QUALITY ASSURANCE PROJECT PLAN ADDENDUM II

ADDITIONAL AND UNCHARACTERIZED SITES OPERABLE UNIT CRAB ORCHARD NWR MARION, ILLINOIS - WILLIAMSON COUNTY

January 25, 2007

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QAPP Addendum Crab Orchard NWR December, 2006

QUALITY ASSURANCE PROJECT PLAN ADDENDUM II ADDITIONAL AND UNCHARACTERIZED SITES OPERABLE UNIT CRAB ORCHARD NWR MARION, ILLINOIS, WILLIAMSON COUNTY

Prepared by NewFields Companies, L.L.C., ENTRIX, Inc., & Conestoga-Rovers & Associates

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LIST OF ACRONYMS/ABBREVIATIONS

QAPP	Quality Assurance Project Plan
NWR	National Wildlife Refuge
RI/FS	Remedial Investigation/Feasibility Study
DCL	DataChem Laboratories, Inc.
QA	Quality Assurance
COC	Chain of Custody
QA/QC	Quality Assurance/Quality Control
SOP	Standard Operating Procedures
CSM	Client Services Manager
M/MD	Pairs of Matrix Samples
MS/MSD	Spiked Matrix Samples
RPF	Relative Percent Difference
LCS	Laboratory Control Sample
MS	Matrix Spike
RI	Remedial Investigation
MDL	Method Detection Limit
USFWS	United States Fish and Wildlife Service
QC	Quality Control
CLP	Contract Laboratory Program
GC/FPD	Gas Chromatograph Flame Photometric Detector
COD	Coefficient of Determination
CCV	Continuing Calibration Verification
LIMS	Laboratory Information Management System
SDG	Sample Delivery Group
GC/MS	Gas Chromatography Mass Spectrometer
EDD	Electronic Data Deliverable
FSP	Field Sampling Plan
STL	Severn Trent Laboratories
CRA	Conestoga Rovers and Associates
RL	Reporting Limits

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1.0 QAPP ADDENDUM II

This Quality Assurance Project Plan (QAPP) Addendum II is intended to be used in conjunction with the EPA-approved Quality Assurance Project Plan, Crab Orchard Additional and Uncharacterized Sites Operable Unit, Crab Orchard National Wildlife Refuge (NWR), Marion Illinois, Williamson County, FINAL April 2006 (Entrix 2006) which was developed for the Crab Orchard National Wildlife Refuge Additional Uncharacterized Sites Remedial Investigation/Feasibility Study (RI/FS). This QAPP addendum was prepared to specifically address the addition of White Phosphorous analyses and to supply information on the laboratory that will support this analysis. This QAPP addendum is also being utilized to address the following issues:

- Additional compounds being added to the Dioxin/Furan list.
- Adjustments made to Figure 2.1 of the QAPP; Project Organizational Chart

2.0 ADDITION OF A NEW LABORATORY

2.1 Laboratory Responsibilities

DataChem Laboratories, Inc. (DCL) has been selected as the analytical laboratory for the White Phosphorous analyses portion of this project. Certificates for pertinent accreditation can be found in Appendix B. DCL is located at:

960 West LeVoy Drive Salt Lake City, Utah 84123 (801) 904-4302

The various Quality Assurance (QA) and management responsibilities of key project personnel are defined below:

DataChem Laboratories Inc. - Program Manager (PM) - Kevin Griffiths

The DCL Project Manager will communicate directly with the ENTRIX Project Manager and will be responsible for the following:

• Scheduling sample analyses with the laboratory;

- Verifying chain of custody (COC) and accepted samples versus the Project QAPP;
- Relaying technical issues with the ENTRIX Project Manager;
- Overseeing data completeness;
- Overseeing preparation of final data package; and
- Approving final data package prior to distribution.

DataChem Laboratories Inc., - QA Officer - Robert P.Di Rienzo, CQA

The DCL QA Officer will be responsible for the following:

- Overview laboratory QA;
- Overview Quality Assurance/Quality Control (QA/QC) documentation;
- Overview in-house COC;
- Review laboratory corrective actions;
- Conduct detailed data review, if corrective action warrants;
- Review Laboratory Standard Operating Procedures (SOPs)

DataChem Laboratories - Organics Laboratory Manager - Richard W. Wade

Responsibilities of the DCL Organics Laboratory Manager or his designee include:

- Manage the staff responsible for sample preparation and analysis;
- Supervises and trains analytical personnel and provides technical management for laboratory methods and procedures in the organics section. Prepare laboratory SOPs for the section;
- Tracks samples and ensures that sample data are reported on time.
- Provides technical expertise in maintenance and configuration of analytical instrumentation in regards to the organics section.

The DCL Technical Staff will be responsible for sample preparation and analysis, scheduling sample analysis, identification of corrective actions and archival of extracts. The analyst is responsible for the initial data review, followed by a review from a peer. The Laboratory Manager reviews the data for final approval. The Client Services Manager (CSM) and Project Manager reviews for completeness. The QA Officer will review any corrective action necessary.

3.0 Quality Assurance Objectives

3.1 Tracking and Evaluation of Accuracy and Precision

3.1.1 Definitions

Precision is the degree of agreement among repeated measurements of the same characteristic (analyte, parameter, etc.) under the same or similar conditions (USEPA, 2000).

Accuracy is the extent of agreement between an observed value (sample results) and the accepted, or true, value of the parameter being measured (USEPA, 2000).

3.1.2 Laboratory Objectives

Assessment of the precision (repeatability) of an analytical measurement is based upon repeated analysis of equivalent samples of known or unknown composition. DCL relies upon the analysis of pairs of matrix samples (M/MD) or spiked matrix samples (MS/MSD) to assess precision. The range of the pair is expressed as a relative percent difference (RPD). Control limits for the accuracy and precision charts are calculated assuming a normal distribution of results. A set of historical data points is used to calculate the mean value, two standard warning limits, and three standard deviation control limits. The establishment and updating of control charts is described in DCL SOP QC-DC-001. Establishing and updating control limits.

Assessment of the accuracy of an analytical measurement is based upon the analysis of samples of known composition. DCL relies upon the analysis of Laboratory Control Samples (LCS) and Matrix Spike (MS) samples to track accuracy. The percent recovery relative to the expected value is calculated and plotted on an accuracy chart (x chart) for tracking. The data generated demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. The (%R) is calculated according to the following formula (USEPA, 2000):

%*R* = <u>Spiked Sample Concentration – Unspiked Sample Concentration</u> X 100 Concentration of Spike Added

SOPs for laboratory analyses are provided in Appendix A and contain the required accuracy, precision, sensitivity of the analyses.

3.2 Completeness

3.2.1 Definition

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions.

3.2.2 Laboratory Completeness Objectives

Laboratory completeness is a measure of the amount of valid measurements obtained from all the measurements taken in the project. In this case, completeness refers to all measurements that correspond to the White Phosphorous analyses. The equation for completeness is presented below. Laboratory completeness for this project, and these analyses, should be 90 percent or greater.

> Completeness = <u>(number of valid measurements)</u> X 100 (Number of measurements planned)

3.3 Representativeness

3.3.1 Definition

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition.

3.3.2 Measures to Ensure Representativeness of Laboratory Data

Representativeness in the laboratory is ensured by using the proper analytical procedures, meeting sample holding times and analyzing and assessing field duplicate samples.

3.4 Comparability

3.4.1 Definition

Comparability is an expression of the confidence with which one data set can be compared with another. Comparability is also dependent on similar QA objectives.

3.4.2 Measures to Ensure Comparability of Laboratory Data

The criteria for laboratory data comparability will be to ensure that the analytical methods used for the Remedial Investigation (RI) sampling and analysis events are comparable to the methods used for previous sampling events, if applicable.

3.5 Sensitivity

3.5.1 Definition

Sensitivity is the ability of the method or instrument to detect the contaminant of concern and other target compounds at the level of interest.

3.5.2 Sensitivity of Laboratory Data

DCL will evaluate and monitor method and instrument sensitivity through the development of laboratory method detection limits (MDL). A laboratory fortified blank, a blank that is spiked at the quantitation limit, is used in the development of the MDLs. MDL verification samples are analyzed at ½ the target value of the MDL study. The SOP for MDL development has been provided in Appendix A of this QAPP Addendum and includes formulas for calculating analytical sensitivity. MDL studies are conducted on an annual basis, a standard in the laboratory industry. Since MDLs may nominally change throughout the duration of the project, USFWS will be informed via the QCSR if changes in MDLs impact the reporting limits (RL).

The level of quality control (QC) effort provided by the laboratory will be Level IV, CLP-Like (or equivalent). Please note that CLP is simply referenced as such. SW-846 methods will be used.

4.0 Analytical Method

4.1.1 White Phosphorus Analysis by 7580

The instrument used for analyses of White Phosphorus (P4) is a Gas Chromatograph with a Flame Photometric Detector (GC/FPD) with a phosphorus lens. The concentration of P4 is calculated using peak area (or height) with an external standard calibration procedure. Six standards of varying concentration (five for linear fit) are analyzed to tabulate a peak area response versus the concentration in the standard. The results are used to prepare a quadratic or linear calibration curve. The initial calibration acceptance criterion using linear curve fitting is that the correlation coefficient ® must be equal to or greater than 0.99 or if using a quadratic curve fitting is that the coefficient of determination (COD) must be greater than or equal to 0.99. The instrument calibration is verified at the beginning and end of each sequence and after ten samples. The acceptance criteria for the continuing calibration verification (CCV) is 15% of target value.

5.0 Quantitation Limits and ESV'S

Method detection limits, reporting limits, and ecological benchmarks can be found in this QAPP Addendum in Table 1.0.

6.0 Laboratory Data Reduction, Verification, and Reporting

Data reduction, verification, and reporting are accomplished though extensive use of a Laboratory Information Management System (LIMS). The DCL LIMS is a commercial automated data handling system that incorporates a relational database with additional custom programming to interface with laboratory instruments and produce reports required by DCL clients. It is maintained by the DCL computer support staff and updated as necessary.

6.1 Data Reduction

Data reduction consists of identifying the pertinent set of calibration standards, specifying the type of calibration to use, and calculating analytical results from the calibration equation. The actual calculations are performed by either the instrument software or the DCL LIMS after the transfer of the raw data to the system. Linear calibrations of the use of response factors are preferred for the reduction of data. DCL policy is to utilize the simplest appropriate equation that produces a good fit with the data.

6.2 Ensuring Accuracy of Calculations and Transcriptions

All of the software used for data reduction, verification, and reporting is documented and validated by the DCL computer support staff according to the following SOPs: DCL SOPs LAB-101, "Computer Program Testing", LAB-102 "Computer Programs Documentation". A continuing effort is made to increase the use of automated data handling, improve efficiency, and minimize human error.

Along with the use of the LIMS for data reduction and handling, DCL also employs a peer review system to ensure quality of analytical reports. Peer review procedures are specified in DCL SOP XX-DC-023 "Peer Review". An analyst familiar with the analytical method used to produce the results (peer reviewer), reviews each report. The peer reviewer verifies that the calibration standards, type of calibration, and sample set with associated QC samples were selected correctly. The peer reviewer also verifies any manual transcriptions and calculations.

6.3 Verification of Quality Control

The analyst is responsible to evaluate the QC results (method blank, surrogate recovery, LCS, matrix spike, and duplicate results). The peer reviewer is responsible to verify that QC results have been evaluated correctly and that necessary actions have been taken. Peer review procedures are specified in the DCL SOP XX-DC-023 "Peer Review". The peer review is considered complete when all issues raised by the peer reviewer have been resolved.

6.4 Reporting

When the peer review is completed, a report is generated. The reports that are generated by DCL meet the following requirements:

- The report identifies the method used. If the method is modified, it is noted as "modified" in the report.
- Any abnormal sample conditions such as deviations from hold time, irregularities in preservation, or other situations that might affect the analytical results.
- The contents of the report shall include:
 - 1. The report title with the name, address, and telephone number of the laboratory.
 - 2. The name of the client or project and the client identification number.
 - 3. Description and laboratory identification number.
 - 4. The dates of the sample collection, sample receipt, sample preparation, and analysis.
 - 5. The time of sample preparation and/or analysis if the required hold time for either activity of 48 hours or less.
 - 6. A method identifier for each method, including methods for preparation steps

- 7. The MDL of minimum reporting limit for the analytical results.
- 8. The analytical results with qualifiers as required.
- 9. A description of any quality control failures and deviations from the accepted method.
- 10. The signature and title of the individual(s) who accept responsibility for the content of the report.
- 11. The date the report is issued.
- 12. Clear identification of any results generated by a subcontract laboratory.
- 13. Page numbers and total number of pages.

The DCL Project Manager will review final reports for compliance with client requirements.

- 1. Case Narrative:
- Any deviations from intended analytical strategy
- Laboratory lot number/sample delivery group (SDG)
- Numbers of samples and respective matrices
- QC procedures utilized and also references to the acceptance criteria
- Laboratory report contents
- Project name and number
- Condition of samples 'as-received'
- Discussion of whether or not sample holding times were met
- Discussion of technical problems or other observations which may have created analytical difficulties
- Discussion of any laboratory QC checks which failed to meet project criteria
- Signature of the QA Manager
- 2. Chemistry Data Package
- Case narrative for each analyzed batch of samples
- Summary page indicating dates of analyses for samples and laboratory QC checks
- Cross referencing of laboratory sample to project sample identification numbers
- Data qualifiers to be used should be adequately described
- Sample preparation and analyses for samples

- Sample results
- Raw data for sample results and laboratory QC samples
- Results of (dated) initial and continuing calibration checks, and gas chromatography mass spectrometer (GC/MS) tuning results
- MS and MS duplicate recoveries, laboratory control samples, method blank results, calibration check compound, and system performance check compound results
- Labeled (and dated) chromatograms/spectra of sample results and laboratory QC checks
- Preparation factors and logbook notations

The laboratory shall also prepare and verify an electronic data deliverable (EDD). The format of the EDD shall be in the approved Region 5 format.

a. For this investigation, DCL will provide a standard or 21-day turn-around-time for the analytical data package and EDD. The 21-day timeframe begins the day the DCL receives a given sample for analysis.

7.0 **PREVENTIVE MAINTENANCE**

Preventative Maintenance Procedure

7.1 For Preventative Maintenance information, please see Section 5.3 of the DCL QA Manual found in Appendix C and the corresponding SOP which is found in Appendix A of this QAPP Addendum.

8.0 SAMPLING PROCEDURES

The sampling procedures to be used in this investigation have been selected to achieve the goals of the data quality objectives outlined in Section 1 of the QAPP. The RI/FS Work Plan and Field Sampling Plan (FSP) outline all the sampling procedure information.

8.2 Sample Containers, Sample Preservation and Maximum Holding Times

Samples collected for analysis will be contained and preserved in accordance with USEPA approved procedures.

All sample containers used for sample collection and analysis for this project will be prepared according to the procedures contained in the USEPA document, Specifications and Guidance for Obtaining Contaminant-Free Sample Containers, dated December 1992. This document specifies the acceptable types of containers, the specific cleaning procedures to be used before samples are collected, and QA/QC requirements relevant to the containers and cleaning procedures. DCL and/or Severn Trent Laboratories (STL) will supply all sample containers utilized for this investigation. If field personnel observe any cracked, dirty, or the appropriate preservative missing in the sample bottles, those bottles will be discarded and DCL and/or STL will be notified of the problem to prevent its re-occurrence.

8.2.1 Sample Containers for Solid Matrix

Solid matrix samples for this investigation include soils and sediments. These samples will be submitted to DCL for the following analyses: White Phosphorous

a. Solid samples for White Phosphorus are placed into a 4 oz. glass jar with as little head space as possible and preserved at 4 degrees C+2 degrees C, with a 30-day holding time before extraction and 5 days thereafter for analyses.

8.2.3 Sample Bottle Decontamination

Sample bottles will be decontaminated in accordance with the USEPA document "Specifications and Guidance for Obtaining Contaminant-Free Sample Containers, December 1992".

8.3 Sample Handling, Packaging and Shipment

The sampling team will assist the ENTRIX and/or Conestoga Rovers & Associates (CRA) Field Supervisor with the preparation of samples being run for White Phosphorous to be shipped to DCL. Following sample collection, the exterior of the sample containers will be decontaminated near the sampling location. Sample documentation and packaging will be performed in accordance with the procedures outlined in A Compendium of Superfund Field Operations Methods (USEPA, 1988). Samples will be packaged for shipment as outlined in the FSP.

8.4 Change in Scope to Dioxin/Furan Compound List in QAPP.

The compound list that will be analyzed and reported by the West Sacramento STL laboratory will include the compounds found in Tables 2.0, 3.0, and 4.0 of this QAPP Addendum.

Tables

Table 1 Method Detection and Reporting Limits for White Phosphorous in a Solid Matrix Crab Orchard National Wildlife Refuge NPL Site - AUS OU

Solid:

		Method	
General Chemistry	Units	Detection Limits ¹	Reporting Limits ¹
White Phosphorous	ug/kg	0.0721	0.5

ug/kg = micrograms per kilograms

¹Method Detection Limits and Reporting Limits based on Data Chem Laboratories results

Table 2

Ecological Benchmarks, Method Detection Limits and Reporting Limits for Dioxins in a Solid (Non-Sediment) Matrix Crab Orchard National Wildlife Refuge NPL Site - AUS OU

Solia (Noli-Seullient)				
	¹ Method	¹ D	² COPEC	² COPEC
	Detection	Reporting	Screening	Screening
	Limits	Limits	Direct	Ingestion
Constituent (Method SW846 8290)	(ng/kg)	(ng/kg)	(ng/kg)	(ng/kg)
2,3,7,8-TCDD	0.5	1	5000000	0.805000000
1,2,3,7,8-PeCDD	2.5	5	N/A	N/A
1,2,3,4,7,8-HxCDD	2.5	5	N/A	N/A
1,2,3,6,7,8-HxCDD	2.5	5	N/A	N/A
1,2,3,7,8,9-HxCDD	2.5	5	N/A	N/A
1,2,3,4,6,7,8-HpCDD	2.5	5	N/A	N/A
OCDD	5	10	N/A	N/A
2,3,7,8TCDF	0.5	1	N/A	N/A
1,2,3,7,8-PeCDF	2.5	5	N/A	N/A
2,3,4,7,8-PeCDF	2.5	5	N/A	N/A
1,2,3,4,7,8-HxCDF	2.5	5	N/A	N/A
1,2,3,6,7,8-HxCDF	2.5	5	N/A	N/A
2,3,4,6,7,8-HxCDF	2.5	5	N/A	N/A
1,2,3,7,8,9-HxCDF	2.5	5	N/A	N/A
1,2,3,4,6,7,8-HpCDF	2.5	5	N/A	N/A
1,2,3,4,7,8,9-HpCDF	2.5	5	N/A	N/A
OCDF	5	10	N/A	N/A

Solid (Non-Sediment)

mg/kg - milligrams per kilogram

NA - Not applicable

¹Method Detection Limits and Reporting Limits based on Severn Trent Laboratories results.

²Benchmark values derived from URS DRAFT Problem Formulation Document (August, 2005).

Table 3 Ecological Benchmarks, Method Detections Limits and Reporting Limits for Dioxins in a Sediment Matrix Crab Orchard National Wildlife Refuge NPL Site - AUS OU

Sediment

	¹ Method		² COPEC	² COPEC
	Detection	¹ Reporting	Screening	Screening
	Limits	Limits	Direct	Ingestion
Constituent (Method SW846 8290)	(ng/kg)	(ng/kg)	(ng/kg)	(ng/kg)
2,3,7,8-TCDD	0.5	1	5000000	NC,b
1,2,3,7,8-PeCDD	2.5	5	N/A	N/A
1,2,3,4,7,8-HxCDD	2.5	5	N/A	N/A
1,2,3,6,7,8-HxCDD	2.5	5	N/A	N/A
1,2,3,7,8,9-HxCDD	2.5	5	N/A	N/A
1,2,3,4,6,7,8-HpCDD	2.5	5	N/A	N/A
OCDD	5	10	N/A	N/A
2,3,7,8TCDF	0.5	1	N/A	N/A
1,2,3,7,8-PeCDF	2.5	5	N/A	N/A
2,3,4,7,8-PeCDF	2.5	5	N/A	N/A
1,2,3,4,7,8-HxCDF	2.5	5	N/A	N/A
1,2,3,6,7,8-HxCDF	2.5	5	N/A	N/A
2,3,4,6,7,8-HxCDF	2.5	5	N/A	N/A
1,2,3,7,8,9-HxCDF	2.5	5	N/A	N/A
1,2,3,4,6,7,8-HpCDF	2.5	5	N/A	N/A
1,2,3,4,7,8,9-HpCDF	2.5	5	N/A	N/A
OCDF	5	10	N/A	N/A

mg/kg - milligrams per kilogram

NA - Not applicable

NC,b - Ingestion screening value is not calculated, however, is automatically considered a COPEC if decided based upon bioaccumulation potential.

¹Method Detection Limits and Reporting Limits based on Severn Trent Laboratories results.

²Benchmark values derived from URS DRAFT Problem Formulation Document (August, 2005).

Table 4 Ecological Benchmarks, Method Detections Limits and Reporting Limits for Dioxins in an Aqueous Matrix Crab Orchard National Wildlife Refuge NPL Site - AUS OU

Aqueous

	¹ Method		² COPEC	
	Detection	¹ Reporting	Screening	² COPEC
	Limits	Limits	Direct	Screening
Constituent (Method SW846 8290)	(pg/L)	(pg/L)	(pg/L)	Ingestion (pg/L)
2,3,7,8-TCDD	5	10	500000	NC,b
1,2,3,7,8-PeCDD	25	50	N/A	N/A
1,2,3,4,7,8-HxCDD	25	50	N/A	N/A
1,2,3,6,7,8-HxCDD	25	50	N/A	N/A
1,2,3,7,8,9-HxCDD	25	50	N/A	N/A
1,2,3,4,6,7,8-HpCDD	25	50	N/A	N/A
OCDD	50	100	N/A	N/A
2,3,7,8TCDF	5	10	N/A	N/A
1,2,3,7,8-PeCDF	25	50	N/A	N/A
2,3,4,7,8-PeCDF	25	50	N/A	N/A
1,2,3,4,7,8-HxCDF	25	50	N/A	N/A
1,2,3,6,7,8-HxCDF	25	50	N/A	N/A
2,3,4,6,7,8-HxCDF	25	50	N/A	N/A
1,2,3,7,8,9-HxCDF	25	50	N/A	N/A
1,2,3,4,6,7,8-HpCDF	25	50	N/A	N/A
1,2,3,4,7,8,9-HpCDF	25	50	N/A	N/A
OCDF	50	100	N/A	N/A

ug/L = micrograms per liter

NA - Not applicable

NC,b - Ingestion screening value is not calculated, however, is automatically considered a COPEC if decided based upon bioaccumulation potential.

¹Method Detection Limits and Reporting Limits based on Severn Trent Laboratories results.

²Benchmark values derived from URS DRAFT Problem Formulation Document (August, 2005).

Figures



Figure 2.1 - Project Organization Chart Rev: 1-2007

Appendix A

Laboratory SOPs

LIST OF SOPs

SOP023	Peer Review
SOP101	Computer Program Testing
SOP102	Computer Program Documentation
SOP7580	Analytical Determination of White Phosphorous (P4) using EPA Method 7580
SOP MDL	Calculation of Method Detection Limit
SOP 001 Sample Receiving	Sample Receipt and Log-in (Environmental)
SOP LAB002	Preventive Maintenance for Analytical Instrumentation

DATACHEM LABORATORIES, INC.

STANDARD OPERATING PROCEDURE APPROVAL SHEET

SOP TITLE: Peer Review

DOCUMENT CONTROL NUMBER: XX-DC-023 – Revision 3

EFFECTIVE DATE: April 15, 2003

APPROVALS:

MANAGER	Date
QA MANAGER	 Date
LAB DIRECTOR	Date

STANDARD OPERATING PROCEDURE

PEER REVIEW

1.0 SCOPE AND APPLICATION

- 1.1 This Standard Operating Procedure (SOP) summarizes the peer review process used by DataChem Laboratories, Inc. (DCL) to verify the accuracy of data generated by the laboratory. These procedures are used by those individuals responsible for peer review of data.
- 1.2 This SOP process does not supersede any client, method, or other SOP QC requirements. Criteria listed in this SOP shall be used when method, SOP, or client criteria are unavailable.
- 1.3 This SOP does not include any data package review. Refer to SOP XX-DC-020, "Deliverable and Data Package Preparation and Review," or the specific project protocol worksheet (PPW).

2.0 **RESPONSIBILITY**

- 2.1 Technical operations managers are responsible to train reviewers to the procedures outlined in this SOP.
- 2.2 It is the responsibility of the peer reviewer to check data in accordance with Section 4.0 and 5.0 of the SOP. Upon signature of the report, the peer reviewer certifies compliance with this SOP. It is also the reviewer's responsibility to ensure that appropriate evaluation criteria are used as defined in the PPW. The evaluation criteria are prioritized as follows:
 - 2.2.1 As defined by the client or regulation or program.
 - 2.2.2 As defined by a published or promulgated method.
 - 2.2.3 As defined by the DCL method SOP or default procedures.

3.0 DEFINITIONS

- 3.1 Peer Review is defined as a checking procedure by a peer chemist or analyst who is knowledgeable concerning the analytical requirements of a specific method.
- 3.2 Acronyms

CCB	=	Continuing Calibration Blank
CCC	=	Calibration Check Compounds
CCV	=	Continuing Calibration Verification
COC	=	Chain of Custody

DataChem Laboratories, Inc. XX-DC-023 – Revision 3 Revised: April 15, 2003 Page 2 of 10

GC	=	Gas Chromatography
GC/MS	=	Gas Chromatography/Mass Spectrometry
HPLC	=	High Performance Liquid Chromatography
ICB	=	Initial Calibration Blank
ICV	=	Initial Calibration Verification
ICP	=	Inductively Coupled Plasma
LCS	=	Laboratory Control Sample
LOD	=	Limit of Detection
LOQ	=	Limit of Quantitation
MD	=	Matrix Duplicate
MDL	=	Method Detection Limit
MS	=	Matrix Spike
MSD	=	Matrix Spike Duplicate
PPW	=	Project Protocol Worksheet
PQL	=	Practical Quantitation Limit
SPCC	=	System Performance Check Compounds
QC	=	Quality Control

4.0 GENERAL PROCEDURES

- 4.1 The following procedures shall be used to review all data prior to submission of the final report. For each operational section, specific review procedures are listed in Sections 5.1 to 5.6 of this SOP.
 - 4.1.1 Method performed with modifications/deviations noted.
 - 4.1.2 Instructions in Project Protocol Worksheet (PPW) are followed.
 - 4.1.3 Analytical Report form completed and signed by analyst.
 - 4.1.4 Notebooks reviewed and signed.
 - 4.1.4.1 Sample Preparation/Extraction Logs
 - 4.1.4.2 Standards Logs
 - 4.1.4.3 Instrument Logs
 - 4.1.5 LOD/LOQ entered and meets the requirements of the client.
 - 4.1.6 Instrument QC in compliance with deviations noted.
 - 4.1.6.1 Calibration, CCV, ICV, CCC, SPCC, and Internal Standards
 - 4.1.6.2 GC/MS Tuning
 - 4.1.7 Method QC in compliance with deviations noted.

