Idaho
National
Engineering
Laboratory

INEL-96/0133
Revision 0
August 1996

Analytical Chemistry Laboratory Quality Assurance Project Plan for the Transuranic Waste Characterization Program

S. J. Sailer

Uncontrolled Copy

LOCKHEED MARTIN

INEL-96/0133
Revision 0

Analytical Chemistry Laboratory Quality Assurance Project Plan for the Transuranic Waste Characterization Program

S. J. Sailer

Published August 1996

Idaho National Engineering Laboratory
Lockheed Martin Idaho Technologies Company
Idaho Falls, Idaho 83415

Prepared for the
U.S. Department of Energy
Assistant Secretary for Environmental Management
Under DOE Idaho Operations Office
Contract DE-AC07-94ID13223

Analytical Chemistry Laboratory Quality Assurance Project Plan for the Transuranic Waste Characterization Program

INEL-96/0133

Approved By:

Team Leader, National TRU Program — Date

Manager, DOE-CAO Quality Assurance — Date

DOE-ID Operations Office — Date

for — 8/28/96

M. J. Connolly
INEL Site Project Manager — Date

8/27/96

G. R. Hayes
INEL Site Quality Assurance Officer — Date

8/29/96

R. L. Hand
LMITCO Analytical Laboratories Department Manager — Date

This document has been prepared in accordance with the *Transuranic Waste Characterization Quality Assurance Program Plan*, April 30, 1995, CAO-94-1010, Revision 0.

Analytical Chemistry Laboratory Quality Assurance Project Plan for the Transuranic Waste Characterization Program

INEL-96/0133

Reviewed by:

8/28/96

T. L. Clements, Jr., Manager
LMITCO TRU Waste Programs Department

Date

08/29/96

S. J. Sailer
LMITCO Analytical Laboratories Department
Quality Assurance Officer

Date

M. K. Huffalu

LMITCO Quality Systems and Services

8/27/96

Date

ABSTRACT

This Quality Assurance Project Plan (QAPjP) specifies the quality of data necessary and the characterization techniques employed at the Idaho National Engineering Laboratory (INEL) to meet the objectives of the Department of Energy (DOE) Waste Isolation Pilot Plant (WIPP) Transuranic Waste Characterization Quality Assurance Program Plan (QAPP) requirements. This QAPjP is written to conform with the requirements and guidelines specified in the QAPP and the associated documents referenced in the QAPP. This QAPjP is one of a set of five interrelated QAPjPs that describe the INEL Transuranic Waste Characterization Program (TWCP). Each of the five facilities participating in the TWCP has a QAPjP that describes the activities applicable to that particular facility.

Waste characterization data will be collected at the INEL to support regulatory compliance programs associated with the WIPP facility. These regulatory compliance programs include an assessment and certification of the WIPP repository performance, the preparation of permit applications and a variance petition, and an evaluation of existing transportation restrictions.

This QAPjP describes the roles and responsibilities of the Idaho Chemical Processing Plant (ICPP) Analytical Chemistry Laboratory (ACL) in the TWCP. Data quality objectives and quality assurance objectives are explained. Sample analysis procedures and associated quality assurance measures are also addressed; these include: sample chain of custody; data validation; usability and reporting; documentation and records; audits and assessments; laboratory QC samples; and instrument testing, inspection, maintenance and calibration. Finally, administrative quality control measures, such as document control, control of nonconformances, variances and QA status reporting are described.

CONTENTS

ABSTRACT vii

ACRONYMS xix

DEFINITIONS xxiii

1. PROGRAM MANAGEMENT 1

1.1 Program Organization and Responsibilities 1

1.1.1 Analytical Laboratories Department Manager 1
1.1.2 Analytical Laboratories Department Quality Assurance Officer 1
1.1.3 Analytical Operations Supervisor 3
1.1.4 Organic Analysis Section Supervisor 3
1.1.5 Inorganic Chemistry Section Supervisor 3
1.1.6 Organic Analysis Laboratory Technical Leader 3
1.1.7 Spectrochemistry Technical Leader 4
1.1.8 Special Analysis Technical Leader 4
1.1.9 Chemists, Instrument Operators and Sample Preparation Specialists 5
1.1.10 Sample Custodian 5
1.1.11 Data and Records Coordinators 5
1.1.12 Instrument Custodians 5
1.1.13 Quality Control Chemists 6
1.1.14 Computer Support Personnel 6

1.2 Program Documents 6

1.2.1 Key Documents 6
1.2.2 Document Review, Approval, and Control 8

1.3 Problem Definition and Background 10

1.4 Program Description 10

1.5 Data Quality Objectives 10

1.6 Indoctrination and Training 13

1.7 Records Management 13

1.7.1 Control of Records 13
1.7.2 Disposition of Records 13
1.7.3 Requests for QA Records 17

1.8 Procurement 17

1.9 Work Processes 17

1.9.1 Control of Processes 17
1.9.2 Identification and Control of Items 21
1.9.3 Computer Hardware and Software 21

2. ASSESSMENT AND OVERSIGHT 22

2.1 Audits 22

2.2 Corrective Actions 22

2.2.1 Nonconformances 22
2.2.2 Operational Variances 25

2.3 Quality Improvement 25

2.3.1 Trend Analysis 25
2.3.2 Control Charts 27

2.4 Management Assessments 27

2.5 Independent Assessments 27

2.6 QA Reports to Management 29

2.7 Performance Demonstration Program 30

2.8 Procedure Performance Demonstration 30

3. DATA VALIDATION, USABILITY, AND REPORTING 31

3.1 Data Review, Validation, and Verification Requirements 31

3.1.1 Data Generator Review 32
3.1.2 Independent Technical Review 32
3.1.3 Technical Supervisory Review 33
3.1.4 Quality Assurance Officer Review 33

3.2 Validation Methods 38

3.2.1 Precision 38
3.2.2 Accuracy 39
3.2.3 Method Detection Limits 40
3.2.4 Instrument Detection Limits (IDLs) 41
3.2.5 Completeness 41

3.2.6 Comparability 42

3.3 Reconciliation with Data Quality Objectives 42

3.4 Data Reporting Requirements 42

4. MEASUREMENT AND DATA ACQUISITION 43

4.1 Facilities and Workplaces 43

4.1.1 CPP-602 Facility 43
4.1.2 CP-627 Facility 43
4.1.3 Remote Analytical Laboratory (CPP-684) 43

4.2 Equipment Calibration 43

4.3 Equipment Maintenance 44

5. SAMPLING PROCESS DESIGN 46

6. DRUM AND SAMPLE HANDLING AND CUSTODY REQUIREMENTS 47

6.1 Sample Container Labeling and Chain-of-Custody 47

6.2 Sample Receiving 48

6.3 Sample Handling and Storage Requirements 52

6.4 Sample Disposal 52

7. HEADSPACE GAS SAMPLING 57

8. SAMPLING OF SOLID PROCESSING RESIDUES AND SOILS 58

9. RADIOASSAY 59

10. RADIOGRAPHY 60

11. HYDROGEN AND METHANE ANALYSIS 61

12. GAS VOLATILE ORGANIC COMPOUND ANALYSIS 62

13. TOTAL VOLATILE ORGANIC COMPOUND ANALYSIS 63

13.1 Quality Assurance Objectives 63

13.1.1 Precision 63

13.1.2 Accuracy 63
13.1.3 Method Detection Limits (MDLs) 63
13.1.4 Program Required Quantitation Limits (PRQLs) 63
13.1.5 Completeness 65
13.1.6 Comparability 65
13.1.7 Representativeness 65

13.2 Methods Requirements 65

13.2.1 Criteria for Standards 66
13.2.2 Criteria for GC/MS Qualitative and Quantitative Analysis 66
13.2.3 Criteria for GC Qualitative and Quantitative Analysis 66

13.3 Quality Control 67

13.3.1 Method Performance Samples 67
13.3.2 Laboratory Control Samples (LCSs) 67
13.3.3 Laboratory Blanks (LBSs) 68
13.3.4 Matrix Spikes (MSs) and Matrix Spike Duplicates (MSDs) 69
13.3.5 Surrogate Compounds 69
13.3.6 Blind Audit Samples 69

13.4 Instrument Testing, Inspection, and Maintenance Requirements 69

13.4.1 GC/MS Instrument Requirements 69
13.4.2 GC Instrument Requirements 70

13.5 Instrument Calibration and Frequency 70

13.5.1 GC/MS Calibration 70
13.5.2 GC Calibration 72

13.6 Data Management 72

13.6.1 Data Reduction 73
13.6.2 Data Validation 73
13.6.3 Data Reporting 73

14. TOTAL SEMIVOLATILE ORGANIC COMPOUND ANALYSIS 105

14.1 Quality Assurance Objectives 105

14.1.1 Precision 105
14.1.2 Accuracy 105
14.1.3 Method Detection Limits 105
14.1.4 Program Required Quantitation Limits 105
14.1.5 Completeness 105

14.1.6 Comparability 106
14.1.7 Representativeness 107

14.2 Methods Requirements 107

14.2.1 Criteria for Standards 107
14.2.2 Criteria for GC/MS Qualitative and Quantitative Analysis 107

14.3 Quality Control 108

14.3.1 Method Performance Samples 108
14.3.2 Laboratory Control Samples (LCSs) 108
14.3.3 Laboratory Blanks (LBs) 109
14.3.4 Matrix Spike (MSs) and Matrix Spike Duplicates (MDSs) 110
14.3.5 Surrogate Compounds 110
14.3.6 Blind Audit Samples 110

14.4 Instrument Testing, Inspection, and Maintenance Requirements 110

14.5 Instrument Calibration and Frequency 111

14.5.1 GC/MS Calibration 111

14.6 Data Management 112

14.6.1 Data Reduction 112
14.6.2 Data Validation 113
14.6.3 Data Reporting 113

15. TOTAL METALS ANALYSIS 133

15.1 Quality Assurance Objectives 133

15.1.1 Precision 133
15.1.2 Accuracy 133
15.1.3 Instrument Detection Limits 133
15.1.4 Program Required Quantitation Limits 133
15.1.5 Completeness 134
15.1.6 Comparability 134
15.1.7 Representativeness 134

15.2 Methods Requirements 135

15.2.1 Criteria for Standards 136
15.2.2 Criteria for ICP-AES Qualitative and Quantitative Analysis 136
15.2.3 Criteria for GFAA Qualitative and Quantitative Analysis 136

15.2.4 Criteria for Mercury (CVAF & CVAA) Qualitative and Quantitative Analysis 136

15.3 Quality Control 137

15.3.1 Method Performance Samples 137
15.3.2 Laboratory Control Samples 137
15.3.3 Laboratory Blanks 138
15.3.4 Matrix Spikes and Matrix Spike Duplicates 139
15.3.5 Blind Audit Samples 139

15.4 Instrument Testing, Inspection, and Maintenance Requirements 139

15.5 Instrument Calibration and Frequency 139

15.5.1 ICP-AES Calibration 141
15.5.2 GFAA Calibration 141
15.5.3 Mercury Calibration 142

15.6 Data Management 142

15.6.1 Data Reduction 142
15.6.2 Data Validation 142
15.6.3 Data Reporting 149

REFERENCES 163

FIGURES

1-1. ACL organization and responsibilities for TWCP support 2

2-1. Example of a nonconformance report 24

2-2. Example of a TWCP record of variance 26

2-3. Example of ACL control chart 28

3-1. Example of the ALD QAO data review checklist for total purgeable VOC analysis 34

3-2. Example of the ALD QAO data review checklist for total nonhalogenated VOC analysis 35

3-3. Example of the ALD QAO data review checklist for total SVOC analysis 36

3-4. Example of the ALD QAO data review checklist for total metals analysis 37

6-1. Example of a TWCP Chain-of-Custody form 50

6-2. Example of TWCP Sample Receiving and Custody Review Checklist 51

6-3. Example of ACL internal tracking COC form 53

6-4. Example of ACL internal sample tracking form 54

6-5. Example of an SPO data validation notification form 56

13-1. Example of independent technical review checklist for total purgeable VOC analysis 74

13-2. Example of independent technical review checklist for total nonhalogenated VOC analysis 76

13-3. Example of ACL data report cover page 78

13-4. Example of a sample identification table 79

13-5. Example of total purgeable VOC Analysis Data Sheet 80

13-6. Example of total purgeable VOC TIC analysis data sheet 82

13-7. Example of LCS reporting form for total purgeable VOC analysis 83

13-8. Example of matrix spike/matrix spike duplicate reporting form for total purgeable VOC analysis 84

13-9. Example of surrogate spike reporting form for total purgeable VOC analysis 85

13-10. Example of blank results reporting form for total purgeable VOC analysis 87

13-11. Example of LB summary reporting form for total purgeable VOC analysis 88

13-12. Example of method blank summary reporting form for total purgeable VOC analysis 89

13-13. Example of BFB tune reporting form for total purgeable VOC analysis 90

13-14. Example of internal standard area reporting form for total purgeable VOC analysis . . . 91

13-15. Example of CCAL reporting form for total purgeable VOC analysis 92

13-16. Example of CCAL Internal Standard Area/RT reporting form for total purgeable VOC analysis 93

13-17. Example of ICAL reporting form for total purgeable VOC analysis 94

13-18. Example of MDL reporting form for total purgeable VOC analysis 95

13-19. Example of total nonhalogenated VOC analysis data sheet 96

13-20. Example of LCS reporting form for total nonhalogenated VOC analysis 98

13-21. Example of matrix spike/matrix spike duplicate reporting form for total nonhalogenated VOC analysis 99

13-22. Example of LB reporting form for total nonhalogenated VOC analysis 100

13-23. Example of CCV reporting form for total nonhalogenated VOC analysis 101

13-24. Example of analyte identification form for total nonhalogenated VOC analysis 102

13-25. Example of ICAL reporting form for total nonhalogenated VOC analysis 103

13-26. Example of MDL reporting form for total nonhalogenated VOC analysis 104

14-1. Example of independent technical review checklist for total SVOC analysis 114

14-2. Example of total SVOC analysis data sheet 116

14-3. Example of TIC analysis data sheet for total SVOC analysis 118

14-4. Example of LCS reporting form for total SVOC analysis 119

14-5. Example of matrix spike/matrix spike duplicate reporting form for total SVOC analysis 120

14-6. Example of surrogate spike reporting form for total SVOC analysis 121

14-7. Example of LB results reporting form for total SVOC analysis 122

14-8. Example of LB summary reporting form for total SVOC analysis 123

14-9. Example of DFTPP tune reporting form for total SVOC analysis 124

14-10. Example of internal standard area reporting form for total SVOC analysis 125

14-11. Example of CCAL reporting form for total SVOC analysis 127

14-12. Example of CCAL Internal Standard Area/RT reporting form for total SVOC analysis 128

14-13. Example of ICAL reporting form for total SVOC analysis 131

14-14. Example of MDL reporting form for total SVOC analysis 132

15-1. Example of independent technical review checklist for ICP-AES total metals analysis 143

15-2. Example of independent technical review checklist for GFAA total metals analysis . . . 145

15-3. Example of independent technical review checklist for CVAF/CVAA total metals analysis 147

15-4. Example of Total Metals Analysis Data Sheet 150

15-5. Example of LCS reporting form for total metals analysis 151

15-6. Example of matrix spike/matrix spike duplicate recovery reporting form for total metals analysis 152

15-7. Example of matrix spike duplicate RPD reporting form for total metals analysis 153

15-8. Example of ICB/CCB/LB reporting form for total metals analysis 155

15-9. Example of ICV/CCV reporting form for total metals analysis 156

15-10. Example of interference check reporting form for ICP-AES total metals analysis 157

15-11. Example of post-digestion spike recovery reporting form for total metals analysis . . . 158

15-12. Example of serial dilution reporting form for total metals analysis 159

15-13. Example of Method of Standard Additons reporting form for total metals analysis . . . 160

15-14. Example of high standard verification check reporting form for total metals analysis . . 161

15-15. Example of IDL reporting form for total metals analysis 162

TABLES

1-1. Cross-reference between sections of ACL QAPjP, basic requirements of ASME NQA-1, and DOE Order 5700.6c criteria 7

1-2. Requirements for review, approval, and control of ACL documents 9

1-3. Target analytes for homogeneous solid and soil/gravel analyses 11

1-4. Minimum training and qualification requirements 14

1-5. ACL records for the TRU Waste Characterization Program 15

1-6. ACL procedures used in support of TWCP 18

4-1. Primary and backup instrument and equipment systems used for TWCP support 45

6-1. Sample handling requirements for solid process residues and soils 55

13-1. Total volatile organic compounds target analyte list and QA objectives 64

13-2. Summary of laboratory QC samples and frequencies for total VOC analysis 68

13-3. 4-bromofluorobenzene key ions and abundance criteria 70

13-4. GC/MS and GC/FID calibration requirements for total VOC analysis 71

14-1. Total semivolatile organic compounds target analyte list and QA objectives 106

14-2. Summary of laboratory QC samples and frequencies for total SVOC analysis 109

14-3. DFTPP key ions and abundance criteria 111

14-4. GC/MS calibration requirements for total SVOC analysis 112

15-1. Total metals target analyte list and QA objectives 134

15-2. Summary of laboratory QC samples and frequencies for total metals analysis 138

15-3. Summary of calibration requirements and analysis QC for total metals analyses 140

ACRONYMS

AA	atomic absorption
ACL	Analytical Chemistry Laboratory
ACLP	Analytical Chemistry Laboratory Procedure
ACMM	Analytical Chemistry Methods Manual
ACS	Analytical Computer System
AEDL	Applied Engineering and Development Laboratory
Ag	silver
ALARA	As Low As Reasonably Achievable
ALD	Analytical Laboratories Department
ANL-W	Argonne National Laboratory-West
ANSI	American National Standards Institute
As	arsenic
ASME	American Society of Mechanical Engineers
ASTM	American Society for Testing and Materials
Ba	barium
Be	beryllium
BFB	4-bromofluorobenzene
°C	degrees Centigrade
%C	percent completeness
CA	corrective action
CAO	Carlsbad Area Office
CAS	Chemical Abstract Service
CCAL	continuing calibration
CCB	continuing calibration blank verification
CCC	calibration check compound
CCP	Chemical Processing Plant
CCV	continuing calibration verification
Cd	cadmium
CFR	Code of Federal Regulations
COC	chain of custody
Cr	chromium
CV	coefficient of variation
CVAA	cold vapor atomic absorption
CVAF	cold vapor atomic fluorescence
%D	percent difference
DFTPP	decafluorotriphenylphosphine
DOE	U.S. Department of Energy
DOE-ID	U. S. Department of Energy Idaho Operations Office
DQO	data quality objective
DRC	data and records coordinators
EICP	extracted ion current profile

EPA	U.S. Environmental Protection Agency
FID	flame ionization detector
ft	foot
ft^2	square foot
g	gram
GC	gas chromatography
GC/FID	gas chromatograph/flame ionization detector
GC/MS	gas chromatograph/mass spectrometer
GFAA	graphite furnace atomic absorption
HEPA	high efficiency particulate air
Hg	mercury
hr	hour
ICAL	initial calibration
ICB	initial calibration blank verification
ICP	inductively coupled plasma
ICP-AES	inductively coupled plasma atomic emission spectrometry
ICPP	Idaho Chemical Processing Plant
ICSA	interference check sample, part A
ICSAB	interference check sample, part A+B
ICV	initial calibration verification
ID	identification
IDL	instrument detection limit
IECF	interelement interference correction factor
INEL	Idaho National Engineering Laboratory
ISA	internal standard area
kg	kilogram
L	liter
LB	laboratory blank
LCS	laboratory control sample
LMITCO	Lockheed Martin Idaho Technologies Company
MCP	Management Control Procedure
MDL	method detection limit
mg/kg	milligram per kilogram
mg/L	milligram per liter
MMDDYY	month-day-year format
MPS	method performance sample
MS	matrix spike
MSA	method of standard additions
MSD	matrix spike duplicate
μg	microgram

μg/L	microgram per liter
NARA	National Archives and Records Administration
Nci/g	nanocuries per gram
NCR	nonconformance report
NCSL	National Conference of Standards Laboratories
NEIC	National Enforcement Investigations Center
ng	nanograms
Ni	nickel
NIST	National Institute of Standards and Technology
NH-VOCs	nonhalogenated volatile organic compounds
NQA	Nuclear Quality Assurance
NTP	National TRU Program
P&T	purge and trap
Pb	lead
PDD	Program Description Document
PDP	Performance Demonstration Program
PFTBA	perfluorotributylamine
ppb	parts per billion
ppm	parts per million
PRDL	Program Required Detection Limit
PRQL	Program Required Quantitation Limit
Pu	plutonium
QA	Quality Assurance
QAO	Quality Assurance Officer
QA/QC	Quality Assurance/Quality control
QAPjP	Quality Assurance Project Plan
QAPD	Quality Assurance Program Description
QAPP	Quality Assurance Program Plan
QC	Quality Control
QE	quality engineer
%R	percent recovery
r	correlation coefficient
RAL	Remote Analytical Laboratory
RCRA	Resource Conservation and Recovery Act
RCT	Radiological Control Technician
RF	response factor
RPD	relative percent difference
RRF	relative response factor
RRT	relative retention time
%RSD	percent relative standard deviation
RSD	relative standard deviation
RT	retention time
RWMC	Radioactive Waste Management Complex

SA	spike added
Sb	antimony
Se	selenium
SOP	standard operating procedure
SPCC	system performance check compound
SPM	site project manager
SPO	site project office
SQAO	site quality assurance officer
SR	sample result
SSR	spiked sample result
SVOC	semivolatile organic compound
TC	toxicity characteristic
TICs	tentatively identified compounds
TL	technical leader
Tl	thallium
TP	technical procedure
TRU	transuranic
TWCP	Transuranic Waste Characterization Program
U	uranium
UCL_{90}	Upper 90-percent confidence limit
V	vanadium
VOC	volatile organic compound
VTSR	validated time of sample receipt
WIPP	Waste Isolation Pilot Plant
$\bar{X}$	mean value
Zn	zinc

DEFINITIONS

Accuracy—The degree of agreement between a measured value and an accepted reference or the true value. Accuracy is determined as the percent recovery (%R).

Aliquot—A measured portion of a field sample taken for analysis.

Analysis date/time—The date and military time (24-hour clock) of the introduction of the sample, standard, or blank into the analysis system.

Analyte—The element, ion, or compound an analysis seeks to determine; the constituent of interest.

Analytical batch—A suite of samples of similar matrix that are processed as a unit, using the same analytical method within a specific time period. An analysis batch must not exceed 20 samples, all of which must be received by the laboratory within 14 days of the validated time of sample receipt (VTSR) of the first sample of the batch.

Analytical method—The sample preparation and instrumentation procedures or steps that must be performed to estimate the quantity of an analyte in a sample.

