor consistency of instrument response through the analysis of a standard after at least every 20 sample analyses.

- Demonstrate system cleanliness through the analysis of at least one daily reagent blank.

7.2.3 Pesticides and PCBs - GC/EC

- The initial calibration curve must have an RSD of ≤20% with a continuing calibration of ≤15%. (RSDs are calculated based on guidance found in SW846, Method 8000, Section 7.4.4.2.). Alternately, the linear regression performed must have a correlation coefficient greater than 0.995.

- Generation of five point calibration curves for all analyzed compounds monthly, prior to any sample analysis, or as stated in the analytical method. Recalibration is done when continuing calibration is not met and the compound of interest is present in the sample.

- Maintain sample response within linear range of instrument by dilution.

7.2.4 Metals - ICAP

- Analysis of at least one standard and a blank.
- Verification of system cleanliness and baseline maintenance through the analysis of a continuing calibration blank (CCB) after every ten samples. Detected metals must be less than Contract Required Detection Limit (CRDL).
- Verification of instrument stability through the analysis of a continuing calibration verification (CCV) standard after every ten samples. Recovery must be 90-110%.
- Determination of instrument performance by the analysis of an interference check sample (ICSAB) at the beginning and end of each run, or twice in an 8 hour shift with limit of 80% - 120%.
- Maintain sample concentration within the linear range of the instrument by dilution.

7.2.5 AA Furnace and Cold Vapor Mercury Analyzer

- Initial and continuing calibration must be recovered at 80-120% for the Cold Vapor Mercury Analyzer and 90-110% for AA.
- Verification of system cleanliness by a CCB analysis after every ten samples.
- Construction of at least a three point calibration curve for each element prior to the analysis of any sample set.
- Maintain sample response within linear range of instrument by dilution.

7.2.6 pH and Ion-Selective Electrodes

- Construction of a three point (2 point for pH) calibration curve prior to the analysis of any sample.
- Maintain sample response within linear range of instrument by dilution.

- Verification of cleanliness of the analytical system through the analysis of a reagent blank (where applicable).

- Verification of instrument consistency through the analysis of a standard/buffer after the analysis of every ten samples (where applicable).

7.2.7 Lachat Auto Analyzer

- Construction of a three point calibration curve prior to the analysis of any sample. The initial calibration curve must have an RSD of ≤20% and r^2 of ≥0.995.

- Monitor for the introduction of any interferents through the analysis of a reagent blank, prior to any sample analysis.

- Maintain sample response with the linear range by dilution.

- Verification of the consistency of instrument response through the analysis of a lab control standard (LCS) after every twenty sample analyses for DQO level D. DQO levels C and E require a LCS after every 10 samples.

7.3 Calibration Records

Records for periodically calibrated equipment (balances, thermometers, pipettes) must be kept by the Section Supervisor and include as appropriate:

- Identification number of equipment and type of equipment, or assigned unique equipment number.

- Calibration frequency and acceptable tolerances.

- Identification of calibration procedure used.

- Date calibration was performed.

- Identity of ORTEK personnel and/or external agencies performing calibration.

- Reference standards used for calibration (to be stored separately from any samples or reagents).

- Calibration data.

- Certificates or statements of calibration provided by manufacturers and independent service personnel traceable to NIST.

- Information regarding calibration acceptance or failure and any repair of failed equipment.

- Date of when next scheduled calibration is due.

Section Supervisors are responsible for ensuring recalibration is completed on time and documented.

For instruments and equipment that are calibrated when used (as often as daily), calibration consists of determining instrumental response against known standards or the preparation of a standard response curve. Records of these calibrations are maintained by ORTEK in two ways:

- The calibration data are kept with the affected analytical sample data (government projects, GC/MS).

- A log book or raw data folder contains all calibration data (Lachat, ICAP, AA, GC).

The first method provides response factor information directly with analytical data, for easy data validation by the Section Supervisor and the raw data package is complete. However, when samples from several projects are processed together, the location of the calibration data must be referenced in each affected project file.

The second method provides an ongoing record of the calibration undertaken for a specific instrument, and enables easier detection of trends indicating instrument problems. However, to verify the analytical data, the log must be used in conjunction with the raw data.

8.0 ANALYTICAL PROCEDURES

Analytical procedures should be selected by the client in consultation with regulatory agencies and ORTEK to meet the DQOs and detection limits necessary. Whenever possible ORTEK uses EPA approved methods and incorporates them into an ORTEK specific method SOP. Copies of these SOPs are kept in each section by the Section Supervisor. All method SOPs are written in the format listed in Figure 15.

8.1 Detection Limits

ORTEK defines the detection limit of a method as the quantity of analyte which results from the lowest differential between a signal caused by the analyte and that of random noise. Practical Quantitation Limits (PQLs) are defined as 5-10 times this signal. Metals instrument detection limits (IDL) for DQO Level D analyses by CLP protocols are determined quarterly by spiking distilled water at a concentration 3-5 times the anticipated IDL. This solution is analyzed 7 times on three nonconsecutive days and the standard deviation calculated. The IDL is determined as 3 times this standard deviation. PQLs for organic analytes are determined statistically at least annually for methods listed in Table 7 using the criteria contained in Federal Register Vol.49, No. 209, October 26, 1984, Appendix B to Part 136. For SW846 metals analyses, values above the method detection limit (MDL) will be reported. MDLS are determined from 7 low level digested distilled water spikes.

Organic analytes present in concentrations below PQLs WILL NOT be reported as present in the sample when using this analytical technique. A detection limit quantity is reported as a "less than" value(<) or "U" value. This less than or "U" value does not indicate that an analyte is not present in a sample but only that its presence is at levels below PQL. For results produced by US EPA CLP organic methods, values which are below required detection limits, but can still be quantified, are reported as estimated concentrations using a "J" qualifier. For results produced by US EPA CLP inorganic methods, values above the IDL but below the contract required detection limit (CRDL) are reported with a "B" qualifier. Values below the IDL are qualified with a "U" code.

Current IDLs and MDLs are available upon request. Actual limits achievable in "real" world samples vary based on dilution requirements, background interferences, sample concentration factors and cleanup techniques. Data from IDL and MDL studies are reviewed by the QA Officer and are kept in each section by the Section Supervisor.

TABLE 7 ***ORTEK ANALYTICAL METHOD CAPABILITIES***

ANALYTE	REFERENCE METHODS			ANALYTE	REFERENCE METHODS			
WET CHEMISTRY	EPA 600/4-79-020	SW 846	STD METHODS 18TH EDITION	METALS				
Alkalinity	310.1		2320	Aluminum	CLP			6010
Ammonia	350.2		4500-NH_3	Antimony	CLP	204.2	7041	6010
BOD	405.1		5210	Arsenic	CLP	206.2	7060	6010
COD	410.4		5220	Barium	CLP			6010
Chloride	325.2 Mod.		4500-Cl	Beryllium	CLP			6010
	325.1							
Conductivity	120.1		2510	Cadmium	CLP	213.2	7131	6010
Fecal Coliform	909C		9222D	Calcium	CLP			6010
Total Coliform	Colilert		9221B	Chromium, total	CLP	218.2	7191	6010
Cyanide	335.2		4500-CN^-	Chromium, hex			7196	
		9010						
Flashpoint	340.2	1010		Cobalt	CLP			6010
Fluoride	340.2		4500-F^-	Copper	CLP	220.2		6010
Hardness	130.2		2340	Iron	CLP			6010
Total Kjeldahl Nitrogen	351.2 (351.3)		4500-N^{org}	Magnesium	CLP			6010
Nitrate -N	353.2/353.3Mod		4500-NO_3^-	Manganese	CLP			6010
Nitrite -N	353.2/353.3Mod		4500-NO_2^-	Mercury	CLP	245.1	7470	7471*
Nitrate + Nitrite -N	353.2/353.3Mod			Nickel	CLP			6010
Ortho-Phosphate	365.3							
pH	150.1	9045	4500-H^+	Potassium	CLP			6010
Total Phenol	420.2	9065	5530	Selenium	CLP	270.2	7740	6010
Oil & Grease	413.1	9071	5520	Silver	CLP	272.2	7761*	6010
Total Phosphorus	365.4		4500-P	Sodium	CLP			6010
TDS	160.1		2540C	Thallium	CLP	279.2	7841	6010
TOC	415.1	9060A	5310	Lead	CLP	239.2	7421	6010
TOX		9020A	5320	Molydenum				6010
TS	160.3		2540B	Vanadium	CLP			6010
TSS	160.2		2540D	Zinc	CLP			6010
TVS	160.4		2540E	Boron				6010
				Lithium				6010
Sulfate	375.2		4500-SO_4^{2-}	TCLP			1311	
Sulfate			4500-S^{2-}	SPLP Extraction			1312	3005

* Modified

TABLE 7 ORTEK ANALYTICAL METHOD CAPABILITIES

ANALYTE	REFERENCE METHODS			ANALYTE	REFERENCE METHODS			
WET CHEMISTRY	EPA 600/4-79-020	SW 846	STD METHODS 18TH EDITION	METALS				
Sulfide	376.1	9030	SW 846	Metals Digestion	3005	3050	3020	3010
TCLP Extraction			1311	Metals Digestion (continued)	CLP	MCAWW		
SPLP Extraction			1312					

TABLE 7 ORTEK ANALYTICAL METHOD CAPABILITIES

ANALYTE(S)	REFERENCE METHODS					
ORGANIC CHEMISTRY						
Volatile Organics	CLP	8240	624	5030	8021	8260
Semivolatile Organics	CLP	8270	625			
Pesticides/PCB's	CLP	8080	608			
Halogenated Volatiles		8010	601			
Aromatic Volatiles		8020	602			
Chlorinated Herbicides		8150				
Polynuclear Aromatic Hydrocarbons		8310	610			
TRPH		418.1	9073			
TPH-Gasoine (purge & trap)		California DHS	8015			
TPH-Diesel (extractable)		California DHS	8015			
ZHE Extraction		1311				
Aqueous Sample Extraction		3510	3520			
Solid Sample Extraction		3550				
Sample Clean-up		3610	3620	3630	3640	3660

REFERENCES TO TABLE 7

References

909 C: Standard Methods for the Examination of Water and Waste water, 16th and 18th edition.

100-300 series: Methods for Chemical Analysis of Water & Wastes, EPA-600/4-79-020

1000-8000 series: Test Methods for Evaluating Solid Waste, 3rd edition. SW-846 Update II

CLP: US EPA Contract Laboratory Program Statement of Work Inorganics ILM, 3/90, US EPA Contract Laboratory Program Statement of Work-Organics OLM, 1/91.