- 4.1.7.1 Method Blanks, Laboratory Control Samples, Surrogates, and Tracers
- 4.1.8 Matrix QC in compliance with deviations noted.
 - 4.1.8.1 Matrix Duplicate
 - 4.1.8.2 Matrix Spike and Matrix Spike Duplicate
- 4.1.9 Hold times met.
- 4.1.10 Solutions and standards expiration dates checked.
- 4.1.11 Units and conversions accurately assigned and clearly defined.
- 4.1.12 Sample Results:
 - 4.1.12.1 Example calculations provided and checked.
 - 4.1.12.2 Relative Retention Times checked.
 - 4.1.12.3 Confirmation analysis run.
 - 4.1.12.4 Manual integrations checked.
- 4.1.13 Internal COC is complete.

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5.0 OPERATIONAL SECTION PROCEDURES

5.1 Inorganic Chemistry Technical Peer Review

- Solutions standardized if required by method
- Calibration standards analyzed
- Standards traceability checked and meets criteria
- Standard curve coefficient evaluated
- ICVs analyzed and meet acceptance criteria
- CCVs analyzed and meet acceptance criteria
- ICBs, CCBs analyzed and meet acceptance criteria
- CCB/CCV frequency met
- Method/Preparation Blanks analyzed and meet acceptance criteria when performed
- MSs, MSDs, and/or MDs analyzed and meet acceptance criteria when performed
- LCSs analyzed and meet acceptance criteria when performed
- Method deviations and reanalysis noted when performed
- Preparation and analysis hold times met
- Dilution factors noted
- Notebook pages transcription accuracy and completeness checked
- Calculations checked
- Report forms are complete and accurate
- MDLs/PQLs entered and meet requirements
- Internal COC completed

5.2 Atomic Absorption Spectroscopy Technical Peer Review

- Calibration standards analyzed and checked
- Standards traceability checked
- Standard curve coefficient evaluated and meets criteria
- ICVs analyzed and meet acceptance criteria
- CCVs analyzed and meet acceptance criteria
- ICBs, CCBs analyzed and meet acceptance criteria
- CCB/CCV frequency met
- Method preparation blanks analyzed and meet acceptance criteria
- MSs, MSDs, and MDs analyzed and calculations checked; applicable action based on recoveries has been taken.
- LCSs analyzed and meet acceptance criteria
- Method QC recoveries meet acceptance criteria for those performed.
- Method deviations and reanalysis noted when performed
- Preparation and analysis hold times met
- Sample dilution factors noted on reports
- Notebook pages transcription accuracy and completeness checked
- Preparation and analysis calculations checked
- Report forms are complete and accurate.
- Precision of injections checked
- Reanalysis checked, documented, and reported
- MDLs/PQLs entered and meet requirements
- Internal COC completed

5.3 ICP Technical Peer Review

- Calibration standards analyzed and checked
- Standards traceability checked
- Standard curve coefficient evaluated and meets criteria
- ICVs analyzed and meet acceptance criteria
- CCVs analyzed and meet acceptance criteria
- ICBs, CCBs analyzed and meet acceptance criteria
- CCB/CCV frequency met
- Method preparation blanks analyzed and meet acceptance criteria
- MSs, MSDs, and MDs analyzed and calculations checked; applicable action based on recoveries has been taken.
- LCSs analyzed and meet acceptance criteria
- Method QC recoveries meet acceptance criteria for those performed
- Method deviations and reanalysis noted when performed
- Preparation and analysis hold times met
- Sample dilution factors noted on reports
- Notebook pages transcription accuracy and completeness checked
- Preparation and analysis calculations checked
- Report forms are complete and accurate.
- Precision of injections checked
- Reanalysis checked, documented, and reported
- Check dilutions for interferences
- Check Serial Dilutions
- MDLs/PQLs entered and meet requirements
- Internal COC completed

5.4 Ion Chromatography Technical Peer Review

- Calibration Standards analyzed
- Standards traceability checked
- Standard curve coefficient evaluated and meets criteria
- ICVs analyzed and meet acceptance criteria
- CCVs analyzed and meet acceptance criteria
- ICBs, CCBs analyzed and meet acceptance criteria
- Retention Time Windows checked
- CCB/CCV frequency met
- Method preparation blanks analyzed and meet acceptance criteria
- MSs, MSDs, MDs analyzed and meet acceptance criteria when performed
- LCSs analyzed and meet acceptance criteria
- Method deviations and reanalysis noted when performed
- Preparation and analysis hold times met
- Dilution factors noted
- Notebook pages transcription accuracy and completeness checked
- Preparation and analysis calculations checked
- Report forms are complete and accurate.
- MDLs/PQLs entered and meet requirements
- Internal COC completed
- Manual integrations checked

5.5 Chromatography (GC and HPLC) Technical Peer Review

- Calibration Standards analyzed
- Standards traceability checked
- Initial Calibration within method or project criteria
- ICVs analyzed and meet acceptance criteria
- CCVs analyzed and meet acceptance criteria
- Method Blanks analyzed and meets acceptance criteria
- Retention Time Windows checked
- For method 8081A, Endrin/DDT Breakdown is checked for compliance
- Surrogate recoveries checked and appropriately addressed
- All samples bracketed by valid CCV
- MS, MSD, MD recoveries checked and appropriately addressed
- LCSs analyzed and meet acceptance criteria
- Analysis deviations and reanalysis noted when performed
- Preparation and analysis hold times met
- Dilution factors noted
- Notebook pages and spreadsheets transcription accuracy and completeness checked
- Preparation and analysis calculations checked
- Report forms are complete and accurate
- Preparation deviations and repreparations noted when performed
- MDLs/PQLs entered and meet requirements
- Internal COC completed
- Manual integrations checked

5.6 GC/MS Technical Peer Review

- GC/MS Tuning passed criteria (BFB or DFTPP)
- Standards traceability checked
- Initial Calibration passed criteria
- Continuing Calibration passes criteria
- Method Blanks analyzed and meets acceptance criteria
- Review of spectral assignments
- Relative Retention Time checked
- Internal Standards checked
- Surrogate recoveries checked when performed
- Sample Frequency within 12 hours of successful tune
- Method preparation blanks analyzed and meet acceptance criteria
- MSs and MSDs analyzed and meet acceptance criteria
- LCSs analyzed and meet acceptance criteria
- Method deviations and reanalysis noted when performed
- Preparation and analysis hold times met
- Dilution factors noted
- Notebook pages and spreadsheets transcription accuracy and completeness checked
- Preparation and analysis calculations checked
- Report forms are complete and accurate.
- Preparation deviations and repreparations noted when performed
- MDLs/PQLs entered and meet requirements
- Internal COC completed
- Manual integrations checked
6.0 **REFERENCES**

- 6.1 DCL SOP XX-DC-020, "Deliverable and Data Package Preparation and Review."
- 6.2 DCL SOP XX-DC-006, "Chain of Custody and Laboratory Tracking."
- 6.3 DCL SOP QC-DC-006, "Nonconformance/Corrective Action Report (NC/CAR) Procedures."
- 6.4 DCL SOP XX-DC-019, "Standards Purity, Preparation, Traceability, and Verification."

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DATACHEM LABORATORIES, INC.

STANDARD OPERATING PROCEDURE APPROVAL SHEET

SOP TITLE:	Computer Program Testing				
DOCUMENT CONTR	OL NUMBER: _		LAB-101		
EFFECTIVE DATE: _		February 15, 20	02		
APPROVALS:					
MANAGER				Date	
QA MANAGER				Date	
LAB DIRECTOR				Date	

STANDARD OPERATING PROCEDURE

COMPUTER PROGRAM TESTING

1.0 INTRODUCTION

1.1 Computer programs utilized by DataChem Laboratories (DCL) must be tested and verified to document that the program functions as designed and that no anomalies to the designed output will be generated.

2.0 SCOPE

2.1 This Standard Operating Procedure (SOP) document defines procedures and requirements for testing computer programs used at DCL for the management, manipulation, and reporting of critical data.

3.0 APPLICATION

- 3.1 The test and verification procedures presented in this document apply to computer programs and modules used at DCL for the following:
 - 3.1.1 Generation, manipulation, and recording of analytical data and invoicing information
 - 3.1.2 Entry of critical sample tracking information used in the generation of worklists, analytical sequences, report forms and other procedures which impact item 3.1.1. Critical information includes:
 - 3.1.2.1 Customer
 - 3.1.2.2 Customer sample identification
 - 3.1.2.3 Assigned laboratory numbers
 - 3.1.2.4 Date of receipt
- 3.2 The test verification procedures presented in this document do not apply to the following types of computer programs:
 - 3.2.1 Programs used for generating management reports and displays
 - 3.2.2 Programs used to enter, track, and report informational data
- 3.3 For the purpose of this document, the term computer program includes independent program modules, independent computer programs, and computer programs integrated through program management systems.

DataChem Laboratories, Inc. LAB-101 Date: February 15, 2002 Page 2 of 5

4.0 TEST REQUIREMENTS

- 4.1 Test requirements and the criteria for the acceptance of test results shall be specified as part of the initial design of the computer program or module. These requirements and criteria shall be outlined by Computer Support and approved by the organization(s) requesting and/or using the computer program.
- 4.2 Tests will include, as appropriate, the following:
 - 4.2.1 Verification tests
 - 4.2.2 Hardware integration tests
 - 4.2.3 In-use test
- 4.3 All tests will be performed in a controlled environment using inputs and outputs applicable to the design and specifications of the program. Models, methods, and assumptions used to test the software shall be identified and documented by the individuals assigned the responsibility of testing.
- 4.4 Software verification and hardware integration testing shall be performed by qualified members of the DCL staff who were not involved in the development of the software and who do not report directly to the DCL manager responsible for the development of the software.
- 4.5 Final approval of test results is the responsibility of Quality Assurance personnel. Assistance from other organizations within DCL may be requested in order to ensure that acceptance criteria have been satisfied.

5.0 VERIFICATION TESTS

- 5.1 Verification tests shall be designed to document the capability of the computer program to generate the correct result for any input within the range of the design specifications for the program.
 - 5.1.1 Single program modules shall be tested to ensure that the inputs to the module produce the specified output. When feasible, error-trapping routines should exclude inputs outside the design specification.
 - 5.1.2 Complex programs might require a series of tests which include, as appropriate:
 - 5.1.2.1 Testing of individual program modules
 - 5.1.2.2 Tests to verify proper communication between program modules
 - 5.1.2.3 Testing of preliminary program versions
 - 5.1.2.4 Testing of final program version

DataChem Laboratories, Inc. LAB-101 Date: February 15, 2002 Page 3 of 5

5.2 Regardless of the complexity of the program or the number of testing phases, verification testing shall demonstrate that the program produces the required results under the constraints of the design specification.

6.0 HARDWARE INTEGRATION TESTS

6.1 Hardware integration tests shall be designed to document the capability of the program to accept inputs from or provide outputs to the specified hardware. As with verification testing, the program (and modules) shall be tested against the range of specified inputs and outputs. Preliminary testing can be performed using inputs or outputs by a proxy rather than the actual hardware item; however, final testing must be performed using the actual hardware to be integrated.

7.0 IN-USE TESTS

- 7.1 Upon completion and acceptance of verification and integration testing, the computer program will be placed into service. Test problems and associated correct results that demonstrate acceptable functionality of the computer program shall be developed and documented. These test problems shall be run whenever a significant change in the operating environment occurs, such as:
 - 7.1.1 Installation on a computer system other than the system(s) specified in the design specification
 - 7.1.2 Significant revision of the operating system
 - 7.1.3 Significant changes in hardware
- 7.2 Applications that are subject to failures or drift that could affect performance shall be subjected to tests to verify proper functioning of the system. These tests shall be performed in accordance with a schedule specified in the design specifications.

8.0 TEST PROCEDURES

- 8.1 Prior to program development, test procedures or plans shall be specified. These procedures or plans may be modified during program development with the concurrence of the requesting organization.
- 8.2 Test procedures and plans shall include the following items, as applicable:
 - 8.2.1 Required tests and test sequences
 - 8.2.2 Identification of phases where testing shall occur
 - 8.2.3 Designed ranges of inputs
 - 8.2.4 Anticipated outputs for a given set of inputs
 - 8.2.5 Requirements and procedures for testing logic branches

DataChem Laboratories, Inc. LAB-101 Date: February 15, 2002 Page 4 of 5

- 8.2.6 Requirements for integration of hardware
- 8.2.7 Acceptance criteria for a given test
- 8.2.8 Reports, records, formatting, and conventions used to document the test

9.0 TEST RESULTS

- 9.1 All required tests shall be documented. Preliminary tests used as part of program development do not require documentation.
- 9.2 Verification tests shall be evaluated by Quality Assurance personnel with the assistance of other personnel as appropriate.

10.0 TEST RECORDS

- 10.1 Verification and hardware integration tests shall be recorded on form DCL-COM-3, "DCL Computer Program Modification/Verification," which is attached to DCL SOP LAB-103, "Computer Software Control." Additional testing specifications, test problem listings, data, and results should be attached to form DCL-COM-3. The data that shall be recorded on the form are:
 - 10.1.1 Date of test
 - 10.1.2 Computer program tested
 - 10.1.3 Computer hardware tested
 - 10.1.4 Other hardware pertinent to the test
 - 10.1.5 Test equipment and calibrations, where applicable
 - 10.1.6 Individual(s) performing the test
 - 10.1.7 Test problems performed
 - 10.1.8 Results from test problems
 - 10.1.9 Any deviations from anticipated results or acceptance criteria and action taken as a result of the deviation
 - 10.1.10 Individual(s) evaluating the test results
- 10.2 In-use test records shall document the following:
 - 10.2.1 Date of test
 - 10.2.2 Computer program tested
 - 10.2.3 Computer hardware tested

DataChem Laboratories, Inc. LAB-101 Date: February 15, 2002 Page 5 of 5

- 10.2.4 Other hardware pertinent to the test
- 10.2.5 Test equipment and calibrations, where applicable
- 10.2.6 Individual(s) or system performing the test

11.0 COMMERCIAL SOFTWARE

- 11.1 Software obtained from outside DCL is not directly subject to the testing specified in this document. It is anticipated that the supplier has completed testing of the product in accordance with a similar test program. If possible, a letter attesting to the fact that computer program testing has been performed shall be obtained from the supplier.
- 11.2 In-use tests and procedures specified by the supplier shall be performed in accordance with a predetermined schedule.
- 11.3 When appropriate, in-use testing procedures shall be developed by DCL to verify that the program produces the anticipated results from a standardized set of inputs.

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DATACHEM LABORATORIES, INC.

STANDARD OPERATING PROCEDURE APPROVAL SHEET

SOP TITLE: Computer Program Documentation

DOCUMENT CONTROL NUMBER: LAB-102

EFFECTIVE DATE: _____ February 15, 2002

APPROVALS:

MANAGER	 Date
QA MANAGER	 Date
LAB DIRECTOR	Date

STANDARD OPERATING PROCEDURE

COMPUTER PROGRAM DOCUMENTATION

1.0 INTRODUCTION

1.1 Computer programs and modules are generally complex files of code written in specific computer languages. Though the code itself is the only complete documentation of the functionality of the program, it is desirable to provide documentation, both internal and external to the code, which describes the computer program and its purpose, as well as that of program modules and segments.

2.0 SCOPE

2.1 This document describes procedures for documenting computer programs and code developed by DataChem Laboratories (DCL).

3.0 APPLICATION

- 3.1 The computer program documentation procedures described in this document are applicable to all computer program systems, computer programs, and modules developed by DCL.
- 3.2 Computer programs supplied by sources outside DCL, which function as part of a computer program system, are subject to the procedures for documenting the purpose of the program.
- 3.3 Regardless of the following exclusions, all computer programs that have the potential of generating, deleting, or modifying critical data shall be documented.
- 3.4 These procedures do not apply to commercial software that does not function in conjunction with DCL-developed programs.
- 3.5 These procedures do not apply to DCL-developed software that is written for "one-time" use, such as unique reports.
- 3.6 These procedures do not apply to DCL-developed software that is intended only for the use of the writer, and not for distribution to other individuals or organizations within DCL.

4.0 PROGRAM DOCUMENTATION

- 4.1 Each computer program system, computer program, or general use module shall be documented external to the actual code files. As a minimum, this documentation shall include the following:
 - 4.1.1 Title of computer program system, program, or module
 - 4.1.2 Individual responsible for overseeing the development
 - 4.1.3 Brief statement of the purpose of the development

DataChem Laboratories, Inc. LAB-102 Date: February 15, 2002 Page 2 of 3

- 4.1.4 Requesting organization or individual
- 4.1.5 Specific reference to the programming request
- 4.1.6 Date development began
- 4.1.7 Individual assignments in the development effort
- 4.1.8 Date development completed
- 4.1.9 Version number of the main program and version number(s) and title(s) of subordinate programs or modules
- 4.1.10 Reference to Computer Program testing test requirements and acceptance criteria (DCL SOP LAB-101)
- 4.1.11 Results of Computer Program Testing (DCL SOP LAB-101)
- 4.2 Documentation shall be maintained in an organized manner that allows rapid access and retrieval of information. Documentation may be maintained in computer files in addition to hard copy output. Hard copy output will be maintained under direction of the Computer Support manager and shall be available to DCL employees upon request. Documentation shall provide identification of the applicable software version and verification that no unauthorized changes have been made.
- 4.3 Individual computer programs and modules referenced in item 4.1.9 shall be individually documented under the provisions of this document.

5.0 PROGRAM CODE DOCUMENTATION

- 5.1 Each program or module written by DCL shall be documented within the code file.
- 5.2 Each separate code file shall have a header containing the following minimum information:
 - 5.2.1 Name of the file
 - 5.2.2 Title of the program or module
 - 5.2.3 Title and name of any parent file
 - 5.2.4 Title(s) and name(s) of any child file(s)
 - 5.2.5 Version number and date
 - 5.2.6 Programmer name
 - 5.2.7 Language
 - 5.2.8 Statement as to whether the file contains non-standard code

- 5.3 Comments shall be entered at the beginning of each major code segment, subrouting, or similar structure, which identify the purpose of the segment. Additional comments that clarify the purpose of the segment, the approach taken to code the segment, or other information that may assist another individual to interpret the code should be included.
- 5.4 Within each code segment comments shall be added to clarify the operation of individual lines of code that may be ambiguous, identify non-standard code, or provide other guidance to an individual reading the code.

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DATACHEM LABORATORIES, INC.

STANDARD OPERATING PROCEDURE APPROVAL SHEET

SOP TITLE: _____Analytical Determination of White Phosphorus (P4) using EPA method 7580

DOCUMENT CONTROL NUMBER: OP-SW-7580 – Revision 1

EFFECTIVE DATE: ______ December 1, 2004

APPROVALS:	
MANAGER	 Date
QA MANAGER	 Date
LAB DIRECTOR	 Date

STANDARD OPERATING PROCEDURE

ANALYTICAL DETERMINATION USING EPA METHOD 7580

1.0 SCOPE AND APPLICATION

- 1.1 This SOP provides instructions for the determination of white phosphorus in water and sediment/soil samples.
- 1.2 White phosphorus in pure form is a colorless or white, transparent, crystalline solid that has been used in poisons, smoke screens, matches, and fireworks, and has been used as a raw material in the production of phosphoric acid. It has been used in smoke-producing munitions since World War I. White phosphorus is thermodynamically unstable in the presence of atmospheric oxygen. As a result, until recently, the prospect of long-term environmental contamination from smoke munitions was considered unlikely. However, a catastrophic die-off of waterfowl at a US military facility has been traced to the presence of P4 in salt marsh sediments, and lead to the realization that P4 can persist in anoxic sedimentary environments.
- 1.3 This SOP is based on a gas chromatographic procedure and is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatographic systems and skilled in the interpretation of the chromatograms and their use as a quantitative tool.

2.0 MODIFICATIONS FROM EPA METHOD 7580

2.1 No substantial modifications from the promulgated method have been made. Specific amounts and volumes are specified as well as actual columns and instrumentation utilized.

3.0 SUMMARY OF METHOD

- 3.1 A 30 mL water sample is extracted once with 3.0 mL isooctane. A 1.0-µL aliquot of the extract is analyzed by GC/FPD.
- 3.2 Wet soil or sediment samples are analyzed by extracting a 40 g wet-weight aliquot of the sample with a mixture of 10.0 mL of reagent water and 10.0 mL of isooctane. The extraction is performed in a glass jar on a shaker for 18 hours. A 1.0-µL aliquot of the extract is analyzed by GC/FPD.
- 3.3 The concentration of P4 in the extract is calculated using peak area (or height) and an external standard calibration procedure. The sample concentration is determined from the extract concentration using the final volume of the sample extract, sample volume (water samples) or sample weight (soils/sediments). Results from soils and sediments are reported on a wet weight basis.

4.0 SAFETY

4.1 P4 should be treated as a potential health hazard, and exposure should be minimized.

Each laboratory is responsible for maintaining awareness of OSHA regulations regarding safe handling of chemicals used in this method. Additional references to laboratory safety are available for the information of the analyst.

- 4.2 Because P4 will spontaneously combust in air, caution should be taken. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.
- 4.3 Refer to: DCL SOP LAB-005, "General Laboratory Safety and Chemical Hygiene" and the Safety Manual and Chemical Hygiene Plan of DataChem Laboratories (DCL).

5.0 INSTRUMENTATION AND EQUIPMENT

- 5.1 A gas chromatograph equipped with a flame photometric detector with a phosphorus lens, on column split/splitless injector, auto sampler, and TurboChrom Pro data system, or equivalent.
 - 5.1.1 Columns
 - 5.1.1.1 This method uses a 30-m x 0.53-mm ID with 1.5 μm film thickness DB-I. Other columns can be substituted if deemed necessary if the substitute columns meet the QC requirements of the Method.
 - 5.1.2 Instrument Parameters: The temperature program is set to facilitate adequate separation of all analytes. Gas flows are also set to facilitate separation and sensitivity. Temperature programs and flows depend on the actual column used. Suggested initial parameters are:
 - 5.1.2.1 Column flow 5 to 10 mL/min. using helium as the carrier gas. Detector flows- hydrogen 75 mL/min., air 90 mL/min.
 - 5.1.2.2 Temperatures:

Injectors – On column

Detectors - 250 °C

Oven - 80 °C for 6 minutes

- 5.1.2.3 Injection Volume approximately 2 μL splitless, open split valve at 45 seconds (0.75 minutes).
- 5.2 40 mL VOA vials with Teflon lined septa for water extractions.
- 5.3 120mL jars with Teflon lined lids for soil extractions.
- 5.4 Syringes: 10 µL thru 10 mL.
- 5.5 Vials: Glass 2-mL capacity with aluminum Teflon-lined crimp caps.
- 5.6 Platform shaker or equivalent.

- 5.7 Analytical balance.
- 5.8 Forceps for handling P4.
- 5.9 Razor blades or scalpels for cutting P4.

6.0 SAMPLING HANDLING REQUIREMENTS

- 6.1 All samples must be stored at 4 °C \pm 2 °C. The temperature must be checked daily and recorded.
- 6.2 Samples and extracts must not be stored with the analytical standards.
- 6.3 Water samples must be analyzed within 5 days of the date of collection. Soil samples must be extracted within 30 days.
- 6.4 Refer to: DCL SOP QS-DC-001, "Sample Receipt and Log-In (Environmental)" and the Environmental Quality Assurance Program Plan of DCL, Appendix 14.8, "Sample Preservation and Holding Times".

7.0 PRACTICAL QUANTITATION LIMITS (PQLs)

7.1 The practical quantitation limits for Method 7580 are 0.05 μ g/L for water and 0.5 μ g/kg for soil.

8.0 INTERFERENCES

8.1 No chromatographic interferences with this SOP have been observed, in part due to the selectivity of the flame photometric detector in the phosphorous mode.

9.0 **REAGENTS**

- 9.1 Solvents: Toluene and Isooctane, pesticide-grade or equivalent.
- 9.2 Reagent Water: ASTM Type II water free of interfering compounds.

10.0 STANDARDS

- 10.1 Stock Standard Solutions
 - 10.1.1 A stock solution is prepared by weighing an aliquot of white phosphorous (P4), transferring the P4 to a volumetric flask and diluting to volume in toluene. The P4 is transferred to the solvent quickly to minimize exposure to the air.
 - 10.1.2 Alternatively, commercially prepared stock standards or mixes may be used and can be at any concentration if they are certified by the manufacturer or by an independent source.
- 10.2 Intermediate Standards

- 10.2.1 Intermediate standards are prepared as needed through dilution in toluene of the stock standard solutions from section 10.1 above.
- 10.3 Working Standards
 - 10.3.1 Working standards at a minimum of five concentration levels for linear calibration (a minimum of six levels for quadratic fit) are prepared through dilution of the intermediate standards with isooctane. The concentration of the low standard must be at the concentration equal to the PQL. The remaining concentration levels correspond to the expected range of concentrations found in actual samples or define the working range of the GC.
- 10.4 Storage and Expiration
 - 10.4.1 All standards and spiking solutions must be stored in the dark at $4 \degree C \pm 2 \degree C$ in screw-cap bottles or test tubes.
 - 10.4.2 Concentrated Stock Solutions have an expiration date of <u>one year six months</u> from preparation. The vendor's expiration date may be used for purchased solutions.
 - 10.4.3 All intermediate solutions have an expiration period of up to six months from preparation, but must not exceed the expiration date of the parent solution from which the intermediate is prepared.
 - 10.4.4 Working standards have an expiration period of up to <u>six two</u> months but must not exceed the expiration of the intermediate or stock solutions from which the working standards are prepared.