Analytical sample—Any solution or media introduced into an instrument on which an analysis is performed, excluding instrument calibrations, initial calibration verifications, and continuing calibration verifications. Note that the following are all defined as analytical samples: field samples, duplicate samples, matrix spike and matrix spike duplicate samples, laboratory control samples, laboratory blanks, post-digestion spike samples, interference check samples and serial dilution samples.

Assessment—The evaluation process used to measure the performance or effectiveness of a system and its elements. In this QAPjP, assessment is an all-inclusive term used to denote any of the following: audit, performance evaluation, management systems review, peer review, inspection, or surveillance.

Audit—A planned and documented investigative evaluation of an item or process to determine the adequacy and effectiveness, as well as compliance with established procedures, instructions, drawings, or other applicable documents.

Background correction—A technique used to compensate for variable background contribution to the instrument signal in the determination of spectrometric determination of trace metals.

Blind audit sample—A sample of known composition provided as a single-blind sample to the analytical laboratory. Used by DOE to evaluate analytical laboratory performance.

Calibration—The comparison of measuring and test equipment or a measurement standard of unknown accuracy to a measurement standard of known accuracy in order to detect, correlate, report, or eliminate, by adjustment, any variation in the accuracy of the instrument being compared. The term calibration is also used to refer to the standardization process used with analytical instrumentation whereby an analytical curve is established relating instrument response (signal) to analyte amount or concentration.

Calibration blank—A volume of acidified deionized/distilled water used to determine the calibration zero-response for spectrometer calibration.

Calibration standards—A series of known-concentration standard solutions used to establish instrument response during calibration.

Chain-of-custody—A set of procedures established to ensure that sample data integrity is maintained.

Comparability—A qualitative parameter expressing the confidence with which one data set can be compared with another. Sample data should be comparable with other measurement data for similar samples and sample conditions.

Completeness—The percentage of measurements made that are judged to be valid measurements. Valid measurements are those obtained when analytical systems were in control, i.e., all calibration, verification, interference and non-matrix checks met acceptance criteria.

Condition adverse to quality—An all-inclusive term used in reference to any of the following: failures, malfunctions, deficiencies, defective items, and nonconformances. A significant condition adverse to quality is one that, if uncorrected, could have a serious effect on safety or operability.

Continuing calibration standards—Analytical standards measured periodically to verify the calibration of the analytical system.

Control limits—A range within which specified measurement results must fall to be compliant. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring that noncompliant data be flagged.

Corrective action— Measures taken to rectify conditions adverse to quality and, where necessary, to preclude repetition. This requires a systematic problem-solving approach using data to draw conclusions about likely reasons for a problem.

Correlation coefficient—A number (r) that indicates the degree of dependence between two variables (e.g., concentration and absorbance). The more dependent they are, the closer the value to one. Determined by least squared analysis.

Data quality objectives (DQO)—Statements that explain the purpose of collecting the data. DQOs may contain qualitative and quantitative statements that describe the overall level of uncertainty that a decisionmaker is willing to accept in results derived from environmental data. DQOs are determined based on the end uses of the data to be collected.

Data reduction—Operations necessary to convert data from the raw form to a final form as required by the customer.

Day—Unless otherwise specified, day shall mean calendar day.

Field sample—A portion of material received for analysis that is contained in a single container and identified by a unique DOE sample number.

Holding time—The maximum permissible time allowed between time of sample collection and time of preparation or analysis.

Independent assessment—A quality assurance program assessment that is conducted by an independent group or organization, having authority and freedom from the line organization, to evaluate the scope, status, adequacy, programmatic compliance, and implementation effectiveness of the quality assurance program.

Independent standard—A standard that is composed of analytes from a different source (i.e., different manufacturer) than that used for initial calibration standards.

Initial calibration—Analysis of analytical standards at different specified concentrations; used to define the quantitative response, linearity, and dynamic range of the instrument to target analytes.

Instrument calibration—Analysis of analytical standards for a series of different specified concentrations; used to define the quantitative response of an instrument to target analytes.

Instrument detection limit (IDL)—The minimum signal that an instrument can detect with 99-percent confidence that the analyte concentration is greater than zero.

Interference check sample (ICS)—A quality control sample containing interferants that is used to evaluate the adequacy of interferant compensation algorithms.

Interferants—Substances that affect the analysis for the compound or element of interest.

Laboratory blank—An analyte-free matrix that undergoes preparation and analysis processes identical to those used on field samples. The laboratory blank is used to document contamination resulting from the laboratory sample preparation and analytical process.

Laboratory control sample (LCS)—A control sample of known composition that is prepared and analyzed with field samples using the same analytical procedures.

Laboratory duplicate—A second aliquot of a sample that is treated the same as the original sample to determine the precision of the analytical procedure.

Linear range—The concentration range over which the analytical curve remains linear.

Matrix—The predominant material of which the sample to be analyzed is composed, e.g., water, soil, sludge. Matrix is not synonymous with phase (solid or liquid).

Matrix checks—Those quality control samples that provide information on how the sample matrix affects achievable analytical method accuracy and precision. Matrix checks include matrix spikes, matrix spike duplicates, laboratory duplicates, post-digestion spikes, serial dilutions, surrogate spikes and internal standards.

Matrix modifier—Salts used in atomic absorption techniques to lessen the effects of chemical interferences, viscosity, and surface tension.

Matrix spike (MS)—An aliquot of a field sample to which known amounts of target analytes are added (i.e., spiked) and is then carried through all sample preparation and analysis procedures. Results of the matrix spike are calculated as percent recovery of the spiked analytes, and indicate potential bias of the method due to the actual sample matrix.

Matrix spike duplicate (MSD)—A second matrix spike prepared on a sample that is then carried through all sample preparation and analysis procedures. In addition to determining percent recovery, results of the matrix spike duplicate are calculated as relative percent difference (RPD) between matrix spike and matrix spike duplicate results. The RPD indicates potential effects of the actual sample matrix on method precision.

Metals—For the purposes of the TWCP, those elements listed in Table 15-1 used to satisfy program requirements.

Method detection limit (MDL)—The minimum concentration of a substance that can be measured and reported for a given method with 99% confidence that the analyte concentration is greater than zero.

Method of Standard Additions (MSA)—The addition of three increments of a standard solution (spikes) to sample aliquots of the same size. Measurements are made on the original and after each addition. The slope, x-intercept and y-intercept are determined by least-squares analysis. The analyte concentration is determined by the absolute value of the x-intercept. Ideally, the spike volume is low relative to the sample volume (<10% of the volume). MSA may counteract matrix effects, but it will not compensate for spectral effects.

Method performance samples (MPS)—Samples of known composition and analyte concentration that are used to determine a laboratory's ability to perform an analysis method in conformance with program data quality assurance objectives.

Narrative—The portion of the data package that includes descriptive documentation of any problems encountered in processing the samples, corrective actions taken, and problem resolution.

Nonconformance—A deficiency in characteristic, documentation, or procedure that renders the quality of an item, process, or service unacceptable or indeterminate. Examples of nonconformance include physical defects, test failures, missing times, incorrect or inadequate documentation, and deviations from prescribed processing, inspection test procedures, or other specifications. Each nonconformance must reach final resolution or disposition.

Nonconformance dispositions—Actions taken to resolve a nonconformance.

NCR coordinator—An individual designated by cognizant line management to coordinate submittal and resolution of nonconformance reports (NCRs). The NCR coordinator works with the cognizant quality engineer to ensure that all NCRs are properly dispositioned and closed. The NCR coordinator for the ACL is the ALD quality assurance officer.

Operational variance—An operational variance is an approved and controlled change from routine, approved procedures or plans. This change does not affect the ability to achieve specified performance standards or quality requirements.

Percent difference (%D)—The absolute difference between an initial measurement value and a subsequent measurement, expressed as a percentage of the initial measurement.

Percent solids—The proportion of solid in a soil or sludge sample, determined by drying an aliquot of the sample.

Post-digestion spike—An aliquot of digested sample (for metals analysis) to which known amounts of analyte are added. Comparison of post-digestion spike recovery with that of the matrix spikes indicates whether observed matrix effects are occurring during digestion or during determinative analysis.

Precision—A measure of mutual agreement among individual measurements of the same property made under prescribed similar conditions; often expressed as a standard deviation or relative percent difference.

Procedure—A document that specifies or describes how an activity is to be performed. The document contains detailed, step-by-step descriptions of the sequence of actions to be followed in order to perform a given task. If followed in sequence, a procedure provides enough information that a trained person could complete the covered task without additional information.

Product—The end result of a work process, e.g., the deliverables in a project or program.

Program—A program is typically made up of technology-based activities, projects, and support organizations. A program may contain one or more projects or support organizations.

Program required detection limit (PRDL)—The maximum values for instrument detection limits permissible under the TWCP QAPP.

Program required quantitation limit (PRQL)—Minimum level of analyte quantitation acceptable under the TWCP QAPP.

Project—A project is a unique effort within a program with a scheduled beginning, intermediate and ending milestones, and prescribed cost.

Purge and trap—An analytical technique used to isolate volatile (purgeable) organic compounds by stripping the compounds from water or soil/solid with a stream of inert gas, trapping the compounds on a porous polymer trap, and thermally desorbing the trapped compounds onto the gas chromatographic column.

Quality—The totality of features and characteristics of a product or service that bear on its ability to satisfy stated or implied needs and expectations of the user.

Quality assurance (QA)—All those planned and systematic actions necessary to provide confidence that a facility, structure, system, or component will perform satisfactorily and safely in service.

Quality assurance objectives—The characteristics of data that are associated with their ability to satisfy a given purpose or objective. The characteristics of major importance are accuracy, precision, completeness, representativeness, and comparability.

Quality control (QC)—The overall system of technical activities that measure the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer.

Recovery—The numerical ratio of the amount of analyte measured by the laboratory method to that of the known amount of analyte. Usually expressed as a percent recovery (%R).

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

Review/data review—The process used to ensure that the proper reduction of raw data has been accomplished.

Root cause—That cause that, if corrected, would prevent recurrence of the occurrence under consideration and similar occurrences. It is a cause that has generic implications to a broad group of possible occurrences, and it is the most fundamental cause that can logically be identified and corrected. There may be a series of causes that can be identified, one leading to another. This series should be pursued until the more fundamental, correctable cause has been identified.

Run—A continuous analytical sequence consisting of prepared samples and all associated quality assurance measurements as required by the TWCP QAPP.

Sample—A portion of material to be analyzed that is contained in a single container and identified by a unique sample number.

Sample custodian—An individual assigned and authorized to receive, track, and dispose of samples.

Sample number—A unique identification number that is designated for each sample. The sample number appears on all sample reports that document information on that sample. Each sample is assigned a field sample number at the time of collection and a laboratory sample number at the time of laboratory receipt.

Sampling batch—A suite of samples of a similar matrix collected consecutively using the same sampling equipment within a specific time period. A sampling batch can be up to 20 samples, all of which must be collected within 14 days of the first sample in the batch.

Semivolatile organic compound (SVOC)—For the purposes of the TWCP, those SVOCs listed in Table 14-1 and any additional compounds tentatively identified by the SVOC analytical procedures used to satisfy program requirements.

Serial dilution—The dilution of a sample by a factor of five for metals analysis. When corrected by the dilution factor, the diluted sample must agree with the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferants or non-linear calibration.

Standard—A mixture of known analyte concentration used to establish or check instrument calibration.

Stock standard—A standard solution which can be diluted to derive other standards.

Target analytes—Those analytes for which analysis is required by the TWCP.

Tentatively identified compounds (TICs)—Nontarget compounds identified by GC/MS mass spectral library searches. These reported concentrations have a higher associated uncertainty than the reported target analyte concentrations.

Traceability—The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for quality of the project.

Trend analysis—A process whereby performance data are collected, organized, displayed, and evaluated for changes over a period of time.

Total metals—Analyte elements that have been digested using a hot-acid leach prior to analysis.

Validated time of sample receipt (VTSR)—The date and time when a sample or group of samples is received at the laboratory, as recorded on the field COC documentation. The VTSR is used to determine analytical batches and data report due dates.

Validation—An activity that demonstrates or confirms that a process, item, data set, or service satisfies the requirements defined by the user. Validation is documented by signature release.

Variance—A measure of the dispersion of a series of results around their average. It is the sum of the squares of the individual deviations from the average of the results, divided by the number of results minus one.

Verification—The act of authenticating that a process, item, data set, or service is, in fact, that which is claimed. Data verification is the process used to confirm that all review and validation procedures have been completed.

Volatile organic compound (VOC)—For the purposes of the TWCP, those VOCs listed in Table 13-1 and any additional compounds tentatively identified by the VOC analytical procedures used to satisfy program requirements.

Wet weight—the weight of a sample aliquot including moisture (undried).

Analytical Chemistry Laboratory Quality Assurance Project Plan for the Transuranic Waste Characterization Program

1. PROGRAM MANAGEMENT

The Transuranic (TRU) Waste Characterization Program (TWCP) quality assurance program plan (QAPP) (DOE, 1995a) requires each U.S. Department of Energy (DOE) facility participating in the program to develop and implement a quality assurance project plan (QAPjP) that addresses all pertinent requirements specified in the QAPP. This facility QAPjP is the Analytical Chemistry Laboratory (ACL) document intended to satisfy this requirement, and is a supplement to the Idaho National Engineering Laboratory (INEL) Site Project Office (SPO) QAPjP (LMITCO, 1996).

1.1 Program Organization and Responsibilities

The ACL is a laboratory within the Analytical Laboratories Department (ALD), Applied Engineering and Development Laboratory (AEDL) Branch, of Lockheed Martin Idaho Technologies Company (LMITCO), which manages the INEL for the DOE Idaho Operations Office (DOE-ID). The ACL supplies laboratory services to the SPO in support of the TWCP. A functional organization chart for the ACL, showing lines of authority and communication for TWCP support, is presented in Figure 1-1.

1.1.1 Analytical Laboratories Department Manager

The ALD Manager is the manager of the Analytical Chemistry Laboratory (ACL). The ALD manager is responsible for managing laboratory operations and product quality. ALD manager responsibilities include, but are not limited to, personnel training, equipment and systems maintenance, laboratory safety, working with customers to identify project-specific requirements, monitoring status of work, implementing preventive and corrective actions (CA) and cost control. The ALD manager is responsible for the timely reporting of data to the SPO and for ensuring that the data meet the client's specifications. The ALD manager reviews all ACL procedures and the ACL TWCP QAPjP, and manages the implementation of ACL project responsibilities. The ALD manager also participates in business assessments of ACL operations.

1.1.2 Analytical Laboratories Department Quality Assurance Officer

The ALD quality assurance officer (QAO) is responsible for the oversight of all ACL quality assurance (QA) activities associated with TWCP. The ALD QAO reports directly to the ALD manager and is organizationally independent of ACL production activities. The QAO's responsibilities for TWCP support include routine interfacing with the ALD manager and staff members on the development, documentation, and evaluation of ACL quality assurance/quality control (QA/QC) procedures and policy. The QAO is responsible for drafting the ACL QAPjP, and reviews all ACL quality procedures, implementing procedures and analytical methods. The QAO issues

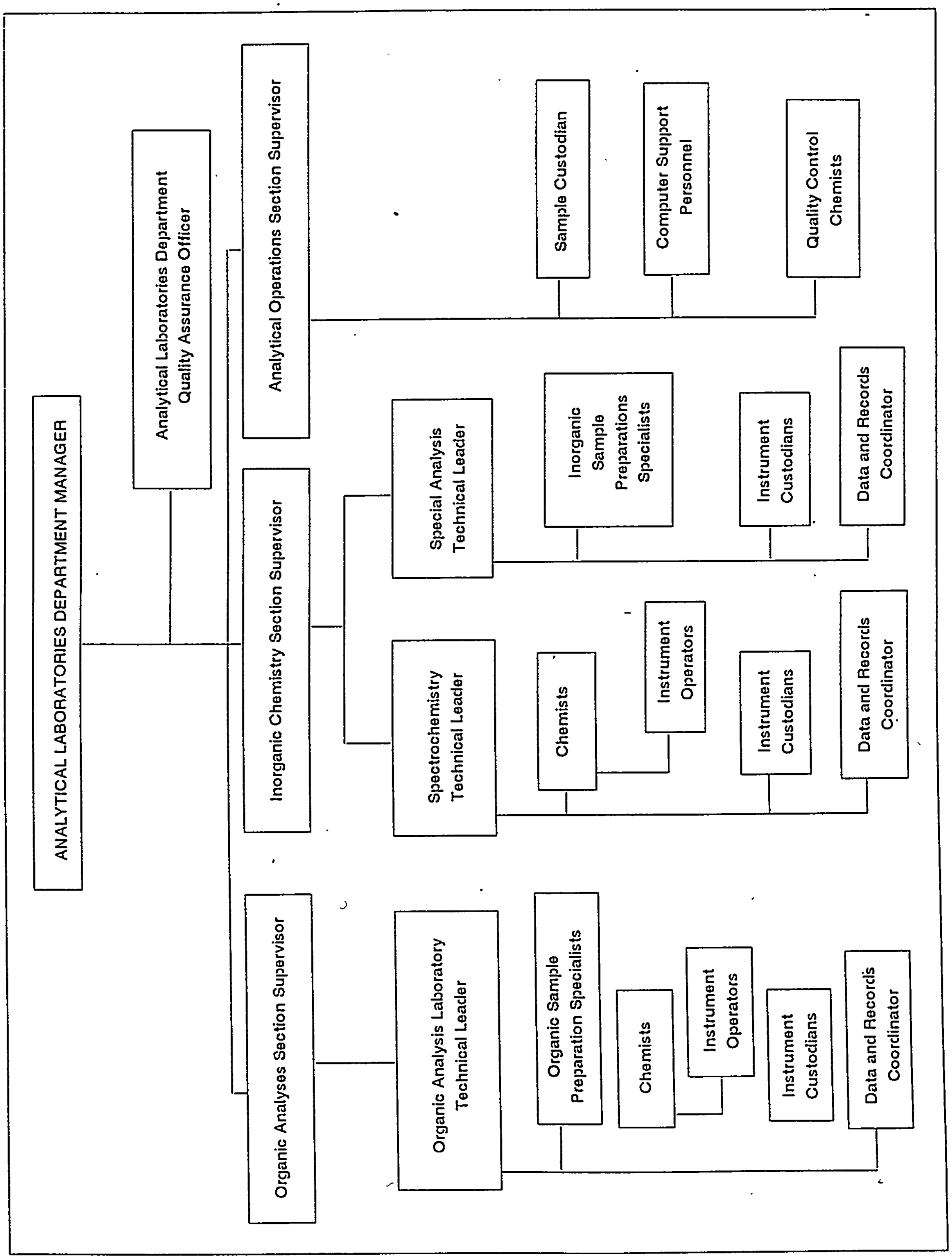


Figure 1-1. ACL organization and responsibilities for TWCP support.

quarterly QA reports to the site quality assurance officer (SQAO), reviews and releases TWCP data packages, monitors and documents method performance, trains staff in TWCP-related QA/QC requirements, coordinates nonconformance reporting and CAs, and reports problems and findings to the ALD manager and to the INEL SQAO and INEL site project manager (SPM). The ALD QAO also performs independent assessments of ACL operations.

1.1.3 Analytical Operations Supervisor

The Analytical Operations supervisor is responsible for overseeing and managing analytical support functions at the ACL, including computer systems configuration, control and maintenance; routine quality control oversight, project management coordination, sample tracking, and waste disposal. The Analytical Operations Supervisor reports directly to the ALD manager.

1.1.4 Organic Analyses Section Supervisor

The Organic Analyses Section supervisor is responsible for managing all organic analyses activities at ALD. The Organic Analyses Section supervisor is responsible for ensuring generation of technically valid data, coordinating and scheduling work, and training section personnel. The Organic Section supervisor is responsible for ensuring that QC practices meet the specifications of the ACL QAPjP and ACL methods, and for informing the ALD QAO and ALD manager of any concerns pertaining to data quality. The Organic Analyses Section Supervisor ensures that all organic analyses data have received proper technical review (see Section 3) before release to the organic data and records coordinator (DRC) for report generation, and may review and approve organic data packages and reports. The Organic Analyses Section Supervisor reports directly to the ALD manager.

1.1.5 Inorganic Chemistry Section Supervisor

The Inorganic Chemistry Section supervisor is responsible for managing all inorganic analyses activities at ALD. These activities include spectrochemical analyses and special analyses (classical analyses and sample preparation). The Inorganic Chemistry Section supervisor is also responsible for managing the Remote Analytical Laboratory (RAL), the ACL analytical hot cell facility. The Inorganic Chemistry Section supervisor is responsible for ensuring generation of technically valid data, coordinating and scheduling work, and training section personnel. The Inorganic Chemistry Section supervisor is responsible for ensuring that QC practices meet the specifications of the ACL QAPjP and ACL methods, and for informing the ALD QAO and ALD manager of any concerns pertaining to data quality. The Inorganic Chemistry Section Supervisor ensures that all inorganic and RAL data have received proper technical review (see Section 3) before release to the spectrochemistry or special analyses DRCs for report generation, and may review and approve inorganic data packages and reports. The Inorganic Chemistry Section supervisor reports directly to the ALD manager.

1.1.6 Organic Analysis Laboratory Technical Leader

The Organic Analysis Laboratory Technical Leader (TL) is responsible for overseeing routine analytical operations in the organic analysis laboratory at ACL. The Organic Analysis Laboratory TL is responsible for development and implementation of laboratory procedures and associated methods for volatile organic compound (VOC) analysis and semivolatile organic compound (SVOC) analysis, and is responsible for implementing corrective actions to address analytical problems revealed by data review, QA monitoring, and audits. The Organic Analysis Laboratory TL works with the Organic Analyses Section Supervisor to coordinate work personnel and ensure that all organic analysis staff members are qualified and trained. The TL is responsible for ensuring that project QA/QC requirements are implemented and following during sample analysis. The TL is responsible for ensuring that complete raw data are generated for each analysis and that all data receive independent technical review (see Section 3) before release to the organic DRC for report generation. The TL may review and approve organic data packages and reports. The Organic Analysis Laboratory TL reports to the Organic Analyses Section supervisor.

The Organic Analysis Laboratory TL fulfills the qualification and responsibilities of the GC/MS and GC technical supervisors, as defined in Table 1-4 of the TWCP QAPP.

1.1.7 Spectrochemistry Technical Leader

The Spectrochemistry TL is responsible for overseeing routine spectrochemical analytical operations at ACL. The Spectrochemistry TL is responsible for development and implementation of laboratory procedures and associated methods for metals analysis, and is responsible for implementing corrective actions to address analytical problems revealed by data review, QA monitoring, and audits. The Spectrochemistry TL works with the Inorganic Chemistry Section Supervisor to coordinate work personnel and ensure that all spectrochemical analysis staff members are qualified and trained. The TL is responsible for ensuring that project QA/QC requirements are implemented and followed during sample analysis. The TL is responsible for ensuring that complete raw data are generated for each analysis and that all data receive independent technical review (see Section 3) before release to the spectrochemistry DRC for report generation. The TL may review and approve spectrochemical data packages and reports. The Spectrochemistry TL reports to the Inorganic Chemistry Section supervisor.