* Modified

8.2 Variance From Analytical Methods/ORTEK SOPs

Analyses will be performed in accordance with the ORTEK SOPs derived from the methods cited in Table 7 unless specific project/client requirements dictate a modification. The modification must be documented in accordance with Figure 15 in an ORTEK Laboratory Operations SOP.

If an existing SOP needs modification, the analyst will prepare a memo to the QA Officer stating what changes are prepared and the justification for change. The Section Supervisor and QA Officer must review and approve these changes prior to implementation. A revised SOP will then be distributed.

FIGURE 15
FORMAT FOR ORTEK METHOD STANDARD OPERATING PROCEDURE (SOP)

0. Approval and Distribution Cover Sheet

1. Scope and Application
   - 1.1 Analytes
   - 1.2 Detection limit (instrument and method)
   - 1.3 Applicable matrices
   - 1.4 Working linear range
   - 1.5 Approximate analytical time (e.g., five minutes, two days) and throughput (# samples/shift)

2. Summary of Method
   - 2.1 Generic description of method and chemistry

3. Comments
   - 3.1 Interferences/corrective action
   - 3.2 Helpful hints

4. Safety Issues

5. Sample Collection, Preservation, Containers, and Holding Times
   - 5.1 Minimum sample volume necessary for analysis
   - 5.2 Storage location and disposal concerns

6. Apparatus
   - 6.1 Instrument and operating parameters, instrument logbook format

7. Reagents and Standards
   - 7.1 Shelf life, source, disposal.

8. Procedure (detailed step-by-step)
   - 8.1 Sample preparation
   - 8.2 Calibration
   - 8.3 Analysis
   - 8.4 Documentation logbook format, any bench forms used

9. QA/QC Requirements
   - 9.1 QC samples what kind, when, how prepared.
   - 9.2 Acceptance criteria
   - 9.3 Corrective action required
   - 9.4 QC checklist, control charts

10. Calculations
    - 10.1 Examples, forms used

11. Reporting
    - 11.1 Reporting units
    - 11.2 Reporting limits
    - 11.3 Significant figures and reporting values below detection limits.

11.4 LIMS data entry
11.5 Data package contents/list

12. References
12.1 Method source
12.2 Deviations from reference method and rationale

13. Method Detection Limit Data and Protocol

14. Documentation

14.1 Examples of bench log books used
14.2 ORTEK forms used

9.0 DATA REDUCTION, VALIDATION & REPORTING

Data reduction is the process of compiling all pertinent results, calibration records, and QC data, and producing a report that is accurate and meets the client's requirements. Data validation is the process of reviewing data generated against a pre-established set of criteria to determine its validity. Data reporting is the process of producing the results in a format suitable to the client and ensuring that it accurately represents the results of the reduction and validation processes. The inter-relationship of these activities is presented in Figure 16.

9.1 Data Reduction

The ORTEK laboratory uses the Telecation Smartlab® computerized Laboratory Information Management System (LIMS) to accomplish several data acquisition activities: laboratory sample log-in, sample result archival, sample status and tracking, and final report generation. This system is summarized below:

- Smartlab® assigns individual laboratory identification numbers and prints labels for each sample. Sample data input includes client sample ID, analytical test methods required, matrix, turnaround time, collection date and holding time requirements.

- Smartlab® assimilates the sample data and generates backlog reports for each section of the laboratory for scheduling and prioritizing analyses. These reports identify the analytical parameters, the method, the turnaround time requested and critical holding time considerations.

- Analysts enter their completed sample analytical results into Smartlab®. The section manager reviews the run and if data are acceptable, approves the run. Approval of the run removes the sample analyses from the backlog as completed and a final report is generated.

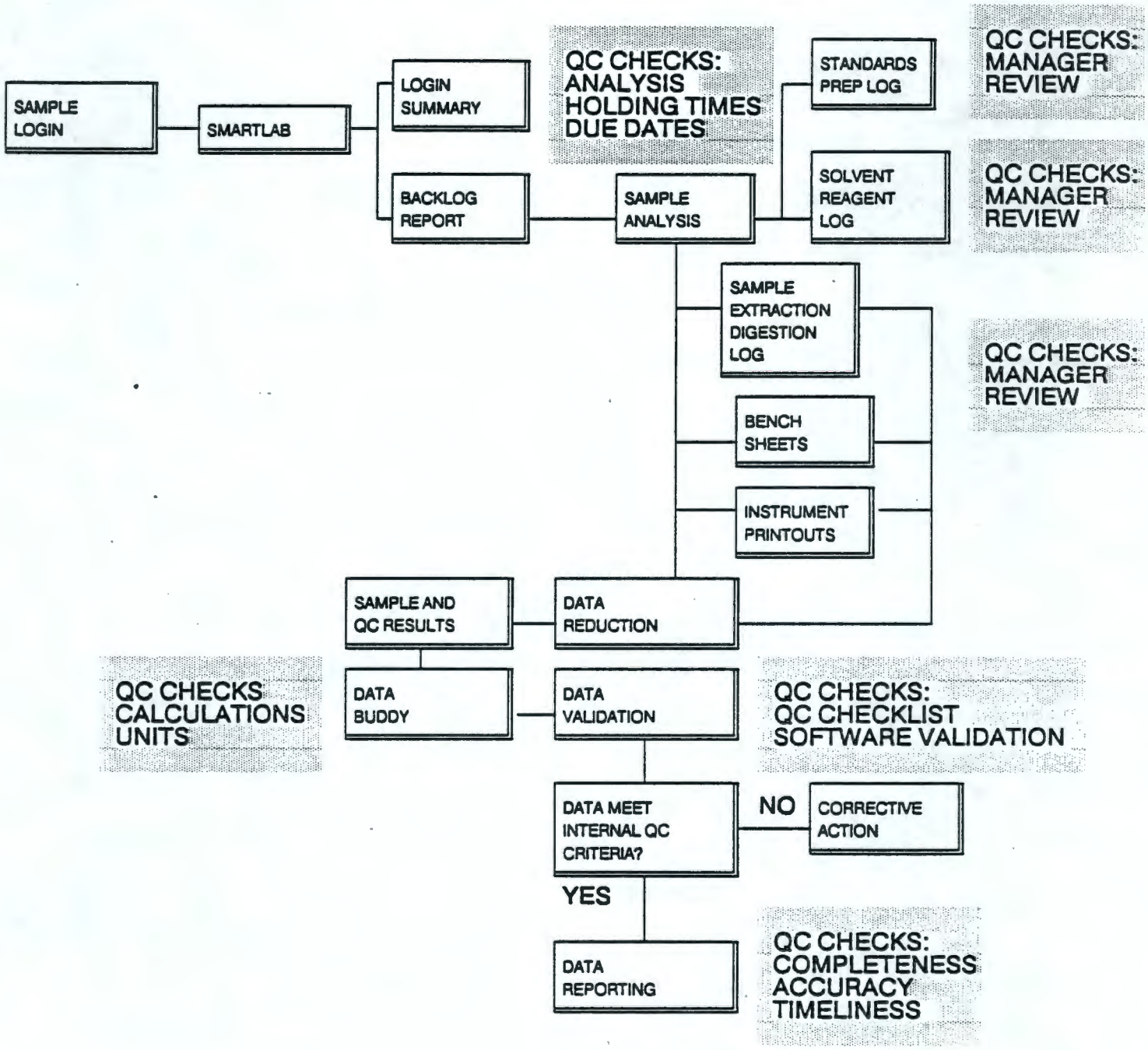
- CLP data package forms are generated through independent software systems. When the entire CLP Sample Delivery Group (SDG) is complete it is removed from the LIMS backlog.

9.1.1 Raw Data Generation

All analytical data are generated either by computer data reduction systems (GC, GC/MS,ICAP, AA) or by manual calculation (Wet Chemistry). Manually calculated data are entered into spiral bound logbooks, and into Smartlab®.

FIGURE 16

LABORATORY DATA PROCESSING FLOW CHART



All extractions, sample preparation, standards preparation and instrument runs are also entered into bound logbooks. Copies of these pages related to each project are sent with the data for review for QC level D. Each set of analytical data is therefore traceable to specific lots of standards, digestion or extraction dates and instrument runs. Data are generated by the analyst in one of the following ways:

- By manual computation of results directly on a data sheet or on calculation pages attached to the data sheets.
- By entering raw data into the computer for processing.
- By direct acquisition and processing of raw data by the computer.

If data are manually generated by the analyst, all steps in the computation will be specified, including equations used and the source of input parameters such as response factors, dilution factors, and sample weights/volumes. The analyst will sign and date in ink each page of calculations.

If data are directly acquired by the computer from the instrument and a printout is supplied, the analyst will verify that the following can be traced to the raw data: calibration results, response factors, QC sample results and numerical values used for detection limits. Units and correct sample numbers must be checked.

Each section of the laboratory uses a checklist format to verify that all applicable samples and QC were analyzed. These checklists are presented in Figures 17 - 20. Each SOP is to be consulted for the applicable QC limits, the calculations/equations to be used, the appropriate number of significant figures and the correct reporting units.

9.1.2 Raw Data Verification

A "data buddy" system allows for the review of calculations done by each analyst by an independent analyst. This check is to include the math, checking the dilution factors against the final result and verifying that proper units are reported. SOP OP-17 details the procedure to be used.