11.0 CALIBRATION AND STANDARDIZATION

- 11.1 Calibration and Standardization
 - 11.1.1 Prepare and analyze a minimum of six standards of varying concentration (five for linear fit). One standard should contain the method analytes at the PQL for each compound. The standards should bracket the concentration range expected in samples.
 - 11.1.2 Analyze each calibration standard and tabulate peak area response versus the concentration in the standard. The results can be used to prepare a calibration curve for each compound.
- 11.2 Calibration Criteria
 - 11.2.1 An initial calibration curve is constructed by the TurboChrom data system for P4 using the concentration and peak areas (or peak heights) of the initial calibration standards at a minimum of six different concentration levels for quadratic and five for linear. The calibration curve is constructed using a linear or quadratic fit of the data.

- 11.2.2 The initial calibration acceptance criterion using linear curve fitting is that the correlation coefficient (r) must be equal to or greater than 0.99.
- 11.2.3 The initial calibration acceptance criterion using quadratic curve fitting is that the coefficient of determination (COD) must be greater than or equal to 0.99.
- 11.3 Continuing Calibration Verification (CCV)
 - 11.3.1 A CCV is analyzed at the beginning, and end of each sequence and after every ten samples.
 - 11.3.2 Acceptance criteria for the CCV is that the result for the CCV quantitated against the curve is within 15% of the target value.
 - 11.3.3 Samples must be bracketed with CCV standards that meet the 15% criterion.
- 11.4 Refer to: the Environmental Quality Assurance Program Plan of DCL, Appendix 14.7, "Summary of Calibration and Corrective Action".

12.0 SAMPLE PROCEDURE

- 12.1 Water
 - 12.1.1 Water samples must be extracted within five days of collection and analyzed within thirty days from preparation.
 - 12.1.2 Place 30 mL of water sample in a 40 mL vial, add 3.0 mL of isooctane and shake for five minutes.
 - 12.1.3 Transfer the isooctane extract to an autosampler vial with Teflon lined cap, for analysis.
- 12.2 Soil
 - 12.2.1 <u>Soil samples do not have an method established holding time. Samples are</u> <u>extracted within thirty days and analyzed within thirty days from preparation.</u> <u>must be extracted within fourteen days of collection and analyzed within 30 days</u> <u>from preparation.</u>
 - 12.2.2 Weigh out 40 grams of soil sample and transfer to a 120 mL amber jar with Teflon lined caps. Add 10 mL of water and 10mL of isooctane and shake for 18 hours.
 - 12.2.3 Transfer the isooctane extract to an autosampler vial with Teflon lined cap for analysis.
- 12.3 Analysis
 - 12.3.1 Inject 2 μ L of each standard, QC blank and field sample into the gas chromatograph.

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13.0 CALCULATIONS

13.1 Typically quadratic curve fitting is used for the initial calibration standards. This part of the procedure is identical for soil and water matrices. The general quadratic equation is:

 $y = instrument response = ax^2 + bx + c,$

where x is the concentration and a, b, and c are parameters determined by the best fit to the calibration data.

Note:
$$x = \left[\frac{-b \pm \sqrt{b^2 - 4a(c - y)}}{2a}\right]$$

13.2 The concentration of the sample is expressed in μ g/L for water samples:

Conc. Sample = $(D)(C_s)$

Where:

- D = Dilution factor, if dilution was made for the sample prior to analysis. If no dilution was made, D = 1; dimensionless.
- $C_s = Sample concentration in \mu g/L.$

14.0 QUALITY CONTROL

- 14.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of CCV, method blank, laboratory control sample LCS, matrix spike and matrix spike duplicate (MS/MSD), and to evaluate and document data quality. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.
 - 14.1.1 An MDL study will be performed annually.
 - 14.1.2 Each day of analysis, the analyst must analyze a method blank to demonstrate that interferences from the analytical system are under control before any samples are analyzed. In general, background interferences coeluting with method analytes should be below half the PQL.
 - 14.1.3 The laboratory must, on an ongoing basis, demonstrate through the analyses of laboratory control samples (LCS) that the operation of the measurement system is in control. The frequency of the LCS analyses is one for each sample batch of up to twenty field samples. The LCS is prepared by spiking the reference blank matrix with the matrix spiking solution and then extracting and analyzing as a sample.
 - 14.1.4 The LCS acceptance criterion is 75 to 125% recovery.

- 14.1.5 A matrix spike and matrix spike duplicate will be analyzed with each sample batch of up to 20 field samples.
- 14.2 The analyst must establish the ability to achieve low detection limits and generate acceptable accuracy and precision using this method before analysis of samples.
- 14.3 Refer to: DCL SOP XX-DC-018, "Evaluation of Quality Control Data" and the DCL Environmental Quality Assurance Program Plan, Section 10, "Quality Control Procedures", Section 11, "Data Reduction, Verification, and Reporting", Section 12, "Corrective Action", Appendix 14.7, "Summary of Calibration and Corrective Action", and Appendix 14.10, "Batch QC and Corrective Action Flowcharts". Nonconformance procedures are in accordance with DCL SOP LAB-020, "Nonconformance/Corrective Action Report (NC/CAR) Procedures".

15.0 REPORTING RESULTS

- 15.1 All results are reported on a DataChem Laboratories Analytical Report Form or equivalent in either $\mu g/L$.
- 15.2 Quality Control Data must be submitted with each analytical batch.

16.0 PREVENTIVE MAINTENANCE

16.1 Refer to: DCL SOP LAB-002, "Preventive Maintenance for Analytical Instrumentation".

17.0 WASTE MANAGEMENT

17.1 Refer to: DCL SOPs LAB-004, "Hazardous Waste Handling and Disposal" and LAB-005, "General Laboratory Safety and Chemical Hygiene".

18.0 DEFINITIONS

- 18.1 Refer to: the Environmental Quality Assurance Program Plan of DCL, Appendix 14.12, "Definitions and Terms".
- 18.2 Method Blank An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The method blank is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.
- 20.3 Continuing Calibration Verification (CCV) A solution of one or more method analytes, surrogates, internal standards, or other test substances used to check calibration with respect to a defined set of criteria.
- 18.4 Laboratory Control Sample (LCS) An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

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- 18.5 Matrix Spike and Matrix Spike Duplicate (MS or MSD) An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS or MSD are analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS/MSD corrected for background concentrations.
- 18.6 Working Standard (WS) A solution prepared from the primary dilution standard solution and stock standard solutions of the internal standards and surrogate analytes. The WS solutions are used to calibrate the instrument response with respect to analyte concentration.
- 18.7 Initial Calibration Verification (ICV) The ICV is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check primary standards with externally prepared test materials.

DATACHEM LABORATORIES, INC.

STANDARD OPERATING PROCEDURE APPROVAL SHEET

SOP TITLE: Calculation of Method Detection Limit

DOCUMENT CONTROL NUMBER: LAB-024 Revision 1

EFFECTIVE DATE: _____ November 1, 2002

APPROVALS:

MANAGER	 Date
	Data
QA MANAGER	 Date
LAB DIRECTOR	 Date

STANDARD OPERATING PROCEDURE

CALCULATION OF METHOD DETECTION LIMIT

1.0 INTRODUCTION

- 1.1 "The Method Detection Limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte." [40CFR Pt 136, Appendix B]
- 1.2 An MDL is the result of statistical manipulation of data obtained from the analysis of samples in accordance with a specific method. Thus, it is only valid for a specific analyte contained in a specific matrix when analyzed according to a specific method. If any of these variables change (e.g., sample matrix), there will likely be a deviation from the calculated MDL. Thus, MDLs are provided as an indication of what can be achieved by applying a given method to a general sample matrix.

2.0 SCOPE

- 2.1 The calculation of an MDL is applicable to all environmental sample matrices routinely analyzed at DataChem Laboratories (DCL) unless the methodology is not amenable to the calculation (e.g., pH determinations, gravimetric analyses).
- 2.2 Methods for which DCL requests certification from a state or organization generally require MDL calculations unless such are specifically excluded by the certifying body or unless the methodology is not amenable to the calculation.
- 2.3 DataChem Laboratories often undertakes projects that require the determination of MDLs in accordance with the procedures presented in this Standard Operating Procedure (SOP).
- 2.4 Method Detection Limits for both organic and inorganic environmental analysis are determined annually unless otherwise specified by project guidelines.
- 2.5 For Industrial Hygiene analyses MDLs are determined only for projects and programs explicitly requiring such.

3.0 APPLICATION

- 3.1 This Standard Operating Procedure (SOP) describes the calculation of an MDL in accordance with the requirements of 40 CFR 136 Appendix B. EPA Manual SW-846 contains a similar procedure for calculation of MDL; it is less stringent in some aspects. MDLs calculated in accordance with the procedures outlined in this SOP satisfy all the requirements of either approach. DCL policy regarding MDL Studies is listed in the Appendix to this SOP.
- 3.2 To determine MDLs, the computer program used in the calculations must in all cases be validated to ensure accuracy. DCL has a validated computer program in place to

determine MDLs. Outside vendor software that has been validated can be used. HAND-CALCULATED MDLs ARE NOT ACCEPTED.

3.3 MDLs are calculated in accordance with the procedures in this SOP unless DCL management approval for the less stringent SW-846 procedures is obtained. If approval for SW-846 procedures is obtained, the MDLs calculated shall not be applied to any method other than the SW-846 Method (e.g., any MDL calculation following SW-846 procedures for Method 8080 *must not* be reported for a Method 608 analysis).

4.0 **PROCEDURE**

- 4.1 Estimate the MDL for each analyte using one of the following approaches; obviously, the judgment of the analyst is an important factor in this estimation:
 - 4.1.1 The MDL from a previous MDL determination
 - 4.1.2 The concentration value that corresponds to a signal/noise ratio in the range of 2.5 to 5
 - 4.1.3 The concentration equivalent to three times the standard deviation of replicate determinations using reagent water or other appropriate sample medium as the matrix
 - 4.1.4 The region of the curve where there is a significant change in sensitivity
 - 4.1.5 Analytical judgment based on experience with similar analytes in the matrix
- 4.2 Prepare sample matrix and perform sample analysis.
 - 4.2.1 For water matrix, laboratory reagent (blank) water shall be used.
 - 4.2.2 For other matrices, analyze a portion of the proposed sample matrix according to the method. The proposed matrix can be specially prepared, or it can be a previously analyzed sample. The default matrix for soils is Ottawa sand.
 - 4.2.2.1 If the proposed sample matrix contains an analyte concentration between one and five times the estimated MDL, it is acceptable for MDL determinations.
 - 4.2.2.2 If the measured level of an analyte in the proposed sample matrix is less than the estimated detection limit, add a known amount of analyte to adjust the level of the analyte to between one and five times the estimated detection limit.
 - 4.2.2.3 If the analyte concentration in the proposed sample matrix is greater than five times the estimated detection limit, consider one of two options:
 - 4.2.2.3.1 If possible, select another sample of the same matrix with a lower level of analyte.

- 4.2.2.3.2 If the analyte level does not exceed 10 times the MDL of the analyte in reagent water, the proposed sample matrix can be used for determining the MDL.
- 4.2.2.4 It might be difficult to meet all MDL requirements for the 10 x rule. If the analyte concentration in the proposed sample matrix is below the determined MDL, or if it exceeds 10 times the MDL of the analyte in reagent water, the MDL value determined for this sample matrix must be reviewed by Quality Assurance personnel. The following equation summarizes criteria pertinent to the determination of the MDL:

MDL \leq Target Concentration in test samples $\leq 10 \text{ x MDL}$.

Contact QA personnel for specific guidance. If 80% of all analytes meet the 10 x rule and no analyte is greater than 20x, the pertinent determined MDL is deemed acceptable. When standard-traceable solutions are used for the Air Force Center for Environmental Excellence (AFCEE), the determined MDL must not be more than one-half the AFCEE Reporting Limit (RL).

- 4.2.3 Prepare a minimum of seven aliquots of sample matrix for the MDL determination.
- 4.2.4 Prepare and analyze each aliquot as a separate sample in strict accordance with the method.
 - 4.2.4.1 If desired, two aliquots can be processed initially. The results of this analysis can be used to evaluate the estimation of the MDL.
 - 4.2.4.1.1 If the MDL appears to be accurate, proceed with the analysis of the other five aliquots, and use all seven results to determine the MDL.
 - 4.2.4.1.2 If the MDL does not appear to meet pertinent criteria, reestimate the MDL and reevaluate the test samples proposed for the MDL determination (Steps 4.1 and 4.2).
- 4.2.5 Calculate the analyte concentration in each aliquot according to the method and determine results in the units specified in the method for reporting or which are applicable to the sample matrix.
- 4.3 Environmental testing methods only: An MDL verification sample is extracted with each MDL study. The concentration will be at ½ the target values used in the MDL study. Data generated by this MDL verification sample will be evaluated and included in the MDL study data package. Any detectable result is acceptable for the MDL verification sample. Non-detected results are assessed using the data systems to determine if the signal is at least 3 times the noise level. Additionally, Quality Assurance personnel or Technical Directors are contacted for determination of the impact of non-detects and for approval/rejection of this verification.

- 4.4 From the final results, calculate the MDL.
 - 4.4.1 Calculate the variance (S^2) and the standard deviation (S) of the replicate measurements according to the following:

$$S^{2} = \frac{1}{n - 1} \left[\begin{array}{ccc} n \\ \sum \\ i \end{array} X^{2} \\ i \end{array} - \left(\begin{array}{ccc} n \\ \sum \\ i \end{array} X^{2} \\ i \end{array} - \left(\begin{array}{ccc} n \\ \sum \\ i \end{array} X^{2} \\ i \end{array} \right)^{2} / n \end{array} \right]$$

 $S = (S^2)^{\frac{1}{2}}$

4.4.2 Compute the MDL as follows:

 $MDL = t_{(n-1, 1-\alpha = 0.99)}(S)$

where: $t_{(n-1, 1-\alpha = 0.99)}$ = the student's t value for 99% confidence (see Table 1).

- 4.5 Negative values are possible with some instrumentation. For example, the ICP/MS instrumentation gives valid negative values. The negative values are above the baseline noise but calculated as negative since the calibration equation does not pass though the origin. Since these and other negative values are determinations above the background noise they are valid results and should be used to assess the precision of the analytical method. Therefore all results positive or negative should be used to calculate S as listed in section 4.4.1.
- 4.6 Calculated MDLs must be evaluated as to reasonableness and may be adjusted upward if ICB, CCB, Rinses or Method Blanks have routine contamination to invalidate MDL studies. This data must be presented to QA personnel to adjust MDL upward.

5.0 RECORDS

- 5.1 Upon completion of an MDL determination, all data used to determine the MDL shall be placed in data package envelopes and delivered to Quality Assurance personnel. All data pertinent to the current MDL analyses are kept on file by QA personnel. Data relative to outdated MDLs are stored in DCL archives.
 - 5.1.1 The following information shall be placed in the data package, as applicable:
 - 5.1.1.1 Copies of all applicable laboratory notebook pages. The following information shall be included in the notebook:
 - 5.1.1.1.1 Estimated MDL for each analyte
 - 5.1.1.1.2 Results of preliminary analysis of the sample matrix
 - 5.1.1.1.3 Results of the analysis of each aliquot

- 5.1.1.1.4 Calculation of the variance and the MDL
- 5.1.1.1.5 Any other information normally recorded in the notebook for this analytical method
- 5.1.1.2 All chromatograms, instrument printouts, and other raw data
- 5.1.1.3 Copies of applicable pages from all standard logs or notebooks containing information relative to the preparation of standards or spiked samples
- 5.1.1.4 Any deviations from method procedures, reason for the deviation, and approvals of the deviations by manager(s) and QA personnel
- 5.1.1.5 Summary sheet showing all calculations performed
- 5.1.1.6 Any other data normally included in a data package for this analysis
- 5.1.2 The data package shall be identified on the front as an MDL Study. In addition, the following information shall be recorded on the front of the data package.
 - 5.1.2.1 Method
 - 5.1.2.2 Preparation method
 - 5.1.2.3 Instrument
 - 5.1.2.4 Matrix
 - 5.1.2.5 Analyte(s)
 - 5.1.2.6 Date(s) of study
 - 5.1.2.7 Responsible analyst
- 5.1.3 An MDL Determination Summary Sheet shall be prepared that documents the results of the MDL determination. Quality Assurance personnel shall file the original of the summary sheet in a readily accessible area.

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Number of Replicates	Degrees of Freedom (n – 1)	$t(n-1, 1-\alpha = 0.99)$
3	2	6.965
4	3	4.541
5	4	3.747
6	5	3.365
7	6	3.143
8	7	2.998
9	8	2.896
10	9	2.821
11	10	2.764
12	11	2.718
13	12	2.681
14	13	2.650
15	14	2.624
16	15	2.602
17	16	2.583
18	17	2.567

TABLE 1 STUDENT'S T VALUES AT THE 99 PERCENT CONFIDENCE LEVEL

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APPENDIX



Subject:	Policy on Method Detection Limit Studies for Environmental Testing Methods		
Date:	February 27, 2004		
From:	Brent Stephens, Bob Di Rienzo		
To:	Laboratory Staff		

This policy took effect on June 1st, 2000

"The Method Detection Limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte." [40CFR Pt 136, Appendix B]

An MDL is the result of statistical manipulation of data obtained from the analysis of samples in accordance with a specific method on a specific instrument. Thus, they are only valid for a specific analyte contained in a specific matrix when analyzed according to a specific method on a specific instrument. If any of these variables change, sample matrix, method, instrument, there will be a deviation from the calculated MDL. Thus, MDLs are provided as an indication of what can be achieved by applying a given method on a specific instrument to a general sample matrix. DCL uses DI water, silica sand or appropriate sampling media for MDLs as general sample matrices.

DataChem Laboratories, Inc. is required by NELAC, regulating agencies like Utah, AFCEE, Navy and USCOE, and by contract with clients to have valid MDLs on all instruments when analyzing samples. *Valid MDL studies are as essential to quality analytical data as is calibration verification*. Valid MDL studies are required every 12 months for all environmental testing methods unless a shorter frequency is specifically required by the reference method. Therefore, required MDLs must be valid on all instruments for the methods. It is the responsibility of the analyst to make sure valid MDL studies have been completed and are current. MDL studies will no longer be the responsibility of the QA department to ensure compliance with this policy. The responsible analyst can request QA to get a MDL study logged in to the LIMS system at any time within the required 12 months. The calculation of a MDL is addressed is the DCL SOPs Lab-024.

DATACHEM LABORATORIES, INC.

STANDARD OPERATING PROCEDURE APPROVAL SHEET

SOP TITLE: _____ Sample Receipt and Log-In (Environmental)

DOCUMENT CONTROL NUMBER: _____ QS-DC-001 – Revision 14

EFFECTIVE DATE: October 2, 2006

 Date
 Date
Date

STANDARD OPERATING PROCEDURE

SAMPLE RECEIPT AND LOG-IN (ENVIRONMENTAL)

1.0 SCOPE

- 1.1 DataChem Laboratories (DCL) Sample Receipt and Log-in procedures provide direction for documenting the condition of a sample shipment and for creating and maintaining a strict chain-of-custody for each sample. Direction is also provided for the initiation of laboratory sample tracking procedures.
- 1.2 This SOP addresses the requirement of NELAP 5.13f, ensuring client confidentiality of final results via transmission utilizing telephone, telex, facsimile, or other electronic or electromagnetic means. The DCL staff shall follow the procedures documented in this Standard Operating Procedure (SOP) and section 1.5 of the Quality Assurance Program Plan (QAPP) to meet this standard and preserve confidentiality.
- 1.3 This SOP is applicable to environmental and radiological samples.
- 1.4 An additional SOP, QS-EP-100, "EPA Sample Receipt and Logging," has been developed to address specific receipt and log-in requirements for EPA samples.

2.0 **RESPONSIBILITY**

- 2.1 The Sample Receipt Technician or designee is responsible for the receipt and logging of samples received at DCL.
- 2.2 The Radiation Safety Officer (RSO) is responsible for training Sample Receipt personnel in correct procedures for receipt of radiological samples.
- 2.3 The assigned DCL Project Manager is responsible for submitting a Project Protocol Worksheet (PPW) for each project to sample receiving personnel. This shall be done preferably before samples arrive at the laboratory, but at least within 24 hours following receipt. The PPW shall include precise and complete instructions pertaining to parties authorized to receive final results via any means. Any subsequent request for client results by unauthorized parties shall require written permission from the authorized client representative.

3.0 SAMPLE RECEIPT PROCEDURES (STANDARD)

- 3.1 Sample Receipt personnel must wear a laboratory coat, safety glasses, and protective gloves while unpacking and handling samples.
- 3.2 Upon delivery of shipping coolers to DCL, the Sample Receipt Technician receives the shipping coolers and examines them to document whether they arrived in acceptable condition. After a visual inspection, the custodian checks the coolers against the shipping documentation to determine whether the appropriate number of shipping coolers was

delivered. Monitors are present in the sample receipt area to detect levels of radiation as described below.

- 3.2.1 Coolers that register an activity dose rate of <.05 mR/hr or <5 x background are received following the nonradioactive procedures outlined in sections 3.3 3.10 of this SOP.
- 3.2.2 Coolers labeled "Radioactive" or that register an activity dose rate of >.05 mR/hr or >5 x background are received following the procedures outlined in section 4.0 of this SOP.
- 3.2.3 If DCL was not notified by the client to expect radioactive samples, the Sample Receipt Technician shall notify the cognizant DCL Project Manager of the measured activity dose rate of the cooler. The Project Manager contacts the client to obtain information concerning the samples.
- 3.2.4 If a cooler registers a dose rate >0.5 mR/hr, the Sample Receipt Technician shall notify the RSO and the DCL Project Manager immediately. Do not open this cooler.
- 3.3 The Sample Receipt Technician must assign a unique consecutive number to each shipping cooler. This number is used to track the condition of the shipping cooler and temperature and condition of the samples contained in each shipping cooler. The cooler number is recorded in a cooler number logbook to ensure there are no duplications of the cooler number; the number is also recorded on the DCL Client-Related Information Report (CRIR).
 - 3.3.1 The Sample Receipt Technician shall, when applicable, sign, date/time, and mark as received, the air bill accompanying the cooler(s).
 - 3.3.2 The Sample Receipt Technician shall include a hard copy of any email from after hours delivery as specified in section 6.1 of this SOP.
- 3.4 The Sample Receipt Technician shall complete a CRIR for each client's samples received in one shipment and record the following information:
 - Client name
 - Project/Task/Site (if available)
 - Time/Date of receipt
 - Number of coolers received
 - Condition of the custody seals, coolers, ice, and samples
 - Cooler temperature and location where the temperature was taken
 - ♦ Activity of coolers
 - pH as applicable (see Table 1)
 - Residual chlorine check (Methods 8270C, 8310, 8081, 8151, and 8330)

NOTE: One report can be used for up to nine coolers from an individual client.

3.5 The cooler must be opened in a hood in the sample receiving area. The temperature is taken using a calibrated thermometer. Record on the CRIR the reading indicated on the

thermometer, whether a temperature control was present, and the conditions of the ice in the cooler. If no control is provided, record the average temperature of the samples. The temperature requirements are $2 \degree C - 6 \degree C$. If any cooler temperature is not within project-specific guidelines, the temperature is indicated on the CRIR along with the client ID numbers of the affected samples (if there is more than one cooler in the shipment).

- 3.6 The Sample Receipt Technician removes the enclosed documentation from the shipping cooler and checks the sample containers received against the field chain-of-custody document(s) to note discrepancies. Discrepancies are noted in the *Problem* section of the CRIR.
- 3.7 The Sample Receipt Technician signs the field chain-of-custody in the appropriate *Received by* section and inserts the date and time of receipt.
- 3.8 The Sample Receipt Technician inspects the sample containers and records whether any samples are broken, leaking, or unacceptable. Also, pH is checked using narrow-range pH paper (see Table 1). Results are recorded on the CRIR.
 - 3.8.1 pH Procedure
 - 3.8.1.1 Place the liquid sample in a hood, mix well, and unscrew the cap. Pour a small aliquot into a disposable plastic beaker. Place a small piece of appropriate narrow-range (1 7 or 7 14) pH paper in the beaker and compare the color to the posted color chart. Acceptable pH ranges vary according to the required analysis. Refer to Table 1 for the correct range for each type of sample. If the pH is out of range, note the sample number and approximate pH on the CRIR.
 - 3.8.2 Residual chlorine procedure (Methods 8270C, 8310, 8081, 8151, and 8330)
 - 3.8.2.1 Place the liquid sample in a hood, mix well, and unscrew the cap. Pour a small aliquot into a disposable plastic beaker. Place a residual chlorine test strip in the beaker and verify the presence/absence of residual chlorine. If residual chlorine is present, note the sample number and record the problem on the CRIR.
 - 3.8.3 VOA Headspace
 - 3.8.3.1 Visually inspect all VOA bottles for headspace (air bubbles). Any VOA sample with air bubbles greater than 0.5 cm in diameter shall be noted on the CRIR. Determination of headspace is a visual estimation using 0.5 cm as an approximate value and not an exact measurement. A handy visual criterion is a comparison to the size of a pea.
- 3.9 If problems are noted, the Sample Receipt Technician makes a copy of the CRIR.
 - 3.9.1 The original is given immediately to the cognizant DCL Project Manager, who contacts the client for resolution.

3.9.2 The Project Manager returns the original report to the Sample Custodian, with directions for handling the problems, within 24 hours of report receipt so that samples can be processed in a timely manner.

3.9.2.1 If sample preservation is required by the client the appropriate reagent identification will be recorded on the CRIR in the section labeled "BRIEFLY DESCRIBE THE PROBLEM AND THE ACTION TAKEN".

- 3.9.3 If no discrepancies are found, the original CRIR is filed with the field chain-ofcustody document(s).
- 3.10 Upon completion of the inspection process, the samples are placed in the appropriate refrigerator.
 - 3.10.1 Samples including potential gasoline and diesel contamination samples, with the exception of volatiles, are placed on a laboratory cart in refrigerator R-33-1.
 - 3.10.2 Volatile samples are placed in a designated refrigerator within the VOA lab along with storage blanks (when required). See SOP XX-EP-200 Section 7.1.3.