The Spectrochemistry TL fulfills the qualifications and responsibilities of the atomic spectrometry technical supervisors, as defined in Table 1-4 of the TWCP QAPP.

1.1.8 Special Analysis Technical Leader

The Special Analysis TL is responsible for overseeing inorganic sample preparations and special analytical procedures at ACL. The Special Analysis TL is responsible for development and implementation of laboratory procedures and associated methods, and is responsible for implementing corrective actions to address analytical problems revealed by data review, QA monitoring, and audits. The Special Analysis TL works with the Inorganic Chemistry Section Supervisor to coordinate work personnel and ensure that all special analysis staff members are qualified and trained. The TL is responsible for ensuring that project QA/QC requirements are implemented and followed during sample preparation and analysis. The TL is responsible for ensuring that complete raw data are generated for each analysis.

1.1.9 Chemists, Instrument Operators and Sample Preparation Specialists

Chemists, Instrument Operators and Sample Preparation Specialists are the scientists, analysts and technicians responsible for performing sample preparation and analyses, data reduction, and reporting in accordance with project requirements. Chemists fulfill the role of operator, as defined in Table 1-4 of the TWCP QAPP, and independently operate analytical equipment, perform troubleshooting and maintenance, and interpret data. Chemists may act as independent technical reviewers. Instrument operators operate analytical equipment, perform troubleshooting and maintenance, and interpret data under the direction of a chemist. Instrument operators cannot act as independent technical reviewers. Sample preparation specialists are responsible for performing all preparation steps necessary to prepare samples for analysis (e.g., digestions and extractions). Chemists, instrument operators and sample preparation specialists are responsible for following analysis and QC procedures specified in analytical methods, and documenting any deviation from TWCP QAPP specifications. They are responsible for critically observing and evaluating all analytical procedures and bringing any practices or occurrences that might affect the reliability of analytical data to the attention of the appropriate TL, supervisor, and ALD QAO. Chemists and sample preparation specialists may be responsible for writing analytical methods at the direction of the TL or supervisor.

1.1.10 Sample Custodian

The sample custodian (or trained alternates) receives and maintains custody of samples delivered to the laboratory (see Section 6), and ensures that all samples receive appropriate preservation within the laboratory, secure storage, and complete record keeping. The sample custodian documents all discrepancies in sample documentation received with the samples and reconciles the discrepancies with the responsible sampling organization. The sample custodian reports to the Analytical Operations Section Supervisor.

1.1.11 Data and Records Coordinators

A data and records coordinator (DRC) is assigned to each of the organic analysis, spectrochemistry, and special analysis groups. The DRCs assemble the final data reports and data packages. After ensuring that the data have been reviewed and approved, the DRCs distribute and archive the data reports. The DRCs also assist the ALD QAO in maintaining and controlling all project records.

1.1.12 Instrument Custodians

Each analytical instrument in ACL is assigned to an instrument custodian. The instrument custodian is responsible for ensuring that the instrument is calibrated per ACL and project requirements, and that preventive maintenance is routinely performed and documented. The instrument custodian is also responsible for maintaining an inventory of critical spare parts to ensure proper instrument operation. The instrument custodian is responsible for training other staff members in the use of the instrument.

1.1.13 Quality Control Chemists

Quality control (QC) chemists are those scientists and technicians who are responsible for maintaining the QC laboratory. All stock standards are procured or prepared by QC chemists in the QC laboratory. The QC chemists are responsible for maintaining and documenting traceability of all stock standards and working standards. QC chemists report to the Analytical Operations Section Supervisor.

1.1.14 Computer Support Personnel

Computer support personnel are responsible for programming and maintaining the Analytical Computer System (ACS). Computer support personnel are responsible for controlling access to the system and ensuring that all system applications are verified (see Section 1.9.3). Computer support personnel report to the Analytical Operations Section Supervisor.

1.2 Program Documents

ACL facility requirements for TWCP support are implemented through this QAPjP, ACL procedures, and LMITCO procedures.

1.2.1 Key Documents

The ACL QAPjP describes the operational requirements and procedures that will be used by the ACL to satisfy TWCP objectives for organization, management, communications, data quality, and documentation. It implements the applicable requirements specified in the TWCP QAPP, the INEL SPO QAPjP, the DOE Carlsbad Area Office (DOE-CAO) quality assurance program document (QAPD) (DOE, 1996a), DOE Order 5700.6C (DOE, 1991), American Society of Mechanical Engineers (ASME) Nuclear Quality Assurance (NQA)-1 (ASME, 1994), and U.S. Environmental Protection Agency (EPA) QA/R-5 (EPA, 1994a). This QAPjP follows the guidelines and format specified in the TWCP QAPP; those sections that cover topics nonapplicable to ACL responsibilities are left blank. A cross-reference of the contents of this QAPjP with NQA-1 and DOE Order 5700.6C requirements is provided in Table 1-1. Review, approval, and control requirements for the ACL QAPjP are provided in Section 1.2.2.

Management control procedures (MCPs), ACL standard operating procedures (SOPs) and Analytical Chemistry Laboratory procedures (ACLPs) are used to provide written instruction and implementation detail for ACL operational and administrative functions defined in the ACL QAPjP. Analytical Chemistry Methods Manual (ACMM) methods are used to provide written instruction and implementation detail for the analytical functions defined in the ACL QAPjP. MCPs follow the format specified by LMITCO company requirements. ACLPs and ACMM methods follow a specific format that includes, as a minimum, a statement of the purpose and scope of the procedure and a step-by-step description of the applicable operations, administrative or analytical process. Review, approval, and control requirements for MCPs and ACL ACMM methods and ACLPs are provided in Section 1.2.2.

Table 1-1. Cross-reference between sections of ACL QAPjP, basic requirements of ASME NQA-1, and DOE Order 5700.6C criteria.

Section of ACL QAPjP	Basic Requirement of ASME NQA-1		DOE Order 5700.6C Criteria	
Front section	6	Document Control	4	Documents and Records
1. Program Management	1 2 3 4 5 6 7 9 10 14 17	Organization QA Program Design Control Procurement Document Control Instructions, Procedures & Drawings Document Control Control of Purchased Items and Services Control of Processes Inspection Inspection, Test, and Operating Status QA Reports	1 2 3 4 5 6 7 8	Program Personnel Training and Qualification Quality Improvement Documents and Records Work Processes Design Procurement Inspection and Acceptance Testing
2. Assessment and Oversight	2 15 16 18	QA Program Control of Nonconforming Items Corrective Action Audits	1 3 9 10	Program Quality Improvement Management Assessment Independent Assessment
3. Data Validation, Usability, and Reporting	3 6 9 17	Design Control Document Control Control of Processes QA Records	4 5 6	Documents and Records Work Processes Design
4. Measurement and Data Acquisition	12 14	Control of Measuring and Test Equipment Inspection, Test, and Operating Status	8	Inspection and Acceptance Testing
5. Sampling Process Design[a]	—		—	
6. Drum and Sample Handling and Custody Requirements	3 8 13	Design Control Identification and Control of Items Handling, Storage, and Shipping	5 6 8	Work Processes Design Inspection and Acceptance Testing
7. Headspace Gas Sampling[a]	—		—	
8. Sampling of Solid Processing Residues and Soils[a]	—		—	
9. Radioassay[a]	—		—	
10. Radiography[a]	—		—	
11. Hydrogen and Methane Analysis[a]	—		—	
12. Gas Volatile Organic Compound Analysis[a]	—		—	

Table 1-1. (continued).

Section of ACL QAPjP	Basic Requirement of ASME NQA-1	DOE Order 5700.6c Criteria
13. Total Volatile Organic Compound Analysis	3 Design Control 5 Instructions, Procedures, and Drawings 9 Control of Processes 10 Inspection 11 Test Control 12 Control of Measuring and Test Equipment 14 Inspection, Test, and Operating Status	5 Work Processes 6 Design 8 Inspection and Acceptance Testing
14. Total Semivolatile Organic Compound Analysis	3 Design Control 5 Instructions, Procedures, and Drawings 9 Control of Processes 10 Inspection 11 Test Control 12 Control of Measuring and Test Equipment 14 Inspection, Test, and Operating Status	5 Work Processes 6 Design 8 Inspection and Acceptance Testing
15. Total Metals Analysis	3 Design Control 5 Instruotions, Procedures, and Drawings 9 Control of Processes 10 Inspection 11 Test Control 12 Control of Measuring and Test Equipment 14 Inspection, Test, and Operating Status	5 Work Processes 6 Design 8 Inspection and Acceptance Testing
References	5 Instructions, Procedures, and Drawings	5 Work Processes

---

a. This section in not applicable to ACL activities supporting TWCP.

1.2.2 Document Review, Approval, and Control

The normal review and approval requirements followed for ACL quality documents and procedures (MCPs, SOPs, ACLPs, ACMM methods) are identified in Table 1-2. All of the requirements identified in Table 1-2 will be adhered to for applicable ACL documents. Review and approval of each document is documented on signature pages included in each document or in the files of the LMITCO Document Control Center responsible for maintaining and issuing the document.

All new or revised ACL-specific procedures are reviewed by the ALD manager, appropriate supervisors and technical leaders, and the ALD-QAO. Review by LMITCO quality, safety and environmental representatives is also obtained, as required. Final review and approval of procedures are by the ALD manager. Each ACL-controlled document contains a document assignment page that assigns the document to a named individual or office, indicates the controlled document copy number, and instructs the document assignee on how to maintain the document and enter changes.

Table 1-2. Requirements for review, approval, and control of ACL documents.

Responsible individual or office[b]	Requirement[a]						
	Original Review	Original Approval	Change Review	Change Approval	Annual Review[c]	Biennial Review[d]	Distribution
Team Leader National TRU Program	1	1	—	—	—	—	—
DOE-CAO QA	1	1	1	1	—	—	—
DOE-ID	1	1	1	1	—	—	—
SPM	1	1	1	1	1	—	1
SQAO	1	1	1	1	—	—	—
ALD Manager	1,2,3	1,2,3	1,2,3	1,2,3	—	2	2,3
ALD QAO	1,2,3,4	—	1,2,3,4	—	1	2	—
Analytical Operations Supervisor	1,2,3,4[e]	4[e]	1,2,3,4[e]	4[e]	—	2	4[e]
Functional Supervisor[f]	1,2,3,4[e]	4[e]	1,2,3,4[e]	4[e]	—	2	4[e]
Functional Technical Leader[g]	1,3,4	—	1,3,4	—	—	—	—
Method author/process owner	3,4	—	3,4	—	—	3,4	—

---

a. The numbers listed above refer to the following types of documents:

1 = ACL QAPjP
2 = ACL operational MCPs/SOPs
3 = Analytical Chemistry Methods Manual (ACMM)
4 = Analytical Chemistry Laboratory Procedures (ACLPs)

b. The following list explains the acronyms in this table:

DOE-CAO QA = DOE Carlsbad Area Office Quality Assurance Manager
DOE-ID = DOE-Idaho Operations Office
SPM = site project manager
SQAO = site QAO

c. ACL QAPjP only

d. MCPs/SOPs, ACMM, ACLPs

e. Analytical Operations Section Supervisor reviews/approves/distributes general laboratory ACLPs. Supervisors of the appropriate technical function approve those specific to their areas

f. Organic Analyses Section Supervisor or Inorganic Chemistry Section Supervisor, as appropriate

g. Organic Analysis Laboratory TL, Spectrochemistry TL, or Special Analysis TL, as appropriate.

Revisions of controlled documents are identified by a consecutive revision number and the date of the revision on the document title page and page headers within the document. All required approval signatures are included in the ACL QAPjP; approval signatures for other controlled documents are included in the procedure or on file at the responsible LMITCO Document Control Center. Each revision transmittal is assigned a sequential issue number, which indicates the number of revisions the document has undergone. A record of revisions will accompany each change transmittal to indicate the number of changes to the document. ACL SOP R.1.22, "Control of the Analytical Chemistry Methods Manual," describes the procedures for controlling the ACMM methods and manual. MCP-2001, "Control of the Analytical Chemistry Laboratory Procedures," describes procedures for controlling ACLPs. Control of MCPs is described in the LMITCO Program Description Document (PDD)-11, Records Management.

1.3 Problem Definition and Background

This section addresses the Waste Isolation Pilot Plant (WIPP) facility; it is not addressed in the ACL QAPjP because it is explained in Section 1.3 of the TWCP QAPP.

1.4 Program Description

The project responsibility of the ACL in the TWCP is the analysis of homogeneous solids and soil/gravel samples for hazardous waste characteristics. Total analyses data are generated for the target analytes listed in Table 1-3. All data generated for the TWCP program will be Quality Level III as defined in LMITCO MCP-540, *Assignment of Quality Levels*.

1.5 Data Quality Objectives

The ACL's primary function in the TWCP is to analyze homogeneous solids and soil/gravel samples for total VOCs, SVOCs and metals. These analyses results will be used by the TWCP to support DOE's WIPP program. WIPP data needs, as stated in the TWCP QAPP, are associated with four regulatory compliance programs: Performance Assessment; Land Disposal Restrictions; General Waste Analysis (Resource Conservation and Recovery Act [RCRA]); and Transportation of Radioactive Waste. In summary, applicable data quality objectives (DQOs) for homogeneous solids and soil/gravel sample analyses for the listed regulatory programs are as follows:

Performance Assessment

- No DQOs applicable to homogenous solids and soil/gravel analysis.

Land Disposal Restrictions

- To compare the upper 90-percent confidence limit (UCL_{90}) values for the mean measured contaminant concentrations in a waste stream to the specified regulatory levels (40 CFR Part 261, Subpart C); that is, to determine if a waste stream exhibits a toxicity characteristic (TC).

Table 1-3. Target analytes for homogeneous solid and soil/gravel analyses.

	Target Analytes	CAS#[a]
VOLATILE ORGANIC COMPOUNDS		
1.	Acetone	67-64-1
2.	Benzene	71-43-2
3.	Bromoform	75-25-2
4.	Butanol (1-butanol)	71-36-3
5.	Carbon disulfide	75-15-0
6.	Carbon tetrachloride	56-23-5
7.	Chlorobenzene	108-90-7
8.	Chloroform	67-66-3
9.	1,2-Dichloroethane	107-06-2
10.	1,1-Dichloroethylene (1,1-Dichoroethene)	75-35-4
11.	Ethylbenzene	100-41-4
12.	Ethyl ether	60-29-7
13.	Isobutanol	78-83-1
14.	Methanol	67-56-1
15.	Methyl ethyl ketone (2-butanone)	78-93-3
16.	Methylene chloride (dichloromethane)	75-09-2
17.	1,1,2,2-Tetrachloroethane	79-34-5
18.	Tetrachloroethylene (Tetrachloroethene)	127-18-4
19.	Toluene	108-88-3
20.	1,1,1-Trichloroethane	71-55-6
21.	1,1,2-Trichloroethane	79-00-5
22.	Trichloroethylene (Trichloroethene)	79-01-6
23.	Trichlorofluoromethane	75-69-4
24.	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon-113)	76-13-1
25.	Vinyl chloride	75-01-4
26.	m-Xylene[b]	108-38-3
27.	o-Xylene	95-47-6
28.	p-Xylene[b]	106-42-3
SEMIVOLATILE ORGANIC COMPOUNDS		
29.	2-methylphenol[c] (o-cresol)	95-48-7
30.	3-methylphenol[c,d] (m-cresol)	108-39-4
31.	4-methylphenol[c,d] (p-cresol)	106-44-5
32.	1,4-Dichlorobenzene	106-46-7
33.	ortho-Dichlorobenzene	95-50-1
34.	2,4-Dinitrophenol	51-28-5
35.	2,4-Dinitrotoluene	121-14-2
36.	Hexachlorobenzene	118-74-1
37.	Hexachloroethane	67-72-1
38.	Nitrobenzene	98-95-3
39.	Pentachlorophenol	87-86-5
40.	Pyridine	78-83-1
METALS		
41.	Antimony	7440-36-0
42.	Arsenic	7440-38-2
43.	Barium	7440-39-3
44.	Beryllium	7440-41-7
45.	Cadmium	7440-43-9
46.	Chromium	7440-47-3
47.	Lead	7439-92-1
48.	Mercury	7439-97-6
49.	Nickel	7440-02-0
50.	Selenium	7782-49-2

Table 1-3. (continued).

	Target Analytes	CAS#[a]
51.	Silver	7440-22-4
52.	Thallium	7440-28-0
53.	Vanadium	7440-62-2
54.	Zinc	7440-66-6

---

a. CAS# = Chemical Abstract Service number

b. These individual isomers are not separated during analysis and are quantitated and reported as the m-xylene/p-xylene combination

c. These analytes are identified in the TWCP QAPP as total cresols (CAS# 1319-77-3)

d. These individual isomers are not separated during analysis and are quantitated and reported as the 3-methylphenol/4-methylphenol (m/p-cresol) combination.

- To compare the UCL_{90} values for the mean measured concentrations of spent solvent VOCs to the program required quantitation limits (PRQLs); that is, to determine if a waste stream is listed for the presence of spent solvents (40 CFR Part 261, Subpart D).
- To report the average concentrations, standard deviation, UCL_{90}, and number of samples collected for hazardous constituents in a waste stream, as specified in 40 CFR 261, Appendix VIII.

General Waste Analysis

- To compared the UCL_{90} values for the mean measured contaminant concentrations in a waste stream to the specified regulatory levels (40 CFR Part 261, Subpart C); that is, to determine if a waste stream exhibits a toxicity characteristic (TC).
- To compare the UCL_{90} values for the mean measured concentrations of spent solvent VOCs to the program required quantitation limits (PRQLs); that is, to determine if a waste stream is listed for the presence of spent solvents (40 CFR Part 261, Subpart D).

Transportation of Radioactive Waste

- No DQOs applicable to homogenous solids and soil/gravel analysis.

Specific quality assurance objectives were established for each ACL analytical method to ensure that the measurement results satisfy the program data needs. These quality assurance objectives are listed in Sections 13, 14 and 15 of this QAPjP, and they address calibration, contamination, detection limits, precision, accuracy, and completeness.

1.6 Indoctrination and Training

The generation of reliable data by a laboratory requires that all analyses are conducted and reviewed by knowledgeable and trained personnel. The ACL requires a chemist, instrument operator or sample preparation specialist to successfully complete a prescribed sequence of training objectives before that individual is permitted to independently conduct any analyses, data review, or data reporting activities for TWCP. The appropriate section supervisor or technical leader shall record training accomplishments and qualifications achieved by their personnel. ACL SOP R.1.23, "Analytical Chemistry Training and Testing Program," describes the specific procedures and documentation requirements for ACL staff training, and covers program indoctrination, initial qualification and evaluation, and continuing qualification requirements. ACL staff independently conducting work in support of TWCP are also required to fulfill the minimum training and qualification requirements for TWCP analyses as specified in Table 1-4 of the TWCP QAPP. A cross-reference between ACL minimum qualifications and training for positions defined in Section 1.1 and those specified in the TWCP QAPP is provided in Table 1-4.

1.7 Records Management

1.7.1 Control of Records

Control procedures for ACL records are described in ACL SOP R.1.21, *Record Management for Analytical Chemistry*. This SOP provides instruction for control, disposition and storage of paper and electronic records. TWCP records are separated into files based on subject matter and requirements of the TWCP QAPP. The project file categories used to store ACL records for TWCP are listed in Table 1-5.

Table 1-5 also identifies which TWCP records are quality records. All quality records are stored in 2-hour rated fire-proof cabinets. Copies of all analytical records are maintained in accordance with National Enforcement Investigations Center (NEIC) guidelines (EPA 1991). Analytical records are maintained in the ACL TWCP project files in a location remote from the laboratories.

ACL records are stored in file cabinets in the ACL buildings at the Idaho Chemical Processing Plant (ICPP) within the site boundaries of the INEL. The ICPP is a controlled access facility. Access to ACL laboratories is restricted to DOE contractor employees and approved visitors. (Refer to Section 6.3 in this QAPjP for ACL facility security controls.) Access to TWCP project files is restricted to the ALD manager, the ALD QAO, the section supervisors, and the DRCs.

1.7.2 Disposition of Records

Records for the TWCP are currently unscheduled, meaning that a legal retention period has not been determined by DOE-CAO and approved by the National Archives and Records Administration (NARA). Until NARA schedules are approved, the minimum retention for all ACL TWCP quality

Table 1-4. Minimum training and qualification requirements.

QAPP position	QAPP Minimum Training and Qualifications Requirements	ACL-specific Title	ACL Minimum Training and Qualification Requirements
GC Technical Supervisor	B.S. or equivalent experience and 6 months previous applicable experience	Organic Analysis Laboratory Technical Leader	B.S. and 6 months previous applicable experience
GC Operator	B.S. or equivalent experience and 6 months previous applicable experience	Chemist	B.S. or equivalent experience and 6 months previous applicable experience; method-specific on-the-job training
GC/MS Technical Supervisor	B.S. or equivalent experience and 1 year applicable experience	Organic Analysis Laboratory Technical Leader	B.S. and 1 year previous applicable experience; 1 year independent spectral interpretation
GC/MS Operator	B.S. or equivalent experience and 1 year independent spectral interpretation or demonstrated expertise	Chemist	B.S.or equivalent experience and 1 year independent spectral interpretation or demonstrated expertise; method-specific on-the-job training
Atomic Emission Spectroscopy Technical Supervisor	B.S. and specialized training in atomic emission spectroscopy and 2 years applicable experience	Spectrochemistry Technical Leader	B.S. and specialized training in atomic emission spectroscopy and 2 years applicable experience
Atomic Emission Spectroscopy Operators	B.S. or equivalent experience and 1 year applicable experience	Chemist	B.S. or equivalent experience and 1 year applicable experience
Atomic Absorption Spectroscopy Technical Supervisor	B.S. or equivalent experience and 1 year applicable experience	Spectrochemistry Technical Leader	B.S. or equivalent experience and 1 year applicable experience
Atomic Absorption Spectroscopy Operators	B.S. or equivalent experience and 1 year applicable experience	Chemist	B.S. or equivalent experience and 1 year applicable experience
—	—	Instrument Operator	Post-high school chemistry classes or equivalent experience; specialized training in GC,GCMS, ICP-AES or Atomic Absorption/fluorescence operation; method-specific and instrument-specific on-the-job training
—	—	Sample Preparation Specialist	Post-high school chemistry classes or equivalent experience; method-specific and equipment-specific on-the-job training

Table 1-5. ACL records for the TRU Waste Characterization Program.