FIGURE 17

ORTEK ***WET CHEM STANDARD QC CHECK LIST***			
ORTEK BATCH #:	CLIENT:		
# OF SAMPLES:	WATER	SOIL	OTHER
LAB #'S			
MET HOLD TIME: ☐ YES ☐ NO			
PARAMETER:			
DATE ANALYZED:			
QC SUMMARY:			
A) CHAIN OF CUSTODY CHECKED ☐			
B) INITIAL CALIBRATION DATE [/ /93]			
C) DAILY CALIBRATION [/ /93] ☐ PASSED ☐ NOT REQUIRED			
D) LABORATORY BLANK ☐ < MDL BLANK CORRECTION ☐			
E) SPIKE RECOVERY _____%			☐ PASSED
F) DUPLICATE (% ERROR) _____%			☐ PASSED
CASE NARRATIVE			
ANALYST:		DATE:	
APPROVED BY SUPERVISOR:		DATE:	

FIGURE 18

ORTEK METALS QC CHECK LIST

ORTEK BLANK #	METHOD	# SAMPLES	WATER	SOIL	OTHER
MET HOLD TIME ☐ YES ☐ NO	DATE ANALYZED:			INITIALS	

QC SUMMARY:

	Al	Sb	As	Ba	Be	Cd	Ca	Cr	Co	Cu	Fe	Pb	Mg	Mn	Hg	Ni	K	Se	Ag	Na	Tl	V	Zn
STD PREPARED																							
ICV PASSED																							
CAL PASSED																							
ICB PASSED																							
P B PASSED																							
SPIKE PASSED																							
DUP PASSED																							
LCS PASSED																							
ICSA																							
ICSAB																							
BS/BSD																							
		SAME BATCH					BATCH #						NOT REQUIRED										
MAN.S.PASSED																							
		SAME BATCH					BATCH #						NOT REQUIRED										

CASE NARRATIVE:

ANALYST SIGNATURE: DATE:

FIGURE 19

GC QC CHECK LIST

ORTEK BATCH #:	CLIENT:		
# SAMPLES:	WATER	SOIL	OTHERS
DATE RECEIVED:	MET HOLDING TIME: ☐ YES ☐ NO		
METHOD:	DATE ANALYZED:		

QC SUMMARY:

A) CHAIN OF CUSTODY CHECKED ☐

B) INITIAL CALIBRATION DATE [/ /93]

C) DAILY CALIBRATION [/ /93] ☐ PASSED

D) LABORATORY BLANK ☐ < MDL

E) SURROGATE RECOVERY ☐ PASSED

F) SPIKE RECOVERY ☐ PASSED

G) BS/BSD ANALYZED FROM

☐ SAME BATCH ☐ BATCH _____ ☐ NOT REQUIRED

H) MS/MSD ANALYZED FROM

☐ SAME BATCH ☐ BATCH _____ ☐ NOT REQUIRED

I) LIMS CHECKED AGAINST COMPUTER PRINT OUT ☐

CASE NARRATIVE:

ANALYST:	DATE:
APPROVED BY:	DATE:

FIGURE 20

GC-MS QC CHECK LIST						
BATCH #:			CLIENT:		DATE RECEIVED:	
METHOD:			INSTRUMENT ID:		LIMS RUN #:	
# SAMPLES:			WATER	SOIL	TCLP	OTHER
YES	NO	N/R				
☐	☐		CHAIN-OF-CUSTODY CHECKED			
			HOLD TIMES MET			
☐	☐		EXTRACTION			
☐	☐		ANALYSIS			
			INITIAL CALIBRATION			
☐	☐		1st I	/ /93	☐ SAME BATCH	☐ OTHER
☐	☐		2nd IC	/ /93	☐ SAME BATCH	☐ OTHER
			DAILY CALIBRATIONS (Including BFB or DFTPP)			
☐	☐	☐	1st CAL	/ /93	☐ SAME BATCH	☐ OTHER
☐	☐	☐	2nd CAL	/ /93	☐ SAME BATCH	☐ OTHER
☐	☐	☐	3rd CAL	/ /93	☐ SAME BATCH	☐ OTHER
☐	☐		4th CAL	/ /93	☐ SAME BATCH	☐ OTHER
			DAILY BLANKS PASS			
☐	☐		1st BLANK	/ /93	☐ SAME BATCH	☐ OTHER
☐	☐		2nd BLANK	/ /93	☐ SAME BATCH	☐ OTHER
☐	☐		3rd BLANK	/ /93	☐ SAME BATCH	☐ OTHER
☐	☐		4th BLANK	/ /93	☐ SAME BATCH	☐ OTHER
☐	☐		INTERNAL STANDARD AREAS PASS (If no explain below)			
☐	☐	☐	BS/BSD PASS		☐ SAME BATCH	☐ OTHER
☐	☐	☐	MS/MSD PASS		☐ SAME BATCH	☐ OTHER
☐	☐	☐	CONTROL CHARTS PLOTTED			
CASE NARRATIVE ITEMS:						
ANALYST:						DATE:
DATA BUDDY:						DATE:
APPROVED BY:						DATE:

9.2 Data Validation

ORTEK data are validated during collection and after generation by a series of steps described in SOP OP-27 that minimizes the possibility of reporting results that do not meet client DQOs. These steps include assuring all software used is accurate in accordance with SOP OP-15, the instrument is properly calibrated and the method used is not biased.

9.2.1 Software

As computer software is used to acquire, process and report data, periodic demonstration that it is operating correctly is required. This consists of comparing its performance against known results. SOP OP-15 details how specifically this will be accomplished, and a summary is described below:

- If the program has been prepared external to ORTEK and is accepted by regulatory agencies as an "industry standard," independent verification is not required. Industry standard programs are defined as those which are widely used throughout the environmental lab community (i.e. Formaster, Smartlab®) and are brought into ORTEK and used without modification.

- For programs that are developed within ORTEK and externally prepared programs that are modified by ORTEK, complete checking of performance is required. Checking is dependent upon the function of the software and could include:

  - For software that only performs numerical manipulation, sample sets of numbers for which the results are known must be processed and compared. In this case, known results are usually generated by performing hand calculations using the same equations and procedures as the software. Verification of the software must test all options of the program. Problems must test both the theory, or basis for computation and the ability of the software to store and manage files.

  - Software that performs as part of instrument operation should be verified by processing reference materials through the instrument

system. Processed instrument response should be compared against the standards used.

Software will be verified whenever modification is made. The test problems used to provide initial verification will be reprocessed and the results compared to demonstrate that performance of the software is unchanged. If software performance has changed, the effect of the change upon intended function and since last verification will be assessed. Effect must be determined on a case-by-case basis for the scope and impact of incorrectly reported results. If necessary, the data will be reprocessed and recipients of affected data reports notified.

Software verification shall be documented by the individual performing the work, signing and dating in ink the computer output, and supporting calculations. If test problems are used, the input will be marked with check marks to indicate correct usage and the output checked to indicate acceptable comparison. If reference materials are used as the basis for verifying instrumental related software, the "true" values or certificates will be included with the output.

9.2.2 Instrument Calibration

All instruments used in the generation of analytical results must be properly calibrated in accordance with the ORTEK Method SOP. No sample data can be generated on an instrument until the requirements for initial calibration are fulfilled (i.e. correlation coefficient RRF, %RSD). Calibration results must be reviewed and approved by the Section Supervisor as indicated by his signature on the QC checklist.

9.2.3 Analytical Method Assessment

The QC checklist items used to validate data that indicate how valid the methods performed are: lab method and holding blank results, blank spike (BS)/blank spike duplicate (BSD) recoveries, and laboratory control standard recoveries. The results of these QC checks are not dependent on sample matrix interferences and must be within acceptance limits listed in Section 10 and SOP OP-26 in order for the analysis to be considered valid. Field blank and trip blank results, surrogate spike recoveries, matrix spike/matrix spike duplicate

recoveries and field or lab duplicate RPD are all sample collection or matrix dependent and do not necessarily indicate if the data was generated by a valid method on a properly calibrated instrument. Results out of acceptance limits are noted on the data result sheet or case narrative to alert the client of a possible sample collection or matrix problem.

Figure 21 presents the order of data validation and the actions required. All QC data are then reviewed by the Section Supervisor to ensure that the proper number, type of QC samples and appropriate limits were used. The Section Supervisor indicates his review by signature of the QC checklist. In addition, 10% of the project files generated each month are reviewed by the QA Officer to assess if the data validation process is being followed.

9.3 Data Reporting

- The format and content of a data report are dependent upon project needs, such as whether or not a CLP data package, case narrative, or QA Summary is required, client or contract requirements, and government reporting formats. ORTEK is flexible and does not specify a report format, but all reports must meet the requirements in SOP OP-10 which are summarized below:

  - Data are presented in tables whenever possible.

  - All result sheets and/or a cover letter/case narrative are signed by the Laboratory Manager. This signature indicates that the data has been reviewed for:

    - Completeness - results for all parameters requested are present; detection limits, units, dates, and sample descriptions are complete and correct.

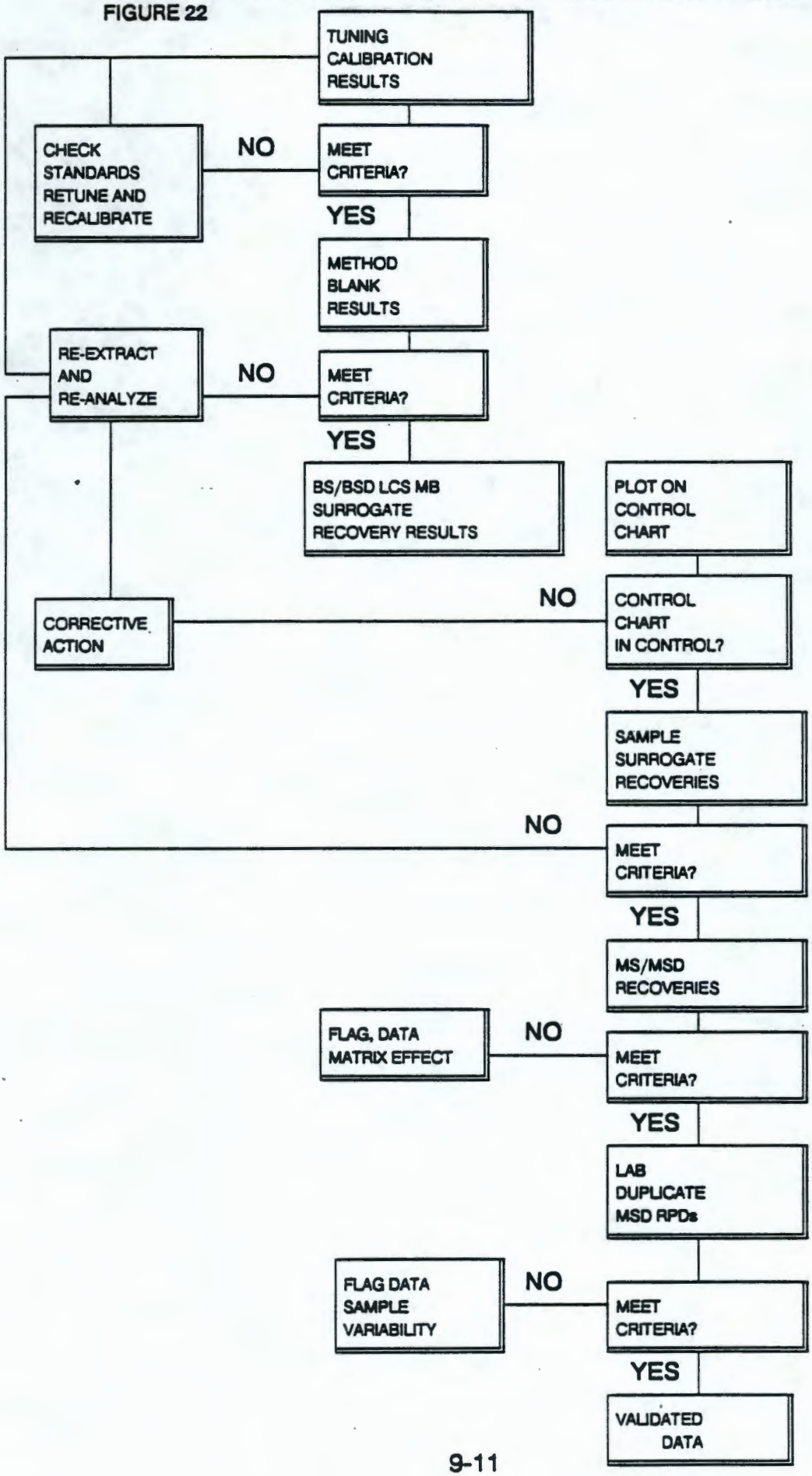
    - Consistency - all parameters are reviewed for internal consistency (hexavalent chromium $\leq$ total chromium, TKN $\geq$ NH_3-N, TS $\geq$ TSS, total metals $\geq$ dissolved metals).