4.0 SAMPLE RECEIPT PROCEDURES (RADIOLOGICAL)

- 4.1 Any samples designated as radioactive by project management on the PPW or by the RSO on the "Authorization for Radioactive Sample Receipt" or any cooler labeled "Radioactive" or registering an activity dose rate of >.05 mR/hr or >5 x background must be received under the radiological sample receipt procedures.
 - 4.1.1 The Sample Receipt Technician must immediately inspect the coolers to ensure that they are not damaged or leaking.
 - 4.1.1.1 Upon detecting a damaged or leaking cooler, the Sample Receipt Technician detains the delivery carrier and summons the RSO or designee to perform removable contamination surveys.
 - 4.2 The Sample Receipt Technician places the <u>un-opened</u> coolers or containers in the radiation laboratory.
 - 4.3 The radiological sample receiving personnel must complete the proper documentations as required for environmental samples specified in this SOP or the DCL SOP IH-GL-006 for industrial hygiene samples.
 - 4.4 If the samples require radioactive materials inventory tracking (RMITS) as designated by the radioactive sample approval procedures, See DCL SOP WA-DC-002, a copy of complete paperwork will be given to the RSO.

5.0 SAMPLE LOG-IN PROCEDURES

5.1 For long-term projects, the original PPWs are filed alphabetically in the Sample Receiving area.

- 5.2 The Sample Custodian assigns a unique DCL identification number to each of the samples. The DCL sample number is recorded on the field chain-of-custody adjacent to the corresponding client ID number.
 - 5.2.1 The DCL identification numbers are stream-specific alphanumeric numbers and are assigned sequentially in increasing order.
 - 5.2.2 The DCL sample identification numbers are printed on labels with unique alphanumeric numbers and client ID numbers. They are affixed to the sample container without covering up vital information. This step provides verification of samples received.
 - 5.2.3 DCL identification numbers and client sample numbers are also recorded on the DCL chain-of-custody form.
- 5.3 The Sample Custodian initiates the DCL chain-of-custody document(s) and a DCL work order. This is accomplished by entering data from the field chain-of-custody into the computer to generate the forms.
- 5.4 After completing the DCL chain-of-custody, a different Sample Technician or designee visually and electronically compares the field chain-of-custody and PPW with the DCL chain-of-custody for the following items:
 - Client data (e.g., PPN, rush/non-rush, due date)
 - ♦ Date/Time sampled
 - Field ID numbers and DCL numbers
 - Site ID/Customer ID (IRDMIS information if applicable)
 - Types of analyses or analytes requested
 - Number of bottles submitted per analyte for each sample
 - Disposal Information: If Project Management permission is required prior to disposing of Field Samples, the chain-of-custody will be stamped in the upper right hand corner indicating "Disposal Permission Required".
 - 5.4.1 If an error is noted, the paperwork is immediately corrected.
 - 5.4.2 Upon completion of verification, the person doing the verification initials and dates the *Verified* section of the DCL chain-of-custody.
 - 5.4.3 After electronic verification is completed for the entire sample set, a pre-invoice is generated and given to the Project Manager.
- 5.5 The DCL sample labels are applied to the pertinent bottles.
 - 5.5.1 Ensure that DCL labels are verified against client sample numbers and are placed on the correct bottles in a location as close as possible to the client ID without covering any vital information on the original bottle label.
- 5.6 The samples are placed on the shelves in the appropriate refrigerator (R-33-1, or R-24-2).

- 5.7 The Sample Receipt Technician signs the DCL chain-of-custody in the first *Relinquished by* space, including the date, time, and storage location.
- 5.8 A copy of the completed and verified DCL chain-of-custody is made to accompany the work order and filed with the original field chain-of-custody and air bill in a central location in the Sample Receiving area.
- 5.9 The Sample Receipt Technician files the following documentation in the analyst file cabinet located in Sample Receipt.
 - DCL Chain-of-Custody (original)
 - ◆ PPW (copy)
 - Field Chain-of-Custody (copy for IRDMIS sets only)
 - DCL Cooler Receipt Checklist (copy only if a problem is noted on the CRIR)

6.0 AFTER-HOURS, HOLIDAY, AND SATURDAY SAMPLE RECEIPT

- 6.1 The responsible party shall perform the following sample receiving activities:
 - 6.1.1 Perform <u>visual</u> inspection of container and contents only for items specified on the CRIR (Exhibit 7.2) for any anomalous conditions.
 - 6.1.2 Immediately upon opening, perform temperature reading of interior of container.
 - 6.1.3 Remove any water, ice, or packing material from container.
 - 6.1.4 Review all paperwork submitted with the samples and sign any Chain of Custody (CoC) documents, making sure all samples listed on the CoC are accounted for.
 - 6.1.5 Document receipt by sending an email to all pertinent project managers, sample receiving, and operations managers detailing items 6.1.1, 6.1.2, any anomalies from 6.1.4 and any additional appropriate information pertaining to the deliveries.
 - 6.1.6 Samples and appropriately signed submittal paperwork are left in shipping containers and placed in the walk-in cooler in sample receipt area (R-33-1).
- 6.2 Receipt of samples known or suspected to be potentially radioactive must be coordinated in advance with trained and qualified personnel available to process the receipt of these samples. If samples known or suspected to be potentially radioactive are delivered without prior arrangement, the containers are to be placed, unopened, in the area designated for such samples. Notification is then to be made via telephone and email to the responsible project manager and operations manager. No further action is to be taken.
- 6.3 On a project specific basis, other project specific requirements for sample receipt may be performed if requested by the client through instructions given by the project manager. These task may include but not limited to: chemical preservation, COC procedures, storage requirements, paperwork, CRIR completion and sample sorting.

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7.0 EXHIBITS

- 7.1 Example Project Protocol Worksheet
- 7.2 DataChem Laboratories Client-Related Information Report (CRIR)
- 7.3 DataChem Laboratories Chain-of-Custody
- 7.4 Sample Work Order
- 7.5 Radiological Survey and Screening Report
- 7.6 Action Levels Pertaining to Surveys Performed upon the Receipt of Radioactive Material(s)
TABLE 1. PRESERVED WATER SAMPLES FOR PH CHECK UPON RECEIPT

Analyte	pН
Metals	< 2
Cyanide	> 12
Sulfide	>9
Ammonia	< 2
Total Phenolics	< 2
TPH – Method 418.1	< 2
COD	< 2
TKN	< 2
NO ₃ /NO ₂	< 2
Oil & Grease	< 2
Total Phosphorus	< 2
TOC	< 2
Gross A/B, Gamma Spec	< 2

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EXHIBIT 7.1 PROJECT PROTOCOL WORKSHEET EXAMPLE



PROJECT PROTOCOL WORKSHEET FAX -- YES/NO Final by: FedEx/USPS

Project Name: Contract/PO#: Account: 08001 Site ID: Set Info: Billing ID:

 Project Manager:
 [ext]

 e-Mail:
 Aurum - Tigger

 Sample Stream:
 E

 Disposal Code:
 R-D-30R-N

 MW Screen:
 Kenter

 MS/MSD/MD:
 CLIENT/DCL/METHOD

 Solids:
 NONE/TOTAL/MOISTURE

 Reporting Basis:
 AS RECEIVED/DRY

 Batch with other clients samples:
 YES/NO

 Report TICs:
 N/A

 Detection Limits:
 DCL /CLIENT

 QA/QC Requirements:
 METHOD/CLIENT

		PPN:	
Client Contact: Name- Company- Street- City/State- Telephone- Fax-	0 0		
Invoice Contact:	Same		
Bill for field samp	oles only:	YES (plus MS/MSD)	
Type of report:			
Report by: Case Narrative: Raw Data: No. of copies:	Fraction/Se YES/NO YES/NO 1	et/SDG	

Prep, Analysis and Reporting:

Sample Receipt Instructions:

Parameter	Matrix	Units	Sec. Code	Inst. Code	DCL SOP or Method	Analyte List	Hold Code	Verbal TAT	Mail TAT	Analyte Group	Price Code

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EXHIBIT 7.2 DCL CLIENT-RELATED INFORMATION REPORT (CRIR) DATACHEM LABORATORIES CLIENT-RELATED INFORMATION REPORT (CRIR) COOLER OR CONTAINER INFORMATION CHECKLIST (Fill In or Circle)

Client Name		JLEK OK		K INFORMA	Project	/Task/Site				
Data/Tima	of Possint:				Numbe	r of Cooler	Dessived		<u> </u>	
Date/Time (of Receipt:				Number of Coolers Received:					
Condition o	f Coolers:	Accept	able/Unacce	eptable	Temperature Control: Present/Not Included				uded	
Custody Sea	als:	Present Integt/	t/Absent/NA		Locatio	Location Temp Taken: Control/Between Samples				
Tamper Evi	dent:	Ves/No	NA		nroject	Are all temperatures within				
Ice Present:	dent.	Yes/No	/NA		Are all	applicable	nHs within	165/100/104		
100 1 1000111		Frozen	/Melted/NA		specific	c guidelines	?	Yes/No/NA		
pH Check	Metals	Yes	Yes/No/NA Total Phenolics		ics	Yes/No/NA	NO3/N	02	Yes/No/NA	
Performed:	Cyanide	Yes	/No/NA	TPH - 418.1		Yes/No/NA	Oil & O	Grease	Yes/No/NA	
	Sulfide	Yes	No/NA	COD		Yes/No/NA	A Total P	hosphorous	Yes/No/NA	
	Ammonia	Yes	/No/NA	TKN		Yes/No/NA	A TOC		Yes/No/NA	
Residual	8270	Yes	No/NA	8310		Yes/No/NA	8330		Yes/No/NA	
Check	8081	Y es/	NO/NA	8151		Yes/INO/INA	1			
Performed:								I		
<u>Received</u>	DCL Cooler No.	Temp.	Received	DCL Cool	er No.	Temp.	Received	DCL Cooler No.	Temp.	
1	C05 -	°C	4	C05 -		°C	7	C05 -	°C	
2	C05 -	°C	5	C05 -		°C	8	C05 -	°C	
3	C05 -	°C	6	C05 -		°C	9	C05 -	°C	
Taken By:		Signatur	e Cum			Printed	Name		Date	
D Missing	Castar		CLIEF	- /D - 441		MATION		Lu Cii t Ci		
	Cooler		sing Sample	S/Dotties		literio Not	Mat	Volume	imple	
	Deportuork		rroot Pottlo	Turno		dual Chlorir	De Brocont	☐ Chain of Cust	odv	
	Incorrect Bottle		ler Tempera	tures Out		Space in Bottles				
Labels	Incontect Bottle	of R	ange	lules Out	Conter:					
			U					EPA Custody	Seal:	
BRIEFLY DESCRIBE THE PROBLEM AND THE ACTION TAKEN:										
Faxed to Cli	ient? YES		No	(if	yes, attac	h Fax Cove	er Sheet)			
]	Response	Required	Within	24 Hour	'S			
			Pr	OJECT MA	NAGEN	1ENT				
PROJECT N	MANAGER COMM	ENTS:								
DCL Project	Manager:	Printed Name	Ret	urned to Samp	le Receipt	by:	Signature	Date:		

SLC/CRIR.doc

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Revised 1/02/04

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EXHIBIT 7.3 DCL CHAIN-OF-CUSTODY

Check box if there is a continuation page $\Box_{\rm Form:\ COFC1.01-SWV2.00}$

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EXHIBIT 7.4 SAMPLE WORK ORDER

		Sample	Work (Orde	r			
QC Clea	rance:				DCL	Root Set ID:		
Project	Manager:				DCL	Lab. Name:		
Client:	5				Tota	Total # Samples:		
Account: SDG:		Sample Entry:			ole Entry:			
Project/	Task:				Secti	on: est Sampling Da	•	
Date Re Date for	ceived: Mailing Report:				Prep	aration Type:	le.	
Rep.	Environmental Inorganic.	Latest Prep. Date	Latest Anal. Date	No. of Samp.	Storage Location	Analysis/Prep. Method	Inst.	Matrix

 Analytes Requested
 Prep. Date
 Anal. Date
 Samp. Location
 Method
 Inst.
 Matrix

 01
 02
 02
 03
 04
 04
 05
 04
 05
 05

Special Instructions:

Section Manager:

Other Sections Receiving Sample Portions:

DataChem Laboratories/ 960 West LeVoy Drive / Salt Lake City, Utah 84123 Form: W01.04-SWV2.12 PRINTED 4/14/1999 14:46

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EXHIBIT 7.5 RADIOLOGICAL SURVEY AND SCREENING REPORT

RADIOLOGICAL SURVEY AND SCREENING REPORT

To be filled in by Sample Receiving	
DCL Set ID Number	Account Number
Client	Number of Shipping Containers
(use additional forms if gre	ter than 3 containers are received)
Activi	y (mR/hr)
Shipping Container Contac	<u>1 Meter</u> <u>Comments</u>
(3)	
Activi	y (mR/hr)
Shipping <u>Container</u> DCL Sample Numbers	Contact Comments
Survey Instrument Type	Survey Instrument ID
	Signature/Date
To be filled in by the Radiochemistry Labor	tory
Primary Isotope(s)	
A	tivities
DCL Sample Alpha B	ta Total α+β Gamma Mass
<u>Numbers (pCi/g) (pCi/sample) (pCi/g)</u>	pCi/sample) (pCi/g) (pCi/sample) (g) Calegory
· · · · · · · · · · · · · · · · · · ·	Signature/Date
To be filled in by the RSO or Designee	(Above) DCL Sample Category (I, II, or III)
·	and the second
P PLC TOROUT V.C.D	

EXHIBIT 7.6 ACTION LEVELS PERTAINING TO SURVEYS PERFORMED UPON THE RECEIPT OF RADIOACTIVE MATERIAL(S)

INITIAL SURVEY

INSTRUMENT:	Portable dose rate survey meter (Ludlum Model 19, Eberline Model RO-3C).
<u>APPLICATIONS</u> :	 Survey the following: Any package displaying "Radioactive I" or "Radioactive II" labels Any package that has potential to exhibit radioactivity, as indicated by labeling, paperwork, or origin of shipment
<u>ACTION LEVELS</u> :	 Above background reading at external surface of package or container: Record readings in logbook. ≥ 0.5 mR/hr at external surface of package: Notify RSO or designee to check for proper labeling. ≥ 0.1 mR/hr at external surface of individual sample container: Notify RSO or designee to handle samples.
	WIPE TESTS
INSTRUMENT:	Ludlum Model 2929 Dual-Channel Scintillation Counter
<u>APPLICATIONS</u> :	 Wipe test the following: Any package displaying "Radioactive I" or "Radioactive II" labels Any package that induced a significant response (i.e., 5 x background) on the portable survey meter Any <u>damaged</u> package that has potential to exhibit radioactivity, as indicated by labeling, paperwork, or origin of shipment.
<u>ACTION LEVELS</u> :	 > 20 dpm (alpha) or > 200 dpm (beta/gamma), above background, removable contamination per 100 cm²: Decontaminate in accordance with R.S. 11.0 until below the action level on the next wipe. If unable to decontaminate below the action level, place in a sealed bag and label "CONTAMINATION." 22,000 dpm above background, removable contamination per 100 cm², where: <u>dpm = Gross cpm - cpm (bkg)</u> <u>efficiency</u>
	 Notify the following: RSO and Safety personnel (ext. 366) Final delivering carrier Utah Division of Radiation Control (538-6734) Nuclear Regulatory Commission, Regional Office (Region IV) at (817) 860-8100

IMPORTANT:

- 1. The preceding wipe tests must be performed within three hours of package receipt during normal working hours (8:00 a.m. to 5:00 p.m.), and within 18 hours of receipt during the evening/graveyard shifts. Packages received during weekends or holidays must be screened within the first three hours of the first work day following that weekend or holiday.
- 2. Enter all readings in the survey logbook. Submit "Radiological Survey and Screening Report," along with sample weight and client supplied data, to the RSO or designee for categorization of samples.

Revised 11/03/00

DATACHEM LABORATORIES, INC.

STANDARD OPERATING PROCEDURE APPROVAL SHEET

SOP TITLE:	Preventive Main	tenance for Analytical	Instrumentation
DOCUMENT CONTR	ROL NUMBER:	LAB-002 – R	evision 3
EFFECTIVE DATE:		November 3, 2005	
APPROVALS:			
MANAGER			Date
MANAGER			Date
QA MANAGER			Date
LAB DIRECTOR			Date

STANDARD OPERATING PROCEDURE

PREVENTIVE MAINTENANCE FOR ANALYTICAL INSTRUMENTATION

1.0 SCOPE AND APPLICATION

- 1.1 This Standard Operating Procedure (SOP) provides procedures for preventive maintenance of specific laboratory instrumentation. Calibration and maintenance procedures for balances and refrigerator thermometers are documented in DataChem Laboratories (DCL) SOPs QC-DC-003, "Balances," and QC-DC-004, "Calibration of Thermometers." Calibration procedures for pipettors are found in DCL SOP XX-DC-001, "Calibration of Pipettors."
- 1.2 Table 1 of this SOP (Preventive Maintenance Schedule and Parts List) provides the requirements and activities for specific instruments.

2.0 **DEFINITIONS**

2.1 Preventive Maintenance – Any repair activity or procedure performed on instrument hardware or electronics for the purpose of ensuring the continued quality performance of the instrument.

3.0 RESPONSIBILITIES

- 3.1 Each analyst/technician is responsible for maintenance of equipment immediately prior to analytical testing. Activities and data related to instrument maintenance shall be documented in the Instrument Maintenance Logbook. The analyst/technician is responsible for such documentation.
- 3.2 Operations managers are responsible for (1) ensuring maintenance in accordance with Table 1 of this SOP; (2) notifying the manufacturer or vendor service engineer or representative when outside service is required; (3) ensuring maintenance in accordance with manufacturer specifications; (4) maintaining an adequate supply of selected critical spare parts to minimize instrument downtime; and (5) ensuring that maintenance activities are documented and in compliance with DCL SOP XX-EP-600, "Documentation – Maintaining Instrument Records, Notebooks, and Logbooks."
- 3.3 Operations managers and project managers shall investigate whether or not defective equipment has affected any previously reported test results and implement required corrective measures. Corrective action, in such cases, shall include notification of pertinent clients of any previously reported results that are problematical.
- 3.4 Internal auditing of maintenance records is the responsibility of DCL Quality Assurance (QA) personnel.

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4.0 GENERAL PROCEDURES

- 4.1 Maintenance schedules for analytical instruments shall be those recommended by the instrument manufacturer, unless DCL experience dictates otherwise. Requirements for preventive maintenance procedures for specific laboratory instrumentation are listed in Table 1 of this SOP Preventive Maintenance Schedule and Parts List.
- 4.2 If an instrument does not meet calibration requirements of a specific analytical method or performs unsatisfactorily, it shall not be utilized for sample analysis until repairs are completed and pertinent quality control (QC) criteria are satisfied. Out-of-Service equipment or instrumentation shall be isolated or clearly labeled or marked until it has been repaired and shown by calibration, use, or test to perform correctly. DCL operations managers and project managers shall investigate whether defective equipment has affected previously reported test results. Clients shall be notified of any affected test results.
- 4.3 An instrument identification number is assigned to each instrument by the cognizant operations manager; it is clearly posted on each instrument. A complete list of pertinent equipment and instruments is maintained.
- 4.4 Records are kept for all maintenance performed, including external servicing when required. Documentation is recorded or filed by the person who performs the maintenance procedure. (Refer to DCL SOP XX-EP-600, "Documentation Maintaining Instrument Records, Notebooks, and Logbooks.")
- 4.5 Critical spare parts are listed in Table 1. Each operations manager maintains an adequate supply of critical spare parts for pertinent instrumentation to minimize instrument downtime.

5.0 PREVENTIVE MAINTENANCE PROCEDURES FOR GRAPHITE FURNACE ATOMIC ABSORPTION SPECTROPHOTOMETERS

- 5.1 A number of instrumental variables are assessed as part of preventive maintenance: instrument warm-up, gas flow, lamp intensity, slit width, and wavelength. Preventive maintenance procedures include a minimum warm-up period of 30 minutes. The hollow cathode lamp, or electrodeless discharge lamp, is aligned to produce the maximum emitted light to the detector; the inert gas flow inside the furnace is optimized to ensure maximum sensitivity.
- 5.2 The digital readout values obtained for the standard curve of each element are checked to ensure that they are within a specified range. If readings are excessively low, the operator checks gas flows, cell alignment, wavelength, photomultiplier voltage, and lamp intensity before initiating analysis. Atomization chamber, optical lenses, quartz cells, and reduction flasks are cleaned according to manufacturer instruction whenever excessive electronic noise is apparent, or whenever indicated by visual inspection. Tygon[®] tubing is replaced when deterioration is apparent.

6.0 PREVENTIVE MAINTENANCE PROCEDURES FOR GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS) INSTRUMENTATION

- 6.1 Refer to specific DataChem Laboratories (DCL) documentation for details. Two examples are listed in the following.
 - 6.1.1 Refer to DCL SOP OV-DC-002 for GC/MS Volatile instrumentation, "Preventative Maintenance for Hewlett Packard 5971 and 5972 Mass Selective Detectors and 5890 Gas Chromatograph/Volatile Organic GC/MS."
 - 6.1.2 Refer to DCL SOP OS-DC-002 for GC/MS Semivolatile instrumentation, "Preventative Maintenance for Hewlett Packard 5971 and 5972 Mass Selective Detectors and 5890 Gas Chromatograph."

7.0 PREVENTIVE MAINTENANCE PROCEDURES FOR GAS CHROMATOGRAPHY (GC) INSTRUMENTATION

7.1 Septum replacement: Remove the septum nut. Remove the old septum and replace it with a new one. Replace the septum nut, hand tighten, and then tighten 1/4 turn more. Do not over-tighten the septum nut.

WARNING: The injection port and septum nut can be very hot.

- 7.2 Injection port liner replacement: Remove the split/splitless weldment assembly, exposing the top of the injection port liner. Lift the liner out of the injection port. Clean or replace it with a new liner packed loosely with a small plug of deactivated glass wool. Replace the O-ring at the top of the liner. Replace the weldment assembly, ensuring that the O-ring seals around the insert.
- 7.3 Detector maintenance: Refer to the manufacturer maintenance manual for specific maintenance procedures for each type of detector. Routine maintenance includes, as required: cleaning and/or replacing jets and collectors; replacing O-rings, seals, lamps, and reaction tubes.

8.0 PREVENTIVE MAINTENANCE FOR GEL PERMEATION CHROMATOGRAPHY (GPC) SYSTEMS

- 8.1 Visual inspection of the column: The column is visually inspected for discoloration or channeling that can occur from extended use. When the column is extremely discolored or does not pass calibration, prepare a new column as follows:
 - 8.1.1 GPC Column Preparation: The instructions listed below for GPC column preparation are for Bio Beads. Alternate column packing can be used if: (1) the column packing has equivalent or better performance than Bio Beads and meets technical acceptance criteria for GPC calibration and associated checks of calibration; and (2) the column packing does not introduce contaminants/artifacts that interfere with the analysis of pesticide and semivolatile compounds. Follow manufacturer instructions for preparation of the GPC column packing.

- 8.1.1.1 Weigh 70 g of Bio Beads (SX-3). Transfer the beads to a one-liter bottle with a Teflon[®]-lined cap or a 500-mL separatory funnel with a large-bore stopcock; add approximately 300 mL of methylene chloride. Swirl the container to ensure the wetting of all beads. Allow the beads to swell for a minimum of two hours. Maintain sufficient solvent to cover the beads. If a guard column is used, repeat the above procedures with 5 g of Bio Beads in a 125-mL bottle or a beaker, using 25 mL of methylene chloride.
- 8.1.1.2 Turn the column upside down from its normal position and remove the inlet bed support plunger (the inlet plunger is longer than the outlet plunger). Position and tighten the outlet bed support plunger as near to the end of the device as possible.
- 8.1.1.3 Raise the end of the outlet tube to keep the solvent in the GPC column, or close the column outlet stopcock. Place a small amount of solvent in the column to minimize the formation of air bubbles at the base of the poured column packing.
- 8.1.1.4 Swirl the bead/solvent slurry to obtain a homogeneous mixture and, if wetting was effected in a quart bottle, quickly transfer the mixture to a 500-mL separatory funnel with a large-bore stopcock. Drain the excess methylene chloride directly into the waste beaker, and start draining the slurry into the column by placing the separatory funnel tip against the column wall. This approach minimizes bubble formation. Swirl occasionally to keep the slurry homogeneous. Drain sufficiently to fill the column. Place the tubing from the column outlet into a waste beaker located below the column, open the stopcock (if attached), and allow the excess solvent to drain. Raise the tube to stop the flow, and close the stopcock when the top of the gel begins to look dry. Add sufficient methylene chloride to just rewet the gel.
- 8.1.1.5 Wipe any remaining beads and solvent from the inner walls of the top of the column with a laboratory tissue. Loosen the seal slightly on the other plunger assembly (long plunger) and insert it into the column. Make the seal just tight enough so that any beads on the glass surface are pushed forward, but loose enough so that the plunger can be pushed forward.

CAUTION: Do not tighten the seal if beads are between the seal and the glass surface; this can damage the seal and cause leakage.

8.1.1.6 Compress the column as much as possible without applying excessive force. Loosen the seal and gradually pull out the plunger. Rinse and wipe the plunger. Slurry any remaining beads and transfer them into the column. Repeat the step in Section 8.1.1.5 and reinsert the plunger. If the plunger cannot be inserted and pushed in without allowing beads to escape around the seal, continue compression of the beads without tightening the seal, and loosen and remove the plunger as described. Repeat this procedure until the plunger is inserted successfully.

- 8.1.1.7 Push the plunger until it meets the gel and then compress the column bed approximately four centimeters.
- 8.2 Solvent lines and junctions shall be inspected monthly. Check lines and junctions for cracks, crimps, or other signs of wear. Replace old lines and junctions as necessary.
- 8.3 Valving units shall be inspected quarterly. Check the valves and ports for leaks or corrosion. Check with the manufacturer for replacement procedures.
- 8.4 Detector maintenance: Refer to manufacturer manual for specific maintenance procedures for each type of detector. Routine maintenance includes inspection of preparation cells and the mercury lamp.

9.0 PROCEDURE FOR CRITICAL SPARE PARTS

9.1 A supply of selected critical spare parts shall be maintained in each operational unit of the laboratory to minimize downtime. Selected critical spare parts are listed in Table 1 of this SOP.

10.0 PROCEDURES FOR SERVICE CONTRACTS OR ALTERNATE SERVICE ARRANGEMENTS

10.1 Instrumentation is maintained by a tandem of manufacturer service contracts and DCL inhouse personnel. It is the responsibility of the analyst to perform routine maintenance and repairs. For instruments not under service contract: after it is determined by the cognizant operations manager that an instrument problem is beyond the scope of the analyst and other DCL personnel, service from the appropriate vendor is solicited.