Project File Category	Record Type	Retention Period[a]	Quality Record (yes/no) Nonpermanent	Lifetime	Record Storage Location[b]	Record Dual Storage Location
Audits and Assessments	DOE audit plans, reports, responses, corrective actions, and final closure	Not scheduled	Yes	No	ACL, CPP-602	Not necessary
	Company assessment and plans, reports, responses, corrective actions, and final closure (internal and external to ACL)	Not scheduled	Yes	No	ACL, CPP-602	Not necessary
Equipment and Materials	Analytical instrument and spare parts lists, maintenance schedules	Not scheduled	No	No	ACL, CPP-602	Not required
	Instrument and equipment calibrations	Not scheduled	Yes	No	ACL, CPP-602	Not necessary
	Computer program verification testing records	Not scheduled	Yes	No	ACL, CPP 602	Not necessary
Forms	Program-related form templates	Non-record	No	No	ACL, CPP-602	Not required
Method Performance	PDP Data reports and raw data	Not scheduled	Yes	No	ACL, CPP-602	Not necessary
	PDP scoring reports	Not scheduled	No	No	ACL, CPP-602	Not required
	Method performance demonstration data	Not scheduled	Yes	Yes	ACL, CPP-602	Not necessary
	MDL/IDL demonstration data	Not scheduled	Yes	Yes	ACL, CPP-602	Not necessary
Presentations, Patents and Papers	Program-related presentations, patents and papers, and related documents	Not scheduled	No	No	ACL, CPP-602	Not required
Procedures (laboratory)	Quality procedures and technical implementing procedures (MCPs, SOPs, ACMM methods, ACLPs); originals and revisions	Not scheduled	Yes	No	ACL, CPP-602	Not necessary
Program Correspondence	Incoming and outgoing general correspondence, memoranda, and telephone records (segregated and filed with applicable file category)	Not scheduled	No	No	ACL, CPP-602	Not required
	QA correspondence	Not scheduled	Yes	No	ACL, CPP-602	Not necessary
Program Requirements Documents	ACL QAPjP original and revisions	Not scheduled	Yes	No	ACL, CPP-602	Not necessary
Personnel Qualification and Training	Personnel assignments, resumes and signature lists	Not scheduled	No	No	ACL, CPP-602	Not required
	Staff QA training records and method training records	Not scheduled	Yes	No	ACL, CPP-602	Not necessary

Table 1-5. (continued).

Project File Category	Record Type	Retention Period[a]	Quality Record (yes or No) Nonpermanent	Lifetime	Record Storage Location[b]	Record Dual Storage Location
Quality	Audit and assessment corrective actions	Not scheduled	Yes	No	ACL, CPP-602	Not necessary
	Nonconformance reports and process deficiency resolution reports	Not scheduled	Yes	No	ACL, CPP-602	Not necessary
	Operational variances	Not scheduled	Yes	No	ACL, CPP-602	Not necessary
	Quality assurance reports to management	Not scheduled	Yes	No	ACL, CPP-602	Not necessary
	Quality improvement documents and records	Not scheduled	Yes	No	ACL, CPP-602	Not necessary
Requisitions	Program-related requisitions and associated documentation	Not scheduled	No	No	ACL, CPP-602	Not required
Safety and Health	Hazard analyses	Not scheduled	No	No	ACL, CPP-602	Not required
Sample Tracking	Shipping records, sample disposal records	Not scheduled	No	No	ACL, CPP-602	Not required
	Field COC	Not scheduled	No	Yes	ACL, CPP-602	SPO, TSB
	Internal (laboratory) COC	Not scheduled	No	Yes	ACL, CPP-602	Not necessary
Waste Disposal	Laboratory waste stream documentation	Not scheduled	No	No	ACL, CPP-602	Not required
Work Scope	Reference and background documentation	Non-record	No	No	ACL, CPP-602	Not required
	Work releases, work scopes, cost accounting, status reports	Not scheduled	No	No	ACL, CPP-602	Not required
Logbooks	Raw analytical data	Not scheduled	Yes	No	ACL, CPP-602	Not necessary
Data Reports	Summary data reports (include sample results, COC, and review/validation documentation)	Not scheduled	No	Yes	ACL, CPP-602	SPO, TBS
	Data package files (raw analytical data)	Not scheduled	Yes	No	ACL, CPP-602	Not necessary

---

a. No TWCP records schedule has been issued by DOE-CAO and approved by NARA. Until NARA schedules are approved, minimum retention for all ACL TWCP QA records is life of the TWCP project at the INEL plus 6 years.

b. ACL TWCP project files are maintained in fire-rated filing cabinets. Therefore, dual record storage is not necessary.

SPO = INEL TWCP Site Project Office.

records is life of the TWCP project at INEL plus 6 years. ACL TWCP records will be transmitted to the INEL project manager as requested.

1.7.3 Requests for QA Records

Access to all TWCP QA records is limited to the ALD manager, the section supervisors, the ALD QAO, and the DRCs. All requests for QA records must be directed to one of these individuals. Original documents shall not be taken from the file storage area without permission from one of the listed individuals, and copying and distribution of such documents must have their authorization.

1.8 Procurement

The ACL's procedures for procurement of materials, equipment, and services are described in the LMITCO company procedure MCP-592, *Acquisition of Goods and Services*, which describes the practices for preparation and review of procurement requests, source verification including vendor QA, ordering, receiving, inspection and testing, evaluation, record keeping, and acceptance or rejection of materials or service.

1.9 Work Processes

1.9.1 Control of Processes

All aspects of ACL operations are controlled by the key documents listed in Section 1.2.1; the ACL TWCP QAPjP, MCPs/SOPs, ACLPs, and ACMM methods. Company and ACL MCPs, SOPs, and ACLPs document the implementing procedures for the operation and administrative activities and requirements specified in the ACL TWCP QAPjP. ACL SOPs and MCPs are similar in nature and serve the same function; SOPs (R.x.x) are those procedures originated under previous contractors that have not yet been reissued in LMITCO format (MCPs). ACMM methods document the implementing procedures for the analytical activities and requirements specified in the ACL TWCP QAPjP. The individual ACL MCPs/SOPs, ACLPs, and ACMM methods involved in this project are listed in Table 1-6. MCP-2001, "Control of the Analytical Chemistry Laboratory Procedures," and ACL SOP R.1.22, "Control of the Analytical Methods Manual," specify the format, content, and preparation requirements for ACL ACLPs and ACMM methods, respectively. The format and content of MCPs is specified in LMITCO STD-1, "Procedure Writing Standard."

Analytical methods in the ACMM are the key guidance documents for sample analysis activities at the ACL. ACMM methods use the following general format:

- Title
- Abstract
- Applicability
- Discussion

Table 1-6. ACL procedures used in support of TWCP.

Type of Procedure	Procedure Number	Title
LMITCO MCP	MCP-4	Business Assessment
	MCP-592	Acquisition of Goods and Services
	MCP-538	Control of Nonconforming Items
	MCP-598	Process Deficiency Resolution
ACL MCP	MCP-2002	Analytical Chemistry Chain of Custody
	MCP-2001	Control of the Analytical Chemistry Laboratory Procedures
	MCP-2004	Waste Disposal at the Analytical Chemistry Laboratory
SOP	R.1.20	Analytical Chemistry Quality Control Program
	R.1.21	Record Management for Analytical Chemistry
	R.1.22	Control of the Analytical Chemistry Methods Manual
	R.1.23	Analytical Chemistry Training and Testing Program
	R.1.24	The Management of Samples in Analytical Chemistry
	R.1.29	Software Control in the Analytical Chemistry Section
	R.1.47	Logs and Roundsheets
ACLP	0.02	Method of Standard Additions
	0.27	Refrigerated Storage Cabinet Control Procedure
	1.01	Preparation of Quality Control Reagents and Standards
	2.012	Glassware Cleaning for Environmental Samples
	2.05	Control, Distribution and Use of Spectrochemical Standards
	2.06	Pipette Calibration Verification Procedures for Spectrochemistry
	2.25	Warm ICP Radiological Controls and Routine Maintenance

Table 1-6. (continued).

Type of Procedure	Procedure Number	Title
ACMM	2350	Determination of Arsenic and Selenium in Environmental Samples Using Graphite Furnace Atomic Absorption Spectrometry
	2900	Determination of Trace Metals in Environmental Samples by ICP Emission Spectrometry
	7801	Determination of Mercury by Cold-Vapor Atomic Absorption Spectrophotometry
	7802	Determination of Mercury by Cold-Vapor Fluorescence Spectrophotometry
	8909	Microwave Assisted Digestion of Homogeneous Solids and Soil/Gravel
	8969	Determination of Percent Solids
	9260	Volatile Organic Compounds by Gas Chromatography Mass Spectrometry (GC/MS): Capillary Column Technique
	9261	Determination of Total Volatile Organic Compounds in Homogeneous Solids and Soil/Gravel by Gas Chromatography/Mass Spectrometry (GC/MS)
	9271	Determination of Semivolatile Organic Compounds in TRU Waste Characterization Samples
	9441	Determination of Nonhalogenated Volatile Organic Compounds by Gas Chromatography/Flame Ionization Detector (GC/FID)
	9501	Sample Preparation of TRU Waste Characterization Samples for Organic Analysis

- Safety Precautions
- Apparatus and Reagents
- Procedure
- Calculations
- References

- Supplemental Information
- Signature/approval block.

Calibration procedures and quality control (QC) requirements and specifications are addressed in subsections of the procedure section.

ACLPs are the key ACL guidance documents for laboratory operational procedures (e.g., glassware cleaning, refrigerator temperature monitoring) that do not directly involve the analysis of samples. ACLPs use the following format:

- Title
- Purpose
- Scope
- Definitions
- Procedures
- References
- Approvals.

MCPs are the key guidance documents for other operational and administrative tasks at ACL. MCPs are written in the following LMITCO-mandated format:

- Introduction
    - Purpose
    - Scope and Applicability
- Precautions and Limitations
- Prerequisites
- Instructions
- Records
- Source Requirements
- Appendices.

The ACL uses several means of communication to ensure that the laboratory is successful in supporting all pertinent TWCP requirements. Within the laboratory, implementation and routine operational requirements shall be communicated to applicable staff through distribution of this QAPjP and through written procedures. All of these documents are controlled (see Section 1.2.2) and are issued to ACL staff on an individual basis, depending on staff assignment, task responsibilities, and work location. Changes in project requirements shall be implemented by revision to this QAPjP and applicable procedures, and the revisions shall be distributed to individuals assigned controlled copies of documents.

Laboratory staff shall be kept informed of the laboratory's performance and commitments (e.g., sample analysis schedule, assessments, audits and visits) to the project through distribution of SPO project status reports and quarterly QA reports (see Section 2.6). Any special occurrences that require specific activities by laboratory staff shall be communicated to those involved by the ALD manager or appropriate section supervisor, and all such communications shall be documented with a copy stored in the project files. The ALD manager holds regularly-scheduled staff meetings with supervisors, technical leaders (as necessary) and the ALD QAO to ensure that appropriate inter-departmental communication occurs.

Any occurrences (e.g., equipment failure) that impact the schedule of routine deliverables or laboratory capabilities to perform project duties shall be immediately communicated by the ALD manager or appropriate section supervisor to the SPM, SQAO, and ALD QAO. These communications shall be documented on paper (e.g., recorded in a memo of conversation or a FAX), and a copy shall be placed in the project files. Special occurrences and their impact on project activities shall also be documented and summarized in the quarterly QA report.

1.9.2 Identification and Control of Items

Identification and control of items and materials begins with the procurement process. LMITCO company procedures (see Section 1.8) describe the processes for source verification, ordering, receiving, inspection and testing, record keeping, and if necessary, return to source. Nonconforming items are identified and controlled as described in LMITCO MCP-538, *Control of Nonconforming Items*, and MCP-598, *Process Deficiency Resolution*. Items and materials once installed or in use are controlled in accordance with detailed analysis method procedures for the applicable analysis systems (see Sections 4, 13, 14, and 15). Samples, standards, and waste are identified and controlled as described in applicable ACL ACMM methods, MCPs/SOPs, and ACLPs (see Sections 6, 13, 14, and 15).

1.9.3 Computer Hardware and Software

The Analytical Computer System (ACS) is used for information and data management at ACL. All computer hardware used to support TWCP is tested before use and is not used for program work if it does not satisfy manufacturer specifications and program requirements. Computer software and hardware/software configurations used, acquired or developed to support TWCP are verified, validated, tested and the test results documented as described in ACL SOP R.1.29., "Software Control in the Analytical Chemistry Section," which implements QAPD and NQA-1, Part II, Subpart 2.7 requirements. A complete inventory list of computer software and hardware/software configurations used for TWCP data processing is maintained in ACL TWCP project files.

2. ASSESSMENT AND OVERSIGHT

2.1 Audits

The ACL shall participate in and conduct both external audits and internal assessments of its management and analytical systems to verify compliance with all aspects of the TWCP QAPP, DOE-CAO QAPD, INEL SPO QAPjP and ACL QAPjP. The objectives of these audits are to verify compliance with the TWCP QA/QC requirements and to determine the effectiveness of the ACL quality program.

2.2 Corrective Actions

In compliance with ASME NQA-1, Element 16, conditions adverse to the QA/QC requirements specified in this document shall be promptly identified and corrected as soon as practical. The cause of any adverse condition that affects compliance with the QA/QC requirements shall be determined and action shall be taken to preclude its recurrence. The identification, cause, and CAs for conditions that do not comply with the quality requirements for this project must be documented and reported to appropriate levels of management as indicated throughout this section.

The ACL corrective action system is part of the LMITCO system as described in MCP-538, *Control of Nonconforming Items* and MCP-598, *Process Deficiency Resolution*, and is based upon detecting and correcting deficiencies before they become serious enough to cause data loss or the release of erroneous data. There are three general categories of CA:

- Immediate (i.e., in-process) action to correct or repair nonconforming data or equipment
- Long-term action to eliminate causes of nonconformance and to take measures to preclude recurrence
- Operational variances that are approved and controlled changes to approved plans and procedures.

Immediate CA is the process of correcting nonconforming equipment or analysis activities at the time of analysis before reportable data are generated. This type of CA is the responsibility of the person performing the procedure and is documented in log books or on raw data. A nonconformance report (NCR) is not required unless the problem impacts reported data.

2.2.1 Nonconformances

Nonconformances are uncontrolled and unapproved deviations from an approved plan, procedure, or expected result. Nonconforming items and activities are those that do not meet the project requirements, procurement document criteria, or approved work procedures. In compliance with ASME NQA-1, Element 15, nonconforming items shall be identified and segregated, and the affected organization(s) shall be notified.

Nonconformances will normally be identified by internal assessments or external audits, customer complaints, equipment malfunction, performance evaluation studies, data quality assessments, or any activities involved in analysis or data reporting for the TWCP. When a nonconforming item is discovered or suspected, the occurrence shall be reported immediately and documented as described in MCP-538, using an NCR like the one in Figure 2-1. All ACL personnel engaged in TWCP support shall be responsible for detecting and reporting nonconformances. All NCRs shall be submitted to the quality engineer (QE) assigned to the SPO for dispositioning and tracking. The QE makes the determination to proceed with the NCR or to initiate the Process Deficiency Resolution (PDR) process described in MCP-598. The ALD QAO serves as the laboratory point-of-contact for the NCR/PDR process.

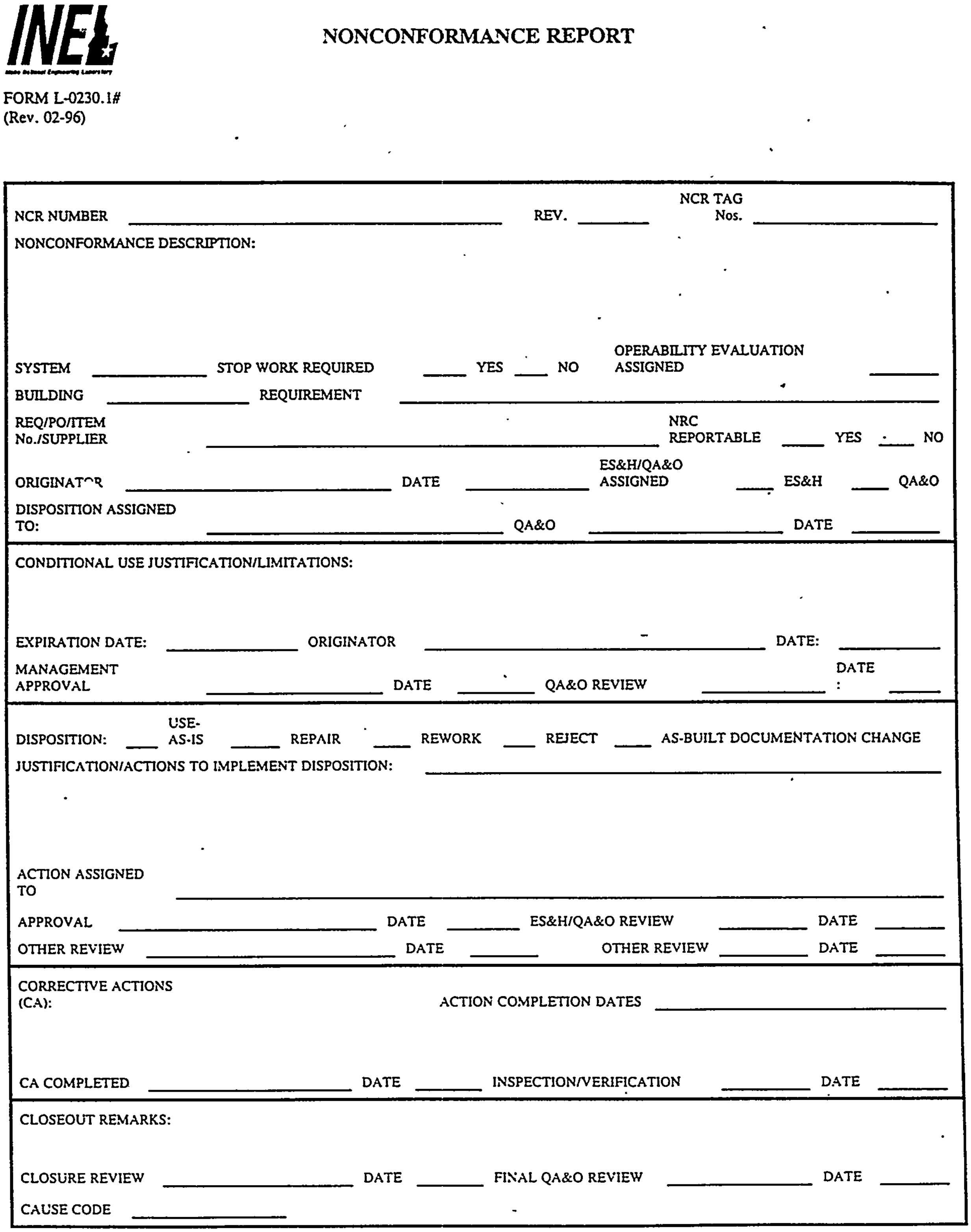
Each NCR shall contain, as a minimum, the following information:

- NCR number
- Identification of the individual(s) originating the NCR
- Description of the nonconformance
- Disposition of the nonconformance
- Cause of the nonconformance (if known)
- Action required (if any) to prevent recurrence
- Schedule for completing the CA (if required)
- An indication of the potential ramifications and overall useability of the data, if applicable.
- Any required approval signatures.

Nonconformances and process deficiencies shall be reported to the SQAO in accordance with the INEL SPO QAPjP. This will be accomplished by sending copies of the initiated and completed NCR/PDR to the SQAO.

In cooperation with the SPO QE, the ALD QAO or designee will determine if the nonconformance requires CA by ACL. As a minimum, nonconformances that are significantly adverse to quality or that are recurring are subject to CA. If a nonconformance requires CA, the responsible individual (usually the appropriate technical leader or section supervisor) institutes and schedules CA procedures, including identification of remedial and investigative actions, root causes, and actions to preclude recurrence.

The ALD QAO will inform the SPO QE and the SQAO of the status of corrective actions (i.e., completion and whether or not the CA was satisfactory).



INEL

NONCONFORMANCE REPORT

FORM L-0230.1#
(Rev. 02-96)

NCR NUMBER ____ REV. ____ NCR TAG Nos. ____

NONCONFORMANCE DESCRIPTION:

SYSTEM ____ STOP WORK REQUIRED ____ YES ____ NO OPERABILITY EVALUATION ASSIGNED ____

BUILDING ____ REQUIREMENT ____

REQ/PO/ITEM No./SUPPLIER ____ NRC REPORTABLE ____ YES ____ NO

ORIGINATOR ____ DATE ____ ES&H/QA&O ASSIGNED ____ ES&H ____ QA&O

DISPOSITION ASSIGNED TO: ____ QA&O ____ DATE ____

CONDITIONAL USE JUSTIFICATION/LIMITATIONS:

EXPIRATION DATE: ____ ORIGINATOR ____ DATE: ____

MANAGEMENT APPROVAL ____ DATE ____ QA&O REVIEW ____ DATE: ____

DISPOSITION: ____ USE-AS-IS ____ REPAIR ____ REWORK ____ REJECT ____ AS-BUILT DOCUMENTATION CHANGE

JUSTIFICATION/ACTIONS TO IMPLEMENT DISPOSITION: ____

ACTION ASSIGNED TO ____

APPROVAL ____ DATE ____ ES&H/QA&O REVIEW ____ DATE ____

OTHER REVIEW ____ DATE ____ OTHER REVIEW ____ DATE ____

CORRECTIVE ACTIONS (CA): ACTION COMPLETION DATES ____

CA COMPLETED ____ DATE ____ INSPECTION/VERIFICATION ____ DATE ____

CLOSEOUT REMARKS:

CLOSURE REVIEW ____ DATE ____ FINAL QA&O REVIEW ____ DATE ____

CAUSE CODE ____

Figure 2-1. Example of a nonconformance report.

2.2.2 Operational Variances

An operational variance is an approved and controlled change to TWCP-related plans or procedures. The need for a variance is caused by the identification of improvement opportunities or unusual or nonroutine occurrences that affect operations, but not the ability to achieve the performance standards or quality requirements specified in the TWCP QAPP or TWCP QAPjPs.

When a need to deviate from established procedures is identified, it is the responsibility of the person performing the work to initiate a variance. When a variance is required, the person identifying the need for the variation completes a record of variance (Figure 2-2), which is then approved by the ALD manager and the ALD QAO. The approved record of variance must be submitted to the SPM and SQAO for review. INEL will obtain DOE-CAO concurrence with any variance that may impact the overall quality of reported data. A record of variance must be completed and approved before initiation of the activity to document the variation from normal, approved procedures. An anticipated close-out action is assigned for each variance. The ALD QAO monitors the status of active variances, closes them when close-out actions have been completed and submits the closed variance to the SQAO.

2.3 Quality Improvement

The ACL TWCP project staff meets periodically (as needed) as a quality improvement team to continually assess project work processes and safety conditions, identify needed improvements, assign responsibilities for making improvements, and monitor progress on improvement actions. All ACL staff are responsible for notifying appropriate supervisors and TLs when they identify needed revisions or improvements to procedures and methods.