    - Sample identification number used by ORTEK and the sample identification provided to the laboratory by the client.

    - Chemical parameters analyzed, reported values, units of measurement, analytical method used dates prepared/extracted and analyzed.

DATA VALIDATION PROCESS FLOW

FIGURE 22



- Detection limit of the analytical procedure if less than the detection limit is reported.
- Data for a chemical parameter reported with consistent significant figures.
- Results of Quality Control sample analyses if requested.
- Explanation of any out-of-control events that affect data quality (holding times, preservatives, surrogates).
- Explanation of any data qualifiers used.

Any results faxed to clients or verbally transmitted are considered preliminary until a formal hard copy is received. Fax transmittal sheets and telephone conversation records, regarding transmittal of results, must be kept in the project file.

10.0 INTERNAL QC CHECKS AND FREQUENCY

This section describes the types of QC samples which are prepared by ORTEK and routinely analyzed with client samples to demonstrate that ORTEK is operating within known precision and accuracy, representativeness, completeness and comparability limits. Table 8 summarizes these QC samples and indicates their frequencies, and applicabilities. Control limits are updated on an ongoing basis and are available upon request. When internal control limits are exceeded during analysis, an Out-of-Control Form (Figure 22) must be completed. Client supplied QC checks (field blanks, rinsate blanks, field duplicates, splits, collocated samples) are not included.

10.1 Precision QC Samples

Precision is defined as the reproducibility of analytical measurements. It is a quantitative measure of the variability of a group of measurements compared to their average value, and is dependent on sampling and analytical error.

10.1.1 Lab Duplicate (LD)

A sample is split by ORTEK and both aliquots are analyzed separately to assess method precision. The relative percent difference (RPD) is calculated. Metals and wet chemistry sections use this QC sample.

Frequency: One per 10 samples or daily, whichever is more frequent.

Limits: Less than 20% RPD water, less than 20% RPD soil. Analyte, matrix and concentration dependent.

10.1.2 Blank Spike Duplicate (BSD)

This sample is prepared as in 10.2.6. The RPD is calculated between the Blank Spike (BS)/BSD pair. If the variability between the BS/BSD exceeds limits, the analytical system is out of control and too unstable to provide valid sample data. Any associated "real" samples must be reextracted and reanalyzed.

Frequency: One per 20 samples or daily whichever is more frequent.

Limits: Less than 20% RPD. Analyte and matrix dependent.

Table 8	INTERNAL QC CHECKS & FREQUENCY										
ANALYTE	BLANKS			DUPLICATES & SPIKES						CONTROL STD	
	TB	PS	MB*	LD	BS/BSD	MS/MSD	MS	SS	AS	IS	LCS
ALKALINITY	--	-	1/10	1/10	1/20	--	--	--	--	--	1/10
AMMONIA	--	P	1/10	1/10	1/20	--	1/10	--	--	--	1/10
BOD	--	--	DAILY	1/10	1/20	--	--	--	--	--	1/10
COD	--	P	1/10	1/10	1/20	--	1/10	--	--	--	1/10
CHLORIDE	--	--	1/10	1/10	1/20	--	1/10	--	--	--	1/10
CONDUCTIVITY	--	--	1/10	1/10	--	--	--	--	--	--	1/10
COLIFORM	--	--	1/10	1/10	--	--	--	--	--	--	--
CYANIDE	--	P	1/10	1/10	1/20	--	1/10	--	--	--	1/10
FLASHPOINT	--	--	--	1/10	--	--	1/10	--	--	--	1/10
FLUORIDE	--	--	1/10	1/10	1/20	--	1/10	--	--	--	1/10
HARDNESS	--	--	1/10	1/10	1/20	--	1/10	--	--	--	1/10
KJELDAHL NITROGEN	--	P	1/10	1/10	1/20	--	1/10	--	--	--	1/10
NITRATE-N	--	P	1/10	1/10	1/20	--	1/10	--	--	--	1/10
NITRITE-N	--	P	1/10	1/10	1/20	--	1/10	--	--	--	1/10
NITRATE AND NITRITE	--	--	1/10	1/10	1/20	--	1/10	--	--	--	1/10
PH	--	--	--	1/10	--	--	--	--	--	--	1/10
PHENOL, TOTAL	--	P	1/10	1/10	1/20	--	1/10	--	--	--	1/10
OIL & GREASE	--	P	1/10	1/10	1/20	--	1/10	--	--	--	1/10
TOTAL PHOSPHORUS	--	P	1/10	1/10	1/20	--	1/10	--	--	--	1/10
TDS	--	--	1/10	1/10	--	--	--	--	--	--	--

Table 8	INTERNAL QC CHECKS & FREQUENCY										
ANALYTE	BLANKS			DUPLICATES & SPIKES						CONTROL STD	
	TB	PS	MB*	LD	BS/BSD	MS/MSD	MS	SS	AS	IS	LCS
TS	--	--	--	1/10	--	--	--	--	--	--	--
TSS	--	--	1/10	1/10	--	--	--	--	--	--	--
TVS	--	--	1/10	1/10	--	--	--	--	--	--	--
SULFATE	--	--	1/10	1/10	1/20	--	1/10	--	--	--	1/10
SULFIDE	--	--	1/10	1/10	1/20	--	--	--	--	--	1/10
TAL METALS (CLP)	--	P	1/20	1/20		--	1/20	--	AA=100% CV=NA	--	1/20
TCLP and OTHER METALS (SW846)	--	P	1/20	1/20	1/20	--	1/20	--	AD	--	1/10
TRPH	--	PS	1/10	1/10	1/20	--	1/10	--	--	--	1/10
TCL VOLATILE	1/C	P	1/20	--	1/20	1/20	--	100%	--	100%	1/20
TCL SEMIVOLATILES	--	S	1/20	--	1/20	1/20	--	100%	--	100%	1/20
TCL PCB/PESTICIDE	--	S	1/20(1)	--	1/20	1/20	--	100%	--	--	1/20
GC VOLATILES	1/C	PS	1/20	--	--	1/20	--	100%	--	--	--
TPH - GRO	--	--	1/20	--	--	1/20	--	--	--	--	--
TPH - DRO	--	S	1/20	--	1/20	--	--	--	--	--	--
HERBICIDES	--	S	1/20	--	1/20	1/20	--	--	--	--	1/20
ZHE VOLATILES	1/C	S	1/20	--	--		1/20	100%	--	100%	1/20
TCLP SEMIVOLATILES	--	S	1/20	--	--		1/20	100%	--	100%	1/20
TCLP PESTICIDES	--	S	1/20	--	--		1/20	100%	--	--	1/20
TCLP HERBICIDES	--	S	1/20	--	--		1/20	--	--	--	1/20

(1) Analysis also includes instrument blanks (PIBLK)

TABLE 8 LEGEND	
TB	TRIP BLANK
1/C	ONE PER COOLER RECEIVED
P	PRESERVATIVE CHECK
S	SOLVENT QC CHECK
MB	METHOD BLANK
LD	LAB DUPLICATE
BS/BSD	BLANK SPIKE/BLANK SPIKE DUPLICATE
MS/MSD	MATRIX SPIKE/MATRIX SPIKE DUPLICATE
MS	MATRIX SPIKE
SS	SURROGATE SPIKE(S) ADDED
AS	ANALYTICAL SPIKE (POST DIGEST)
IS	INTERNAL STANDARDS ADDED
LCS	LAB CONTROL STANDARD OR EPA REFERENCE STANDARD
* OR AT LEAST DAILY, WHICHEVER IS MORE FREQUENT	
100% = EVERY SAMPLE SPIKED	

AD Analyst Discretion/SOP Dependent

FIGURE 22

ORTEK ANALYSIS OUT OF CONTROL FORM		
BATCH:	DISTRIBUTION:	(CHECK)
SAMPLE(S):	RECEIVING	
MATRIX:	PREPARATION	
CLIENT:	GC/LC	
DATE REPORTED:	METALS	
	WET CHEM	
REPORTED BY:	GC/MS/EC	

PROBLEM:	
☐ Hold Time missed	☐ Lab blank contaminated
☐ Improper pH of sample	☐ Calibration out
☐ Insufficient sample volume	☐ Check standards out
☐ Custody violated	☐ Surrogates out
☐ Program change	☐ BS/BSD recovery out
	☐ MS/MSD RPD out

ACTION TAKEN:	
Person Notified:	Date of Notification:
Company:	
Requested Action:	☐ Re-extraction for
	☐ Re-digestion for
Date Action Requested:	Turn Time Requested:
Holding Time Expires on:	
PROBLEM RESOLVED: ☐ YES ☐ NO	
Resolved by:	
cc: QA Officer	cc: Client File (Original)

10.1.3 Matrix Spike Duplicate (MSD)

The matrix spike duplicate is prepared as in Section 10.2.7 and the RPD calculated between the MS/MSD pair. If the variability between the MS/MSD exceeds limits, the associated sample data case narrative or report sheet shall contain a note to this effect.

Frequency: One per 20 samples of a similar matrix.

Limits: method and matrix dependent.