11.0 QUALITY CONTROL (QC) LIMITS

- 11.1 Specific QC criteria are outlined in each method or in project-specific directives.
- 11.2 Specific QC criteria are used to verify that an instrument is correctly calibrated. Successful calibration, meeting pertinent criteria, demonstrates that the instrument has been properly maintained.

12.0 INSTRUMENTATION TAKEN OUT OF SERVICE

- 12.1 "Do Not Use" tags similar to Exhibit 18.1 are attached to instrumentation taken out of service until such time as the equipment is either repaired or removed from the laboratory.
- 12.2 Tags are available from Quality Control personnel.
- 12.3 The responsible analyst determines through prior records the effect of prior calibrations or tests. If a determination is made that previous analytical data have been affected, a Nonconformance/Corrective Action Report is initiated.

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13.0 CORRECTIVE ACTION PROCEDURES

- 13.1 In the event that one or more of the procedures required in this SOP have not been followed, a DCL Nonconformance/Corrective Action Request (NC/CAR) shall be submitted by the employee making the discovery.
- 13.2 A copy of the NC/CAR shall be given to the appropriate DCL manager, to QA personnel, and to the pertinent Project Manager. Refer to DCL SOP QC-DC-006, "Nonconformance/ Corrective Action Report (NC/CAR) Procedures."
- 13.3 QA personnel shall follow up to ensure that a resolution of the problem is effected and documented in accordance with DCL SOP QC-DC-006.
- 13.4 Operations managers and project managers shall determine whether or not data previously reported results have been affected by malfunctioning equipment. If data have been affected, the assigned project manager shall notify pertinent clients.

14.0 DOCUMENTATION

14.1 An instrument identification number is assigned to each instrument and is posted on the instrument. A separate Instrument Maintenance Logbook is assigned to the instrument for recording all maintenance activities associated with the instrument, including daily activities. One or more pages in the front of the logbook are designated for signatures. One or more pages directly following the signature page(s) are reserved for recording the following pertinent information: instrument description, manufacturer, model number, and serial number. A schedule of routine maintenance and a list of critical spare parts shall be included. Documentation in the Instrument Maintenance Logbook shall be in accordance with procedures detailed in DCL SOP XX-EP-600, "Documentation – Maintaining Instrument Records, Notebooks, and Logbooks."

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15.0 TABLES

TABLE 1. PREVENTIVE MAINTENANCE SCHEDULE AND PARTS LIST

Instrument	Items Checked/Serviced	Frequency	Critical Spare Parts
Gas Chromatograph	Replace column or column packing, clean detector, clean injection port.	Determined by analyst so that response and the calibration are within required specifications	Column ferrules Injection port liners and O- rings Autosampler syringes Deactivated glass wool
	Replace septa.	As determined by analyst	Columns Column packings
	Replace incoming gas drying cartridges.	When color change is observed	Injection port septa Detector igniters NPD collectors Moisture traps Oxygen traps
Atomic Absorption Spectrophotometer	Perform at least a 3-point calibration, and if readings are low, check the gas flows, burner, cell alignment, wavelength slit width, photomultiplier voltage, and lamp intensity prior to analysis.	Daily, as needed, or when used	Nebulizers, contact rings, graphite tubes, quartz windows
	Change graphite tubes and contact rings.	Daily, as needed, or when used	
	Clean burner heads, nebulizers, quartz cells, and reduction flasks according to manufacturer instructions whenever excessive noise is apparent or whenever indicated by visual inspection.	Daily, as needed, or when used	
	Tygon [®] tubing	6 months, or if deterioration is observed	
	Optical lenses	As needed	
Analytical Balance	Internal weight, train, gears, electronics	Annual service	
Inductively Coupled Plasma/ Atomic Emission	Sample introduction system (aspirator)	Daily, as needed, or when used	Torches, nebulizers, pump tubing, torch collars (bonnets), spray chambers
Spectrophotometer (ICP/AES)	Check pumps and tubing. Clean nebulizer.	Weekly, as needed, or when used As needed	
	Clean sample probe.	As needed	
	Check plumbing.	Daily, or when used	

Instrument	Items Checked/Serviced	Frequency	Critical Spare Parts
Inductively Coupled Plasma/Mass Spectrometer (ICP/MS)	Change pump tubing. Check sampler and skimmer cones for deposits, change and/or clean.	Daily, or as needed Daily, or as needed	Pump tubing Sampler and skimmer cones Extraction lenses Torches Nebulizer
	Check torch for deposits or damage.	Daily	O-rings for cones and spray chamber
	Check roughing pump oil level.	Weekly	Sampling cone retaining
	Check backing pump oil level.	When vacuum shut down Weekly, or as needed	Vacuum oil
	Check water chiller water level.	Monthly, or as needed	Distilled water for chiller
	Clean spray chamber and check O-rings.	Monthly, or as needed	Cone cleaning powder
	Check nebulizer for damage or deposits.	Monthly	
	Check and clean air filter on front of torch compartment.(if	Monthly, or when vacuum shut down	
	Check water filter on back of instrument. (if applicable)	Monthly	
	Check filter on front of laboratory air conditioning unit.	Weekly	Air conditioner filters
Ion Chromatograph	Check filter (inlet).	As needed	Suppressor column
	Check bed support.	When specifications are not met	
	Clean cells.	As needed	
Infrared Spectrophotometer	Polystyrene Test Spectrum	Weekly	Chart paper, pens, set of 10-cm cells set of 1-cm
spectrophotometer	Zero Adjustment	Daily	cells, source coils, ceramic rods
pH Meters	None	None	None
Mercury Analyzer	Change Reagent Tubing Change Filter Change Hg Absorber Check Instrument Sensitivity Clean Cell	As needed As needed Quarterly Daily Every 6 months or when contaminated	Reagent Tubing Filters Absorber New Lamps New or cleaned cell

TABLE 1. PREVENTIVE MAINTENANCE SCHEDULE AND PARTS LIST (continued)

-		-	
Instrument	Items Checked/Serviced	Frequency	Critical Spare Parts
Gas Chromatograph/	Replace column or column	Determined by analyst so	Septa
Mass Spectrometer	packing, clean ion source, and	that response and the	
(GC/MS)	replace filaments.	calibration are within	Single-taper injection port
		required specifications	liners
	Replace foreline pump fluid.	Annually	Ferrules Columns
	Check diffusion pump fluid.	As needed	Syringes Filaments
	Printer maintenance	As needed	Toner cartridges O-rings
	Clean instrument area.	As needed	8
	Replace separator pump fluid (VOA GC/MS systems only).	Annually	
	Change septum (SVOA GC/MS systems only).	As needed as determined by analyst	
	Injection port maintenance (SVOA GC/MS systems only)	As needed as determined by analyst	
Ultraviolet (UV/VIS) Spectrophotometer	Lamp and wavelength check or serviced	As needed or during calibration steps when used	Replacement cells
	Wash, rinse, and dry cells.	Each use	
HPLC	Detector lamps	If baseline is unstable or	Pump seals
		has excessive noise	Switching valve seals
		without flow through the	Check valve seals (inlet and
		cell	outlet)
			Lamps
	Pump seals, check valves, inlet	If HPLC system pressure	Columns
	frits	becomes unstable while	Tubing
		How is isocratic	Ferrules/nuts
	Switching valve seals, injection	If replicate injections are less than 95% of each	
	volume metering device seals	other where chroma-	
		tography is not in question	

TABLE 1. PREVENTIVE MAINTENANCE SCHEDULE AND PARTS LIST (continued)

Flushing the HPLC system: After each use of a buffer or modifier, the HPLC system shall be flushed with 20 mL of water, then 10 mL of 50/50 (acetonitrile or methanol/water). The column can be included in this flushing if applicable.

Water Mobile Phase: Before each use of the HPLC system, the water last used must be replaced with fresh DI water in order to avoid introducing bacteria.

Repairs: All replacement of lamps, seals, or other parts should be performed according to manufacturer instructions.

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16.0 MISCELLANEOUS NOTES AND PRECAUTIONS

16.1 Additional information on preventive maintenance and calibrations is available in DCL SOP XX-EP-700 entitled: "Data Control Systems – Calibration."

17.0 REFERENCES

- 17.1 DCL SOP XX-DC-001, "Calibration of Pipettors"
- 17.2 DCL SOP QC-DC-002, "Refrigeration Units"
- 17.3 DCL SOP QC-DC-003, "Balances"
- 17.4 DCL SOP QC-DC-006, "Nonconformance/Corrective Action Report (NC/CAR) Procedures"
- 17.5 DCL SOP XX-EP-600, "Documentation Maintaining Instrument Records, Notebooks and Logbooks"
- 17.6 DCL SOP XX-EP-700, "Data Control Systems Calibration"

18.0 LIST OF EXHIBITS

18.1 Exhibit 1 – Example Out of Service Instrumentation Tag

EXHIBIT 1



Appendix B

Laboratory Certifications

Certification/Validations/Accreditations

AGENCIES

- AIHA American Industrial Hygiene Association
- AIHA ELLAP Environmental Lead Laboratory Accreditation Program
- AFCEE Air Force Center for Environmental Excellence
- NFESC Naval Facilities Engineering Service Center
- USACE U. S. Army Corps of Engineers—Missouri River Division
- USEPA Contract Laboratory Program
- USEPA Perchlorate Testing in Drinking Water

STATES

- ◆ Alaska (wastewater/hazardous waste) 4/30/05
- ◆ California NELAC (hazardous waste and drinking water) Expires 7/31/05
- Connecticut (water/wastewater/RCRA/Lead Paint/Asbestos) Expires 3/31/06
- Idaho (drinking Water) Expires 11/30/05
- **Iowa** (wastewater) Expires 8/1/06
- **Maryland** (drinking water) Expires 12/31/05
- ♦ Minnesota (RCRA) Expires 1/2/06
- Nevada (wastewater, RCRA and drinking water) Expires 7/31/05
- New Jersey NELAC (hazardous waste/wastewater/drinking water/air) Expires 06/30/05
- Utah NELAC (hazardous waste/wastewater/drinking water) Expires 11/30/05
- Washington (hazardous waste/wastewater) Expires 5/27/05

Proficiency Testing Participation

- Water Pollution (WP) Performance Evaluation Study (NIST Approved)
- Water Supply (WS) Performance Evaluation Study (NIST Approved)
- Soil Samples Performance Evaluation Study (NIST Approved)
- EPA Contract Laboratory Program (CLP) blind audits
- AIHA Proficiency Analytical Testing (PAT) Program
- AIHA Environmental Lead Proficiency Analytical Testing (ELPAT) Program
- AIHA Bulk Asbestos Proficiency Testing Program
- ♦ AIHA Environmental Microbiology Proficiency Analytical Testing (EMPAT) Program
- AIHA Beryllium Proficiency Analytical Testing (BePAT) Program



12/22/2005

Utah Department of Health David N Sundwall, MD Executive Director

Epidemiology and Laboratory Services Patrick F Luedtke, MD. MPH *Director of Public Health Laboratories*

Bureau of Laboratory Improvement David B Mendenhall, MPA, MT (ASCP) Bureau Director

DataChem Laboratories Inc. - Salt Lake Brent E Stephens Director 960 West Levoy Drive Salt Lake City UT 84123

Director,

On the basis of your most recent Proficiency Testing results and continuing compliance with the ELCP requirements, the laboratory listed is certified for environmental monitoring under the Clean Water Act and authorized to perform the following methods, for the analytes and matrix listed:

Non-Potable Water

Inorganics and Me	itals
120.1	Conductance (Specific Conductance, umhos at 25-C)
150 1	pH (Electometric)
160 1	Residue, Filterable (Gravimetric. Dried at 180-C)
160 2	Residue. Non-Filterable (Gravimetric. Dried at 103-105-C)
1664 A	Oil & Grease and Total Petroleum Hydrocarbons
200 7	Metals and Trace Elements in Water
200 7	Aluminum
200 7	Antimony
200 7	Arsenic
200.7	Barium
200 7	Beryllium
200 7	Boron
200 7	Cadmium
200 7	Calcium
200 7	Chromium
200 7	Cobalt
200 7	Соррег
200.7	iron
200 7	Lead
200 7	Magnesium
200 7	Мапдалеѕе
200 7	Molybdenum
200 7	Nickel
200 7	Potassium
200 7	Selenium
200 7	Silver
200 7	Sodium
200.7	Strontium
200 7	Thallium
200.7	Tin

Utah Department of Health The expiration for the laboratory's certification is 11/30/2006. The Utah Environmental Laboratory Certification Program (ELCP) encourages clients and data users to verify the most current certification letter for the authorized method. For further assistance please call. Lorna Ward 801-584-8469



ID # DATA1 Account # 8012667700

Utah!

Inorganics and N	letals
200 7	Titanium
200 7	Vanadium
200 7	Zinc
200 8	Metals And Trace Elements In Water and Wastes
200 8	Aluminum
200 8	Antimony
200 8	Arsenic
200 8	Barium
200.8	Beryllium
200 8	Cadmium
200 8	Chromium
200.8	Copper
200 8	Iron
200 8	Lead
200 8	Manganese
200 8	Molybdenum
200 8	Nickel
200 8	Selenium
200 8	Silver
200 8	Strontium
200 8	Thallium
200 8	Tin
200 8	Vanadium
200 8	Zinc
2340 B	Hardness (Calculation)
245 1	Mercury
300 0	Inorganic Anions In Water By Ion Chromatography
300 0	Chloride
300.0	Nitrate
300 0	Nitrite
300 0	ortho-Phosphate
300 0	Sulfate
310 1	Alkalinity
310 2	Alkalinity
335 4	Cyanide, Total
340 2	Fluoride
350 1	Nitrogen, Ammonia
353.2	Nitrogen, Nitrate-Nitrite
365.1	Phosphorous, All Forms
365 4	Phosphorous, Total
415 1	Organic Carbon. Total
420 4 [1993]	Phenolics, Total
4500 (F-) B	Fluoride (Preliminary Distillation)
4500 (F-) C	Fluoride (Ion-Selective Electrode)
HACH 8000	Chemical Oxygen Demand (COD)



DataChem Laboratories Inc. - Salt Lake Clean Water Act Page 3 of 3

The effective date of this certificate letter is: 12/21/2005

The analytes by method which a laboratory is authorized to perform at any given time will be those indicated in the most recent certificate letter. The most recent certification letter supersedes all previous certification or authorization letters. It is the certified laboratory's responsibility to review this letter for discrepancies. The certified laboratory must document any discrepancies in this letter and send notice to this bureau within 15 days of receipt. This certification letter will be recalled in the event your laboratory's certification is revoked.

All laboratories are required to submit a Corrective Action Report for all failed PT Audit Results to the Bureau of Laboratory Improvement.

Respectfully,

Luedtke, MD Patrick F

Director of Public Health Laboratories Deputy Director of Epidemiology and Laboratory Services





12/7/2005

Utah Department of Health David N Sundwall, MD Executive Director

Epidemiology and Laboratory Services Patrick F Luedtke, MD, MPH *Director of Public Health Laboratories*

Bureau of Laboratory Improvement David B Mendenhall, MPA. MT (ASCP) Bureau Director

DataChem Laboratories Inc. - Salt Lake Brent E. Stephens Director 960 West Levoy Drive Salt Lake City UT 84123

Director,

On the basis of your most recent assessment, Proficiency Testing results and continuing compliance with the ELCP requirements, the laboratory listed is certified for environmental monitoring under the Resource Conservation and Recovery Act and authorized to perform the following methods, for the analytes and matrix listed:

Characteris	tics		
		Non-	
	Onlini	Water	
	50110		h
1010			Ignitability
1110	<u>×</u>		Corrosivity Toward Steel
1311	<u> </u>	<u>×</u>	Toxicity Characteristic Leaching Procedure Metals
1311	<u>×</u>	<u>×</u>	Toxicity Characteristic Leaching Procedure Semi-Volatiles
1311	✓	\checkmark	Toxicity Characteristic Leaching Procedure Volatiles
1312	V	\checkmark	Synthetic Precipitation Leaching Procedure (TCLP Approval)
Sec 7 3 3	\checkmark	v	Reactive Cyanide
Sec 7 3 4	\checkmark	\checkmark	Reactive Sulfide
Sec 8 3	V	\checkmark	Reactivity
Inorganics			
		Non- Dotable	
	Colid	Water	
9012 A	V.	Z	Total and Amenable Cyanide
9030 A	V	1	Acid-Soluble and Acid-Insoluble Sulfides
9034	✓	✓	Acid-Soluble and Acid-Insoluble Sulfides
9040 B		\checkmark	pH
9045 C	~		Soil and Waste pH
9050		\checkmark	Specific Conductance
9060	,	\checkmark	Total Organic Carbon
9066	,	V	Phenolics
9081	\checkmark		Cation-Exchange Capacity of Soil (Sodium Acetate)
9095	V		Paint Filter Liquids Test



The expiration for the laboratory's certification is 11/30/2006 The Utah Environmental Laboratory Certification Program (ELCP) encourages clients and data users to verify the most current certification letter for the authorized method. For further assistance please call. Lorna Ward 801-584-8469



ID # DATA1 Account # 8012667700

46 North Medical Drive • Salt Lake City, UT 84113-1105 • phone (801) 584-8469 • fax (801) 584-8501 www health utah gov/els/labimp/



<u>Metal Dig</u>	estion	M	
		Non- Potable	
	Solid	Water	
2005 4	30110	7	Anid Digestica Total Recoverable or Dissolved Metals
3000 A		1	Acid Direction for Total Metals
3010 A	1	×.	Acid Digestion for Ford Merces
3015 A		<u>×</u>	Arid Dissetion of Sedimente, Sludges and Sells
3050 A	<u>×</u>		Acid Digestion of Sediments, Studges and Solis
3051 A	V.		Microwave Acia Digestion of Sediment, Sludges. Sons & Ons
<u>Metals</u>		A 1	
		Non- Potable	
	Solid	Water	
6010 B	V	~	Aluminum
6010 B	v	~	Antimony
6010 B	~	V	Arsenic
6010 B	Ī	V	Barium
6010 B	J		Benilium
6010 B	-	Ī	Cadmium
6010 B		V.	Calcium
6010 D			Chromium
6010 B		÷	Cabalt
6010 B			Coppar
6010 D	÷		kon
COTO B			
			Leau
6010 B	ž.		Magnesium
6010 B	ž	×	Manganese
6010 B	×.	× ·	MolyDaenum Nichal
6010 B		×.	Nickei
6010 B			Polassium
6010 B	×.	<u>×</u>	Selenium
6010 B	ž	×	Silver
6010 B	× Z		Stantium
6010 B		<u>×</u>	Stontum
6010 B	<u> </u>	<u>.</u>	
6010 B		×	
6010 B	<u>×</u>	<u>×</u>	
6010 B	×	ž	
0010 B	×.	ž	
6020 A	<u>×</u>	×	Authinum
6020 A	×	×	America
6020 A	<u>×</u>	×	Alseine
6020 A		×:	Danum
6020 A	V V	ž	Beryinum
6020 A	×	×	Gaomum
6020 A	<u>×</u>	×.	Unromium Caladi
6020 A	Y	×	Coose
6020 A	× ·	<u>×</u> :	Copper
6020 A	×.	<u>×</u>	Lead
6020 A	Ň	<u>×</u>	Manganese
6020 A	<u>×</u>	<u>×</u>	Noiybaenum
6020 A	<u>×</u>	<u>×</u>	
6020 A	×	× ·	Selenium
6020 A		X	Silver
6020 A	X	V	Inallum

Department of Health

<u>Metals</u>		N 1	
		Non- Potable	
	Solid	Water	
6020 A	0010	7	Tin
6020 A		<u> </u>	Venedium
6020 A		ž	Zine
6020 A	<u>×</u> .	a construction	Zinc Observive Hevevelet
7196 A	<u>×</u>	<u>×</u> :	Chromium, Hexavalent
7470 A		<u>×</u>	Mercury
7471 A	¥		Mercury
<u>Miscellane</u>	ous		
		Non- Potable	
	Solid	Water	
7580	V	V	White Phosphorus (P4)
Organic C	leanup		
<u>organic o</u>	canap	Non-	
		Potable	
	Solid	Water	
3640	\checkmark	\checkmark	Gel Permeation Cleanup
Organic E	xtractic	<u>n</u>	
		Non- Rotable	
	Colid	Water	
2510	30110	7	Separatory Europh Liquid Liquid Extractions
3310		<u> </u>	Continuous Liquid Liquid Extraction
3520		×	Continuous Elquid-Elquid Extraction
3540			
3050 A			Weste Dilution
3580	<u>×</u>	-	Waste Dilution
3010	.	- X *	neauspace
Organic Ir	istrume	Non-	
		Potable	
	Solid	Water	
8011		\checkmark	1,2-Dibromo-3-chloropropane (DBCP)
8011		V	1,2-Dibromoethane (EDB, Ethylene dibromide)
8011		v	EDB and DBCP by Microextraction and Gas Chromatography
8015 B	~	\checkmark	Diesel Range Organics (DROs)
8015 B	~	~	Gasoline Range Organics (GROs)
8015 B	V	\checkmark	Nonhalogenated Organics Using GC/FID
8081 A	V	V	4.4'-DDD
8081 A	V	V	4.4'-DDE
8081 A	V	~	4.4'-DDT
8081 A	V	V	Aldrin
8081 A	~	~	alpha-BHC(alpha-hexachlorocyclohexane)
8081 A	~	~	alpha-Chlordane
8081 A	V	$\overline{\mathbf{v}}$	beta-BHC(beta-hexachlorocyclohexane)
8081 A	~	~	Chlordane (technical)
8081 A	V	~	Chlordane - not otherwise specified
8081 A	V	V	Chlordane total
8081 A	Ī	V	delta-BHC(delta-hexachlorocyclohexane)
8081 A	Ī	$\overline{\checkmark}$	Dieldrin
8081 4	~	V	Endosulfan l
8081 A		~	Endosulfan II
000171		1.00	

(ELCP) e (ELCP) e Department of Health

8081 A

8081 A

V

1

V

V

Endosulfan sulfate

Endrin



Organic Instrumentation		ntation	
	Non-		
	Solid	Water	
0004 4	5010	7	Endrin Aldobudo
0001 A	- Ž	<u>.</u>	Endrin Adenyae
8081 A			
8081 A	× .	×.	gamma-BHC (Lindane, gamma-nexacitorocyclonexane)
8081 A	<u>×</u>	×.	gamma-chloroane
8081 A	<u>×</u>	×	Heptachlor
8081 A	<u>×</u>	×	Heptachior Epoxide
8081 A	×	<u>×</u>	Methoxychlor
8081 A	<u>S</u>	×.	Organochlorine Pesticides
8081 A	<u>×</u>	×	Toxaphene [Chlorinated camphene]
8082	×.	<u>×</u>	Aroclor-1016 [PCB-1016]
8082	<u>×</u>	<u>×</u>	Aroclor-1221 PCB-1221]
8082	2	<u>×</u>	Arocior-1232 [PCB-1232]
8082	<u>×</u>	~	Aroclor-1242 [PCB-1242]
8082	<u>×</u>	2	Aroclor-1248 [PCB-1248]
8082	<u> </u>	<u>×</u>	Aroclor-1254 [PCB-1254]
8082	V	V	Aroclor-1260 [PCB-1260]
8082	V	\checkmark	PCBs
8151 A	Z		2,4.5-TP (Silvex)
8151 A	\checkmark		2,4-D
8151 A	V		2,4-DB
8151 A	×.	عدر استحدد	Chlorinated Herbicides
8151 A	✓		Dalapon
8151 A	\checkmark		Dichlorprop(Dichloroprop)
8151 A	$\mathbf{\overline{\mathbf{v}}}$		Dinoseb (DNBP. 2-sec-butyl-4,6-dinitrophenol)
8151 A	v		MCPA
8151 A	$\overline{\mathbf{v}}$	and and	MCPP
8260 B	V	V	1,1,1,2-Tetrachloroethane
8260 B	V	$\overline{\mathbf{v}}$	1.1.1-Trichloroethane
8260 B	~	V	1.1.2.2-Tetrachloroethane
8260 B	V	V	1,1,2-Trichloro-1,2,2-trifluoroethane
8260 B	V	V	1.1.2-Trichloroethane
8260 B	V	~	1.1-Dichloroethane
8260 B	~	~	t 1-Dichloroethylene (-ethene)
8260 B	Ī		1 1-Dichloropropene
9260 B	Ī		1.2.3-Trichloropropage
0200 D			1.2.4-Trichloropenzene
0200 D	Ī	Ī	
0200 D			1.2-Dibromo-3-chloropropage (DBCP)
0200 D		<u>×</u> .	1,2-Dibromosthana (EDB, Ethylene dibromide)
6200 B	×		1.2 Disblessbergers
8260 B	×		
8260 B	×	×.	
8260 B	<u>×</u>		1,2-Dichloropropane
8260 B	<u>×</u>	Y	1,3,5- i rimetnyibenzene
8260 B	<u> </u>	<u>×</u> :	1,3-Dichlorobenzene
8260 B	×.	<u> </u>	1,3-Dichloropropane
8260 B	<u> </u>	<u>×</u>	1,4-Dichlorobenzene
8260 B	<u>×</u>	<u>×</u>	2-Chloroethyl Vinyl Ether
8260 B	<u> </u>	<u>×</u>	2-Chlorotoluene
8260 B	<u>×</u>	<u>×</u>	2-Hexanone
8260 B	×	×	4-Chlorotoluene
8260 B	v	~	4-Methyl-2-pentanone (MIBK, Isopropylacetone, Hexone)