2.3.1 Trend Analysis

The ACL uses trend analysis to monitor its analytical systems and associated activities. The goals of the trend analysis are as follows:

- To detect quality problems before they become significantly adverse to the quality of the products
- To allow timely initiation of CAs to prevent development of significant quality problems
- To ensure continuous quality improvement.

Control charting (see Section 2.3.2) and frequency histograms are the main techniques ACL uses to conduct trend analysis; among the parameters charted are NCR/PDR characteristics, audit/assessment results, and QC measurement results.

RECORD OF VARIANCE

FACILITY____________________ VARIANCE NO.____________

SUBJECT ACTIVITY:

ITEM(S) AFFECTED: ITEM I.D. #:

PROCEDURE: (check all that apply)

__ Facility QAPjP. Document #__________________. Rev._____. Section(s)______________________
__ Facility Procedure. Number__________________. Rev._____. Step/Section(s)__________________
__ Other____

DESCRIPTION OF VARIANCE:

REASON FOR THE VARIANCE:

SPECIAL EQUIPMENT OR PERSONNEL REQUIRED:

FACILITIES AFFECTED: ____SWEPP ____ECL ____SPO ____ORNL ____ANL-W ____ICPP/ACL

ANTICIPATED CLOSE-OUT ACTION:

DATE:

INITIATOR:____________________ DATE:____________

APPROVALS

Facility Manager	Date	Facility QA Officer	Date

REVIEWS

Site Project Manager	Date	Site QA Officer	Date

CLOSE-OUT

COMMENTS:

Facility QA Officer	Date	Site QA Officer	Date

Figure 2-2. Example of a TWCP record of variance.

2.3.2 Control Charts

Control charts are used by the ACL to monitor trends in analytical performance. A control chart consists of a graph with the vertical axis labeled in units of the analysis or parameter of interest and the horizontal axis labeled in units of time or sequence of results. The upper and lower warning and action control limits, which are statistically determined, may be used as criteria for instituting CA as described in Section 2.2. When the parameter being plotted is the relative percent difference (RPD), range, or relative standard deviation (RSD), the lower limits do not apply (i.e., the minimum value of the RPD, range, or RSD plotted is always zero and the limits plotted are upper limits).

ACL maintains control charts for each analytical method performed in support of TWCP. At a minimum, laboratory control sample (LCS) analyte recoveries are charted for each method for representative analytes. QC measurement data are charted against program QA specifications (see Tables 13-1, 14-1 and 15-1) and against statistical process control limits. The statistical process control limits are used as quality improvement tools only, i.e., an NCR is not issued when they are exceeded unless the TWCP QA specifications are also exceeded for reported data. An example of an ACL control chart is presented in Figure 2-3.

2.4 Management Assessments

The ACL is subject to management assessment activities as described in LMITCO MCP-4, *Business Assessment*. Under this procedure, a business process assessment plan is developed to focus on key areas such as:

- Mission and strategic objectives of the organization
- Employees' role in the organization
- Customer's expectations and degree to which expectations are being met
- Opportunities for improving quality and cost-effectiveness
- Recognition and enhancement of human resource capabilities.

The purpose of the business process assessment is to evaluate, correct, and prevent management problems that might hinder ALD's ability to comply with customer program requirements. These business assessments are performed on a regularly-scheduled basis and the results are used as input to the quality improvement process. Results of business assessments are documented and archived in the project files (see Table 1-5).

2.5 Independent Assessments

The ALD QAO, INEL SPO, or other independent parties shall perform independent assessments in accordance with DOE Order 5700.6C, the SPO TWCP QAPjP, and the TWCP QAPP. These

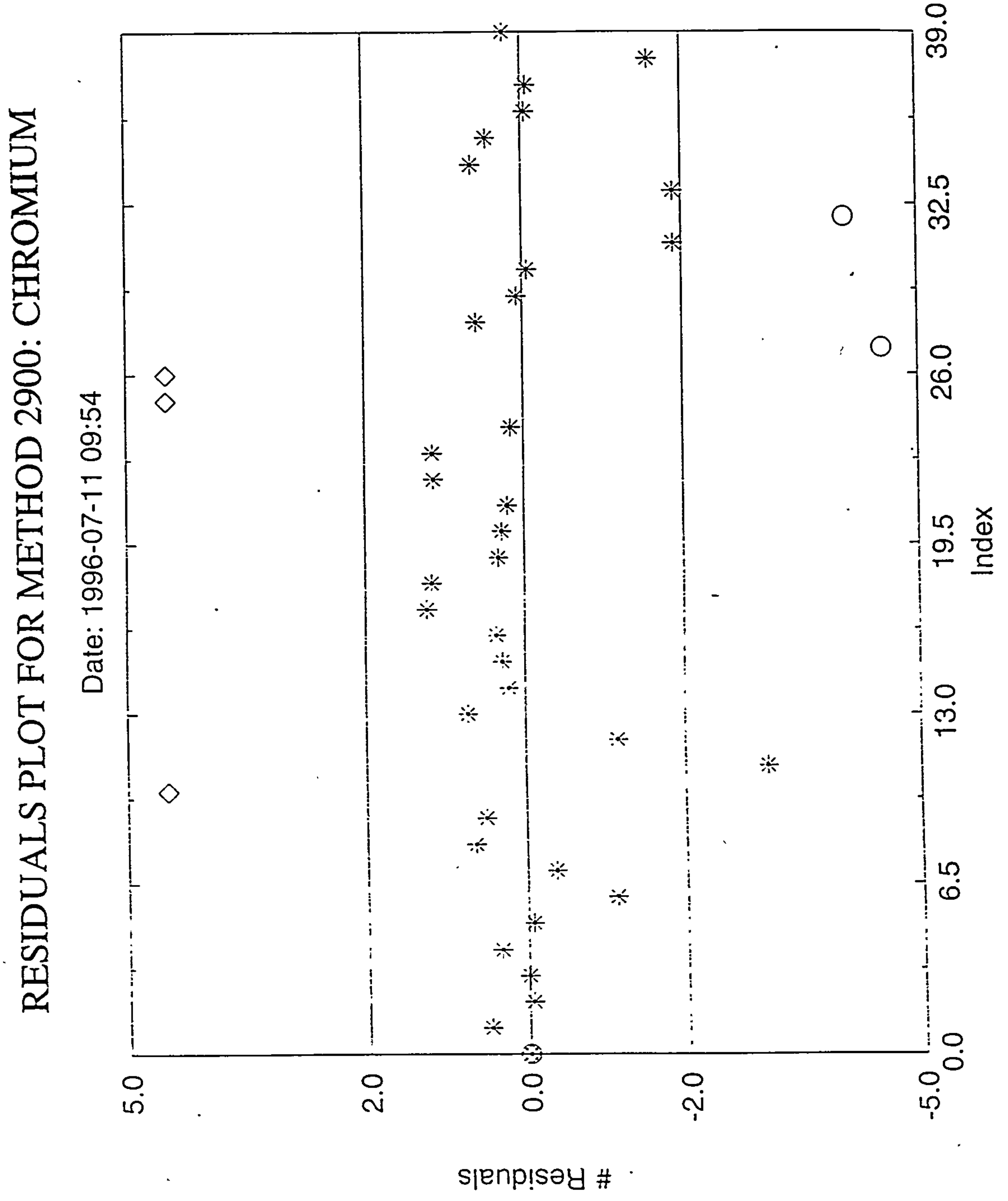


Figure 2-3. Example of ACL control chart.

independent assessments shall be performed on a regularly-scheduled basis for the duration of ACL support to TWCP and shall focus on improving data product quality and process effectiveness. Results of the independent assessments are documented and archived in the project files (see Table 1-5).

Independent assessments by the ALD QAO shall be scheduled so that they occur with sufficient frequency to cover quality program requirements. The intent is that several independent assessments will be conducted throughout the fiscal year, evaluating only a portion of the entire program requirements with each assessment. The scope and detail of the assessments will be consistent with the status and importance of the activity.

At the beginning of each fiscal year, the ALD QAO will prepare an independent assessment schedule describing the activities and tasks to be evaluated and the date(s) of the evaluation. The assessment schedule and scope will be reviewed periodically and revised as necessary to ensure that coverage is complete. As a minimum, at least one independent assessment must be conducted annually by the ALD QAO. If necessary, in response to unusual circumstances, nonconformances, or quality problems, scheduled assessments shall be supplemented by additional assessments of specific subjects.

Upon request from the SPO, the ACL will also participate in independent assessments conducted by organizations external to the Analytical Laboratories Department.

2.6 QA Reports to Management

The purpose of quality assurance reports to project management is to appraise the SPM and SQAO of ACL analytical support and QA activities. Quality assurance reports are prepared by the ALD QAO on a quarterly basis, and include, at a minimum, the following information:

- Status of Data Quality Objectives (e.g., accuracy, precision, completeness) and any associated limitations on data usage
- Identification of any significant or recurring QA/QC problems, recommended solutions, and corrective actions
- Status of all nonconformance reports, process deficiency resolution reports, and operational variances
- Results of any audits, assessments or surveillances conducted during the period
- Status of performance demonstration program participation and results
- Status of method (procedure) performance demonstrations

- Any changes to the ACL QAPjP or other controlled documents and procedures related to TWCP activities.

2.7 Performance Demonstration Program

To demonstrate and document performance characteristics for analytical methods, the ACL shall participate in the Performance Demonstration Program (PDP) described in the TWCP QAPP. The PDP Plan for RCRA Constituent Analysis of Simulated Wastes (DOE 1996c) is administered by the NTP Waste Characterization manager at DOE-CAO.

The PDP is an evaluation of the participating laboratories' ability to conduct the analysis of homogeneous solids and soils/gravels for total VOCs, SVOCs, and metals using approved methods. The evaluation is conducted through the semiannual analysis of single-blind performance samples (i.e., analytes and concentrations are unknown to the laboratory). The PDP samples are analyzed using the same methods and under the same conditions of radioactivity confinement that ACL anticipates using for the analysis of TWCP samples. Only methods used in the PDP will be used to support the analysis of TWCP samples.

If specified criteria for performance are not achieved, problems must be identified, and CA as described in Section 2.2 must be instigated.

2.8 Procedure Performance Demonstration

A demonstration of laboratory precision, accuracy and achievable detection limits is required by Sections 13.3, 14.3 and 15.3 of the TWCP QAPP. This demonstration shall be made by replicate analysis of standards of known concentration, as described in Sections 13.3.1, 14.3.1 and 15.3.1 of this QAPjP.

3. DATA VALIDATION, USABILITY, AND REPORTING

All processes at ACL (i.e., sample receiving and handling, sample analysis, data reduction, data reporting, and data review) are subject to examination to evaluate adherence to project specifications. This examination consists of several layers of technical and QA review. These reviews ensure that all data released by ACL have received scrutiny from qualified independent reviewers and are scientifically sound, appropriate to the method, and completely documented.

All data shall receive analyst review, independent technical review, technical supervisory review, and QAO review. The review processes shall be appropriately documented before data are released from the laboratory.

3.1 Data Review, Validation, and Verification Requirements

Data review ensures that raw data are properly collected, reduced, and reported. Data validation confirms that the data satisfy the requirements defined by the TWCP QAPP and are accompanied by signature release. Data verification authenticates that the data are in fact that which is claimed. Specific personnel responsibilities for these three related functions are determined by job function and training qualifications.

The following minimum data recording requirements for analyses supporting TWCP must be satisfied by the ACL analysis and review process:

- All original data must be signed and dated in black ink by the person generating it.
- All data must be recorded clearly and accurately in laboratory records, bench sheets, or logbooks and include applicable sample identification numbers.
- All changes and additions to original data must be lined out; initialed, and dated by the individual making the change. An explanation of the change or addition must be included if the change or addition deals with rejecting data for use.
- All data must be transferred and reduced from logbooks and laboratory records completely and accurately.
- All laboratory records shall be maintained in permanent files according to NEIC guidelines.
- Data shall be organized into standard formats as outlined in Sections 13.6, 14.6 and 15.6 of this QAPjP.
- All electronic data shall be stored appropriately to ensure that sample and QC data are readily retrievable. Corrections made to hardcopy data must also be made in electronic data files whenever possible.

3.1.1 Data Generator Review

Personnel conducting analyses (i.e., the data generators) are responsible for conducting real-time review and verification of 100% of the data resulting from their activities. This review is documented by the data generator's signature on the first page of each set of raw data. Data generators, either chemists, instrument operators or sample preparation specialists, are accountable for ensuring that all data they generate in support of TWCP are complete, accurate, and compliant with ACL and TWCP requirements. Data generators are responsible for performing all data reduction required prior to independent technical review and reporting, and for notifying the appropriate technical leader or ALD QAO of any problems encountered during analysis and data review that potentially impact data quality.

3.1.2 Independent Technical Review

One hundred percent of the data must receive independent technical review. The reviewer(s) must be a qualified individual other than the data generator (i.e, the appropriate technical leader). The independent technical reviewer must meet the minimum training and qualifications requirements for chemists as defined by Table 1-4, i.e., individuals not qualified to perform independent sample analysis and data interpretation cannot perform independent technical review. The reviewer(s) must signature release the data and, as a consequence, ensure that:

- Data generation and reduction were conducted in a technically correct manner in accordance with the methods used
- Data are reported in the proper units and correct number of significant figures
- Calculations have been verified by a valid calculation program, a spot check of verified calculation programs, or 100% check of all hand calculations
- All deviations from an accepted method and the rationale for the variations have been documented and approved
- The data have been reviewed for transcription errors
- The analytical data documentation is complete and includes raw data, calculation records, calibration records, and QC sample results
- QC sample results are within established program specification limits, and if not, the data have been appropriately qualified
- Analytical sample holding times have been met, or exceptions have been documented.

Independent technical review is required before any data are approved for release. The independent technical review process is documented with a signed checklist (see Figures 13-1, 13-2, 14-1 15-1, 15-2 and 15-3 for method-specific examples). The checklist is archived in the associated data package, and a copy is included in the data reports (see Sections 13.6, 14.6 and 15.6).

If the technical leader is not the analyst or independent technical reviewer, the technical leader then reviews the data forms for accuracy, consistency, and fulfillment of QC criteria. The TL approves the data by initialing and dating it, and also indicates if explanatory information will be accompanying the final report (e.g., comment in narrative required).

3.1.3 Technical Supervisory Review

One hundred percent of the data must receive technical leader or supervisory review. This review must ensure that:

- The data are technically reasonable based on the technique used
- All data have received independent technical review
- The analytical data documentation (i.e., the data package) is complete and includes raw data, data forms, calculation records, QC sample results, narrative comments and COC forms
- Analytical sample holding times were met, or exceptions were documented.

Technical supervisory review must occur after the data have been entered on reporting forms, the data report has been assembled for release, and the data package is ready for archival, and must be completed before the technical supervisor (i.e., technical leader or section supervisor) gives signature release for the data. This review is documented by signature on the data report cover page.

3.1.4 Quality Assurance Officer Review

One hundred percent of the data require review and sign-off by the ALD QAO before release. This review does not technically validate the data, but rather serves as an overall quality evaluation. This review must ensure that:

- Independent technical and technical supervisory reviews have been performed as evidenced by the appropriate signature releases.
- The analytical QC documentation is complete.
- Laboratory QC checks [e.g., laboratory blanks (LBs), LCSs, matrix spikes] have been properly performed. QC criteria that were not met are documented according to requirements of the Sections 2.2.1, 13.3, 14.3 and 15.3 of this QAPjP.
- QA objectives have been met according to the methods outlined in Section 3.2 of this QAPjP.

The review is completed with the aid of method-specific checklists (see examples in Figures 3-1, 3-2, 3-3, and 3-4), which list all program quality requirements pertinent to specific laboratory analyses.

QUALITY ASSURANCE DATA REVIEW CHECKLIST for TWCP METHOD 430.4 (TOTAL PURGEABLE VOCs)
LITCO Analytical Chemistry Laboratory

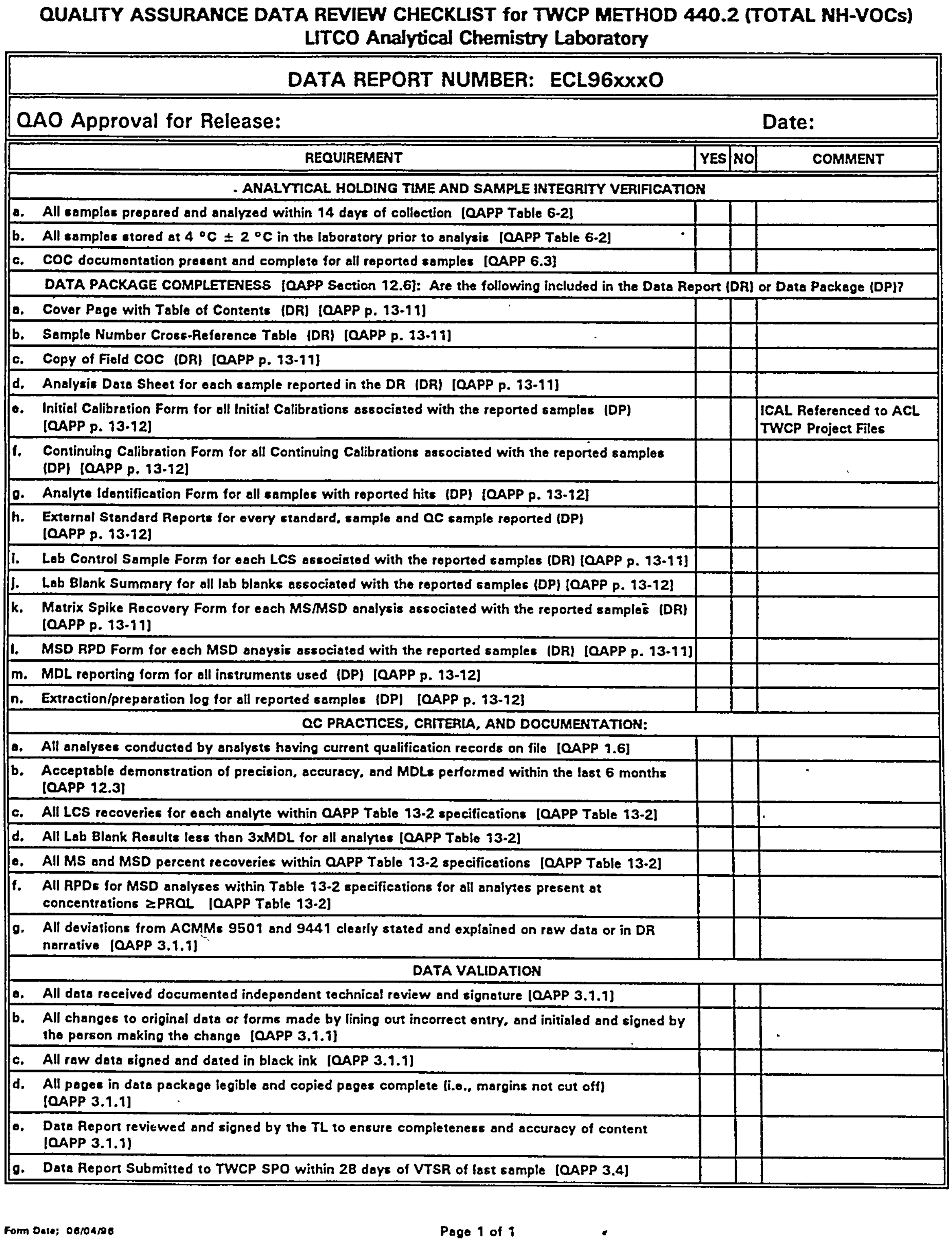
DATA REPORT NUMBER: ACL96xxx0

QAO Approval for Release: Date:

	REQUIREMENT	YES	NO	COMMENT
	ANALYTICAL HOLDING TIME AND SAMPLE INTEGRITY VERIFICATION			
a.	All samples prepared within 14 days and analyzed within 40 days of collection (methanol extracts) or prepared and analyzed within 14 days of collection (non-extracted) [QAPP Table 6-2]			
b.	All reported samples stored at 4 °C ± 2°C in the laboratory prior to analysis [QAPP Table 6-2]			
c.	COC documentation present and complete for all reported samples [QAPP 6.3]			
	DATA PACKAGE COMPLETENESS [QAPP Section 13.6]: Are the following included in the Data Report (DR) or Data Package (DP)?			
a.	Cover Page with Table of Contents (DR) [QAPP p. 13-11]			
b.	Sample Number Cross-Reference Table (DR) [QAPP p. 13-11]			
c.	Copy of Field COC (DR) [QAPP p. 13-11]			
d.	Analysis Data Sheet for each sample in the DR, TICs when appropriate (DR) [QAPP p. 13-11]			
e.	BFB Tune Report for each Initial Calibration and Continuing Calibration associated with the reported samples (DP) [QAPP p. 13-12]			ICAL BFB Tune Referenced to ACL TWCP Project Files
f.	Initial Calibration Form for all Initial Calibrations associated with the reported samples (DP) [QAPP p. 13-12]			ICAL Referenced to ACL TWCP Project Files
g.	Continuing Calibration Form for each Continuing Calibration associated with the reported samples (DP) [QAPP p. 13-12]			
h.	Internal Standard Area Summary Form for daily calibration and associated sample analyses (DP) [QAPP p. 13-12]			
i.	Lab Control Sample Form for each LCS associated with the reported samples (DR) [QAPP p. 13-11]			
j.	Lab Blank Summary for all lab blanks associated with the reported samples (DP) [QAPP p. 13-12]			
k.	Matrix Spike Recovery Form for each MS/MSD associated with the reported samples (DR) [QAPP p. 13-11]			
l.	MSD RPD Form for each MSD analysis associated with the reported samples (DR) [QAPP p. 13-11]			
m.	Surrogate recovery form(s) including all reported samples (DP) [QAPP p. 13-11]			
n.	MDL reporting form for all instruments used [QAPP, p. 13-12]			
o.	Quantitation Reports for every standard, sample, and QC sample reported (DP) [QAPP p. 13-12]			
p.	Mass Spectra for all reported TICs (DP) [QAPP p. 13-12]			
q.	Extraction/preparation log for all extracted samples [QAPP p. 13-12]			
	QC PRACTICES, CRITERIA, AND DOCUMENTATION:			
a.	All analyses conducted by analysts having current qualification records on file [QAPP 1.6]			
b.	Acceptable demonstration of precision, accuracy, and MDLs performed within the last 6 months [QAPP 13.3]			
c.	All LCS recoveries for each analyte within QAPP Table 13-2 specifications [QAPP Table 13-2]			
d.	All Lab Blank Results less than 3xMDL for all analytes [QAPP Table 13-2]			
e.	All MS and MSD percent recoveries within QAPP Table 13-2 specifications [QAPP Table 13-2]			
f.	All RPDs for MSD analyses within Table 13-2 specifications for all analytes present at concentrations ≥PRQL [QAPP Table 13-2]			
g.	All surrogate recoveries within Table 13-2 specifications [QAPP Table 13-2]			
h.	All deviations from ACMMs 9501, 9260, and 9261 clearly stated and explained on raw data or in DR narrative [QAPP 3.1.1]			
i.	All noncompliant QC (cause and data impact) discussed in DR narrative, required NCRs issued [QAPP p. 13-11]			
	DATA VALIDATION			
a.	All data received documented independent technical review and signature [QAPP 3.1.1]			
b.	All changes to original data or forms made by lining out incorrect entry, and initialed and signed by the person making the change [QAPP 3.1.1]			
c.	All raw data signed and dated by generator in black ink [QAPP 3.1.1]			
d.	All pages in data package legible and copied pages complete (i.e., margins not cut off) [QAPP 3.1.1]			
e.	Data Report reviewed and signed by the TL to ensure completeness and accuracy of content [QAPP 3.1.1]			
f.	Data Report Submitted to TWCP SPO within 28 days of VTSR of last sample [QAPP 3.4]			

Form Date: 06/03/96 Page 1 of 1

Figure 3-1. Example of the ALD QAO data review checklist for total nonhalogenated VOC analysis.