10.2 Accuracy QC Samples

Accuracy is defined as the bias of analytical measurements. Sources of bias are the sampling process, field contamination, sample preservation, handling, sample matrix, laboratory sample preparation, and analysis.

10.2.1 Trip Blanks (TB)

Volatile organics samples are susceptible to contamination by diffusion of contaminants through the teflon septum of the sample vial. Trip blanks are prepared by ORTEK in accordance with SOP OP-19, are shipped with the coolers to the client, and are analyzed to monitor possible sample contamination during shipment. Trip blanks accompany the sample bottles through collection and shipment to the laboratory and are stored with the samples. If the trip blanks indicate contamination, the client may decide to flag data for the trip blank concentration or re-sample. Results of trip blank analyses are reported with the corresponding sample analytical data.

Frequency: Two 40 ml. volatile vials per cooler containing volatile samples.

Limits: Check if holding and lab blanks also contain same analytes, report results.

10.2.2 Field Blanks (FB)

A field blank is "pure" water or soil used to demonstrate the absence of contamination during sampling. Deionized, distilled laboratory water, or Ottawa sand supplied by ORTEK on request is placed into sample

containers by the client, packaged, and shipped with the other field samples. If the field blanks indicate possible contamination of the samples depending upon the nature and extent of the contamination, the client may decide to correct sample data for the field blank concentration or resample. Sources of contamination may include: Containers; sample storage facilities; field handling procedures; and sampling equipment. Results are reported with the corresponding sample analytical data.

Frequency: One per 10 field samples or daily (recommended by EPA).

Limits: Not applicable, report results.

10.2.3 Rinsate Blanks (RB)

A rinsate blank is a volume of rinse solution (deionized, distilled lab water or organic solvent) used to rinse a sampling tool which contacts multiple samples. The rinse solution is collected by the client after the tool has collected a sample and has been cleaned, to demonstrate that there is no residual contamination on the tool to carry over into the next sample. If the rinsate blank indicates possible contamination of the succeeding samples, the client may decide to flag data for the rinsate blank concentration or resample. Results of rinsate blank analyses are reported with the corresponding sample analytical data.

Frequency: One per 10 field samples or daily (recommended by EPA).

Limits: Not applicable, report results.

10.2.4 Method Blanks (MB)

A method blank is a volume of laboratory water for water samples, or Ottawa sand for soil/sediment samples carried through the entire analytical procedure. The volume or weight of the blank must be approximately equal to the sample volume or sample weight processed. If the concentration of an analyte in the blank is above the Contract Required Detection Limit (CDRL) or Method Detection Limit (MDL), the sample with the

least concentration analyte must be greater than 10 times the blank concentration, or all samples associated with the blank and less than 10 times the blank concentration must be redigested, reextracted and reanalyzed. No sample values will be corrected for the blank value. Analysis of the blank verifies that method interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware are known and minimal.

Results of method blanks are reported with the data for volatile organics analyses, and kept in the project file for other analyses.

Frequency: One per 20 samples analyzed or daily, whichever is more frequent.

Limits: Less than detection limit or less than 10 x lowest detected sample level for inorganic analytes (Phthalates, methylene chloride, and acetone).

10.2.5 Preservative or Solvent Blank (PS)

This blank consists of the chemical(s) added to the client samples during preservation or extraction. It is prepared and analyzed in accordance with SOP OP-20. Detection of analytes necessitates rejection of the lot.

Frequency: One per each set aside lot of chemical.

Limits: Less than instrument detection limit.

10.2.6 Blank Spikes/Blank Spike Duplicate (BS/BSD)

A blank spike/blank spike duplicate is a volume of laboratory water or Ottawa sand spiked with the analytes or subset of analytes of interest and analyzed using the same procedure as the samples. Recovery of the analyte(s) is plotted on a control chart. Analysis of these samples with acceptable recoveries and no out of control conditions as defined in Section 13 indicates that the laboratory method is in control and acceptable accuracy has been achieved.

Frequency: One per batch of 20 samples or daily, whichever is more frequent.

Limits: As set by SOP OP-21.

10.2.7 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

To determine the accuracy of the method in the real sample matrix, two separate aliquots of a sample are spiked with the analyte or subset of analytes of interest and analyzed with the sample. The percent recovery is calculated and compared against advisory limits cited in each SOP. If the percent recovery is outside of the limits in both samples, a matrix effect may be suspected and the report must contain a note on the matrix effect. Matrix spikes are applicable for wet chemistry and metals analyses, matrix spike duplicates are appicable to organic analysis.

Frequency: One per batch of 20 samples or daily, whichever is more frequent.

Limits: As set by SOP OP-21.

10.2.8 Analytical (Post Digestion Spikes) (AS)

Target metals at a known concentration are added to an aliquot of the sample digest for GFAA analysis just prior to analysis to assess if matrix effects (suppression or enhancement) are present. If results are outside limits, the data report must indicate that the sample exhibited a matrix effect. Further analysis may be required as indicated in the method SOP. For ICAP analyses, if the matrix spike fails and the concentration of the sample does not exceed four times the spike level, a post digest spike must be done at twice the indigenous level or at twice the CRDL, whichever is greater.

Frequency: Every sample for GFAA analysis (CLP protocol).

Limits: 85-115%.

10.2.9 Internal Standards (IS)

A known concentration of organic analyte not expected in environmental samples is added to the sample extract just prior to analysis. It measures instrument performance and is used to normalize data

for quantitation. Reinjection of the sample is done if results are not acceptable.

Frequency: Every real sample, standard and internal QC sample.

Limits: -50 to + 100% area counts.

10.2.10 Surrogate Spikes (SS)

Surrogates are organic analytes that also are not expected to be found in environmental samples and their behavior mimics those of the target analyses. All samples, blanks and internal QC samples are spiked with surrogates prior to purging or extraction for GC or GC/MS analyses. Reanalysis of samples occurs if a specified number of surrogates are outside limits.

Frequency: Every real sample, standard and internal QC sample.

Limits: As contained in ORTEK Method SOP.

10.2.11 Laboratory Control Standards (LCS)

A standard of midpoint concentration on the curve or a known EPA reference standard/ sample is to be analyzed to assess the accuracy of the calibration curve and the stability of the instrument response. For metals and wet chemistry analysis the LCS is the BS. This sample is in addition to the calibration requirements listed in Table 6. For CLP aqueous samples, the LCS is the digested initial calibration verification solution (ICV).

Frequency: One per 20 samples or daily, whichever is more frequent.

Limits: Method and matrix dependent.

10.3 **Representativeness QC Samples**

Representativeness is defined as the degree to which sample data accurately and precisely represent the environmental conditions. It is controlled by selecting proper sampling locations and collecting a sufficient number of samples.

10.3.1 Field Duplicate

If ORTEK is aware of the identity of the field duplicates in the project samples, an assessment of field collection and homogenization techniques and/or site variability can be made by calculating the RPD.

Frequency: One per 10 field samples collected (recommended by EPA).

Limits: Not established.

10.3.2 Collocated Sample

Collocated samples are independent samples collected simultaneously at a given sample location and time. Two charcoal tubes from a common manifold or two surface water samples collected at the same time and depth are examples. These samples indicate the precision attainable over both the field collection and analytical system. Wide variability may indicate the matrix sampled is nonhomogeneous and more points are needed to provide "representative" samples.

Frequency: One per 20 samples (recommended by EPA).

Limits: Not established.

10.4 Completeness QC Samples

Valid data for 100% of all samples analyzed is the completeness goal of the QA program. Since no specific internal QC checks measure completeness, this goal is achieved only if the ORTEK SOPs are followed for calibration, operation and analysis. Acceptable blank spike recoveries will be used to approximate completeness.

10.5 Comparability QC Samples

Comparability is defined as the confidence with which one group of data can be compared with another. It is controlled by using standard sampling and approved EPA analytical techniques.

10.5.1 Split Samples

A split sample is divided into 2 portions in the field and analyzed by ORTEK and an independent laboratory using the same EPA

method. Comparison of results is usually done by the client by calculating RPD.

Frequency: One per 20 samples recommended.

Limits: Client determined.

10.5.2 External Performance Evaluations

ORTEK participates in inter-laboratory round robin studies supplied by the US EPA, Wisconsin DNR and other commercial vendors. A report of the true values and acceptable statistical limits are received and are used to assess each lab section's performance.

Frequency: At least quarterly.

Limits: Study specific.

10.6 Control Charts

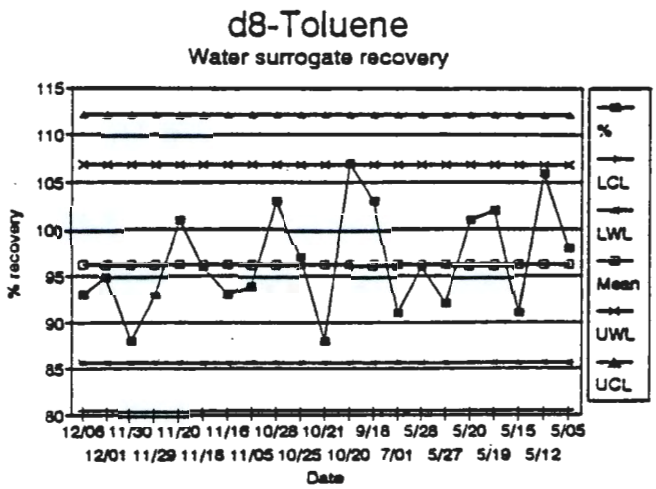
Control charts are a graphic tool to view the statistical performance of a method to enable early detection of out-of-control situations. Blank spike and/or blank spike duplicate percent recoveries for each parameter listed in Table 9 are to be plotted on a Shewhart Control Chart (Figure 23). Limits are set in accordance with SOP OP-21. Analysts are responsible for plotting the points as they are generated.