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Organic Instrumentation		ntation	
		Potable	
	Solid	Water	
8260 B	V	~	Acetone
8260 B	~	V	Acetonitrile
8260 B	~	~	Acrolein (Propenal)
8260 B	V	V	Allvi Alcohol
8260 B	V	V	Benzene
8260 B	V	V	Bromobenzene
8260 B	V	~	Bromochloromethane
8260 B	~	~	Bromodichloromethane
8260 B	\checkmark	$\overline{\mathbf{v}}$	Bromoform
8260 B	V	V	Carbon Disulfide
8260 B	V	~	Carbon Tetrachloride
8260 B	\checkmark	V	Chlorobenzene
8260 B	V	V	Chloroethane
8260 B	V		Chloroform
8260 B	V	✓	cis-1.2-Dichloroethene (-ethylene)
8260 B	V	V	cis-1,2-Dichloroethene (-ethylene)
8260 B	V	~	cis-1,3-dichloropropene
8260 B	\checkmark	V	Cyclohexane
8260 B	V	×.	Dibromomethane
8260 B	V	\checkmark	Dichlorodifluoromethane
8260 B	X	×	Dichlorofluoromethane
8260 B	✓	V	Diethyl Ether
8260 B	×	Z	Ethyl Acetate
8260 B	V	V	Ethyl Ether
8260 B	~	\mathbf{V}	Ethyl Methacrylate
8260 B	×	V	Ethylbenzene
8260 B	~	Υ.	Hexachlorobutadiene
8260 B	×	<u>×</u>	lodomethane (Methyl iodide)
8260 B	_	<u>×</u>	Isopropylbenzene
8260 B	<u>×</u>	<u>×</u>	meta-Xylene
8260 B	<u>×</u>	⊻	Methyl bromide [Bromomethane]
8260 B	Z	<u>×</u>	Methyl chloride [Chloromethane]
8260 B	\mathbf{X}	<u> </u>	Methyl Ethyl Ketone (MEK, 2-Butanone)
8260 B	<u>×</u>	X	Methyl Ethyl Ketone (MEK, 2-Butanone)
8260 B	<u>×</u>	×.	Methyl Isobutyl Ketone
8260 B	<u>×</u>	<u> </u>	Methyl-t-Butyl Ether (MTBE)
8260 B	<u>×</u>	<u>×</u>	Methylene Chloride
8260 B	<u>×</u>	Y	Naphthalene
8260 B	<u>×</u>	<u>×</u>	ortho-Xylene
8260 B	ž	×	para-Xylene
8260 B	×	<u>×</u>	Pentachioroethane
8260 B	<u>×</u>	× ·	sec-Butylbenzene
8260 B	ž	× ·	Styrene
8260 B	<u>×</u>	<u>.</u>	i etrachioroethylene (Perchioroethylene -ethene)
6260 B	× ·	×	rouene
8260 B		ž	trans-1,2-Dichloroethylene (-ethene)
8260 B	<u>×</u>	×.	trans- i.o-ucnioropropyiene (-propene)
8260 B	<u>×</u>		nchoroenene (mchoroennylene)
6260 B	×.	× ·	
0200 B	×	×	
9200 B	<u>v</u>	Y	Vinyi Chiolide

Utah Department of Health



Organic Instrumentation				
			Non- Potable	
	0	hilo	Water	
9260 B			7	Volatile Organic Compounds
0200 0	•	<u> </u>	3	Vulane Total
0200 0	•		-	Xylenes Total
0200 0	•			Ayleries, Total
8270 0	,	×.		1,2,4-Inchlorobenzene
8270 0	,	× -		
8270 0	,		ž	
8270 0	·	×		1,4-Dichlorophonol
8270 0	<i>;</i>	×	ž	2,4,5-Inchiorophenol
8270 0	,	¥	<u>×</u>	2,4,6-menorophenor
8270 C		<u>×</u>	<u>×</u>	2.4-Dichlorophenol
8270 C	5	<u>×</u>		2,4-Dimethylphenol
8270 C	;	×.	×.	
8270 C	;	×	× ·	2,4-Dinitrotoluene (2,4-DNT)
8270 C	;	Y.J.	×	2,6-Dinitrotoluene (2.6-DNT)
8270 C	2	<u>×</u>	×	2-Chloronaphthalene
8270 C	2	<u>×</u>	×.	2-Chlorophenol
8270 C)	Y	×	2-Methyl-4,6-dinitrophenol (4,6-Dinitro-2-methylphenol)
8270 C	2		×	2-Methylphenol (o-cresol. 2-Hydroxytoluene)
8270 C	5	<u>×</u>	<u>×</u>	2-Nitroaniline
8270 C	2	<u>×</u>	<u>×</u>	2-Nitrophenol
8270 C)	<u>×</u>	×.	3.3'-Dichlorobenzidine
8270 0	2	<u>×</u>	<u>×</u>	3-Methylphenol (m-cresol, 3-Hydroxytoluene)
8270 0	2	V	<u>×</u>	3-Nitroaniline
8270 0	5	$\mathbf{\underline{\vee}}$	<u> </u>	4-Bromophenyl Phenyl Ether
8270 0	2	<u>×</u>	<u>×</u>	4-Chloro-3-methylphenol
8270 (2	Ľ	<u>×</u>	4-Chloroaniline
8270 (2	Y	2	4-Chlorophenyl Phenyl Ether
8270 0	0	<u>×</u>	<u>×</u>	4-Methylphenol (p-cresol, 4-Hydroxytoluene)
8270 (2	⊻	<u>×</u>	4-Nitroaniline
8270 0	2	V	<u>×</u>	4-Nitrophenol
8270 0	2	V	×	Acenaphthene
8270 (5	V	<u>×</u>	Acenaphthylene
8270 (5	×	\mathbf{Z}	Anthracene
8270 (0	×	2	Benzo(a)anthracene
8270 (2	V	×	Benzo(a)pyrene
8270 (0	Y	\checkmark	Benzo(b)fluoranthene
8270 (0	~	X	Benzo(g,h,i)perylene
8270 (0	✓	\checkmark	Benzo(k)fluoranthene
8270 (0	V	1	Benzoic Acid
8270 (С	V	V	Benzyl alcohol
8270 (C	✓	×	bis(2-chloroethoxy)methane
8270 (С	✓	V	bis(2-Chloroethyl)ether
8270 (С	✓	V	bis(2-chloroisopropyl)ether
8270	С	V	\checkmark	bis(2-Ethylhexyl) phthalate (DEHP)
8270 (С	V	\checkmark	Butyl Benzyl Phthalate
8270	с	✓.	×.	Carbazole
8270	с	<u>√</u>	~	Chrysene
8270	с	V	V	Di-n-butyl phthalate
8270	с	V	Z	Di-n-octyl Phthalate
8270	с	V	×.	Dibenzo(a,h)anthracene
8270	с	\checkmark	 	Dibenzofuran





Organic I	nstrume	ntation	
		Non- Potable	
	Solid	Water	
8270 C	V	V	Diethvl Phthalate
8270 C	~	$\overline{\mathbf{v}}$	Dimethyl Phthalate
8270 C	V	V	Fluoranthene
8270 C	V	V	Fluorene
8270 C	~	~	Hexachlorobenzene
8270 C	~	~	Hexachlorobutadiene
8270 C	V	V	Hexachlorocyclopentadiene
8270 C	V	V	Hexachloroethane
8270 C	~	~	Indeno(1.2,3-cd)pyrene
8270 C	~	✓	Isophorone
8270 C	~	~	n-Nitroso-di-n-Propylamine
8270 C	~	and a strain of	n-Nitrosodimethylamine
8270 C	~	~	n-Nitrosodiphenylamine
8270 C	\checkmark	~	Naphthalene
8270 C	V.	V	Nitrobenzene
8270 C	✓	V	Pentachlorophenol
8270 C	v	V	Phenanthrene
8270 C	V	\checkmark	Phenol
8270 C	~	X	Pyrene
8270 C	V	×	Pyridine
8270 C	\checkmark	V	Semivolatile Organic Compounds
8310	\checkmark	V	1-Methylnaphthalene
8310	~	V	2-Methylnaphthalene
8310	\mathbf{V}	\checkmark	Acenaphthene
8310	<u> </u>	Υ.	Acenaphthylene
8310	<u>×</u>	<u>×</u>	Anthracene
8310	<u>×</u>	~	Benzo(a)anthracene
8310	×.	<u>×</u>	Benzo(a)pyrene
8310	<u>×</u>	×	Benzo(b)fluoranthene
8310	<u>×</u>	×	Benzo(g,h,i)perylene
8310	×	<u>×</u>	Benzo(k)fluoranthene
8310	×.	×.	Chrysene
8310	ž	×.	Dibenzo(a,h)anthracene
8310	. <u></u>	×	Fluoranthene
8310	X	×	
8310	×	×	Indeno(1,2,3-c.d)pyrene
8310	× ·	v v	Depenthrono
8310		ž	Phenanthiene Delynydear Aromatia Hydrocarboas
0310	10100		Polynucieal Aromatic Hydrocarbons
8330	Ī	-	1 3 5-Trinitrohenzene (1 3 5-TNB)
8330	V	~	1 3-Digitrobenzene (1 3-DNB)
8330	~	~	2.4.6-Trinitrotoluene (2.4.6-TNT)
8330	~	~	2.4-Dinitrotoluene (2.4-DNT)
8330	V	~	2.6-Dinitrotoluene (2.6-DNT)
8330	~	~	2-Amino-4,6-Dinitrotoluene (2-Am-DNT)
8330	V	V	2-Nitrotoluene (2-NT)
8330	~	~	3-Nitrotoluene (3-NT)
8330	V	~	4-Amino-2,6-Dinitrotoluene (4-Am-DNT)
8330	V	~	4-Nitrotoluene (4-NT)
8330	~	\checkmark	Hexahydro-1. 3. 5-tritro-1, 3, 5-triazine (RDX)





Organic Instrumentation Non-			
	Solid	Potabie Water	
8330	✓	✓	Methyl-2,4.6-Trinitrophenylnitramine (TETRYL)
8330	✓	V	Nirtoaromatics and Nitramines
8330	V	V	Nitrobenzene
8330	V	~	Nitroglycerin
8330	V	X	Octahydro-1,3,5,7-Tetranitro-1,3.5,7-Tetrazocine (HMX)
8330	V	~	Pentaerythrite tetranitrate (PETN)
8332	V	×	Nitroglycerine
8332	×	✓	Nitroglycerine By HPLC
<u>Volatile Or</u>	ganic	Preparati Non-	on

	Solid	Potable Water	
5030	V	✓	Purge-and-Trap for Aqueous Samples
5035	V	, and the second	Purge-and-Trap and Extraction for Volatile Organics

The effective date of this certificate letter is: 12/5/2005

The analytes by method which a laboratory is authorized to perform at any given time will be those indicated in the most recent certificate letter. The most recent certification letter supersedes all previous certification or authorization letters. It is the certified laboratory's responsibility to review this letter for discrepancies. The certified laboratory must document any discrepancies in this letter and send notice to this bureau within 15 days of receipt. This certificate letter will be recalled in the event your laboratory's certification is revoked

Respectfully

Patrick F. Luedtke, MD, MPH

Director of Public Health Laboratories Deputy Director of Epidemiology and Laboratory Services







State of Utah JON HUNTSMAN Jr. Governor GARY HERBERT Lieutenant Governor

8/7/2006

Utah Department of Health David N. Sundwall, MD *Executive Director*

Epidemiology and Laboratory Services Patrick F. Luedtke, MD, MPH. *Director of Public Health Laboratories*

Bureau of Laboratory Improvement David B Mendenhall, MPA, MT (ASCP) *Bureau Director*



ID # DATA1 Account # 8012667700

DataChem Laboratories Inc. - Salt Lake Brent E. Stephens 960 West Levoy Drive Salt Lake City UT 84123

Director,

On the basis of your most recent assessment, Proficiency Testing results and continuing compliance with the ELCP requirements, the laboratory listed is certified for environmental monitoring under the Safe Drinking Water Act and authorized to perform the following methods, for the analytes and matrix listed:

Drinking Water

Inorganics and M	<u>Aetals</u>
120.1	Conductivity
150.1	pH
160.1	Residue, Filterable
200.7 [1994]	Vanadium
200.7 [1994]	Zinc
200.7 [1998]	Metals and Trace Elements in Water
200.7 [1998]	Aluminum
200.7 [1998]	Antimony
200.7 [1998]	Arsenic
200.7 [1998]	Barium
200.7 [1998]	Beryllium
200.7 [1998]	Boron
200.7 [1998]	Cadmium
200.7 [1998]	Calcium
200.7 [1998]	Chromium
200.7 [1998]	Cobalt
200.7 [1998]	Iron
200.7 [1998]	Magnesium
200.7 [1998]	Manganese
200.7 [1998]	Molybdenum
200.7 [1998]	Nickel
200.7 [1998]	Selenium
200.7 [1998]	Silver
200.7 [1998]	Sodium
200.7 [1998]	Strontium
200.7 [1998]	Tìn
200.7 [1998]	Titanium
200.7 [1998]	Vanadium
200.7 [1998]	Zinc
200.8 [1994]	Metals And Trace Elements In Water and Wastes









DataChem Laboratories Inc. - Salt Lake Safe Drinking Water Act Page 2 of 4

Inorganics and M	letals
200.8 [1994]	Antimony
200.8 [1994]	Arsenic
200.8 [1994]	Barium
200.8 [1994]	Beryllium
200.8 [1994]	Cadmium
200.8 [1994]	Chromium
200.8 [1994]	Manganese
200.8 [1994]	Nickel
200.8 [1994]	Selenium
200.8 [1994]	Silver
200.8 [1994]	Strontium
200.8 [1994]	Thallium
200.8 [1994]	Vanadium
200.8 [1994]	Zinc
200.8 [1994]	Molybdenum
245.1	Mercury
300.0	Chloride
310.1	Alkalinity
314.0	Perchlorate
335.3	Cvanide
340.2	Fluoride
4500 (E-) B	Fluoride Prelimiary Distillation Step
4500 (F-) C	Fluoride by Ion-Selective Method
Nitrate	
300.0	Nitrata
353.0	
JJJ.Z	Nitiate/Initite
300.0	Nitrito
Organica	Nithe
504 1 [1005]	EDB and DBCB in Water
504.1 [1995]	1.2 Dibromoethano (EDB, Ethylono dibromide)
504,1 [1995]	1.2 Dibromo 3 obleropropago (DBCP)
504.1 [1995]	1,2-2 Trichleropropage
504.1 [1995]	Rurgoshlo Organic Compounds In Water
524.2 [1995]	Purgeable Organic Compounds in Water
524.2 [1995]	Bremehenzene
524.2 [1995]	Bromobleremethano
524.2 [1995]	Bromocnioromethane Bromodickleremethane [Dicklerehrememethane]
524.2 [1995]	Bromodiciniorometriane [Diciniorobiomometriane]
524.2 [1995]	Biomoloim Brememothene [Methyl bremide]
524.2 [1995]	
524.2 [1995]	
524.2 [1995]	sec-Butylbenzene
524.2 [1995]	
524.2 [1995]	Chlorodenzene
524.2 [1995]	
524.2 [1995]	Chlorotorm
524.2 [1995]	
524.2 [1995]	2-Chlorotoluene
524.2 [1995]	4-Gniorotoluene
524.2 [1995]	
524.2 [1995]	
524.2 [1995]	1,3-Dichlorobenzene
524.2 [1995]	1,2-Dichlorobenzene
524.2 [1995]	1,4-Dichlorobenzene

Department of Health

Where ideas connect

DataChem Laboratories Inc. - Salt Lake Safe Drinking Water Act Page 3 of 4

<u>Organics</u>	
524.2 [1995]	Dichlorodifluoromethane
524.2 [1995]	1,1-Dichloroethane
524.2 [1995]	1,2-Dichloroethane
524.2 [1995]	1,1-Dichloroethene
524.2 [1995]	cis-1,2-Dichloroethene
524.2 [1995]	trans-1,2-Dichloroethene
524.2 [1995]	1,2-Dichloropropane
524.2 [1995]	1,3-Dichloropropane
524.2 [1995]	2,2-Dichloropropane
524.2 [1995]	1,1-Dichloropropene
524,2 [1995]	cis-1,3-Dichloropropene
524.2 [1995]	trans-1.3-Dichloropropene [-pylene]
524 2 [1995]	Ethylbenzene
524 2 [1995]	Hexachlorobutadiene
524 2 [1995]	Isopropylbenzene
524 2 [1995]	4-Isopropytoluepe
524 2 [1005]	Nanhthalene
524.2 [1005]	n-Bronylbenzene
524.2 [1005]	Styrene
524.2 [1995]	1 1 2 Tetrachloroethane
524.2 [1995]	1,1,2,2 Tetrachloroethane
524.2 [1995]	Totrachleraothana Lathylana, Barahleraothylana)
524.2 [1995]	
524.2 [1995]	1 0 2 Trichlerchonzono
524.2 [1995]	1,2,3-Trichlershanzana
524.2 [1995]	1,2,4-Trichlereethere
524.2 [1995]	1, 1, 1- Trichlereethane
524.2 [1995]	
524.2 [1995]	
524.2 [1995]	
524.2 [1995]	1,2,4-I rimethylbenzene
524.2 [1995]	1,3,5-Trimethylbenzene
524.2 [1995]	Vinyl Chloride
524.2 [1995]	Total Triholamethanes
524.2 [1995]	Methyl Tert-Butyl Ether (MTBE)
524.2 [1995]	Methylene Chloride [Dichloromethane, DCM]
524.2 [1995]	meta-Xylene
524.2 [1995]	ortho-Xylene
524.2 [1995]	para-Xylene
524.2 [1995]	trans-1,2-Dichloroethene
524.2 [1995]	2-Butanone [Methyl ethyl ketone, MEK]
<u>Pb/Cu</u>	
200.7 [1998]	Copper
200.7 [1998]	Lead
200.8 [1994]	Copper
200.8 [1994]	Lead
<u>Sulfates</u>	
300.0	Sulfate



The expiration for the laboratory's certification is 11/30/2006. The Utah Environmental Laboratory Certification Program (ELCP) encourages clients and data users to verify the most current certification letter for the authorized method. For further assistance please call Lorna Ward 801-584-8469.

46 North Medical Drive • Salt Lake City, UT 84113-1105 • phone (801) 584-8469 • fax (801) 584-8501 www.health.utah.gov/els/labimp/ DataChem Laboratories Inc. - Salt Lake Safe Drinking Water Act Page 4 of 4

The effective date of this certificate letter is: 8/1/2006.

The analytes by method which a laboratory is authorized to perform at any given time will be those indicated in the most recent certificate letter. The most recent certification letter supersedes all previous certification or authorization letters. It is the certified laboratory's responsibility to review this letter for discrepancies. The certified laboratory must document any discrepancies in this letter and send notice to this bureau within 15 days of receipt. This certificate letter will be recalled in the event your laboratory's certification is revoked.

Respectfully, atrich

Patrick F. Luedtke, MD, MPH. Director of Public Health Laboratories Deputy Director of Epidemiology and Laboratory Services



Appendix C

Laboratory QA Manual
QAPP 2006





Quality Assurance Project Plan

January 30, 2006

QUALITY ASSURANCE PROGRAM PLAN OF DATACHEM LABORATORIES, INC.

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DataChem Laboratories, Inc. Quality Assurance Program Plan Revision 9, January 30, 2006 Page 5 of 38

1.0 Introduction

QUALITY ASSURANCE PROGRAM PLAN OF DATACHEM LABORATORIES, INC. QAPP-2006

Address:	960 West LeVoy Drive Salt Lake City, UT 84123-2547
Telephone Numbers:	801-266-7700 Fax: 801-268-9992
Director:	Brent E. Stephens
Quality Assurance Manager:	Robert P. Di Rienzo
Organics Technical Manager	Richard W. Wade
Inorganics Technical Manager	Jeffery S. Ward
Manual Version:	Revision 9
Effective Date:	January 30, 2006
Approval for Implementation:	Concurrence

By: ______ Brent E. Stephens By: _

Robert P. Di Rienzo Quality Assurance Manager

Date:

By:

Richard W. Wade Organics Technical Manager

Laboratory Director

Date:

Date:

By:

Jeffery S. Ward Inorganics Technical Manager

Date:



DataChem Laboratories, Inc. Quality Assurance Program Plan Revision 9, January 30, 2006 Page 6 of 38

1.1 Purpose

This Quality Assurance Program Plan (QAPP) describes the policies, procedures and accountabilities established by the Environmental Laboratory of DataChem Laboratories, Inc. (DCL) to ensure that the environmental and radiochemical results reported from the analysis of air, water, soil, waste, and other matrices are reliable and of known quality. This document describes the quality assurance and quality control procedures followed to generate reliable analytical data.

This QAPP is designed to be an overview of DCL operations. Detailed methodologies and practices are written in DCL Standard Operating Procedures (SOPs). Where appropriate, DCL SOPs are referenced in this document to direct the reader to more complete information. A discussion of DCL SOPs is found in Section 9.2 of this plan, and a list of current SOPs is found in Appendix 14.11.

DCL maintains certifications pertaining to various commercial and government entities; these are listed in Appendix 14.1. Each certification requires that the laboratory continue to perform at levels specified by the programs issuing certification. Program requirements can be rigorous; they include semiannual performance evaluations as well as annual audits of the laboratory to verify compliance.

The State of Utah has primacy in administering certification of this laboratory to perform EPA methods. Thus, the Utah State Health Department certifies DCL to perform EPA methods under Utah Rule R444-14. For that reason, reference is made to Utah Rule R444-14 in this QAPP.

1.2 Quality Assurance Policy

DCL is committed to producing legally defensible analytical data of known and acceptable precision and accuracy for use in compliance with the Safe Drinking Water Act, the Clean Water Act, and the Resource Conservation and Recovery Act. This QAPP is designed to satisfy the applicable requirements of the State of Utah and the United States Environmental Protection Agency (USEPA). DCL complies with the National Environmental Laboratory Accreditation Conference (NELAC) standards.

DCL corporate management has committed its full support to provide the personnel, facilities, equipment, and procedures required by this QAPP.

1.3 Ethics Policy on Waste, Fraud, and Abuse

DCL policy on waste, fraud, and abuse is described in DCL SOP LAB-001, "Ethics and Data Integrity." It is the policy of DCL to generate accurate and reliable data in accordance with contractual and regulatory requirements.

It is also the policy of DCL to perform work for clients in the most efficient manner possible, avoiding waste of resources. It is the role of both DCL management and employees to ensure that work for clients is performed most efficiently and effectively by



properly utilizing DCL purchased materials, equipment, and the time and ability of personnel.

1.4 Quality System

This QAPP and SOPs referenced in this document comprise the DCL Quality System. This Quality System includes all quality assurance policies and quality control procedures. Review of the Quality System is completed on an ongoing basis as described is section 12.6 of this QAPP. The Quality System is based on the required elements as specified in NELAC 2003 Chapter V, section 5.1 through 5.16.

1.5 Client Confidentiality

Documents provided to the laboratory are held in strict confidence by project management staff. Documents pertaining to quality assurance and analytical requirements are reviewed with appropriate managers and staff through the project specific meetings and the project protocol worksheet (PPW). Project related information provided by clients is securely archived using procedures described in the SOP Lab-013 "Archives". The transmittal of final results is specified in the PPW and followed unless specific changes are made to the PPW by the Project Manager assigned to the client/project. Client communication procedures and documentation requirements are listed in the DCL SOP LAB-023 "Client Communication".

1.6 Data Integrity Policies

DCL policy is described in DCL SOP LAB-001, "Ethics and Data Integrity". It is the policy of DCL to generate accurate and reliable data in accordance with contractual and regulatory requirements. It is against DCL policy to improperly manipulate or falsify data or to engage in any other unethical conduct as defined in the DCL Laboratory Ethics SOP. DCL provides mandatory initial and annual refresher training to all employees on SOP Lab-001 "Ethics and Data Integrity".

The pertinent DCL Project Manager must approve deviations from contractual requirements (protocols) and/or SOPs. The Project Manager obtains approval for any such deviations, either in writing or by phone (documented in a phone log) from pertinent contract authorities. In addition, DCL requires that deviations from contractual requirements that might affect data quality be reported to clients. Any employee who knowingly manipulates and/or falsifies data or documents or engages in any unethical conduct is subject to immediate release from employment.

It is also the policy of DCL to perform work for clients in the most efficient manner possible, avoiding waste of resources. It is the role of both DCL management and employees to ensure that work for clients is performed most efficiently and effectively by properly utilizing DCL purchased materials, equipment, and the time and ability of personnel.

DCL employees who are aware of, or reasonably suspicious of, any case of data manipulation, falsification of data, waste of resources, or other unethical practice or



misconduct shall notify the cognizant manager. Under the direction of the laboratory director, every allegation of unethical conduct will be fully investigated.

2.0 Laboratory Organization and Responsibility for Quality Assurance

2.1 Laboratory Organization

The Environmental Laboratory is organized around the functions described in the following sections. Appendix 14.2 of this QAPP contains a detailed organization chart. Each of these organizational elements has specific responsibilities for quality assurance in the laboratory.

2.2 Responsibilities for Quality Assurance

2.2.1 DCL Laboratory Director

The Laboratory Director is responsible to ensure that:

- Employees have sufficient experience and training to perform QAPP-related duties and procedures.
- The necessary facilities and equipment are available to meet the commitments of the laboratory.
- Sample handling, instrument calibration, sample analysis, and related activities are conducted and documented as described in this QAPP, its related Standard Operating Procedures (SOPs), and its referenced methods.
- Routine QC samples are prepared, analyzed, and reviewed as required by this QAPP.
- Regular internal and external audits are conducted and documented to assess compliance with this QAPP.
- Corrective action is initiated and completed to remedy discrepancies or problems identified in any laboratory process.

2.2.2 Quality Assurance

The Quality Assurance Manager reports directly to the President and is responsible to:

- Understand, monitor and evaluate the quality assurance (QA) and quality control (QC) activities described in this QAPP and its references, reporting deficiencies and identifying resource requirements to the Laboratory Director.
- Conduct and document an annual internal audit of laboratory procedures to ensure compliance with this QAPP and its references.
- Conduct an annual review and update of this QAPP and laboratory Standard Operating Procedures (SOPs).
- Arrange for the analysis of QC and performance evaluation (PE) samples.



- Schedule and document the performance of annual MDL studies for QAPP-related methods and analytes.
- Maintain a record of ongoing personnel training for QAPP-related activities, reporting training deficiencies to the Laboratory Director.
- Maintain the laboratory corrective action program.
- 2.2.3 Inorganic Chemistry and Organic Chemistry

The managers of these operations report directly to the Laboratory Director and are responsible to:

- Read, understand and follow this QAPP with its references.
- Ensure that each set of reported results meets the requirements specified in this QAPP and meets the client's requirements as defined in the applicable Project Protocol Worksheet (PPW).
- Ensure that personnel are trained and utilized effectively.
- Ensure that facilities and equipment are maintained and utilized effectively.
- Ensure that supplies are available and utilized effectively.
- Immediately report technical and quality problems to the Laboratory Director.