QUALITY ASSURANCE DATA REVIEW CHECKLIST for TWCP METHOD 440.2 (TOTAL NH-VOCs)
LITCO Analytical Chemistry Laboratory

DATA REPORT NUMBER: ECL96xxxO

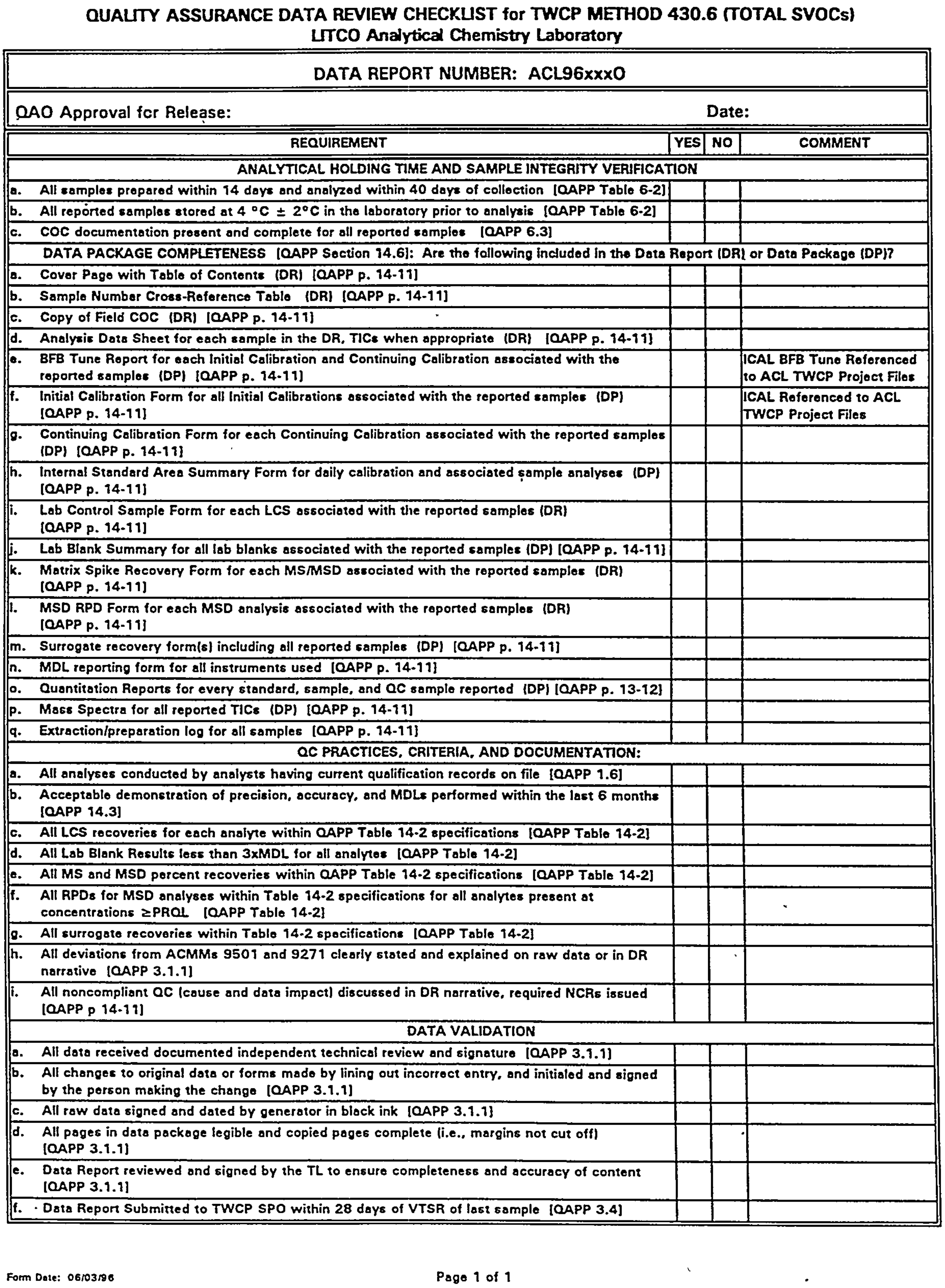
QAO Approval for Release: Date:

	REQUIREMENT	YES	NO	COMMENT
	. ANALYTICAL HOLDING TIME AND SAMPLE INTEGRITY VERIFICATION			
a.	All samples prepared and analyzed within 14 days of collection [QAPP Table 6-2]			
b.	All samples stored at 4 °C ± 2 °C in the laboratory prior to analysis [QAPP Table 6-2]			
c.	COC documentation present and complete for all reported samples [QAPP 6.3]			
	DATA PACKAGE COMPLETENESS [QAPP Section 12.6]: Are the following included in the Data Report (DR) or Data Package (DP)?			
a.	Cover Page with Table of Contents (DR) [QAPP p. 13-11]			
b.	Sample Number Cross-Reference Table (DR) [QAPP p. 13-11]			
c.	Copy of Field COC (DR) [QAPP p. 13-11]			
d.	Analysis Data Sheet for each sample reported in the DR (DR) [QAPP p. 13-11]			
e.	Initial Calibration Form for all Initial Calibrations associated with the reported samples (DP) [QAPP p. 13-12]			ICAL Referenced to ACL TWCP Project Files
f.	Continuing Calibration Form for all Continuing Calibrations associated with the reported samples (DP) [QAPP p. 13-12]			
g.	Analyte Identification Form for all samples with reported hits (DP) [QAPP p. 13-12]			
h.	External Standard Reports for every standard, sample and QC sample reported (DP) [QAPP p. 13-12]			
i.	Lab Control Sample Form for each LCS associated with the reported samples (DR) [QAPP p. 13-11]			
j.	Lab Blank Summary for all lab blanks associated with the reported samples (DP) [QAPP p. 13-12]			
k.	Matrix Spike Recovery Form for each MS/MSD analysis associated with the reported samples (DR) [QAPP p. 13-11]			
l.	MSD RPD Form for each MSD anaysis associated with the reported samples (DR) [QAPP p. 13-11]			
m.	MDL reporting form for all instruments used (DP) [QAPP p. 13-12]			
n.	Extraction/preparation log for all reported samples (DP) [QAPP p. 13-12]			
	QC PRACTICES, CRITERIA, AND DOCUMENTATION:			
a.	All analyses conducted by analysts having current qualification records on file [QAPP 1.6]			
b.	Acceptable demonstration of precision, accuracy, and MDLs performed within the last 6 months [QAPP 12.3]			
c.	All LCS recoveries for each analyte within QAPP Table 13-2 specifications [QAPP Table 13-2]			
d.	All Lab Blank Results less than 3xMDL for all analytes [QAPP Table 13-2]			
e.	All MS and MSD percent recoveries within QAPP Table 13-2 specifications [QAPP Table 13-2]			
f.	All RPDs for MSD analyses within Table 13-2 specifications for all analytes present at concentrations ≥PRQL [QAPP Table 13-2]			
g.	All deviations from ACMMs 9501 and 9441 clearly stated and explained on raw data or in DR narrative [QAPP 3.1.1]			
	DATA VALIDATION			
a.	All data received documented independent technical review and signature [QAPP 3.1.1]			
b.	All changes to original data or forms made by lining out incorrect entry, and initialed and signed by the person making the change [QAPP 3.1.1]			
c.	All raw data signed and dated in black ink [QAPP 3.1.1]			
d.	All pages in data package legible and copied pages complete (i.e., margins not cut off) [QAPP 3.1.1]			
e.	Data Report reviewed and signed by the TL to ensure completeness and accuracy of content [QAPP 3.1.1]			
g.	Data Report Submitted to TWCP SPO within 28 days of VTSR of last sample [QAPP 3.4]			

Form Date: 06/04/96

Page 1 of 1

Figure 3-2. Example of the ALD QAO data review checklist for total purgeable VOC analysis.



QUALITY ASSURANCE DATA REVIEW CHECKLIST for TWCP METHOD 430.6 (TOTAL SVOCs)
LITCO Analytical Chemistry Laboratory

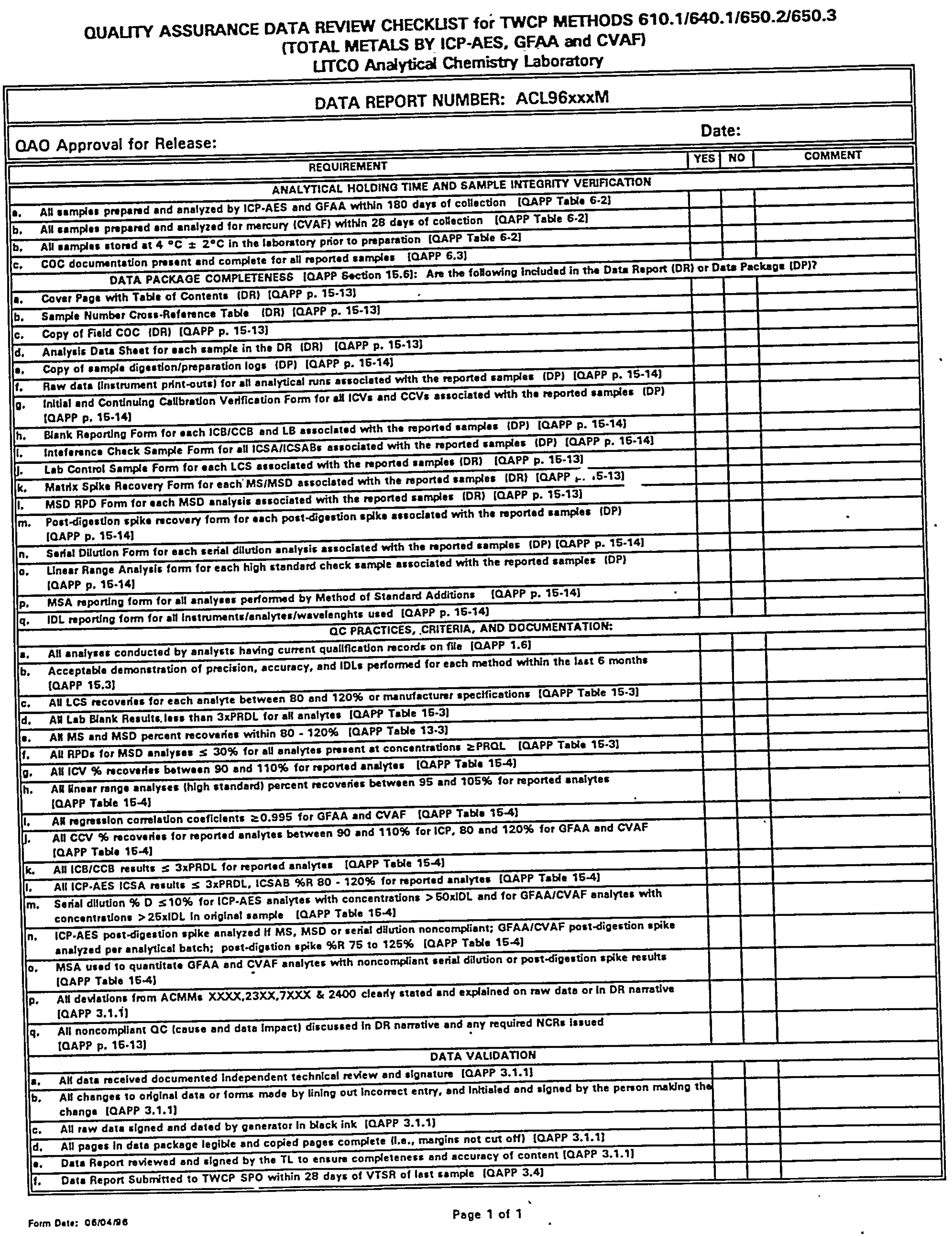
DATA REPORT NUMBER: ACL96xxxO

QAO Approval for Release: Date:

REQUIREMENT	YES	NO	COMMENT
ANALYTICAL HOLDING TIME AND SAMPLE INTEGRITY VERIFICATION			
a. All samples prepared within 14 days and analyzed within 40 days of collection [QAPP Table 6-2]			
b. All reported samples stored at 4 °C ± 2°C in the laboratory prior to analysis [QAPP Table 6-2]			
c. COC documentation present and complete for all reported samples [QAPP 6.3]			
DATA PACKAGE COMPLETENESS [QAPP Section 14.6]: Are the following included in the Data Report (DR) or Data Package (DP)?			
a. Cover Page with Table of Contents (DR) [QAPP p. 14-11]			
b. Sample Number Cross-Reference Table (DR) [QAPP p. 14-11]			
c. Copy of Field COC (DR) [QAPP p. 14-11]			
d. Analysis Data Sheet for each sample in the DR, TICs when appropriate (DR) [QAPP p. 14-11]			
e. BFB Tune Report for each Initial Calibration and Continuing Calibration associated with the reported samples (DP) [QAPP p. 14-11]			ICAL BFB Tune Referenced to ACL TWCP Project Files
f. Initial Calibration Form for all Initial Calibrations associated with the reported samples (DP) [QAPP p. 14-11]			ICAL Referenced to ACL TWCP Project Files
g. Continuing Calibration Form for each Continuing Calibration associated with the reported samples (DP) [QAPP p. 14-11]			
h. Internal Standard Area Summary Form for daily calibration and associated sample analyses (DP) [QAPP p. 14-11]			
i. Lab Control Sample Form for each LCS associated with the reported samples (DR) [QAPP p. 14-11]			
j. Lab Blank Summary for all lab blanks associated with the reported samples (DP) [QAPP p. 14-11]			
k. Matrix Spike Recovery Form for each MS/MSD associated with the reported samples (DR) [QAPP p. 14-11]			
l. MSD RPD Form for each MSD analysis associated with the reported samples (DR) [QAPP p. 14-11]			
m. Surrogate recovery form(s) including all reported samples (DP) [QAPP p. 14-11]			
n. MDL reporting form for all instruments used [QAPP p. 14-11]			
o. Quantitation Reports for every standard, sample, and QC sample reported (DP) [QAPP p. 13-12]			
p. Mass Spectra for all reported TICs (DP) [QAPP p. 14-11]			
q. Extraction/preparation log for all samples [QAPP p. 14-11]			
QC PRACTICES, CRITERIA, AND DOCUMENTATION:			
a. All analyses conducted by analysts having current qualification records on file [QAPP 1.6]			
b. Acceptable demonstration of precision, accuracy, and MDLs performed within the last 6 months [QAPP 14.3]			
c. All LCS recoveries for each analyte within QAPP Table 14-2 specifications [QAPP Table 14-2]			
d. All Lab Blank Results less than 3xMDL for all analytes [QAPP Table 14-2]			
e. All MS and MSD percent recoveries within QAPP Table 14-2 specifications [QAPP Table 14-2]			
f. All RPDs for MSD analyses within Table 14-2 specifications for all analytes present at concentrations ≥PRQL [QAPP Table 14-2]			
g. All surrogate recoveries within Table 14-2 specifications [QAPP Table 14-2]			
h. All deviations from ACMMs 9501 and 9271 clearly stated and explained on raw data or in DR narrative [QAPP 3.1.1]			
i. All noncompliant QC (cause and data impact) discussed in DR narrative, required NCRs issued [QAPP p 14-11]			
DATA VALIDATION			
a. All data received documented independent technical review and signature [QAPP 3.1.1]			
b. All changes to original data or forms made by lining out incorrect entry, and initialed and signed by the person making the change [QAPP 3.1.1]			
c. All raw data signed and dated by generator in black ink [QAPP 3.1.1]			
d. All pages in data package legible and copied pages complete (i.e., margins not cut off) [QAPP 3.1.1]			
e. Data Report reviewed and signed by the TL to ensure completeness and accuracy of content [QAPP 3.1.1]			
f. Data Report Submitted to TWCP SPO within 28 days of VTSR of last sample [QAPP 3.4]			

Form Date: 06/03/96 Page 1 of 1

Figure 3-3. Example of the ALD QAO data review checklist for total SVOC analysis.



QUALITY ASSURANCE DATA REVIEW CHECKLIST for TWCP METHODS 610.1/640.1/650.2/650.3
(TOTAL METALS BY ICP-AES, GFAA and CVAF)
LITCO Analytical Chemistry Laboratory

DATA REPORT NUMBER: ACL96xxxM

QAO Approval for Release:

Date:

	REQUIREMENT	YES	NO	COMMENT
	ANALYTICAL HOLDING TIME AND SAMPLE INTEGRITY VERIFICATION			
a.	All samples prepared and analyzed by ICP-AES and GFAA within 180 days of collection [QAPP Table 6-2]			
b.	All samples prepared and analyzed for mercury (CVAF) within 28 days of collection [QAPP Table 6-2]			
b.	All samples stored at 4 °C ± 2°C in the laboratory prior to preparation [QAPP Table 6-2]			
c.	COC documentation present and complete for all reported samples [QAPP 6.3]			
	DATA PACKAGE COMPLETENESS [QAPP Section 15.6]: Are the following included in the Data Report (DR) or Data Package (DP)?			
a.	Cover Page with Table of Contents (DR) [QAPP p. 15-13]			
b.	Sample Number Cross-Reference Table (DR) [QAPP p. 15-13]			
c.	Copy of Field COC (DR) [QAPP p. 15-13]			
d.	Analysis Data Sheet for each sample in the DR (DR) [QAPP p. 15-13]			
e.	Copy of sample digestion/preparation logs (DP) [QAPP p. 15-14]			
f.	Raw data (instrument print-outs) for all analytical runs associated with the reported samples (DP) [QAPP p. 15-14]			
g.	Initial and Continuing Calibration Verification Form for all ICVs and CCVs associated with the reported samples (DP) [QAPP p. 15-14]			
h.	Blank Reporting Form for each ICB/CCB and LB associated with the reported samples (DP) [QAPP p. 15-14]			
i.	Inteference Check Sample Form for all ICSA/ICSABs associated with the reported samples (DP) [QAPP p. 15-14]			
j.	Lab Control Sample Form for each LCS associated with the reported samples (DR) [QAPP p. 15-13]			
k.	Matrix Spike Recovery Form for each MS/MSD associated with the reported samples (DR) [QAPP p. 15-13]			
l.	MSD RPD Form for each MSD analysis associated with the reported samples (DR) [QAPP p. 15-13]			
m.	Post-digestion spike recovery form for each post-digestion spike associated with the reported samples (DP) [QAPP p. 15-14]			
n.	Serial Dilution Form for each serial dilution analysis associated with the reported samples (DP) [QAPP p. 15-14]			
o.	Linear Range Analysis form for each high standard check sample associated with the reported samples (DP) [QAPP p. 15-14]			
p.	MSA reporting form for all analyses performed by Method of Standard Additions [QAPP p. 15-14]			
q.	IDL reporting form for all instruments/analytes/wavelenghts used [QAPP p. 15-14]			
	QC PRACTICES, CRITERIA, AND DOCUMENTATION:			
a.	All analyses conducted by analysts having current qualification records on file [QAPP 1.6]			
b.	Acceptable demonstration of precision, accuracy, and IDLs performed for each method within the last 6 months [QAPP 15.3]			
c.	All LCS recoveries for each analyte between 80 and 120% or manufacturer specifications [QAPP Table 15-3]			
d.	All Lab Blank Results less than 3xPRDL for all analytes [QAPP Table 15-3]			
e.	All MS and MSD percent recoveries within 80 - 120% [QAPP Table 13-3]			
f.	All RPDs for MSD analyses ≤ 30% for all analytes present at concentrations ≥PRQL [QAPP Table 15-3]			
g.	All ICV % recoveries between 90 and 110% for reported analytes [QAPP Table 15-4]			
h.	All linear range analyses (high standard) percent recoveries between 95 and 105% for reported analytes [QAPP Table 15-4]			
i.	All regression correlation coeficients ≥0.995 for GFAA and CVAF [QAPP Table 15-4]			
j.	All CCV % recoveries for reported analytes between 90 and 110% for ICP, 80 and 120% for GFAA and CVAF [QAPP Table 15-4]			
k.	All ICB/CCB results ≤ 3xPRDL for reported analytes [QAPP Table 15-4]			
l.	All ICP-AES ICSA results ≤ 3xPRDL, ICSAB %R 80 - 120% for reported analytes [QAPP Table 15-4]			
m.	Serial dilution % D ≤10% for ICP-AES analytes with concentrations >50xIDL and for GFAA/CVAF analytes with concentrations >25xIDL in original sample [QAPP Table 15-4]			
n.	ICP-AES post-digestion spike analyzed if MS, MSD or serial dilution noncompliant; GFAA/CVAF post-digestion spike analyzed per analytical batch; post-digstion spike %R 75 to 125% [QAPP Table 15-4]			
o.	MSA used to quantitate GFAA and CVAF analytes with noncompliant serial dilution or post-digestion spike results [QAPP Table 15-4]			
p.	All deviations from ACMMs XXXX,23XX,7XXX & 2400 clearly stated and explained on raw data or in DR narrative [QAPP 3.1.1]			
q.	All noncompliant QC (cause and data impact) discussed in DR narrative and any required NCRs issued [QAPP p. 15-13]			
	DATA VALIDATION			
a.	All data received documented independent technical review and signature [QAPP 3.1.1]			
b.	All changes to original data or forms made by lining out incorrect entry, and initialed and signed by the person making the change [QAPP 3.1.1]			
c.	All raw data signed and dated by generator in black ink [QAPP 3.1.1]			
d.	All pages in data package legible and copied pages complete (i.e., margins not cut off) [QAPP 3.1.1]			
e.	Data Report reviewed and signed by the TL to ensure completeness and accuracy of content [QAPP 3.1.1]			
f.	Data Report Submitted to TWCP SPO within 28 days of VTSR of last sample [QAPP 3.4]			

Page 1 of 1

Form Date: 06/04/96

Figure 3-4. Example of the ALD QAO data review checklist for total metals analysis.