TABLE 9 ***CONTROL CHART PARAMETERS***

ANALYSIS GROUP	MATRIX	COMPOUND(S)	QC SAMPLE CHARTED
Volatiles GC/MS	Soil & Water	d_8 toluene Bromofluorobenzene d_4-1,2-dichloroethane	Method Blank Recovery
Volatiles GC	Water & Soil	Trifluorotoluene	Method Blank Recovery
Semivolatiles GC/MS	Soil & Water	2-fluorophenol 2-fluorobiphenyl d_5-nitrobenzene d_6-phenol d_{14}-terphenyl 2,4,6-tribromophenol	Method Blank Recovery
PCB/Pesticides	Soil & Water	Aroclor 1248 or 1254, dieldrin, 4,4,'-DDT	BS/BSD Recovery
ICAP Metals	Soil & Water	Lead, Cadmium, Chromium	LCS Recovery
Mercury	Soil & Water	Mercury	LCS Recovery
Hexavalent Chromium	Soil & Water	Hexavalent Chromium	BS/BSD Recovery
GFAA Metals	Soil & Water	Arsenic, Selenium, Lead, Thallium	LCS Recovery
TPH-IR	Soil & Water	TPH	BS/BSD Recovery
Cyanide	Soil & Water	Cyanide	BS/BSD Recovery

FIGURE 23

Blank Surrogate Percent Recovery Control Chart



Date	%Recover	LCL	LWL	Mean	UWL	UCL
		80.4	85.7	96.3	106.9	112.2
12/06	93					
12/01	95					
11/30	88					
11/29	93					
11/20	101					
11/18	96					
11/16	93					
11/05	94					
10/28	103					
10/25	97					
10/21	88					
10/20	107					
9/18	103					
7/01	91					
5/28	96					
5/27	92					
5/20	101					
5/19	102					
5/15	91					
5/12	106					
5/05	98					
5/04	102					
5/01	102					

Limits are initially calculated after 20 points are generated. Control charts are updated at least annually, or when 20 additional points have been generated, whichever is more frequent. Old control charts are archived by the QA officer. Out of control conditions are defined as follows:

- One point outside the upper or lower control limit.
- Three consecutive points outside the upper or lower warning limit.
- Eight consecutive points on one side of the centerline.
- Six consecutive points such that each one is larger or smaller than its immediate predecessor.
- Any cyclic pattern seen over time.

Many factors lead to analytical problems that show up in control charts. The symptoms seen on control charts are either due to a shift or trend as described below:

SYMPTOM	POSSIBLE CAUSES
Shift in centerline after updating	Incorrect standard preparation
	Incorrect reagent preparation
	Systematic contamination of system
	Incorrect instrument calibration
	Analyst error
Trend Upwards	Deterioration of standard
	Deterioration of samples
Trend Downwards	Concentration of standard due to evaporation of solvent
	Deterioration of reagents
Increase in variability	Analyst performance (new analysts, deviation from SOP, poor technique)

Once an out of control condition has been identified and documented on an Out-of-Control Form, corrective action must be taken and documented and no additional data generated until the next point is in control. Suggested corrective actions are (in order of completion):

1. Check calculations
2. Check age of spiking solution
3. Make new spiking solution
4. Reanalyze affected batch
5. Check age of stock spike standard
6. Make new stock spike standard
7. Reanalyze affected batch

Control charts are included in all data packages for Navy (NEESA) and HAZWRAP projects (upon request by other clients) and are submitted in the project file to Client Services for transmittal.

11.0 PERFORMANCE AND SYSTEMS AUDITS AND FREQUENCY

Performance audits independently collect data from the QA system using performance evaluation samples and are quantitative. Results are usually expressed as falling within or outside of statistically determined acceptance limits. System audits are the review of the entire data production process and consist of on-site inspection and a review of documentation. System audits are qualitative and consist of an audit report containing any deficiencies. Data audits consist of reviewing client project files for appropriate QC results and documentation from sample receipt through disposal. Data audits are conducted monthly on randomly selected projects or in response to data challenges from clients.

These audits are performed to:

- Determine that contractual/regulatory criteria are met.
- Determine that the ORTEK SOP's and this QA Program Manual are being followed.
- Verify that document control procedures are followed.
- Establish that DQO's are met, including that holding times and report due dates are met, approved methods and SOP's are followed, and stated frequencies and limits for precision, accuracy, representativeness, completeness and comparability are met.
- Serve as a management tool to evaluate the effectiveness and appropriateness of ORTEK's QA Program.

Results of all audits are communicated to the President, Laboratory Manager, and affected Section Supervisors by the QA Officer. Blame is not assigned, but request for investigation of any deficiencies and proposed corrective action and schedule for implementation is requested. Each Section Supervisor is to respond in writing, to document corrective action and avoid repeating of the same deficiency.

11.1 Performance Audits

The QA Officer is responsible for preparation of any internal single blind performance evaluation (PE) samples and insertion of any double blind external (PE) samples into the ORTEK analytical system.

Single blind PE samples are obtained from commercial sources such as Environmental Resource Associates and are used after an external PE has indicated

deficiencies. Once corrective action has been taken and documented by affected sections of the lab, the QA Officer will prepare full volume samples, and "dummy" up sampling documentation such as the chain-of-custody form to disguise the PE sample as a real sample. These samples are submitted to the laboratory as unknown blind samples to determine if analytical systems are in control and meeting client requirements.

Blind PE samples are sent to ORTEK from EPA and DNR. These samples are not disguised as real samples and are usually contained in sealed glass ampules or vials. The QA Officer does not prepare full volume samples from the PE ampules. Each section of the lab is responsible for correctly diluting and spiking samples as indicated by the directions. Blind performance evaluation studies in which ORTEK participates and their frequency are listed below:

U.S. EPA CLP PE Samples	Quarterly, through EPA Region Region V and EPA EMSL-LV
U.S. EPA Water Pollution Studies	Semiannually
Wisconsin Department of Natural Resources Recertification samples	Annually
U.S. Army Corps of Engineers laboratory validation PE samples	Every 18 months

11.2 Systems Audits

Every 3 years, the Wisconsin Department of Natural Resources conducts an on site inspection. Every 18 months prior to recertification, the US Army Corps of Engineers performs an on-site evaluation. Other clients audit ORTEK on a project specific basis.

Quarterly, an internal on-site audit is conducted by the QA Officer. One section of the lab (sample receiving/Document Control, wet chemistry, metals, organics) is chosen each quarter. A predesigned checklist will be used that includes the following items as applicable:

- **Sample Operations**
  - Are stated temperatures for sample storage monitored?
  - Are samples extracted and analyzed within prescribed holding times?
  - Are samples properly logged in, stored, and disposed of?
- **Calibration**
  - Are calibrations performed as required?
  - Are calibration records properly documented in instrument log books, or as part of project data if required?
  - Do calibration results indicate a trend in instrument performance?
- **Preventive Maintenance**
  - Are adequate spare parts available?
  - Do specific instruments have repeated maintenance problems?
  - Is preventive maintenance performed and properly documented?
- **Receipt and storage of standards, chemicals, and gases**
  - Are all reagents, chemicals, and gases purchased for use in the laboratory of adequate grade for the intended use?
  - Are certifications and QC checks done when required?
  - Are they kept beyond stated shelf life?
  - Are they properly prepared, stored and documented?
- **Analytical Methods**
  - Are the methods used appropriate for project requirements?
  - Are detection limit studies available and current?

- Are alternate methods approved for use?
- Are copies of method SOPs current and available?

· **Data Verification**

- Are data processed and validated as prescribed?
- Are control charts updated, with out-of-control conditions noted and corrective action documented?

· **Records Management**

- Are the records of analyses complete and properly identified?
- Is chain of custody fully documented?

The QA Officer will meet with the Laboratory Director, and affected Section Supervisor prior to beginning the audit to discuss what will be audited. At the close of the audit, the findings will be discussed with them. A corrective action plan and implementation schedule will be discussed and agreed upon if deficiencies are found. An audit report will be written by the QA Officer to include:

- Date/location of audit.
- Persons contacted in the lab, specific lab operations/records audited.
- Description of items requiring corrective action and, if possible, the means for correction.
- Due date for completion of corrective action.
- Means of verifying completion of corrective action.
- Review of the Quality Assurance Program implementation in the section.

The audit report will be issued within 10 days after completion of the audit.

The Section Supervisor is responsible for responding to the audit report. The response will be in writing to the QA Officer and will state the corrective action taken or the action underway. If correction can be verified, the Section Supervisor should attach documentation of the corrective action to the audit response. Upon receipt of the audit response, the QA Officer must verify completion of the corrective action.

11.3 Data Audits

Data audits are done monthly by the QA Officer to address the precision, accuracy, representativeness, and completeness of the data. Projects are selected at random or as requested by clients through verbal data challenges or written data verification requests. A memo is written by the QA Officer that details the findings. The Laboratory Director, and Section Supervisors are to respond to deficiencies as requested.

11.4 ORTEK Certifications

The following certifications have been obtained by ORTEK:

- Wisconsin Department of Natural Resources
- State of Washington
- State of Utah
- U.S. Army Corps of Engineers, Missouri River Division
- 8(a) Program, US Small Business Administration
- Disadvantaged/Minority Business Enterprise, City of Madison
- Disadvantaged Business Enterprise (DBE)
  - Wisconsin Department of Transportation
  - Illinois Department of Transportation
- Minority Business Enterprise (MBE)
  - Wisconsin Department of Development
  - Wisconsin Supplier Development Council
  - Joint Certification Program, Milwaukee, WI

Copies of certification documents are available upon written request.

12.0 PREVENTIVE MAINTENANCE

Preventive maintenance (PM) is an organized program of actions (such as equipment cleaning, lubricating, reconditioning, adjustment and/or testing) taken to maintain proper instrument and equipment performance and to prevent instruments and equipment from failing during use. The purpose of PM is to increase reliability of data reported and reduce downtime. ORTEK's preventive maintenance program includes the following:

- Instruments, equipment, and parts that are subject to wear or deterioration without proper periodic maintenance.
- Spare parts that should be readily available to minimize downtime.
- Frequency that maintenance is required and documentation that it was performed.

Implementation of the preventive maintenance program is dependent upon the specific instrument and manufacturer. This manual does not designate specific PM for each instrument and equipment but lists in Table 10 the general practices that are applicable. The equipment SOP includes the PM specific to each instrument.

Each Section Supervisor is reponsible that analysts properly conduct and document PM. Documentation must be recorded in each instrument or maintenance logbook.