2.2.4 Project Managers

Project Managers report directly to the Laboratory Director. Project Managers are especially involved in the production and assurance of quality results. Client communication procedures and documentation requirements are listed in the DCL SOP LAB-023 "Client Communication". They are responsible to:

- Complete and distribute a Project Protocol Worksheet (PPW) for each project before the laboratory starts work on the project.
- Immediately communicate to the laboratory changes made to projects in progress and document these changes in the PPW as appropriate.
- Respond to client requests for information and coordinate responses to client audits.
- Perform a final review to verify that data reports submitted to the client meet all requirements.

2.2.5 Computer Support

Computer Support personnel are responsible to:

- Specify, procure, and maintain all computer hardware and software used at DCL.
- Program and maintain the DCL Laboratory Information Management System (LIMS).
- Perform backups and safely archive stored data.
- Document software produced at DCL.



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2.3 QA Plan Implementation

The Laboratory Director is responsible to ensure that resources for implementation of the QAPP are available and that implementation is expedited. The Quality Assurance Manager is responsible to implement this QAPP and to verify laboratory compliance with it through internal audits and other reviews of performance. A copy of this QAPP is issued to each member of the DCL staff involved in QAPP-related activities. Each member of the laboratory staff is responsible to understand and follow this QAPP, produce results that conform to this QAPP, and meet client requirements. The Quality Assurance Manager has the authority to stop any laboratory process that does not meet the requirements of this QAPP. The Laboratory Director will designate deputies in case of absence of the Technical Directors and/or Quality Assurance Officer.

3.0 Personnel

The DCL environmental laboratory employs sufficient personnel to complete required chemical analyses and support activities. Support activities include personnel recruiting and management, sample receiving and logging, computer programming and data processing, analytical report preparation, equipment procurement, and method development.

3.1 Key Personnel

Key personnel as defined by Utah Rule 444-14, "Rule for Certification of Environmental Laboratories," are identified in the following table with their corresponding DCL titles.

Rule 444-14 Title	DCL Title	Key Individual
Laboratory Director	Laboratory Director	Brent E. Stephens
Laboratory Supervisor	Inorganic Technical Manager	Jeffery S. Ward
Laboratory Supervisor	Organic Technical Manager	Richard W. Wade
Quality Assurance Officer	Quality Assurance Manager	Robert P. Di Rienzo

Appendix 14.3 of this QAPP contains biographies of the key personnel documenting applicable experience.

3.2 Laboratory Staff

In addition to key personnel, the DCL staff members directly responsible for the production of quality analytical results are assigned to the following positions:

3.2.1 Radiation Safety Officer (RSO)

The RSO is responsible for technical aspects and safety issues related to samples received under the DCL radiation license.



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3.2.2 Chemist/Scientist

Chemists and Scientists perform analyses according to specified methods. They exercise technical judgment and review the results of other analysts. They are responsible to implement the requirements of this QAPP and verify its implementation in their review of others' work.

3.2.3 Project Manager/Client Service Representative

Project Managers and Client Service Representatives are responsible for clear, timely communication between clients and the laboratory. They are also responsible to ensure that the requirements of this QAPP and client QA/QC requirements are implemented.

3.2.4 Technician

Technicians work under the direction of a chemist or scientist to perform analyses. They are responsible to implement specific instructions in keeping with this QAPP and client QA/QC requirements. Technicians exercise technical judgment as assigned based upon training and experience.

The education and experience of the DCL staff are summarized in Appendix14.4.

3.3 Training

All DCL staff assigned to perform tasks affecting or relating to environmental testing data quality receive training relative to pertinent areas of responsibility, both prior to performing work on client samples and on an ongoing basis. Such training comes from internal and external sources. The DCL training program specified in the DCL SOP Lab-006 "Training" includes quality training, technical training, safety training, and other training as described in this QAPP. DCL Managers are responsible to ensure that all staff training is initiated, completed, verified, and documented.

The specific training and experience of laboratory personnel are documented in individual training files maintained in accordance with DCL SOP LAB-007, "Record of Training," and include documentation of analytical proficiency through the analysis of QC and PT samples.

3.3.1 Quality Training

The DCL Quality Assurance Manager is responsible to orient new analytical personnel to the DCL QA program, policies, and procedures. This required orientation includes training classes and video presentations, as well as reading and understanding this QAPP. Quality orientations are presented on an as-needed basis as new employees are hired. The quality orientation has two goals: to



communicate information and to emphasize the importance of implementing quality in the laboratory.

3.3.2 Technical Training

Technical training is accomplished through reading SOPs, using other training materials (manufacturer manuals, videos, computer-based instruction), observation of others' performance, and performing tests under direct supervision. When possible, training is verified through the successful analysis of QC samples. The cognizant manager evaluates the acceptability of prior experience and training.

As laboratory SOPs are updated, assigned analysts receive notification. They are required to read the revised SOPs and document that reading in their training files before performing analyses using the revised procedure.

Demonstration of Capability – A demonstration of capability is conducted initially and at least annually for all methods. The initial demonstration of capability includes results from four quality control samples whenever possible. The continuing demonstration of capability includes four consecutive laboratory control samples and/or PT Results.

3.3.3 Safety Training

Managers are responsible for continuous laboratory safety training and ensuring safety awareness in the laboratory. See section 4.7. Training to handle and properly dispose of hazardous waste is provided, as appropriate, for each work area. Quarterly meetings of the Safety Committee provide a forum to identify and resolve safety concerns.

3.3.4 Other Training

The RSO directs training to handle radiological materials and mixed waste samples. All analysts must complete this training satisfactorily before working with radiological materials and samples. Training concerning the use of the computer system and automated data handling systems is conducted by both the cognizant managers and computer support personnel. Management training is conducted by DCL staff or by outside consultants.



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4.0 Facilities

The DCL facility, constructed in 1988 and located at 960 West LeVoy Drive, was designed and built to function as a laboratory. The area used for chemical analyses and associated activities is approximately 25,000 square feet. It is a secure facility with electronically coded card key access for employees; visitors access the facility through a reception area. The floor plan of the DCL building is included in Appendix 14.5.

4.1 Laboratory Areas

Laboratory areas are segregated by HVAC systems to contain contamination and to eliminate potential contamination from specific laboratory areas that require low ambient chemical background levels for successful analysis. The facility is cleaned and maintained to ensure that contamination is minimized and that laboratory systems perform reliably.

4.2 Bench Space

Each area in the laboratory has adequate bench space for instrumentation and for the processes assigned to that area. Frequently, samples are placed on carts to allow efficient processing from preparation through analysis and into storage.

4.3 Storage Space

In addition to the bulk storage areas, each laboratory area has cabinet and under-bench storage. Some areas have walk-in storage as well.

4.4 Lighting

Each laboratory area was built with lighting designed for analytical work. The lighting has been upgraded to achieve more energy efficiency. Emergency lighting is provided in the event of a power failure.

4.5 Air-handling Systems

Laboratory ventilation is provided by single-pass airflow to the individual laboratories. The sample preparation and extraction laboratories are maintained at a negative pressure relative to the rest of the building. Air intakes and exhausts are positioned to reduce cross-contamination by taking advantage of the prevailing winds.

4.6 Laboratory Reagent Water System

Laboratory reagent water is prepared and maintained in a reservoir using a combination of deionization, reverse osmosis, and UV radiation. It is delivered throughout the laboratory by a constantly circulating system constructed of polyvinyl difluoride piping. The water supplied meets or exceeds the specifications for ASTM Type II water. The conductance of the reagent water system is monitored and maintained continuously to keep the reagent water within ASTM specifications.



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4.7 Safety Considerations

The safety plan of DCL is described in detail in the document entitled, "Safety Manual and Chemical Hygiene Plan."

The laboratory is equipped with safety showers and eyewashes. Fume hood face velocities are checked routinely, and maintenance is conducted to ensure correct hood performance.

Safety Showers and eyewashes are inspected in accordance with the applicable OSHA requirements on a yearly basis, not to exceed 12 months.

Fume hoods are performance tested semi-annually using a calibrated anemometer. The Chemical Hygiene Plan in Section 3, Part 2 outlines the fume hood evaluation criteria and procedure in Figure 3.2.2.

All safety inspection records, including equipment calibration and maintenance, are kept on file in the safety office for a minimum of five years.

Liquid waste is handled through three separate waste systems. Most of the drains lead to a conventional sanitary sewer system. Drains located in areas where acids are often used are connected to a glass piping system that leads to a 600-gallon neutralization tank containing limestone; the tank is connected to a 2,000-gallon mixing tank. The effluent from the neutralization tank is directed to the sanitary sewer system. The pH of the effluent from the neutralization tank is monitored continuously to ensure compliance with standards. Drains located in areas of potential organic chemical spills are connected to a separate glass piping system that leads to a 1,000-gallon holding tank. This tank is not connected to the sanitary sewer system. The liquid level of the tank is monitored, and the tank is emptied periodically to dispose of collected wastes in keeping with EPA and DOT regulations.



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5.0 Equipment

5.1 Specifications

A comprehensive list of instrumentation and equipment utilized at DCL is included in Appendix 14.6. Instrument specifications and the date of purchase are listed. Redundant instruments are maintained for particular analyses. The DCL Equipment List is organized by laboratory area with similar items grouped together.

5.2 Calibration Procedures

All instruments are calibrated before use, or the calibration is verified before use. Calibration requirements are detailed in the method SOPs and summarized in Appendix 14.7.

Analytical balance accuracy is checked before use each day and is verified on a regular schedule against NIST-traceable weights. DCL SOP LAB-015, "Balances," describes the DCL balance program.

5.3 Preventive Maintenance, Schedules, and Documentation

Routine maintenance is performed on laboratory instruments and equipment according to manufacturer recommendations. Maintenance is provided under warranty, through service contracts, and by DCL in-house personnel. The DCL approach to preventive maintenance is described in DCL SOP LAB-002, "Preventive Maintenance for Analytical Instrumentation." Records of routine maintenance and emergency maintenance are kept with the instruments in maintenance logbooks according to DCL SOP LAB-030, "Documentation – Maintaining Instrument Records, Notebooks, and Logbooks."

5.4 Calibration of Support Equipment

All support equipment is maintained in proper working order and the equipment is calibrated or verified at least annually or as described by the following SOP:

- Lab-015 "Balances"
- Lab-010 "Refrigerator Units"
- Lab-016 "Calibration Verification of Pipettors"
- Lab-018 "Calibration of Thermometers"



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6.0 Supplies and Services

6.1 Sample Containers

DCL supplies to clients glass or plastic containers with appropriate closures for sample shipping and storage, as required by environmental program regulations, See Appendix 14.8. The sample containers are precleaned when purchased, and they are used only once.

6.2 Laboratory Glassware

The glassware in general use in the laboratory is made of borosilicate unless otherwise specified in the analytical method. Volumetric glassware (pipettes, burettes, volumetric flasks, and graduated cylinders) must meet Class A specifications. Laboratory ware is inspected and cleaned according to the requirements of two DCL SOPs, LAB-011, "Glassware Cleaning for Inorganic Chemistry," and LAB-012, "Glassware Cleaning – Organic Analysis." Laboratory ware not suitable for continued use is discarded. Cleaned laboratory ware is stored in designated clean areas.

6.3 Reagents and Solvents

ACS reagent-grade chemicals and solvents are used unless otherwise specified in the analytical method or SOP. Representative samples of solvent lots are screened by the manufacturer or by DCL before use to ensure necessary purity.

Reagents, solvents, and solutions not stored in containers with commercial labels must be adequately labeled. At a minimum the label must contain the following information: identification of contents, concentration or purity, preparation and expiration dates (as applicable), date of initial opening (as applicable), notification of special storage requirements, and the initials of the responsible person. If it is impractical to record the required information on the label, the label can contain a unique identifier and a reference to a logbook with the necessary information. Additional details are given in DCL SOP LAB-003, "Labeling of Solutions."

To maintain a record of traceability to the source or reference material, lot and other information (as described in SOP XX-DC-019, "Standards Purity, Preparation, Traceability and Verification") are indelibly recorded by the responsible analyst as described in the SOP.

Hazardous reagents, solvents, and solutions are handled in accordance with the DCL Safety Manual and Chemical Hygiene Plan. Hazardous materials are stored in locations that furnish ventilation, fire barriers, and segregation from incompatible materials, as required.

6.4 Analytical Services Procurement



Laboratories contracted to perform analytical services for DCL must maintain quality programs consistent with the quality requirements of DCL. Before a laboratory performs subcontracted work for DCL, the Quality Assurance Manager must verify the acceptability of the quality program. At a minimum, this effort includes verification of necessary certifications and a review of the subcontract laboratory QAPP. It can also include an on-site audit.

Procedures and documentation for using sub-contract laboratories are listed in the DCL SOP LAB-023 "Client Communication". All results provided to DCL by a subcontract laboratory are identified clearly in the analytical report to the DCL client. Under no circumstances will DCL PT samples be sent to a subcontract laboratory.

7.0 Laboratory Practices

7.1 Radioactive Materials

Some of the samples received at DCL are radioactive or potentially radioactive. These samples are handled in accordance with the DCL radioactive materials license.

Potentially radioactive samples are surveyed for external radiation by sample receiving personnel according to DCL SOP QS-DC-001, "Sample Receipt and Logging." Radioactive samples are prepared in laboratory areas under the direction of assigned personnel and analyzed in an area of the laboratory under procedures designed to prevent the transfer of radioactivity out of that area. The handling of radioactive samples at DCL is carried out under the direction of the DCL Radiation Safety Officer (RSO).

7.2 Waste Management

Analysts are trained and laboratory waste is managed according to the following SOPs:

- LAB-004, "Hazardous Waste Handling and Disposal"
- LAB-005, "General Laboratory Safety and Chemical Hygiene"
- EA-DC-002, "Processed Sample Storage & Disposal Control"



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8.0 Laboratory Sample Handling Procedures

8.1 Applicability and Scope

Properly reported sample results begin with efficient and accurate introduction of pertinent information into the laboratory information management system (LIMS). This section describes DCL procedures for sample receipt, log-in, tracking through the laboratory, and disposal of residual materials. These procedures ensure the integrity of results by maintaining an unbroken chain-of-custody for each sample from receipt of the sample material to final disposal of any excess or residual product.

DCL purchases precleaned sample bottles to ship to clients. A table denoting recommended types of bottles, as well as use and descriptions of preservatives, is included in Appendix 14.8.

8.2 Sample Receipt

Procedures for receiving, processing, and storing environmental and radiochemistry samples, and for ensuring continuity of the chain-of-custody are detailed in the following DCL Standard Operating Procedures:

- QS-DC-001, "Sample Receipt and Logging"
- QS-EP-100, "EPA Sample Receipt and Logging"
- ♦ XX-DC-006, "Chain-of-Custody and Laboratory Tracking"
- WA-DC-002, "Acceptance Criteria for Samples Processed Under the Radioactive Materials License"
- 8.2.1 Sample Receiving and Logging

The DCL Sample Receiving area is isolated from areas of the laboratory that perform analysis. The area is equipped with ventilation hoods and adequate bench space to ensure that the sample receiving process is safe, efficient, and not a source of cross-contamination in the laboratory.

DCL SOP QS-DC-001, "Sample Receipt and Logging," specifies the procedures used to document the condition of shipped samples at the time of receipt, maintain the chain-of-custody, and provide internal laboratory sample tracking. When notified that a client is shipping samples to DCL, the cognizant Project Manager completes an internal Project Protocol Worksheet (PPW); this accompanies samples throughout the laboratory to notify each handler of the specific client requirements for that sample. If any discrepancies exist with respect to the fieldgenerated chain-of-custody, client work request, or project requirements, as noted on the internal Project Protocol Worksheet, the cognizant Project Manager is notified. Discrepancies and/or problems with samples are also documented on a Client Related Information Report (CRIR) that is forwarded to the Project Manager to resolve any problems.



Samples requiring acidic or basic preservation are checked for proper pH in the sample receiving area. Note: VOCs are not checked for pH in sample receiving because the pH is checked immediately before analysis or in the case of 5035 after samples are analyzed. The Project Manager is immediately notified and provided with a CRIR if any discrepancies with protocol are found. Samples requiring temperature control are checked, and the temperature is recorded.

When receiving potentially radioactive samples, sample receiving personnel perform a survey on containers as detailed in QS-DC-001, "Sample Receipt and Logging." Survey instruments are calibrated annually or whenever repairs are necessary. Copies of calibration records are maintained by the Radiation Safety Officer (RSO) in the radiation safety file. It is the responsibility of the RSO and assigned DCL personnel to maintain current calibration of the survey equipment.

If samples are classified by the client as radiological samples, screening information is maintained by the RSO. This information is maintained with the DCL Radioactive Materials Inventory Tracking System (RMITS). The client is required to provide screening data before samples are accepted by DCL.

8.2.2 Sample Tracking

Sample handling in the laboratory is tracked using a computer-based Laboratory Information Management System (LIMS) and through the signatures on the hand-carried chain-of-custody documents. After samples are received by the laboratory, as described above, sample receiving personnel enter the sample information into the LIMS. As samples move throughout the laboratory, a status code is assigned and entered into the LIMS by the various analysts working with the sample as explained in DCL SOP XX-DC-006, "Chain-of-Custody and Laboratory Tracking."

When multiple analyses require splitting a sample, the custody documents are copied such that each split can be independently traced to its origin and appropriate entries can be entered into the LIMS.

8.2.3 Sample Storage and Security

Following receipt, environmental samples are stored in accordance with analytical method requirements for storage and preservation. Samples for organic and inorganic analysis are normally stored in a walk-in refrigerator in the sample receipt area. Samples to be analyzed for volatile analytes are stored separately from all other samples in a refrigerator. Samples are stored, under chain-of-custody, in the receiving area until transferred to an analyst to initiate the analytical process.

To maintain facility security and thus sample security, entrance to the DCL facility can be attained only through coded card key access, except at the main business entrance; this is open only during normal business hours and monitored



by a receptionist. All nonemployees are required to sign in with the receptionist at the main entrance.

8.2.4 Sample Disposal

Sample disposal is accomplished in accordance with the following DCL SOPs:

- LAB-004, "Hazardous Waste Handling and Disposal"
- EA-DC-002, "Processed Sample Storage and Disposal Control"
- DCL Safety Manual and Chemical Hygiene Plan (Section 2: Parts 2, 3, and 4)

The responsibility to implement DCL waste disposal procedures is assigned to specific personnel. The cognizant manager supervises the monitoring of waste produced in each laboratory and the training of laboratory personnel to waste disposal procedures. DCL is considered a generator of hazardous waste and abides by the regulations contained in the EPA Resource Conservation and Recovery Act (RCRA).

Laboratory supervisors are responsible for the proper disposal of hazardous waste generated in pertinent work areas. Special care is taken to ensure that all hazardous waste is accumulated in properly labeled containers; hazardous waste is never discarded improperly.

Hazardous liquid chemical waste is accumulated in plastic bottles, glass bottles, or metal five-gallon cans. Each of these containers is properly labeled. When one or two hazardous waste containers are full, designated persons in laboratory operations transfer the waste to 55-gallon steel drums in the Waste Storage Room. The individuals transferring the waste wear personal protective equipment. Each drum is labeled, and special care is taken to ensure that waste chemicals are transferred to the proper drums.

Assigned personnel are responsible for the ultimate disposal of hazardous waste from DCL. This is accomplished through the services of a commercial waste broker. A DCL employee is assigned as the Hazardous Materials Technician. This person is responsible to arrange for the proper transport, storage, and/or disposal of DCL hazardous waste and to:

- Ensure that proper containers and labels are available.
- Monitor the drums.
- Ensure the proper labeling of drums.
- Maintain complete records of the status of all hazardous waste drums.
- Complete documentation of shipments.

Personnel monitor the pH of the building effluent. An automated system is in place to accomplish this. Any unacceptable excursion outside established limits is noted, its cause determined, and corrective action taken.



After analysis, excess sample materials are stored in the long-term sample storage room for the duration of time required by contract. This area is kept locked. Samples are logged in, labeled, and stored so that they are easily retrieved. Samples requiring refrigeration are stored in a refrigerated unit and monitored for temperature requirements.

After the required hold time, samples are properly disposed of by authorized personnel in the manner prescribed by their hazard class, or in a conventional manner in the case of nonhazardous material. Samples are logged out when disposed of by assigned personnel, with the disposal date noted in a logbook.

A radioactive waste disposal log is used to track the disposal of radioactive material. The Radiation Safety Officer maintains records of use and disposal. Disposal of all chemicals is handled by assigned DCL safety personnel according to regulatory requirements as described in detail in DCL SOP LAB-004, "Hazardous Waste Handling and Disposal," and in the DCL Safety Manual and Chemical Hygiene Plan (Section 2: Parts 2, 3, and 4).

8.3 Chain-of-Custody

In order to ensure that legally defensible data are produced at DCL, chain-of-custody procedures have been established and are followed as described in DCL SOP XX-DC-006, "Chain-of-Custody and Laboratory Tracking."

An example of the DCL chain-of-custody form is included in Appendix 14.9. All signatures are in permanent black ink and strikeouts are initialed and dated.

9.0 Analytical Procedures

DCL policy is that all SOPs be compliant with the reference method. In the event that several methods are referenced in an SOP, all procedures must be compliant with all referenced methods. All SOPs include a section describing changes and clarifications from the reference method. In the event that an analytical method is modified, the SOP documentation must include a description of the modification, any justification of the method modification which includes, but is not limited to, method performance and recovery data, any other supporting data, and approval from the Technical Directors, Quality Assurance Officer, and Laboratory Director. In the event that an analytical method must be modified or is modified to perform on specific sample matrices, the modification and reason must be stated in the case narrative. All modified methods will be identified on the analytical report.

9.1 Reference Methods



Reference methods for environmental samples are drawn primarily from the current version of *Test Methods for Evaluating Solid Waste Physical/Chemical Methods* (SW-846), Third Edition. Reference methods for water analysis are taken from *Methods for Chemical Analysis of Water and Wastes*, EPA-600/4-79-020, March 1983 with its updates, and from *40 CFR*, Part 136. To a lesser extent, methods referenced in DCL SOPs come from the current EPA CLP Statements of Work, from ASTM guides, and from *Standard Methods for the Examination of Water and Waste Water*.

9.2 Laboratory SOPs

SOPs are reviewed during the internal audits and updated as necessary. Review of SOP documents is completed in accordance with DCL SOP Lab-027 "Internal Audits and XX-DC-011, "Preparation of SOP Documents", section 4.0.

9.3 Historical Performance Limits

The table is Appendix 14.17 lists all analytical method and preparatory method combinations in which DCL routinely tracks and maintains statistical control limits. The laboratory can perform other methods upon a client request. The approval for use and the establishment of method limits is the responsibility of the Project Managers with approval and input from clients. The limits use will be from referenced sources when ever possible. Current historical control limits are listed in appendix 14.14.

10.0 Quality Control Procedures

Before environmental samples are analyzed, the analytical system must be in a controlled, reproducible state from which results of known and acceptable quality can be obtained. That state is verified through the use of Quality Control (QC) procedures intended to ensure accuracy, precision, selectivity, sensitivity, freedom from interference, and freedom from contamination. The QC procedures performed at DCL include: calibration and calibration verification; analysis and comparison of resultant data to predetermined control limits for method blanks, laboratory control samples, spiked matrix samples, duplicate matrix samples, and surrogates added to samples; analysis of performance evaluation samples; determination of Method Detection Limits (MDLs); and the tracking and evaluation of precision and accuracy. For specific analytical methods, other QC procedures are implemented as required by the method.

These QC procedures are performed and evaluated on a batch basis. An analytical batch must not exceed 20 field samples (to include field-derived samples, such as the matrix spike) that are of a similar matrix type. The samples in a batch are processed together, through each step of the analysis, to ensure that all samples receive consistent and equal treatment. Consequently, results from the batch QC samples are used to evaluate the results for all samples in the batch.

10.1 Calibration and Calibration Verification



Instrument calibration is a QC measure taken to verify selectivity and sensitivity. Calibration of instruments at DCL is accomplished through the use of reference materials of the highest quality obtainable. NIST-traceable reference materials are procured and used if they are available. When NIST-traceable reference materials are not available. certified reference materials from government agencies or reliable vendors are used. In all cases, written records are maintained that allow all analytical results to be traced unambiguously to the reference materials used for calibration. DCL SOP XX-DC-019, "Standards Purity, Preparation, Traceability, and Verification," describes the process and record keeping responsibilities of analysts to ensure that all reagent and reference materials are traceable to their sources. In general, analytical instruments are initially calibrated with standard solutions made from the reference materials at levels appropriate for the analysis. This is called the initial calibration (IC). The IC is verified at the beginning of each analytical sequence with a standard solution independently prepared from a different lot of the reference material, preferably from a different vendor. This step is called initial calibration verification or ICV. At specified intervals throughout the analytical sequence, the calibration is verified again through the analysis of an independently prepared standard solution. This process is called the continuing calibration verification or CCV. If the IC, the ICV, or any CCV fail the criteria in the analytical method, the system is recalibrated. Only results generated under acceptable calibration conditions are reported. Specific calibration procedures are found in the SOPs associated with each method of analysis.

Alternative calibration sequences or procedures will be discussed with clients as per section 3.1.4 and 3.1.5 of the DCL SOP Lab-023 "Client Communication".

Calibration parameters set by the applicable DCL SOP or method reference shall not be exceeded without initiation of a NC/CAR (See DCL SOP Lab-020).

10.2 Analysis of Method Blanks

The method blank (or preparation blank) contains no sample material; it is treated as a sample in every other way. It is analyzed to monitor any contamination to which the analytical batch might have been exposed during analysis. A method blank is analyzed with every analytical batch. An acceptable blank result must be below one-half the Practical Quantitation Limit (PQL) established by DCL for the analytical method, or have a value less than 10% of the concentration found in the sample. Method QC Evaluation of the Method Blank is available in Appendix 14.10. A description of PQL/RL values is described in section 10.8. The DCL PQLs are specified in the analytical method SOPs and are set at the concentration of the lowest calibration standard. Special project requirements can impose a different standard for acceptability of blank results (i.e. Less than 10% of a regulatory limit) and PQL limits (ie.. 3 times the MDL). If the blank results are unacceptable, the samples in the batch are extracted or digested again and reanalyzed within the hold time. If that is not possible, the client is notified and appropriate action is taken.