The QAO signs the signature release on the data report cover page upon completion of the checklist if the data package is compliant with each of the checklist specifications. If the data package is noncompliant with one or more of the project specifications, the QAO evaluates the nature of the noncompliance. If the noncompliance can be rectified by correcting an error or omission in the data package, the data package is returned to the responsible technical leader or DRC for correction. After the corrections have been made and verified by the TL, the data package is resubmitted to the QAO for verification of the corrections.

If the noncompliance cannot be rectified by correcting the data package, the corrective action process described in Section 2.2.1 is initiated. A copy of any initiated NCR must be included in the data report, and the NCR number must be referenced on the data package review checklist.

The completed checklists are retained as part of the data package, and copies are included in associated data reports.

3.2 Validation Methods

3.2.1 Precision

Precision is a measurement of the random error in an analytical measurement process (i.e., the degree of agreement between independent measurements determined by the analysis of replicate samples). QC specifications for the precision of each analytical parameter and analytical method QC indicators are delineated in Tables 13-1, 14-1, and 15-1, and in the applicable ACL analytical methods (ACMM).

When calculated for duplicate sample analyses (i.e., matrix spike duplicates), precision is expressed as the RPD, which is calculated as:

$$RPD\ (\%) = \frac{|\ S - D\ |}{\frac{(S + D)}{2}} * 100 \qquad (3\text{-}1)$$

where

S = first sample value (original, or matrix spike), mg/kg wet weight

D = second sample value (duplicate, or matrix spike duplicate), mg/kg wet weight.

When precision is calculated for three or more replicate determinations, the RSD, also known as the coefficient of variation, expressed in units of percentage, is used. This is an expression of the spread of the data relative to the mean value ($\overline{X}$) of the determinations. The specific formulas used for calculation of the RSD are:

$$\bar{x} = \frac{\sum_{i=1}^{n} x_i}{n} \qquad (3\text{-}2)$$

$$s = \sqrt{\frac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{n - 1}} \qquad (3\text{-}3)$$

and

$$RSD\ (\%) = CV = \frac{s}{\bar{x}} * 100 \qquad (3\text{-}4)$$

where

$\bar{X}$ = mean of n measurements

x_i = result value for the i_{th} measurement

n = total number of measurements

s = standard deviation.

Precision is also measured as the percent difference (%D) between an initial measurement and a subsequent one. The following formula is used to calculate %D:

$$\%D = \frac{|I-S|}{I} * 100 \qquad (3\text{-}5)$$

where

I = initial measurement

S = subsequent measurement

3.2.2 Accuracy

Accuracy (bias) is a measurement of the extent to which a measured value of a quantity (parameter or analyte) agrees with the accepted value of that quantity. QC specifications for the accuracy of analytical results and calibration results are delineated in Tables 13-1, 14-1, and 15-1, and in applicable ACMMs.

For analyses of homogeneous solids and soils/gravel, accuracy is assessed by the analysis of samples of known concentration (e.g., LCSs, calibration standards, field reference standards, or additional QC samples) for the analytes of concern. Accuracy is quantified by calculating the percent recovery (%R) of the known quantity of analyte:

$$R\ (\%) = \frac{V_m}{V_t} * 100 \tag{3-6}$$

where

V_m = measured value (concentration determined by analysis)

V_t = true value (concentration or quantity as calculated or certified by the manufacturer).

For determining the percent recovery of matrix spikes, matrix spike duplicates, and post-digestion spikes the following equation is used:

$$\%R = \frac{SSR - SR}{SA} * 100 \tag{3-7}$$

where

SSR = spiked sample result (mg/kg or matrix spikes and matrix spike duplicate, $\mu g/\ell$ for post-digestion spikes)

SR = result of unspiked sample (mg/kg or matrix spikes and matrix spike duplicate, $\mu g/\ell$ for post-digestion spikes)

SA = amount of spike added (mg/kg or matrix spikes and matrix spike duplicate, $\mu g/\ell$ for post-digestion spikes).

3.2.3 Method Detection Limits

Method detection limits (MDLs) are determined for each organic analyte for each method used by ACL in support of TWCP. These MDLs are determined by (a) conducting replicate analyses of standards at quantities approximately one to five times the estimated MDL, (b) determining the standard deviation, s, of the replicate measurements, and (c) calculating the MDL from:

$$MDL = t_{(n-1,\ 1-\alpha=0.99)} * s \tag{3-8}$$

where

n = number of replicate analyses

$t_{(n-1,\ 1-\alpha=0.99)}$ = t distribution value appropriate to a 99% confidence level (one-tailed) and standard deviation estimate with n - 1 degrees of freedom

s = standard deviation of the data set.

The MDL calculated in this manner represents the minimum amount of a substance that can be measured and reported with 99% confidence that the analyte quantity is greater than zero.

The MDL does *not* represent the analyte quantity for which there is a 99% probability that the analyte will be detected; there is a 50% probability of detection and reporting of the analyte whose actual amount is at the MDL. The analyte quantity at which there is a 99% probability that the analyte will be detected and reported is twice the MDL.

Because MDLs are usually determined using standards in a clean matrix, they represent optimum obtainable performance; MDLs for actual sample matrices are likely to be higher than those determined using clean matrices. Program-required maximum MDLs for homogeneous solids and soils/gravel target analytes for the TWCP are listed in Tables 13-1 and 14-1. ACL-determined MDLs must be less than or equal to the listed values.

3.2.4 Instrument Detection Limits (IDLs)

Instrument detection limits (IDLs) are determined for each metal analyte for each method used by ACL in support of TWCP. These IDLs are determined by (a) conducting seven replicate analyses of standards at quantities approximately one to five times the estimated MDL, on each of three nonconsecutive days, (b) determining the standard deviation, s, of the replicate measurements for each day, and (c) calculating the IDL as 3 times the mean standard deviation, as follows:

$$IDL = 3 * s_x \qquad (3\text{-}9)$$

where

s_x = the average of the standard deviations determined on three nonconsecutive days from measurement of 7 replicate standards.

Because IDLs are usually determined using standards in a clean matrix, they represent optimum obtainable performance; IDLs for actual sample matrices are likely to be higher than those determined using clean matrices. Program-required instrument detection limits (PRDLs) for homogeneous solids and soils/gravel target analytes for the TWCP are listed in Table 15-1. ACL-determined IDLs must be less than or equal to the listed values.

3.2.5 Completeness

The characteristic of completeness is a measure of the amount of valid analytical data obtained compared to the total number of analyses performed. Valid analytical data are those generated when analytical systems were in control, i.e., all calibration verification, interference and non-matrix checks met acceptance criteria. Completeness of the reported data (expressed as a percentage) is calculated as:

$$C\,(\%) = \frac{M_v}{M_t} * 100 \qquad (3\text{-}10)$$

where

M_v = number of measurements judged to be valid

M_t = total number of measurements performed (based upon number of samples submitted).

3.2.6 Comparability

ACL PDP results provide a measure of data comparability between ACL and other participating laboratories. ACLs analytical processes incorporate specific mechanism (see Sections 3.1 and 13.0–15.0) for ensuring comparability of generated data, such as standardized analysis methods and measurement units, traceable standards and data review.

3.3 Reconciliation with Data Quality Objectives

Reconciliation of reported data with the DQOs listed in Section 1.5 is the responsibility of the SPO.

3.4 Data Reporting Requirements

Data reporting requirements define the type of information and the method of transmittal for data transfer from the ACL to the SPO. Specific reporting requirements and examples of reporting forms are provided in Sections 13, 14 and 15 for VOC, SVOC and metals analyses, respectively. Data are transmitted by hard copy data reports to the SPM, in care of the SPO document controller. These analytical data reports shall be submitted to the SPO within 28 days of the validated time of sample receipt (VTSR) of the last sample in the analytical batch or data report. For special requests for analytical services from the TWCP SPO, the schedule and format requirements for data reporting will be determined on an individual basis.

Routinely, sample results for metals and organics (total purgeable VOCs, total nonhalogenated VOCs [NH-VOCs] and total SVOCs) will be reported separately. Each data report is assigned a unique serial number, in the format ACLYYXXXZ, where ACL designates the laboratory, YY is the last two digits of the calendar year, XXX is a sequentially assigned number, and Z indicates the analysis category or method (O for organics and M for metals). If the organic sample results are reported separately by method, the Z suffix will be V for purgeable VOCs, N for NH-VOCs, and S for SVOCs. In the event that sample results from all analysis methods are included in the same report, the Z suffix is dropped.

ACL data reports with a common sequential number (e.g., ACL96001M and ACL96001O) will normally provide data for the same set of field samples. Due to the different number of samples for VOC analysis and the potential for different batching between methods, ACL data reports may not coincide with a single analytical batch (i.e., a single data report may contain data from multiple analytical batches). Each page of the data report is paginated at the bottom. Red ink is used to paginate the original to distinguish it from any subsequent copies made.

The original data reports are archived by the ACL in the TWCP project files. The reports are filed with the associated data package, which contains all raw data, reduced data, and QC sample results for the reported samples. Copies of the data reports are submitted to the SPO. Resubmissions and corrections to data reports are controlled (i.e., assigned a revision number), and must receive the same signature releases as the original data report.

4. MEASUREMENT AND DATA ACQUISITION

4.1 Facilities and Workplaces

The ACL occupies three facilities at the Idaho Chemical Processing Plant (ICPP or CPP); the CPP-602 facility, the CPP-627 facility, and the Remote Analytical Facility [RAL] (building CPP-684). Analytical work in support of the TWCP will be performed in all three facilities. Due to the presence of TRU contaminants, TWCP samples must be handled in containment (i.e., gloveboxes) or in radiological hoods equipped with high efficiency particulate air (HEPA) filters.

4.1.1 CPP-602 Facility

The portion of the CPP-602 facility occupied by ACL includes offices and 9550 ft^2 of laboratory space. Laboratories are equipped with HEPA hoods and gloveboxes for handling radioactively-contaminated samples. Most of the analytical processes in support of the TWCP will be performed in this facility. TWCP functions carried out in the CPP-602 facility include custody-controlled sample storage, quality control/standard preparation (QC laboratory), sample extractions for VOC and SVOC analyses, and analyses for VOCs, SVOCs and metals.

4.1.2 CPP-627 Facility

ACL has an additional 2170 ft^2 of laboratory space in building CPP-627. Microwave digestions for metals are carried out in the hoods and gloveboxes in this facility. Locked refrigerators in the facility are used for custody-controlled sample storage.

4.1.3 Remote Analytical Laboratory (CPP-684)

The RAL contains a 750 ft^2 cold laboratory, a 2400 ft^2 warm laboratory, and a 20x50 ft seven-window hot cell. TWCP functions performed in the warm laboratory include mercury analyses and other metals analyses (backup capability). It is not anticipated that the level of radiological contamination associated with TWCP samples will require hot cell containment.

4.2 Equipment Calibration

Analytical instruments are calibrated by the user with specific method use. Procedures for calibration of these instruments are provided in the specific analytical methods referenced in Sections 13, 14, and 15. When calibration procedures are not method-specific, then separate calibration procedures are included in ACLPs or other procedures. Procedures and criteria for calibration and calibration checks of analytical balances are specified in R.1.20, *Analytical Quality Control*. Requirements and procedures for temperature monitoring of refrigerated sample and standard storage units are addressed in MCP-2002, *Analytical Chain of Custody*, and in ACLP 0.27, *Refrigerated Storage Cabinet Control Procedure*, respectively. Requirements and procedures for verifying pipettor calibration are addressed in ACLP 2.06, *Pipette Calibration Verification Procedures for Spectrochemistry.*

ACL equipment calibration procedures implement requirements of LMITCO MCP-2391, *Calibration Program*. These calibration requirements comply with ASME NQA-1, Element 12 and ANSI/NCSL Z540.1 (ANSI, 1994). Calibrated equipment is identified by a calibration sticker or a similar method. The calibration sticker indicates the latest calibration date or the due date of the next calibration, or that calibration is performed with use. The calibration records are maintained by the organization performing the calibration, and must include the following information:

- Equipment identification/serial number
- Name of device
- Calibration and/or maintenance schedule
- Procedure(s) and revision number for calibration and/or maintenance
- Date and results of last calibration with signature of person performing calibration
- Date for next scheduled calibration
- Facility or organization performing calibration
- Nonconforming conditions related to the equipment (if applicable)
- Corrective actions taken to eliminate nonconforming conditions (if applicable)
- Standards used for calibration with certification papers.

As required by MCP-538, *Control of Nonconforming Items*, equipment that cannot be calibrated or that becomes inoperable during use will be removed from service and isolated to prevent inadvertent use, or it will be tagged to indicate that it is out of calibration. Such equipment must be repaired and recalibrated to the satisfaction of project requirements before it can be used again.

4.3 Equipment Maintenance

Analytical instrumentation undergoes routine preventive maintenance as recommended by the equipment manufacturer or as dictated by experience. Maintenance schedules are established and included in analytical methods (ACMMs) for ACL analytical instrumentation. Maintenance procedures are incorporated by reference to the manufacturer's manuals or other ACL procedures (e.g., ACLPs).

Instrument custodians are appointed for all major instrumentation systems, and are responsible for ensuring that required maintenance is performed and for maintaining an inventory of critical spare parts. Maintenance logbooks are maintained for all systems that document performance of preventive maintenance and repairs. In general, the ACL has sufficient redundant instrument capability to minimize downtime for a given analytical procedure. Primary instrument and equipment plus backup systems used to support TWCP analyses are listed in Table 4-1.

Table 4-1. Primary and backup instrument and equipment systems used for TWCP support.

Analytical Method	Instrument	Primary System	Secondary System
Total purgeable VOCs analysis	GC/MS	Varian Saturn 3 ID: VOA-2	Finnigan Magnum ID: VOA-1
Total nonhalogenated VOCs analysis	GC-FID	Hewlett-Packard 5890 Series II ID: GC-2	Hewlett-Packard 5890 Series II ID: GC-1
Total SVOC extraction	Sonicator	Daigger Model GE375	(not applicable)
Total SVOC extraction	Gel Permeation Chromatograph	ABC AS-2000 ID: GPC-1	(not applicable)
Total SVOC analysis	GC/MS	Finnigan Magnum ID: SV-1	Varian Saturn 3 ID: SV-2
Total metals digestion	Microwave digestor	CEM MDS-2100 WTC Model SP-4	CEM MDS-81D
Total metals analysis	ICP-AES	Jobin Yvon ISA JY-38 Plus ID: ICP-2	Jobin Yvon ISA JY-38 ID: ICP-1
Total metals analysis	GFAA	Thermo Jarrell Ash SH 4000 AA CTF-188 Furnace ID: GFAA-1	Thermo Jarrell Ash SH 4000 AA; CTF-188 Furnace; ID: GFAA-2
Total metals analysis (Hg)	CVAF	PSA Fluorescence Model 10.023/10.0003 ID: CVAF-1	(not applicable)
Total metals analysis (Hg backup)	CVAA	Thermo Jarrell Ash SH-12 AA Atomic Vapor Accessary Model 440 ID: CVAA-8	(not applicable)

5. SAMPLING PROCESS DESIGN

THIS TOPIC IS NOT APPLICABLE TO ACL ACTIVITIES SUPPORTING TWCP.

6. DRUM AND SAMPLE HANDLING AND CUSTODY REQUIREMENTS

An essential part of any sampling and analytical scheme involves the techniques used to ensure the integrity of the sample from point of collection to data reporting. This includes the ability to trace the possession and handling of samples from the time of collection until analysis. ACL maintains COC documentation for all TWCP samples received for analysis or storage. Drum handling and custody requirements do not apply to ACL.

A sample is under custody if one of the following conditions applies:

- It is in the possession of an authorized individual
- It is in the view of an authorized individual, after being in the possession of that individual
- It was in the possession of an authorized individual, and access to the sample(s) was controlled by locking or placement of signed custody seals that prevent undetected access
- It is in a designated secure area, such as a controlled accesss location with complete documentation of personnel access or a radiological containment area (e.g., glove box).

6.1 Sample Container Labeling and Chain-of-Custody

Each TWCP solid sample received at ACL for analysis is identified by a unique 13-character field sample identification number. The field sample ID format is defined in the SPO QAPjP, and uses the following format:

ID XXXXXX Y Z T A N

where:

ID = Idaho sampling site

XXXXXX = 6-digit drum identification (bar code) number

Y = Yth core sample number

Z = zero, if between-core sample compositing is not required
or
= number of the second core sample in the composite

T = Tth subsample in the core sample
or
= C, when single core sample composite is required
or
= B, when between core compositing is required

A = Specified analysis (V = VOCs, S = SVOCs, M = metals)

N = Nth container in the subsample.

Example:

Sample number ID023691101V2 indicates:

ID: = Idaho sample

023691: = from drum 023691

1: = the first core sample

0: = no between-core composite

1: = the first subsample, no composite

V: = VOC analysis sample

2: = second vial in the subsample.

Each sample container is identified by a sample label that contains the following information:

- Field Sample ID
- Analysis
- COC #
- Sampling Batch #
- Time/Date of sample collection
- Weight of sample (if required)
- Remarks
- Signature of person collecting sample.

6.2 Sample Receiving

A detailed description of the sample-handling process within ACL is provided in ACL MCP-2002, *Analytical Chemistry Chain-of-Custody*, and SOP R.1.24, *The Management of Samples in Analytical Chemistry*. A summary of critical components of the process are listed in the remainder of this section.

Every batch of TWCP samples delivered to the ACL must be accompanied by a completed COC form (Figure 6-1). Only the Sample Custodian or designated alternates shall accept custody of TWCP samples delivered to the laboratory.

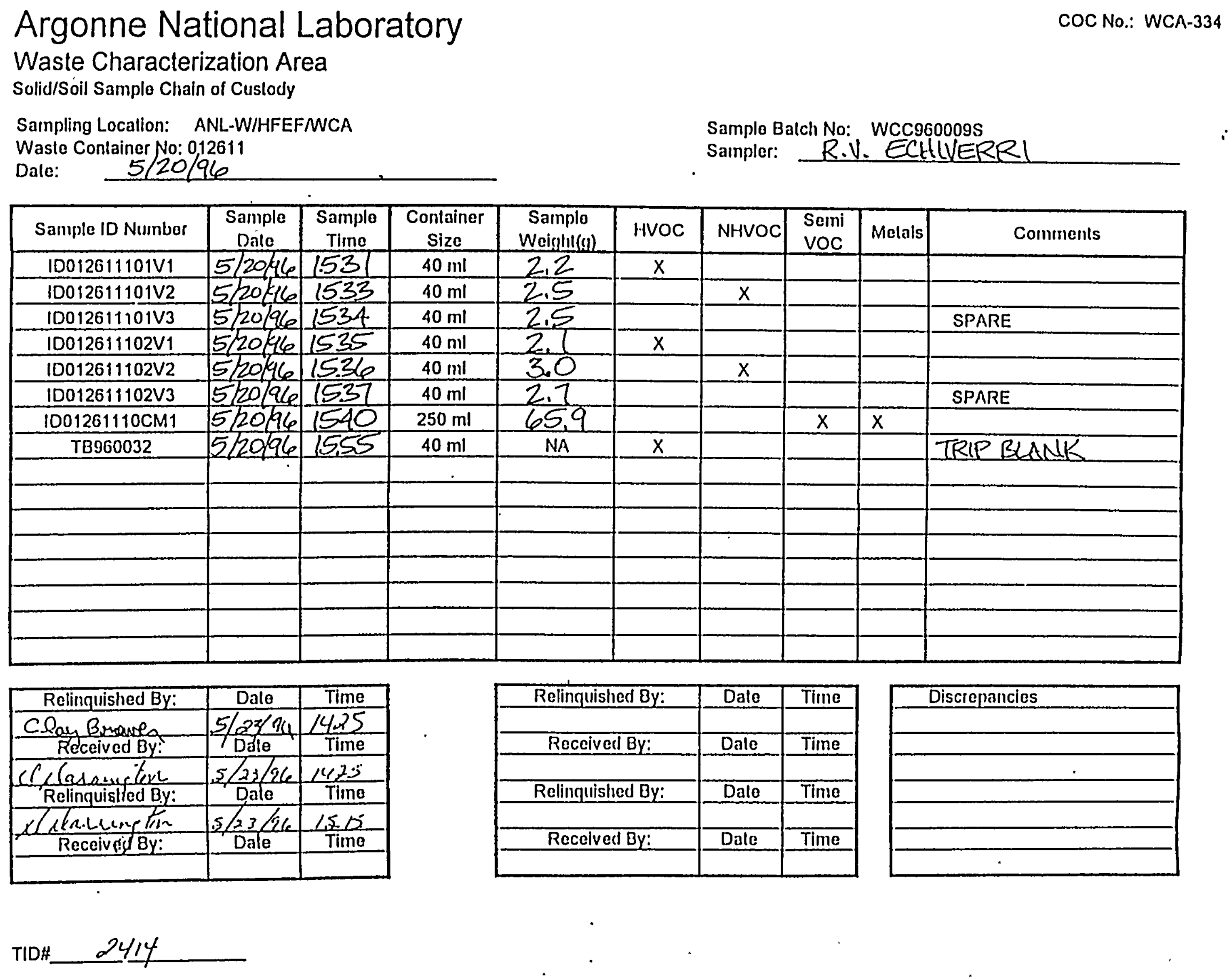
TWCP samples are collected at Argonne National Laboratory-West (ANL-W) and will normally be hand-delivered to front gate at ICPP by INEL staff. Samples to be analyzed by the ACL will never leave the INEL site. Samples will be accepted by the sample custodian or designated alternate from 7:00 a.m. to 4:30 p.m. local time, Monday through Wednesday, and from 7:00 a.m. to 2:30 p.m. on Thursday, unless other arrangements have been made. Details of sample packaging and transportation requirements are provided in a memorandum of understanding between ANL-W and ACL (ANL, 1996).

The COC form with relinquishing signature is shipped inside the sealed shipping container. The sample custodian receives the samples and inspects the custody seal on the shipping container to ensure that it is intact. The sample custodian then arranges for a facility radiological control technician (RCT) for opening the shipping container. The custodian inspects the shipment for completeness and integrity, signs and dates the COC, and returns a copy of the signed COC form to the sample generator at Argonne National Laboratory-West (ANL-W).

During sample receipt inspection and logging, the sample condition is examined and laboratory identification numbers are assigned. Samples are checked for physical damage (e.g., leaking or broken containers) or tampering (e.g., broken custody seal). Temperature within the shipping container at the time it was opened is noted (e.g., cool or ambient). Sample label information is checked for agreement with the COC form. Any discrepancies, problems or unusual circumstances shall be recorded on the COC form or the Sample Receiving and Custody Review Checklist (see example in Figure 6-2). The sample custodian shall contact the sample submitter named on the COC form to resolve discrepancies and document resolution of those problems. Any problems documented, and the resolution of those problems, shall become part of the permanent record and shall be incorporated in the associated data package and discussed in the data report narrative.