TABLE 10	*PREVENTIVE MAINTENANCE GUIDANCE*	
INSTRUMENT	ITEM CHECKED/SERVICED	FREQUENCY
Atomic Absorption Spectrophotometer		
	Burner head	Each shift
	Electrical	Each shift
	Lamps	Each shift
	Nebulizer	Each shift
	Optics	During PM Service Calls
	Graphite tube	As necessary
	Replace graphite tube	As needed
	Replace contact rings	As needed
	Replace quartz windows	As needed
	Clean optics	As needed
	Align background lamp	When changed
Gas Chromatograph		
	EC (Ni-63) wipe test	Semi-annual
		FID monthly
	Change column	As needed
	Change glass wool plug	Weekly
	Clean insert	Weekly
	Replace septum	Daily
	Gas purity check	Upon receipt of new cylinders
	Flow controller	Semi-annually
	Purge and trap	As needed
	Change fuses	As needed
	Reactivate external carrier gas filler dryers	Weekly
	Reactivate flow controller filter dryers	Semi-annually
	Clean and silanize or replace glass liners on injectors	As needed or quarterly
	Clean Detectors a) ECD b) FID, Hall	a) As needed b) As needed or annually
	Clean Purge Vessel	As needed or monthly
	Replace Purge Vessel	As needed

TABLE 10	*PREVENTIVE MAINTENANCE GUIDANCE*	
INSTRUMENT	ITEM CHECKED/SERVICED	FREQUENCY
	Bake Trap	As needed
	Replace Trap	Semi-annually
	Replace carbon filter	Annually
ICAP		
	Sample introduction system	Daily
	Replace o-rings and water filters	As required
	Clean optics	As needed
	Clean torch	As needed
	Change oil and dessicant	Annually
	Check electronics	Daily
	Clean, realign torch	As Required
	Clean nebulizer tips	Daily
	Clean mixing chamber	As Needed
	Replace pump tubing	Daily if left hooked up
Lachat Autoanalyzer		
	Clean and dry random access sampler	Daily
	Clean boats and check placement	Daily
	Clean sensor with cotton swab	Daily
	Spray proportioning pump with silicone, wipe rollers	Every 50 hours, (2500 samples)
	Check pump waste lines	Every 50 hours, (2500 samples)
	Replace injection module flares	Every 500 hours, (25,000 samples)
	Clean unions, replace o-rings	Every 500 hours, (25,000 samples)
	Clean manifold fittings	Every 500 hours, (25,000 samples)
	Replace manifold o-rings	Every 500 hours, (25,000 samples)
	Rewrap coils	Every 500 hours, (25,000 samples)
	Clean and dry colorimeter	As needed
	Run "clean disk" in computer	Every 500 hours
Refrigerators		
	Temperature checked and logged	Daily
Balances		
	Service representative calibration	Annually

TABLE 10	***PREVENTIVE MAINTENANCE GUIDANCE***	
INSTRUMENT	**ITEM CHECKED/SERVICED**	**FREQUENCY**
Deionized/Nanopure Water		
	Conductivity Check	Daily
	Ion exchange bed changed	Weekly
	Replace filters	As needed
GC/MS GC/MS maintenance is the same as GC with the following additions:		
	Mechanical pump oil	Quarterly
	Power Con. air filter	Bi-Weekly
	QEM filter	Bi-Weekly
	Turbo pump oil	Semi-annually
	Water filter (if applicable)	Observe and change as needed
	Computer air filter	Monthly
	Card cage air filter	Monthly
	Source-clean ceramics, polish lenses	As needed
	Clean poles and ceramics on the poles	As needed
	Clean contacts on the component boards	As needed
	Vacuum the component boards	As needed
	Clean all fan screens	Weekly
	Vacuum outside of instrument	Weekly
	Clean grob and replace quartz insert	As needed
	Replace septum	Daily (each shift)
	Injection port liner checked	Daily
	Column maintenance	As needed
Infrared Spectrophotometer		
	Clean cells	Daily
pH Meter		
	Electronics checked	Daily
	Electrolyte changed	Checked weekly, changed when low

13.0 SPECIFIC ROUTINE PROCEDURES TO ASSESS DATA PRECISION, ACCURACY AND COMPLETENESS

The purpose of this section is to describe how data from the QC samples listed in Section 10.0 are treated to determine data quality. Data accuracy and precision are calculated as percent recovery or relative percent difference (RPD). Data completeness is calculated as the overall percent of blank spike samples in control.

13.1 Data Precision Calculation

To determine the precision of the method and/or analyst, a routine program of sample duplicate analyses is performed. These may also be blank spike or matrix spike/matrix spike duplicate pairs. The results of the duplicate analyses are used to calculate the relative percent difference (RPD) which is defined as the difference (range) of each duplicate set, divided by the average value (mean) of the duplicate set, times 100 percent. For duplicate results D_1 and D_2, the RPD is calculated from Equation 13-1:

$$RPD\ \% = \frac{|D_1 - D_2|}{\frac{(D1+D2)}{2}/2} \times 100\ \% \qquad (13\text{-}1)$$

When the RPD is obtained for at least 20 duplicate pairs, the average RPD and the standard deviation are calculated using:

$$m = \frac{\sum_{i=1}^{n} m_i}{n} \qquad (13\text{-}2)$$

$$Sm = \frac{\sum_{i=1}^{n} (m-\bar{m})^2}{n-1} \qquad (13\text{-}3)$$

where

m = the RPD of a duplicate pair,

$\bar{m}$ = the average of the RPD values,

Sm = the standard deviation of the data set of RPD values, and

n = number of RPD values used.

Control limits are calculated from these data as follows:

Upper control limit = $\bar{m} + 3Sm$,

Lower control limit = $\bar{m} + 2Sm$,

Lower warning limit = $\bar{m} - 2Sm$, and

Lower control limit = $\bar{m} - 3Sm$.

Control charts are not kept for RPD statistics. Original limits are distributed to analysts and Section Supervisors by the QA Officer and updated at least annually by the QA Officer. Points are to be plotted as they are generated.

13.2 Data Accuracy Calculation

To determine the accuracy of an analytical method and/or analyst, a sample and blanks are routinely spiked. The results of matrix, matrix spike duplicate, blank spikes, and blank spike duplicates are used to calculate the quality control parameter for accuracy evaluation, the Percent Recovery (%R). Blank spike recoveries and method blank surrogate recoveries are plotted on control charts.

The %R is the observed concentration, minus the sample concentration, divided by the true concentration of the spike, times 100 percent:

$$\%R = \frac{O_s - O_i}{T_i} \times 100\% \qquad (13\text{-}4)$$

where

$\%R$ = the percent recovery,

O_i = the observed spiked sample or blank concentration,

O_s = the unspiked sample or blank concentration, and

T_i = the true concentration of the spike.

The true spike concentration is calculated from Equation 13-5:

$$T_i = \frac{\text{Spike Concentration (mg/L)} \times \text{Volume of Spike (in ml)}}{\text{Volume of Sample [in ml]} + \text{Volume of Spike [in ml]}} \qquad (13\text{-}5)$$

When the percent recovery is obtained for at least twenty blank spike samples, the mean percent recovery and the standard deviation are calculated using the formulas:

$$\overline{\%R} = \frac{\sum_{i+1}^{n} \%R_i}{n} \qquad (13\text{-}6)$$

and

$$S_R = \frac{\sum_{i=1}^{n} (\%R_i - \overline{\%R})}{n=1} \qquad (13\text{-}7)$$

where

R_i = percent recovery,

$\overline{\%R}$ = Mean percent recovery,

$MR_i = 1R_i - (R_i - 1)$ $1_i = 2, 3, \ldots n$

MR_2 = average moving range of 2 successive recoveries,

n = number of results, and

$d_2 = 1.128$.

Control limits are calculated from these data as follows:

Upper control limit = $\overline{\%R} + 3R_2/d_2$,

Upper warning limit = $\overline{\%R} + 2R_2/d_2$,

Centerline = $\overline{\%R}$,

Lower warning limit = $\overline{\%R} - 2R_2/d_2$, and

Lower control limit = $\overline{\%R} - 3R_2/d_2$.

Control charts are kept for blank spike and lab blank surrogate recoveries. All original limits are calculated by the QA Officer, distributed to Section Supervisors and analysts and updated at least annually by the QA Officer. Points are to be plotted as they are generated.

13.3 Data Completeness Calculation

To determine the completeness of an analytical method and/or analyst, blank spikes or method blanks spiked with surrogate compounds are analyzed. The

number of blank spikes (BS) or method blanks (MB) with recoveries within control limits are counted and assessed against the total number analyzed as follows:

$$\% \text{ Completeness} = \frac{\#\text{ BS or MB in control}}{\text{total }\#\text{ BS or MB analyzed}} \qquad (13\text{-}8)$$

13.4 Data Set Assessment

When analysis of a project/batch is completed, the results will be reviewed as described in SOP OP-17 and evaluated in accordance with Figure 20. Briefly, review and evaluation are done for the items listed below in order:

1. Calibration results.
2. Holding times laboratory blank results.
3. Blank spike/lab control standard recoveries.
4. Surrogate spike recoveries.
5. Duplicate sample and Matrix Spike/MSD results.
6. Field/shipping QC results (trip blanks, field blanks and field duplicates).

14.0 CORRECTIVE ACTION

Out of control conditions as documented on ORTEK's Out-of-Control Form (Figure 22) must be corrected. All Out-of-Control Forms must be routed to the QA Officer for determination, if the client needs to be notified.

14.1 Out-of-Control Conditions

An out-of-control condition is an unauthorized deviation from SOPs, methods, or a defect that could lead to the data quality not meeting client needs and/or internal QC check limits. Out-of-control conditions may be caused by lab operations or field operations (documentation not complete, inadequate preservation) that are identified by ORTEK. Out-of-control conditions include, but are not limited to:

- Sample holding time exceeded.
- Sample preservation and/or pH not adequate.
- Sample receiving paperwork not correct.
- Detected analytes in blanks.
- Instrument calibration requirements not met.
- Sample storage requirements not met.
- Chain-of-Custody broken.
- Incorrect sample preparation/analysis used.
- Internal QC sample data outside limits.
- Data recording or transcription errors.
- Failure to document data.
- Data validation errors.

The affected samples/clients and batches are to be identified on the Out-of-Control Form and the QA Officer contacted immediately for consultation on appropriate resolution and responsibility.

14.2 Corrective Action

Corrective action is defined as the effective measure applied to correct and minimize the possibility of recurrence of an out-of-control condition. Examples of corrective actions include, but are not limited to:

- Recalibration.
- Preparation of new standards.
- Preparation of new reagents.
- Reanalysis of samples.
- Additional training of personnel.
- Client notification/consultation (i.e., improper sample preservation, insufficient volume).

14.3 Responsibility for Corrective Action

All employees are responsible for reporting out-of-control conditions that they observe or identify. The employee should initiate the Out-of-Control Form and give it to the Section Supervisor and sign the "Report by" line. They must also indicate under ACTION TAKEN, the person notified, and date. All original (golden rod) Out-of-Control Forms must accompany the project data and be included in the project file. A copy must be sent to the QA Officer for review for trends.