10.3 Analysis of Laboratory Control Samples



A laboratory control sample (LCS) contains the analyte(s) of interest in known concentration(s); it is used to monitor accuracy. It measures the success of the analysis in recovering the analyte(s) of interest from a familiar sample matrix. An LCS is analyzed with every analytical batch. Unless otherwise specified, soil samples and other solid matrices are analyzed with an LCS made of clean sand spiked with the analyte(s) of interest. Water samples and other liquid matrices are analyzed with a method blank spiked with the analyte(s) of interest. The results of the LCS are reported as percent recovery:

% Recovery
$$= \frac{X}{K} \times 100$$

Where: X = Measured value K = Expected value

10.4 Analysis of Spiked Matrix Samples

A known concentration of the analyte(s) of interest is added to a second representative portion of a field sample to prepare a matrix spike. The matrix spike is used to monitor accuracy. It measures the success of the analysis in recovering the analyte(s) of interest from the type of field sample matrix in the batch. A matrix spike is analyzed with every analytical batch. The results are reported as percent recovery.

% Recovery =
$$\frac{(X_s - X_u)}{K} \times 100$$
 Where:

 X_s = Measured value in the spiked sample X_u = Measured value in the unspiked sample K = Expected value

10.5 Analysis of Duplicate Matrix Spike Samples

A duplicate matrix spike sample or duplicate matrix sample is used to monitor the precision (repeatability) of an analysis. If a sufficient amount of the analyte(s) of interest is present in the field sample, a matrix duplicate sample is analyzed directly. If the analyte(s) of interest are not present in a sufficient amount, two additional portions of field sample are spiked with the analyte(s) of interest to ensure that meaningful results are obtained. A pair of duplicate samples (matrix/matrix duplicate or matrix spike/matrix spike duplicate) is analyzed with every analytical batch. The results of the analysis of duplicate samples are reported as relative percent difference (RPD).

$$RPD = \frac{|X_1 - X_2|}{[(X_1 + X_2)/2]} \times 100$$

Where:



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 $|X_1 - X_2|$ = The absolute value of the difference between the two sample values $[(X_1 + X_2)/2]$ = The average of the two sample values

10.6 Analysis of Surrogates Added to Samples

Surrogates are compounds similar to the analyte(s) of interest but that are known *not* to be present in the environment. Examples are fluorinated or deuterated homologues of the organic analyte(s) of interest. When appropriate compounds are available, their use is specified in the analytical method SOP. When surrogates are used, they are added to the calibration solutions and to each field and QC sample in the batch. Surrogate recovery is a measure of the accuracy and selectivity of the method in the sample matrix. Surrogate results are reported as percent recovery.

% Recovery =
$$\frac{X}{K} \times 100$$

Where: X = Measured value K = Expected value

10.7 Analysis of Performance Evaluation Samples

Proficiency testing (PT) samples, also called proficiency testing samples, are prepared by an authorized independent organization outside the laboratory.

They are received and analyzed at regular intervals to monitor laboratory accuracy. DataChem Laboratories sends the PT sample results to the independent organization, where they are evaluated and then forwarded directly from that organization to the State of Utah or other regulatory entity. PT samples are introduced into the regular sample stream of the laboratory and analyzed as routine samples by analysts who regularly perform the method. Laboratory personnel follow all instructions provided by the PT provider. DCL notifies the State of Utah if any changes to the enrollment in certified PT programs occur.

At a minimum, PT samples from an authorized proficiency testing program are generally analyzed at least twice annually for each certified analyte to maintain EPA certification as administered under Utah Rule R444-14-13. The Laboratory Director or the Quality Assurance Manager can institute the analysis of additional PT samples or modify the performance evaluation program as appropriate. The following guidelines are followed by DCL:

- Averaging results is prohibited.
- Only qualified DCL laboratory employees analyze PT samples.
- Results are not discussed with outside entities or other DCL laboratories prior to the deadline for receipt of the results.
- DCL does not subcontract to other laboratories or receive from other laboratories any PT samples.



When a PT sample result is not acceptable, documented corrective action is taken to determine and correct any problem(s) leading to the unacceptable result. Refer to section 12.0 of this QAPP. A corrective action report is available upon request and pertinent reports, report forms, and documentation are stored in accordance with section 13.0 of this QAPP. If a remedial PT sample must be analyzed, only one remedial PT sample for an analyte or independent analyte group can be submitted in any 12-month period.

10.8 Method Detection Limits

The method detection limit (MDL) reflects the sensitivity of an analytical method to the matrix of interest. It is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero in the matrix used for determination of the MDL. The MDL is based upon a standard deviation derived from the analysis of at least seven replicates.

 $MDL = t(n-1, \alpha = 0.99)(s)$

where (s) is the standard deviation derived from at least seven replicates

 $t_{(n-1,\alpha = 0.99)}$ is the one-sided t-statistic for the number of samples used to determine (s)

MDLs in solid and aqueous matrices are determined annually in accordance with DCL SOP LAB-024, "Calculation of Method Detection Limits." This SOP implements the requirements of 40 CFR Part 136, Appendix B, July 1, 1995 edition.

Reporting Limits are set by DCL at the lowest calibration concentration except for methods that require deviation from multiple-point calibration or are not applicable to similar calibration requirements. Practical Quantitation Limits are typically synonymous with Reporting Limits. PQLs can be specified by a client as some multiplier of the MDL determination. In all cases, the Reporting Limit and the Practical Quantitation Limit must be higher than the applicable value derived from the current MDL study and no lower than the lowest calibration concentration, except as designated by the analytical procedure.

10.9 Other Quality Control Procedures

Specific analytical methods might require additional quality control measures. Examples include the verification of GC/MS tuning every 12 hours and the verification of ICP interelement corrections. Both of these QC measures verify method selectivity.

Additional QC measures are implemented as part of the analytical method. The balances at DCL are maintained and checked according to DCL Lab-010, "Balances." The thermometers at DCL are evaluated for future use and calibrated according to DCL SOP Lab-018, "Calibration of Thermometers." Pipettors are maintained and calibrated in keeping with DCL SOP Lab-016, "Calibration of Pipettors."



10.10 Tracking and Evaluation of Accuracy and Precision

Assessment of the accuracy of an analytical measurement is based upon the analysis of samples of known composition. DCL relies upon the analysis of LCS and MS samples to track accuracy. The percent recovery relative to the expected value is calculated and plotted on an accuracy chart (X chart) for tracking. Assessment of the precision (repeatability) of an analytical measurement is based upon repeated analysis of equivalent samples of known or unknown composition. DCL relies upon the analysis of pairs of matrix samples (M/MD) or spiked matrix samples (MS/MSD) to assess precision. The range of the pair is expressed as a relative percent difference (RPD). Control limits for the accuracy and precision charts are calculated assuming a normal (Gaussian) distribution of results. A set of historical data points is used to calculate a mean values, two-standard deviation warning limits, and three-standard deviation control limits. The establishment and updating of control charts is described in DCL SOP QC-DC-001, "Establishing and Updating Control Limits."

When evaluating batch QC the analyst makes a sequence of decisions before reporting sample results regarding calibration, the method blank, LCS, surrogate recovery, matrix spike, and matrix spike duplicate recovery results. Appendix 14.10 contains a set of six flowcharts used by DCL analysts to evaluate batch QC. The first evaluation of QC acceptability is made according to the requirements stated in the analytical method. The second consideration is based upon any special project requirements. The flowcharts then are used to evaluate batch QC in the following order: calibration, method blank results, surrogate recovery results, LCS results, matrix spike recovery results, and duplicate results. Exhibit "MB Flow" (in Appendix 14.10) is a flowchart that summarizes the first set of decisions to be made by the analyst to evaluate the acceptability of method blank results. Exhibit "LCS Flow" is a flowchart that summarizes the second set of decisions to be made by the analyst to evaluate the acceptability of LCS results. Exhibit "MS Flow" is a flowchart that summarizes the set of decisions to be made by the analyst to evaluate the acceptability of matrix spike results. Exhibit "RPD Flow" is a flowchart that summarizes the set of decisions to be made by the analyst to evaluate the acceptability of duplicate results. Table 1 below, "QC Sample Evaluation," summarizes the decisions to be made by the analyst regarding relationships between LCS results, matrix spike results, and duplicate results to complete the evaluation of batch OC.



Table 1 **Inorganic QC Data Evaluation**

LCS Recovery	MS Recovery	MS/MSD or Sample/MD RPD	Blank	Response
+	+	+	+	Samples are reported with no exceptions.
+	+	+	_	See Method Blank Flowchart Appendix 14.10
+	+	_	+	Samples are reported with a flag or note denoting a matrix effect is suspected. See MS/MSD and Duplicate Flowcharts
+	_	+	+	Samples are reported with a flag or note denoting that a matrix effect is suspected. See MS/MSD Flowchart
_	+	+	+	Samples are reprepared and reanalyzed. See LCS Flowchart
+	+	_	_	See Method Blank Flowchart and samples are reported with a flag or note denoting that a matrix effect is suspected. See MS/MSD and Duplicate Flowchart.
	+	+	_	See Method Blank and LCS Flowcharts
	+	_	+	See LCS, MS/MSD and Duplicate Flowcharts.
+	_	+	_	See MS/MSD and Method Blank Flowcharts.
+	_	_	+	See MS/MSD and Duplicate Flowcharts.
	_	+	+	See LCS and MS/MSD Flowcharts.
+		_	_	See Method Blank, MS/MSD and Duplicate Flowcharts.
	+	_	_	Samples are reprepared and reanalyzed.
	_	+	_	Samples are reprepared and reanalyzed.
		_	+	Samples are reprepared and reanalyzed.
	_	_	_	Samples are reprepared and reanalyzed.
		(+) = meets	criteria	(-) = does NOT meet criteria





Table 2Organic QC Data Evaluation

LCS Recovery	MS Recovery	MS/MSD or Sample/MD RPD	Blank	Surrogate	Response
+	+	+	+	+	Samples are reported with no exceptions.
-	+	+	+	+	See LCS Flow Chart Appendix 14.10
+	-	+	+	+	Samples are reported with a flag or note denoting a matrix effect is suspected. See MS/MSD Flowchart
+	+	-	+	+	Samples are reported with a flag or note denoting that a matrix effect is suspected. See MS/MSD and Duplicate Flowcharts
+	+	+	-	+	See Method Blank Flowchart
+	+	+	+	-	See Surrogate Flowchart
-	-	+	+	+	See LCS and MS/MSD Flowchart
+	-	-	+	+	Samples are reported with a flag or note denoting a matrix effect is suspected. See MS/MSD and Duplicate Flowcharts
+	+	-	-	+	See Method Blank Flowchart and samples are reported with a flag or note denoting that a matrix effect is suspected. See MS/MSD and Duplicate Flowchart.
+	+	+	-	-	See Method Blank Flowchart and Surrogate Flowchart
-	+	-	+	+	See LCS Flow Chart and samples are reported with a flag or note denoting a matrix effect is suspected. See MS/MSD and Duplicate Flowcharts.
+	-	+	-	+	See Method Blank Flow Chart and samples are reported with a flag or note denoting a matrix effect is suspected. See MS/MSD and Duplicate Flowcharts.
+	+	-	+	-	See Surrogate Blank Flow Chart and samples are reported with a flag or note denoting a matrix effect is suspected. See MS/MSD and Duplicate Flowcharts.
	_	_	_	-	Samples are reprepared and reanalyzed.

Other situations can occur. Please see the appropriate Method QC Flowchat in Appendix 14.10

(+) = meets criteria (-) = does NOT meet criteria



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In addition to evaluating individual batch QC results against control limits, QC results from successive batches are also evaluated for possible trends. While a trend is not necessarily an out-of-control situation in itself, it can provide an early warning of a condition that can cause the system to go out of control. DCL SOP XX-DC-018, "Evaluation of Quality Control Data," describes in detail the assessment of QC data in the laboratory. The following conditions are trends that initiate action and/or monitoring.

- A series of seven successive points on the same side of the mean
- A series of five successive points going in the same direction
- A cyclical pattern of QC sample results
- Two successive points between warning limits and control limits
- A single QC value outside the control limits

The occurrence of a trend does not invalidate data that are otherwise in control. However, trends do require attention to determine whether a cause can be assigned to the trend so that appropriate corrective action can be undertaken.

11.0 Data Reduction, Verification, and Reporting

Data reduction, verification, and reporting are accomplished through extensive use of a Laboratory Information Management System (LIMS). The DCL LIMS is a commercial automated data handling system that incorporates a relational database with additional custom programming to interface with laboratory instruments and produce reports required by DCL clients. It is maintained by the DCL computer support staff and updated as necessary to accommodate new instrumentation and meet diverse client requirements.

11.1 Data Reduction

Data reduction consists of identifying the pertinent set of calibration standards, specifying the type of calibration to use (e.g., linear, calibration factor, quadratic), and calculating analytical results from the calibration equation. The actual calculations are performed by software residing in the analytical instrumentation or by the DCL LIMS after raw data have been transferred into it. Analyst involvement is limited to selecting standards, the type of calibration, and the sample set to which the calibration is applied.

Linear calibrations or the use of response factors are preferred for the reduction of data. DCL policy is to utilize the simplest appropriate equation that produces a good fit of the data. Other types of calibrations are available if required by the method or made necessary by special circumstances. The types of calibrations available are listed below in Table 2:



Calibration Type	Equation
Linear	y = mx + b
Calibration Factor	y = CFx where CF is the average of the individual
	response factors for each calibration point
Quadratic	$y = a + bx + cx^2$

Table 2: Types of Calibration

11.2 Ensuring Accuracy of Calculations and Transcriptions

All of the software used for data reduction, verification, and reporting is documented and validated by the DCL computer support staff according to DCL SOPs LAB-101, "Computer Program Testing," and LAB-102, "Computer Program Documentation," or by the vendor from whom it is purchased. DCL software is controlled and secured according to DCL SOPs LAB-103, "Computer Software Control," and LAB-104, "Computer Software Security." A continuing effort is made at DCL to increase the use of automated data handling, improve efficiency, and minimize human error.

DCL also relies upon a system of peer review to ensure the quality of analytical reports. Peer review procedures are specified in the DCL SOP XX-DC-023 "Peer Review". An analyst, familiar with the analytical method used to produce the results (peer reviewer), reviews each report. The peer reviewer verifies that the calibration standards, type of calibration, and sample set with associated QC samples were selected correctly. The peer reviewer also verifies any manual transcriptions and calculations. The Manager can perform additional technical review.

11.3 Verification of Quality Control

The analyst is responsible to evaluate the QC results (method blank, surrogate recovery, LCS, matrix spike, and duplicate results) and to take any necessary actions described in section 10.0 of this document. Examples of necessary actions are:

- Reporting sample results with the correct qualifier (e.g., qualifier flag for sample results between the MDL and the PQL)
- Noting unusual situations in the case narrative (For example, although the blank contains an analyte above the PQL, sample results can be reported because all were less than the MDL.)
- Initiating corrective action when required

The peer reviewer is responsible to verify that QC results have been evaluated correctly and that necessary actions have been taken. Peer review procedures are specified in the DCL SOP XX-DC-023 "Peer Review". The peer review is considered complete when all issues raised by the peer reviewer have been resolved. Resolving issues raised by a peer reviewer can involve the manager and the Quality Assurance Manager.



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11.4 Reporting

When the peer review has been completed, a report is generated. In most situations the report is produced from the LIMS. In some cases part or all of the report can be produced from the data system of the analytical instrument. The reports produced by DCL meet the following requirements:

- The report identifies the method used. If the method is modified, it is noted as "modified" in the report.
- Any abnormal sample conditions, deviation from hold time, irregularities in preservation or other situations that might affect the analytical results are noted in the report and associated with the analytical results.
- The contents of the report include:
 - The report title with the name, address, and telephone number of the laboratory
 - The name of the client or project and the client identification number
 - Description and laboratory identification number
 - ✤ The dates of sample collection, sample receipt, sample preparation, and analysis
 - The time of sample preparation and/or analysis if the required hold time for either activity is 48 hours or less
 - * A method identifier for each method, including methods for preparation steps
 - ✤ The MDL or minimum reporting limit for the analytical results
 - ✤ The analytical results with qualifiers as required
 - A description of any quality control failures and deviations from the accepted method
 - The signature and title of the individual(s) who accept responsibility for the content of the report
 - The date the report is issued
 - Clear identification of any results generated by a subcontract laboratory
 - Page numbers and total number of pages

The Project Manager can review final reports for compliance with client requirements. The Quality Assurance Manager periodically reviews a representative selection of reports for compliance with this QAPP. Standard DCL deliverables are produced in accordance with DCL SOP XX-DC-020, "Deliverable and Data Package Preparation and Review."

12.0 Corrective Action

DCL laboratory operations are conducted in accordance with documented internal procedures (such as SOPs and this QAPP) and client-specific provisions communicated through the DCL PPW. When any laboratory process does not meet internal DCL requirements or client-specific provisions, the nonconformance is identified and appropriate corrective action is taken. Corrective action is performed as a part of routine analysis and usually does not require formal documentation. An example of routine corrective action is troubleshooting an instrument and recalibrating it after calibration verification fails. Other corrective action requires formal documentation. An example is consistently poor recovery of analytes from an LCS.



12.1 Individuals Responsible to Take Corrective Action

All DCL staff members are responsible to initiate corrective action as necessary. Each employee is expected to understand laboratory procedures and client requirements governing the work performed and to take prompt action to ensure that those requirements are met. Managers are responsible to determine the extent of the nonconformance and the initial level of corrective action response. The Project Manager is responsible to evaluate the appropriateness of the corrective action response for the client. The Quality Assurance Manager is responsible to oversee the overall effectiveness of the corrective actions taken by the laboratory. The Laboratory Director is responsible to ensure that resources are allocated to correct nonconformances promptly and effectively. Procedures are outlined in DCL SOP LAB-020, "Nonconformance/Corrective Action Report (NC/CAR) Procedures." Appropriate corrective actions are listed in Appendices 14.7 and 14.10 of this QAPP.

12.2 Laboratory Responses to Unacceptable Results

Proficiency testing (PT) samples are prepared by an independent organization outside the laboratory. They are received and analyzed at regular intervals to monitor laboratory accuracy. Any failure to pass a PT sample is reported to the Manager, the Quality Assurance Manager, and the Laboratory Director. It requires documented corrective action. The Manager is responsible to ensure that the corrective action is completed. The Quality Assurance Manager is responsible to review and accept or reject the completed corrective action and its documentation.

Unacceptable results from QC sample analyses that can be addressed as part of the analytical process do not require formal documentation of corrective action. That type of problem and its resolution become part of the information in the laboratory notebook or the instrument maintenance log. Other nonconformances revealed by QC sample results or internal checks, including internal audits, must have documented corrective action. Managers are responsible to ensure that the corrective action is completed. The Quality Assurance Manager is responsible to review and accept or reject the completed corrective action with its documentation.

When a client contacts the laboratory to reveal a failure in the laboratory analytical system, documented corrective action is taken. The Project Manager is responsible to initiate the corrective action. The Manager is responsible to ensure that the corrective action is completed. The Quality Assurance Manager is responsible to review and accept or reject the completed corrective action with its documentation.

12.3 Verification and Documentation of Corrective Action

The DCL SOP governing documented corrective action is LAB-020, "Nonconformance/Corrective Action Report (NC/CAR) Procedures." Verification and documentation of corrective action are implemented in accordance with the SOP.



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12.4 Reports to Laboratory Managers

In addition to the reports described in this section, reports concerning various aspects of quality assurance are furnished to the President and Laboratory Director. The Quality Assurance Manager provides reports of reviews of analytical reports, internal audits, and training. Managers report technical and quality problems directly to the Laboratory Director.

12.5 Internal Audits

Internal audits are conducted in accordance with DCL SOP Lab-027, "Internal Audits."

12.6 Quality System Review by Management

The purpose of Management Review is to conduct a review of the laboratories quality system and testing activities to ensure its continuing suitability and effectiveness. The review will determine if any changes or improvements are necessary to the quality system or laboratory operations. Specific procedures are outlined in the DCL SOP LAB-026 "Procedure for Management Review".

All documentation is retained by the QAO.

13.0 Document Control and Record Keeping

The management and control of documents and records that define laboratory operations and chronicle laboratory activities are necessary to ensure that laboratory data are of known quality, retrievable, reproducible, and defensible. Records that must be maintained, controlled, or managed include sample receiving and chain-of-custody records, sample analysis data records, instrument and other laboratory maintenance records, quality control data, quality assurance documents, and all other records relating to or impacting the quality of analytical data.

The records management system is implemented through several DCL Standard Operating Procedures, including:

- XX-DC-006, "Chain-of-Custody and Laboratory Tracking"
- XX-DC-011, "Preparation of SOP Documents"
- ◆ LAB-021, "Document Control"
- XX-DC-020, "Deliverable and Data Package Preparation and Review"
- ◆ LAB-030, "Documentation Maintaining Instrument Records, Notebooks and Logbooks"
- ◆ LAB-013, "Archives"
- QD-EP-1220, "Document Control and Report Preparation"
- ◆ LAB-007, "Record of Training"

The record system at DCL is designed to the meet regulatory requirements of Utah Rule 444-14. Documentation requirements are met through the implementation of the SOPs noted above.



Examples of documents that are controlled and tracked include:

- Standard Operating Procedures
- Analyst Notebooks
- Instrument Logbooks
- Standards Preparation Logbooks
- Instrument Hard Copy Output (e.g., chromatograms, strip charts)
- Computer Printouts (e.g., raw and processed data)
- Analytical Reports
- Data Packages

13.1 Document Control

Document control procedures are described in DCL SOP LAB-021, "Document Control." Additional information concerning the generation and updating of these controlled documents is contained in DCL SOP XX-DC-011, "Preparation of SOP Documents."

- 13.1.1 Standard Operating Procedures
 - 13.1.1.1 Retention and Distribution

The Quality Assurance Manager is responsible for the retention and distribution of Standard Operating Procedures, in accordance with DCL SOP LAB-021, "Document Control."

13.1.1.2 Revision of SOPs

Assignments are made to the responsible DCL manager or designee to review and update SOPs applicable to the area of responsibility. At times it is also necessary to obtain approval by specific clients before written SOPs can be modified. After revision, the Manger, Quality Assurance Manger, and Laboratory Director must approve the updated SOP. Updated SOPs are then distributed on line and to holders of controlled copies.

13.1.1.3 Retiring of SOPs

If it becomes necessary to retire an SOP, approval of the Laboratory Director, cognizant Manager, and Quality Assurance Manager must be obtained before retirement can take place. After retirement, the SOP is stored in the retired SOP file for future reference.

13.1.1.4 Review of SOPs

Review of all technical SOPs are completed during yearly internal audits. Review of all SOPs are completed on an as needed basis and documented as described in the DCL SOP XX-DC-011.


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13.1.2 QA Program Plans

This QAPP is a controlled document with distribution to all DCL staff members involved in QAPP-related activities. The DCL Quality Assurance Manager can distribute copies of the DCL QAPP to other persons, such as clients and subcontractors. Additionally, quality assurance program documents, project plan documents, and contractual Statement of Work documents generated by a client can be designated as controlled documents at the discretion of the cognizant DCL Project Manager, the DCL Quality Assurance Manager, or the Laboratory Director.

13.1.3 Records of Distribution

The Quality Assurance Manager maintains a record of the distribution of controlled documents. This record includes the document and version numbers, updates, and responsible persons.

13.2 Record Keeping

DCL uses an off-site, commercial record archive facility to retain its records. A filing system is maintained by the archivist to account for documents taken from archives until their return. Detailed pertinent procedures are found in DCL SOP LAB-013, "Archives." The Quality Assurance Manager and, by delegation, assigned DCL personnel, are responsible for the retention, retrieval, and disposition of final records of laboratory data and activities. This includes: data packages, once they are completed; analyst laboratory notebooks and instrument maintenance logs, once submitted for archival; and training records, as established by SOP.

13.2.1 Data Packages

All documentation that pertains to the analysis of a sample or group of samples that are being reported together must be compiled as a data package. SOPs addressing the preparation and control of data packages include:

- ◆ LAB-013, "Archives"
- QD-EP-1220, "Document Control and Report Preparation"
- XX-DC-020, "Deliverable and Data Package Preparation and Review"

Records, or copies of records, that relate to the analysis of field samples are compiled into data packages by the analyst. These data packages are initially stored, generally categorized according to client or project, in open-access files, allowing easy retrieval for review. Data packages are generally maintained in onsite archives until audited by the client or project administrator. Data packages can then be released to the client or archived off-site from the DCL laboratory facility, pending later release to the client. The client and/or regulatory requirements govern the length of time for data package retention. Unless specified by contract, applicable statute, or program, data packages are retained for five years.



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13.2.2 Laboratory Notebooks and Logbooks

Laboratory notebooks and logbooks are retained by DCL for 10 years and are not released to clients. Laboratory notebooks are assigned to specific analysts, who are responsible for their maintenance. If corrections are required, a single-line cross-out and initials and date are entered.

13.2.3 Quality Assurance Records

Quality control sample results data are retained for five years. Records of internal audits, nonconformance reports, and corrective action reports are retained for five years.

13.2.4 Records of Audits and NC/CARs

The Quality Assurance Manager is responsible for maintaining and retrieving all records of audits, both internal and external, proficiency testing results, and nonconformance and corrective action records and reports.

13.2.5 Client Related Information

Project Managers are responsible for maintaining, archiving, and retrieving all contracts, project requirements and QAPPs provided to DCL by clients and related to projects completed by DCL. They are also responsible for the destruction of materials provided on unsuccessful proposals and bidding opportunities. Specific procedures for client communication and required documentation are listed in the DCL SOP LAB-023 "Client Communication".



14.0 Appendices

The following appendices are available upon request. These are dynamic documents; accordingly, they can change without notice or revision to this Quality Assurance Program Plan. Please contact the laboratory on DCL On-Line for current appendices.

- 14.1 Accreditations and Certifications
- 14.2 DCL Organization Chart
- 14.3 Key Personnel
- 14.4 DCL Staff Summary Table
- 14.5 Facilities Floor Plan
- 14.6 Equipment List
- 14.7 Summary of Calibration and Corrective Action Procedures
- 14.8 Sample Preservation and Hold Times
- 14.9 Chain-of-Custody
- 14.10 Batch QC and Corrective Action Flowcharts
- 14.11 SOP List
- 14.12 Definitions and Terms
- 14.13 Analytical Services Provided by DataChem Laboratories, Inc.
- 14.14 Historical Control Limits
- 14.15 Method Detection and Reporting Limits
- 14.16 Marginal Exceedances
- 14.17 DCL Maintained Control Limits
- 14.18 DoD QSM Requirements