Each laboratory Section Supervisor is responsible for documenting and correcting problems that might affect data quality in accordance with the requirements of this section. The Section Supervisor is responsible for stopping work in the event of out-of-control situations, and notifying the QA Officer. With the QA Officer, the Section Supervisor is responsible for determining whether reported problems affect data quality, concurring with the proposed corrective action, and notifying the QA Officer that corrective action has been completed.

The QA Officer is responsible for reviewing Out-of-Control Forms, recommending or approving proposed corrective actions, assessing out of control condition for trends, verifying that corrective action has been completed, distributing and filing out-of-control forms and assisting in resolving disagreements. With the Laboratory Manager and Section Supervisors, the QA Officer also is responsible for determining whether reported problems are serious enough to stop lab operations and establishing schedules for completion of corrective action. The QA Officer is responsible for assisting in resolving disagreements and quality problems, and performing audits of the laboratory for compliance with the corrective action.

14.4 Corrective Action Requested by External Auditors

Results of PE samples and audit reports are sent to affected Section Supervisors and the Laboratory Manager by the QA Officer. If the sample results/audit identify deficiencies that require corrective action, the QA Officer will, in writing, request a turn time on the action and verify that action is taken in order to prepare a written response to the external auditor (if requested). All affected Section Supervisors will receive a copy of the response.

Upon notification by the external auditor of acceptance of the corrective action, the QA Officer will notify the responsible Section Supervisor and the Laboratory Manager. If corrective action is not acceptable, the QA Officer will request, in writing, that additional corrective action be taken. The QA officer will file the records pertaining to the out-of-control conditions with the external audit documents.

Out-of-control and required corrective action can also result from the ongoing laboratory review of lab by the QA Officer. These activities are discussed in Section 11.0.

A summary of out-of-control conditions will be reported monthly to the Laboratory Director. Corrective actions for out-of-control conditions that are detected <u>after</u> data have been reported must also be reported by the QA Officer to the Laboratory Director by a copy of written memos in response to client data verification requests or verbal data challenges.

15.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

If the performance of the laboratory is not reported frequently and concisely to ORTEK management, the effectiveness of the QA program is diminished. Reporting of internal and external systems, performance audits, out-of-control conditions and corrective actions taken are done through three means:

15.1 Weekly Management Meetings

Each week, all Section Supervisors, Marketing Manager, Laboratory Manager, QA Officer and General Manager meet to discuss laboratory operations and personnel issues. Items are solicited for the agenda prior to the meeting and minutes are distributed for the purpose of documenting responsibilities. At these weekly meetings the QA Officer transmits the status of audits, PE sample analyses, out-of-control conditions identified and results of client data challenges.

15.2 Monthly Data Audit Reports

The findings of the QA Officer's random project file review as described in Section 11 is transmitted monthly (by the 10th of the following month) to the Laboratory Manager, General Manager and affected Section Supervisors. A summary of out-of-control conditions reported each month are also included to enable Section Supervisors to assess continuing problem areas. Written results of client data challenges and verifications are also transmitted at least monthly to the Laboratory Manager and affected Section Supervisor.

15.3 Quarterly Internal Lab Audit Results

The written findings of the QA Officer's quarterly audit of a section of the laboratory as described in Section 11 are transmitted to the Laboratory Manager, General Manager and affected Section Supervisor.

15.4 Client Monthly Progress Reports/QA Reports

Major government clients are provided with Monthly Progress Reports (MPRs) upon request, as provided for in the contract. These MPRs follow a format specified by the client. NEESA/HAZWRAP reports must include:

- Project name, number and contract/subcontract number.
- List of client sample numbers, ORTEK sample

numbers, analyses requested.

- Date collected, date extracted and date analyzed.
- Copies of Chain-of-Custody Forms signed by ORTEK.
- New lab methods, equipment or changes in old methods.
- Changes in QA personnel or other personnel (resumes attached).
- Copies of Out-of-Control Forms and Corrective Action Log as they apply to specific project samples.
- Control charts pertinent to project samples.
- External audit results and corrective action plans and written responses.

16.0 DATA DELIVERABLES

The specific data and reporting format necessary is client dependent. DQO levels C,D, and E have a predefined list of data to be included as defined on Table 11. Other client deliverables, unless otherwise specified, consist of the analytical result sheet only. QA Summary Reports (Figure 24) are prepared for a fee by the QA Officer and should be requested by the client prior to sample analysis.

The completeness of data deliverable packages of level C,D, and E are to be checked by the assigned ORTEK Project Manager prior to release to the client. Noncompliant data packages are those with missing information as identified by the QA officer during random data audit review or by the client. The QA Officer will notify the affected Section Supervisor of specific missing items for resolution before re-submittal to the client.

TABLE 11 **DATA DELIVERABLES - LEVEL C**

ANALYTE GROUP	CASE NARRATIVE	CLP FORMS											CONTROL CHART			OTHER REQUIREMENTS
		I	II	III	IV	V	VI	VII	VIII	IX	X	XI-XIV	MB	BS	LCS	
Volatiles-GC/MS	X	X	X	X	X	X	X	X	X				X			CHROMATOGRAMS & MS OF SAMPLES
Semivolatiles-GC/MS	X	X	X	X	X	X	X	X	X				X			CHROMATOGRAPHS & MS OF SAMPLES
PCB-Pesticides	X	X	X	X	X	X	X	X	X	X				X		CHROMATOGRAMS OF SAMPLES
Volatiles-GC	X	X*	X*	X*	X*		X*	X*					X			2nd column, confirmation data, intial and continuing response factors and % D from initial
Metals/CN	X	X	X	X	X	X	X		X		X	X			X	
Wet Chemistry	X	X*		X	X*									X		%RSD from initial calibration

NOTE:
MB = Method blank, surrogate recoveries charted
BS = Blank spike, recovery charted
LCS = Lab control standard, recovery charted

* ORTEK to supply format for review by client

TABLE 11 DATA DELIVERABLES - LEVEL D

ANALYTE GROUP	CASE NARRATIVE	CLP FORMS											CONTROL CHART			OTHER REQUIREMENTS
		I	II	III	IV	V	VI	VII	VIII	IX	X	XI-XIV	MB	BS	LCS	
Volatiles-GC/MS	X	X	X	X	X	X	X	X	X				X			Full CLP package with TICS
Semivolatiles-GC/MS	X	X	X	X	X	X	X	X	X				X			Full CLP package with TICS
PCB-Pesticides	X	X	X	X	X	X	X	X	X	X	X			X		Full CLP package
Volatiles-GC	X	X*	X*	X*	X*		X*	X*					X			As in Level C plus all chromatogram of samples, QC and standards, prep records
Metals/CN	X	X	X	X	X	X	X	X	X	X	X	X			X	
Wet Chemistry	X	X		X*	X*									X		As in Level C plus all absorbances, digest/prep logs and instrument printouts

NOTE:
MB = Method blank, surrogate recoveries charted
BS = Blank spike, recovery charted
LCS = Lab control standard, recovery charted

* ORTEK to supply format for review by client

TABLE 11 **DATA DELIVERABLES - LEVEL B**

ANALYTE GROUP	CASE NARRATIVE	CLP FORMS											CONTROL CHART			OTHER REQUIREMENTS
		I	II	III	IV	V	VI	VII	VIII	IX	X	XI-XIV	MB	BS	LCS	
Volatiles-GC/MS		X			X								X			
Semivolatiles-GC/MS		X			X								X			
PCB-Pesticides		X			X									X		
Volatiles-GC		X*			X*								X			
Metals/CN		X		X											X	
Wet Chemistry		X			X*									X		

NOTE:
MB = Method blank, surrogate recoveries charted
BS = Blank spike, recovery charted
LCS = Lab control standard, recovery charted

* ORTEK to supply format for review by client

FIGURE 24

ORTEK QUALITY CONTROL REPORT

TO:

ATTN:

BATCH ID:

OUR LAB #:

YOUR SAMPLE ID:

SAMPLE MATRIX:

REPORT DATE:

COLLECTION INFORMATION

DATES:

LOCATION:

ANALYTE	LAB BLANK CONC.	LAB DUPLICATE RPD	MATRIX SPIKE % RECOVERY	EPA LAB CONTROL STANDARD RPD	BLANK SPIKE % RECOVERY	BLANK SPIKE DUPLICATE % RECOVERY	SAMPLE NUMBER USED FOR QC

ND = NOT DETECTED

NA = NOT APPLICABLE

RPD = RELATIVE PERCENT DIFFERENCE

BS = BLANK SPIKE

BDS = BLANK SPIKE DUPLICATE

SIGNED: _____ DATE: _____

TITLE: _____

17.0 REFERENCES

This section contains a list of references used during the preparation of this QAPM. For ease of reading, no footnotes or specific reference citations were used in the manual. These references are available from the QA Officer and ORTEK employees are encouraged to consult them to learn more about specific QA/QC policies.

F.M. Garfield, Quality Assurance Principles for Analytical Laboratories, Association of Official Analytical Chemists, Arlington, VA 1984.

US EPA Laboratory Data Validation Guidelines for Evaluating Organics Analyses, February 1, 1988.

US EPA Laboratory Data Validation Guidelines for Evaluating Inorganic Analyses, July 1, 1988.

US EPA Contract Laboratory Program Statement of Work for Organic Analyses, OLM01.1-.6.

US EPA Contract Laboratory Program Statement of Work for Inorganic Analyses, ILM02.01.

US EPA, "Guidelines and Specifications for Preparing Quality Assurance Program Plans," EPA QAMS-004/80, EPA-600/8-83-024.

US EPA, "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans," EPA QAMS-005/80, EPA-600/4-83-004.

US EPA, "Guidelines for the Preparation of Standard Operating Procedures (SOPs) for Field and Laboratory Measurements," US EPA Region V, March 16, 1989.

US EPA, "Final Standard Quality Assurance Project Plan Content Document," US EPA Region V, June 1989.

US ACE, "Chemical Data Quality Management for Hazardous Waste Remedial Activities," Department of the Army, US Army Corps of Engineers, ER-1110-1-263, October 1, 1990.

US EPA, "Content Requirements for Quality Assurance Project Plan," US EPA Region V, January 1989.

DOE, "Requirements for Quality Control of Analytical Data," DOE/HWP-65/R1, July 1990.

US Navy, "Sampling and Chemical Analysis Quality Assurance Requirements for the Navy Installation Restoration Program," NEESA 20.2-047B, June 1988.