Every sample container accepted for analysis by the ACL receives a unique laboratory identification number when logged into the ACS. The laboratory sample ID uses the format XYYYY, where X is the last digit of the calendar year and YYYY is an alphanumeric character sequentially assigned from the beginning of the calendar year (examples: 6AA01, 6AA02...6AA99, 6AB01... 6ZZ99). If multiple containers of a single sample are received, they are designated as bottle 1, 2, 3, etc. Laboratory data reports reference both the laboratory number and the field sample number. The laboratory number (and bottle number, if appropriate) is also written on the sample label.

Samples received at one time are assigned by the ACS to a "log" for tracking, processing, and reporting purposes. The log number uses the following format: YYMMDD-X, where YYMMDD is the date of sample receipt (year/month/day) and X is sequential number starting with 1 assigned to logs received within a calendar day. Analysts are notified via the ACS that samples are in-house and ready for analysis.



Argonne National Laboratory
Waste Characterization Area
Solid/Soil Sample Chain of Custody

COC No.: WCA-334

Sampling Location: ANL-W/HFEF/WCA
Waste Container No: 012611
Date: 5/20/96

Sample Batch No: WCC960009S
Sampler: R.V. ECHEVERRI

Sample ID Number	Sample Date	Sample Time	Container Size	Sample Weight(g)	HVOC	NHVOC	Semi VOC	Metals	Comments
ID012611101V1	5/20/96	1531	40 ml	2.2	X				
ID012611101V2	5/20/96	1533	40 ml	2.5		X			
ID012611101V3	5/20/96	1534	40 ml	2.5					SPARE
ID012611102V1	5/20/96	1535	40 ml	2.1	X				
ID012611102V2	5/20/96	1536	40 ml	3.0		X			
ID012611102V3	5/20/96	1537	40 ml	2.7					SPARE
ID01261110CM1	5/20/96	1540	250 ml	65.9			X	X	
TB960032	5/20/96	1555	40 ml	NA	X				TRIP BLANK

Relinquished By:	Date	Time
Clay Browne	5/23/96	1425
Received By:	Date	Time
C Clarrington	5/23/96	1425
Relinquished By:	Date	Time
C Clarrington	5/23/96	1515
Received By:	Date	Time

Relinquished By:	Date	Time
Received By:	Date	Time
Relinquished By:	Date	Time
Received By:	Date	Time

Discrepancies

TID# 2414

Figure 6-1. Example of a TWCP Chain-of-Custody form.

TWCP SAMPLE RECEIVING & CUSTODY REVIEW CHECKLIST
ICPP Analytical Chemistry Laboratory

Log Number:	Nonconformance Report Initiation Required? Y or N NCR Number:	
Reviewer Signature:	Date:	Form Revision: 0

Instructions: Complete one checklist per log. Enter appropriate response for each question. Each "No" response requires explanation. A "No" response to a question in Bold type may require initiation of an NCR.

	REQUIREMENT	YES	NO	COMMENTS
	1. FIELD CHAIN-OF-CUSTODY (COC)			
a.	**Was a COC form received with each sample shipment?**			
b.	**Were all field samples listed on the accompanying COC form included in the shipment?**			
c.	**Were any samples included in the shipment which were not listed on the COC form?**			
d.	**Have all custody transfers been completely documented by signatures of relinquishers and acceptors, date and time of transfer?**			
e.	Does all sample information (e.g., sample ID, sampling date and time, sample location, analysis requests, sampling batch) listed on the COC correspond with the information on the sample labels?			
f.	Were any corrections on the COC appropriately made (a single line through the incorrect data, correct data written in [not overwritten], initialed and dated)?			
	2. SAMPLE LABELS			
a.	**Was each sample received with a completed sample label?**			
b.	Do the field IDs on the sample labels correspond with those on the COC?			
c.	Was the sampling batch number recorded on each sample label, and does it correspond with that recorded on the COC?			
d.	Are any corrections on the sample label appropriately made (a single line through the incorrect data, correct data written in [not overwritten], initialed and dated)?			
	3. SAMPLE INTEGRITY			
a.	**Were custody seals used on the shipping container or on all individual sample containers?**			
b.	**Were all custody seals intact when the shipment was received by ACL?**			
c.	**Has physical integrity of all samples been maintained (i.e., no cracks)?**			
d.	**Were all RCRA samples preserved during shipment with "Blue Ice" or equivalent cooling mechanism?**			
	4. INTERNAL CHAIN OF CUSTODY			
a.	Were all samples logged into the Analytical Computer System?			
b.	Was all sample information correctly transcribed from the field COC into the Analytical Computer System?			
b.	Were all sample bottles labeled with the Analytical Log Number and Lab Sample ID?			
c.	Was an EPA Sample Log form completed and placed in the appropriate storage location?			
d.	**Was internal tracking maintained on all samples bottles inside the laboratory?**			

The field sampling organization <u>must</u> be contacted if any discrepancies are found in the sample COC and label documentation. This contact must be documented in writing with:

1) the name of person contacting the sampling organization
2) the name of person contacted
3) date and time of contact
4) the resolution of the problem.

COMMENTS:

Rev. 6/96

Figure 6-2. Example of TWCP Sample Receiving & Custody Review Checklist

6.3 Sample Handling and Storage Requirements

After receipt and logging, TWCP samples are stored in the secure custody room (CPP-602-213) or in locked refrigerators in the CPP-627 facility. Access to the custody room and locked refrigerators is limited to the Sample Custodian and designated alternates.

The ACL laboratories are within the security fence at the ICPP; access to the facility is controlled by key cards. Access to the ACL laboratories within the ICPP is controlled by key cards or cipher locks, and is limited to ACL personnel, RCTs, and a finite number of other facility support personnel.

When ready to process samples or conduct analyses, the analyst contacts the sample custodian or designated alternate. The sample custodian then checks the samples out to the analyst; chain-of-custody and sample tracking are maintained in the laboratory through use of internal COC and tracking forms (see Figure 6-3 and 6-4 for examples).

All TWCP samples are assigned to an analytical batch for analysis. An analytical batch consists of a suite of samples of a similar matrix that are processed as a unit, using the same analytical method, within a specified time period. An analytical batch can contain up to 20 samples (excluding laboratory QC and trip blanks), all of which must be received by the laboratory within 14 days of validated time of sample receipt (VTSR) of the first sample in the batch. Analytical batch numbers consist of the log number associated with the first sample received in the batch.

Sample holding times and required preservation/storage conditions are provided in Table 6-1. All holding times begin at time of sample collection, as documented on the COC form.

6.4 Sample Disposal

TWCP samples will not be disposed of (i.e., do not become waste) until associated data reports have been approved by the SPO, and an SPO Data Validation Notification (see example in Figure 6-5) has been received. For this project, disposal of the samples consists of returning the unused portions of the samples to the Radioactive Waste Management Complex (RWMC).

Page__ of __

Analytical Chemistry EPA Sample Log

( ) Log # :

Received For Labs by: ____________________ Date: __________ Time: __________

Condition Samples/Seals: ____________________ No. of Samples: __________

Comments: ____________________

Sample Storage Location: ____________________

Sample Code(s):

Samples/Aliquots removed from custody room

Sample	Volume	Analysis	Date	Time	To	By	Custodian

Samples/Aliquots returned to custody room

Sample	Returned By	Date	Time	To	Custodian

Figure 6-3. Example of an ACL internal COC tracking form.

Analytical Chemistry EPA Sample/Aliquot Tracking

( ) Log # :

From Custody Room 213 Sample Code ____________________

In/Out	Lab	By	Date	Time	Analysis	Comments

Figure 6-4. Example of ACL internal sample tracking form

Table 6-1. Sample handing requirements for solid process residues and soils.

		Holding Times[a]	
Parameter	Preservative	To Preparation	To Analysis
VOCs	Cool to 4° ± 2° C	14 days	40 days[b]
SVOCs	Cool to 4° ± 2° C	14 day	40 days
Metals (except Hg)	Cool to 4° ± 2° C	180 days	NA
Mercury	Cool to 4° ± 2° C	28 days	NA

---

[a] Holding time begins at time of sample collection as documented on the COC form.

[b] 40-day holding time allowable only for methanol extract.

SITE PROJECT OFFICE
DATA VALIDATION NOTIFICATION

Facility(s):______________________________
____

SPO Number____________________

Batches Effected:
Data Packages validated:
________ *Validation with comments:* (See Attachments) ________ Validation completion requires correction of comments and/or disposition of NCR. ________ Comments presented for lessons learned and correction in future packages.
________ *Validation completed:* (Facility may proceed with sample disposal, drum shipment, etc. - as appropriate)
Additional Informational Comments:

Please address any comments or questions to the Site Quality Assurance Office (SQAO) at 526-8605 or 526-9126.

____
Site Project Office Signature/Date

Figure 6-5. Example of SPO data validation notification form.

7. HEADSPACE GAS SAMPLING

THIS TOPIC IS NOT APPLICABLE TO ACL ACTIVITIES SUPPORTING TWCP.

8. SAMPLING OF SOLID PROCESSING RESIDUES AND SOILS

THIS TOPIC IS NOT APPLICABLE TO ACL ACTIVITIES SUPPORTING TWCP.

9. RADIOASSAY

THIS TOPIC IS NOT APPLICABLE TO ACL ACTIVITIES SUPPORTING TWCP.

10. RADIOGRAPHY

THIS TOPIC IS NOT APPLICABLE TO ACL ACTIVITIES SUPPORTING TWCP.

11. HYDROGEN AND METHANE ANALYSIS

THIS TOPIC IS NOT APPLICABLE TO ACL ACTIVITIES SUPPORTING TWCP.

12. GAS VOLATILE ORGANIC COMPOUND ANALYSIS

THIS TOPIC IS NOT APPLICABLE TO ACL ACTIVITIES SUPPORTING TWCP.

13. TOTAL VOLATILE ORGANIC COMPOUND ANALYSIS

This section identifies the required QA elements for the analysis of total VOCs in samples of homogenous solids and soil/gravel.

13.1 Quality Assurance Objectives

The program data quality objectives (DQOs) for total VOC analysis are listed in Section 1.5 of this document. The QA objectives specified in Table 13-1 were developed in order to ensure the generation of total VOC data that meet the program DQOs. Key data quality indicators for laboratory measurements are defined below and the methods to qualitatively and quantitatively assess these indicators are discussed in Section 3 of this QAPjP.

13.1.1 Precision

Precision is assessed by analyzing laboratory matrix spike duplicates (MSDs), replicate analyses of laboratory control samples (LCSs) and Performance Demonstration Program (PDP) blind audit samples. Results from these measurements are compared to the criteria listed in Table 13-1. These QC measurements are used to demonstrate acceptable method performance and to trigger corrective action (CA) when specification limits are exceeded.

13.1.2 Accuracy

Accuracy as %R is assessed for the laboratory operations by analyzing LCSs, matrix spikes (MSs), surrogate compounds, and PDP blind audit samples. Results from these measurements are compared to the criteria listed in Table 13-1. These QC measurements are used to demonstrate acceptable method performance and to trigger CA when specification limits are exceeded.

13.1.3 Method Detection Limits (MDLs)

MDLs are expressed in mg/kg for VOCs. MDLs must be determined every six months, and must be less than or equal to the program-specified maximum values listed in Table 13-1. Detailed procedures for MDL determination, which are based on the method described by Glaser et al. (1981), are included in the appropriate ACMM methods.

13.1.4 Program Required Quantitation Limits (PRQLS)

The capability to quantitate analytes at or below the PRQL concentrations listed in Table 13-1 is demonstrated by semiannual checks of MDL compliance and by setting the concentration of at least one calibration standard below the PRQL for each analyte.

Table 13-1. Total volatile organic compounds target analyte list and QA objectives.

Compound	CAS Number	Precision[a] (%RDS or RPD)	Accuracy[a] (%R)	MDL (mg/kg)	PRQL (mg/kg)	Completeness (%)
Benzene	71-43-2	≤45	37–151	1	10	90
Bromoform	75-25-2	≤47	45–169	1	10	90
Carbon disulfide	75-15-0	≤50	60–150	1	10	90
Carbon tetrachloride	56-23-5	≤30	70–140	1	10	90
Chlorobenzene	108-90-7	≤38	37–160	1	10	90
Chloroform	67-66-3	≤44	51–138	1	10	90
1,2-Dichloroethane	107-06-2	≤42	49–155	1	10	90
1,1-Dichloroethylene	75-35-4	≤250	D-234[b]	1	10	90
Ethylbenzene	100-41-4	≤43	37–162	1	10	90
Methylene chloride	75-09-2	≤50	D-221[b]	1	10	90
1,1,2,2-Tetrachloroethane	79-34-5	≤55	46–157	1	10	90
Tetrachloroethylene	127-18-4	≤29	64–148	1	10	90
Toluene	108-88-3	≤29	47–150	1	10	90
1,1,1-Trichloroethane	71-55-6	≤33	52–162	1	10	90
1,1,2-Trichloroethane	79-00-5	≤38	52–150	1	10	90
Trichloroethylene	79-01-6	≤36	71–157	1	10	90
Trichlorofluoromethane	75-69-4	≤110	17–181	1	10	90
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	≤50	60–150	1	10	90
Vinyl chloride	75-01-4	≤200	D-251[b]	1	4	90
m-Xylene	108-38-3	≤50	60–150	1	10	90
o-Xylene	95-47-6	≤50	60–150	1	10	90
p-Xylene	106-42-3	≤50	60–150	1	10	90
Acetone	67-64-1	≤50	60–150	10[c]	100	90
Butanol	71-36-3	≤50	60–150	10[c]	100	90
Ethyl ether	60-29-7	≤50	60–150	10[c]	100	90
Isobutanol	78-83-1	≤50	60–150	10[c]	100	90
Methanol	67-56-1	≤50	60–150	10[c]	100	90
Methyl ethyl ketone	78-93-3	≤50	50–150	10[c]	100	90

---

a. Criteria apply to PRQL concentrations.

b. Detected; result must be greater than zero.

c. Estimate, to be determined.

MDL = method detection limit (maximum permissible value).
PRQL = program required quantitation limit; calculated from the TC level for benzene assuming a 25 g sample 0.5 L of extraction fluid, and 100 percent analyte extraction.

13.1.5 Completeness

Laboratory analysis completeness is expressed as the number of samples analyzed with valid results (see completeness definition) as a percent of the total number of samples submitted for analysis. Ninety percent of the total number of samples submitted for analysis must result in valid analytical data.

13.1.6 Comparability

The comparability of ACL data sets to those generated by different sites is achieved through the use of standardized methods and traceable standards, and by participation in the PDP.

13.1.7 Representativeness

Representativeness of total VOC analyses is achieved by use of standardized sample handling protocols (e.g., storage, aliquotting) and analysis of laboratory blanks to maintain sample integrity.

13.2 Methods Requirements

ACL uses a combination of gas chromatography (GC) and gas chromatograph mass spectrometry (GC/MS) Methods to perform analyses for total VOCs. The Analytical Chemistry methods Manual (ACMM) methods, listed below, are based on EPA SW-846 (EPA, 1986) and TWCP Methods Manual (DOE, 1996c) protocols:

- ACMM 9260, *Volatile Organic Compounds by Gas Chromatography Mass Spectrometry (GC/MS): Capillary Column Technique*: A gas chromatographic/mass spectrometric technique using purge and trap sample introduction and capillary columns, derived from EPA SW-846 Method 8260A.

- ACMM 9261, *Determination of Total Volatile Organic Compounds in Homogeneous Solids and Soil/Gravel by Gas Chromatography/Mass Spectrometry (GC/MS)*: A TWCP-specific GC/MS method, derived from EPA SW-846 Method 8260A, TWCP Method 430.4, and ACMM 9260.

- ACMM 9441, *Determination of Nonhalogenated Volatile Organic Compounds by Gas Chromatography/Flame Ionization Detector (GC/FID)*: A gas chromatographic technique using megabore capillary columns and FIDs for analysis of aqueous extracts of solid samples, derived from TWCP Method 440.2.

- ACMM 9501, *Sample Preparation of TRU Waste Characterization Samples for Organic Analysis*: Extraction methods for organic analyses, including: methanol extraction for purgeable VOCs, derived from EPA SW-846 Method 8260A and TWCP Method 430.4; aqueous extraction for nonhalogenated VOCs, derived from TWCP Method 440.2; and methylene chloride extraction for SVOCs (see Section 14).

ACL uses ACMM 9441 (GC/FID) for analysis of nonhalogenated VOCs; acetone, butanol, ethyl ether, isobutanol, methanol, and methyl ethyl ketone. ACMM 9261 (GC/MS) is used for all other VOC analytes (purgeable VOCs). ACMM 9261 refers to ACMM 9260 for portions of the procedure that are not TWCP-specific (e.g., purge and trap setup). ACMM 9261 contains all TWCP-specific procedures and QC requirements; in instances of discrepancies between ACMM 9260 and ACMM 9261 requirements, method 9261 requirements take precedent over method 9260. Appropriate extraction protocols for each analytical method are included in ACMM 9501. Detailed operating procedures for these methods, including calibration requirements, sample analysis, and data reduction, are specified in the referenced ACMM methods.

13.2.1 Criteria for Standards

Primary liquid (or gas, if appropriate) standards may be purchased from the best available source for the analytes listed in Table 13-1. Commercially-purchased primary standards must be certified by the manufacturer, and their concentrations must be traceable to the National Institute of Standards and Technology (NIST), EPA, or other nationally-recognized standards. ACL ACMM methods, SOP R.1.20, *Analytical Chemistry Quality Control Program*, and ACLP 1.01, *Preparation of Quality Control Reagents and Standards*, specify detailed preparation and documentation requirements for the preparation of all VOC standards in the laboratory.

13.2.2 Criteria for GC/MS Qualitative and Quantitative Analysis

To be qualitatively identified as an analyte by GC/MS, a sample component must elute within a relative retention time (RRT) window of ±0.06 RRT units and have a mass spectrum that corresponds to the analyte mass spectrum. RRT windows are calculated from the individual analyte retention times (RTs) in the associated continuing calibration (CCAL) standard.

ACL uses internal standards for quantitating analyte concentrations. All instrument tuning and calibration criteria specified in Section 13.4 and 13.5 must be met before performing qualitative analysis of samples. Quantitation of target analytes is based on the integrated abundance from the extracted ion current profile (EICP) of the primary ion. All analytes must be quantitated within the calibration range; multiple dilutions may be required when analyte concentrations exceed the calibration range.

Nontarget compounds are reported as tentatively identified compounds (TICs). For samples containing TICs with total ion current peaks greater than 10% of the nearest (retention time) internal standard, a search of the NIST mass spectral library is performed on the 20 TICs greatest in area count to identify the compound(s). Quantitation of a TIC assumes that the compound's calibration response factor is equal to that of the nearest internal standard. Therefore, TIC concentrations have higher associated analytical uncertainty than do target analyte concentrations.

13.2.3 Criteria for GC Qualitative and Quantitative Analysis

For GC qualitative analysis, RT windows are established for all analytes. Positive analyte identification is achieved by RT confirmation on each of two different columns. The sample component peak must fall within both RT windows for a given analyte for positive identification. RT windows are determined for both columns with each ICAL. For each analyte, windows are calculated as the mean RT of the ICAL standards plus or minus 5%. RT windows are determined for all analytes on each GC column before the analysis of any samples, whenever a new ICAL is performed, or whenever a new GC column is installed.

GC analytes are quantitated against external standards. Quantitation of a given analyte is performed on one of the two columns; the column used for quantitation must be interferant-free in the RT window corresponding to the analyte. All analytes must be quantitated within the calibration range; multiple dilutions may be required when analyte concentrations exceed the calibration range.

13.3 Quality Control

To ensure that data of known and documented quality are generated, the QC criteria specified in this section must be met for all total VOC analyses. The ALD QAO is responsible for monitoring and documenting procedure performance, including the analysis of laboratory control samples, laboratory blanks, matrix spikes and matrix spike duplicates. The ALD QAO, the organic section supervisor, and the organic analysis laboratory TL are responsible for implementing CAs when acceptable procedure performance, as specified in this section, is not met.

Specific QC samples and frequencies are summarized in Table 13-2. Analytical QC samples are associated with field samples through the use of analytical batches (see Section 6.3). Trip blanks are analyzed with the samples associated with them, but do not count towards the size of the analytical batch.

13.3.1 Method Performance Samples

Before the analysis of any samples for TWCP, acceptable method performance must be demonstrated for each method to be used. This demonstration consists of determination of MDLs (see Section 13.1.3), and analysis of method performance samples (MPSs). Initially, seven replicate MPSs (standards containing known concentrations of all analytes) must be analyzed to demonstrate that the criteria specified for precision and accuracy listed in Table 13-1 can be met. If the seven replicates do not meet the criteria, then seven more replicates must be analyzed until the initial procedure performance demonstration criteria are met.

Continuing acceptable procedure performance is demonstrated semiannually by analyzing four replicate MPSs. If the precision and accuracy criteria specified in Table 13-1 are not met for the four replicates, four additional replicates must be analyzed until the criteria in Table 13-1 are met.

13.3.2 Laboratory Control Samples (LCSs)

LCSs are analyzed at a minimum frequency of one per analytical batch. A single LCS may not be shared between two separate analytical batches. LCSs are prepared from commercially purchased primary standards that are independent (i.e., different manufacturer) from those used for instrument calibration. For GC/MS analysis, the LCS must contain at least 10 of the purgeable VOC target analytes listed in Table 13-1. For GC analysis, the LCS must contain all nonhalogenated VOC target analytes. LCSs for purgeable VOCs are made in methanol, and those for NH-VOCs are made in

Table 13-2. Summary of laboratory QC samples and frequencies for total VOC analysis.

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective action
Method performance samples	Seven (7) samples initially and four (4) semiannually	Meet Table 13-1 QA objectives	Repeat until acceptable
Laboratory blanks	One (1) per analytical batch	Analyte concentrations $< 3 \times$ MDL	See Section 13.3.3
Laboratory control samples	One (1) per analytical batch	$80\% \leq \%R \leq 120\%$	See Section 13.3.2
Matrix spikes	One (1) per analytical batch	Meet Table 13-1 %Rs	See Section 13.3.4
Matrix spike duplicates	One (1) per analytical batch	Meet Table 13-1 %Rs and RPDs	See Section 13.3.4
Surrogate compounds	Each analytical sample	Average %R from minimum of 30 samples for a given matrix $\pm$ 3 standard deviations	See Section 13.3.5
Blind audit samples	Samples and frequency controlled by the Solid PDP Plan	Specified in the Solid PDP Plan	Specified in the Solid PDP Plan

water. LCSs are not carried through the sample preparation procedure performed on field samples because the solid extraction procedures are incompatible with the liquid LCSs.

LCS results are acceptable if the recoveries for all analytes are between 80 and 120% (i.e., $80\% \leq \%R \leq 120\%$). If LCS results do not meet this criteria, the LCS may be reanalyzed once; if the results of the rerun LCS meet specifications, sample analysis may continue. If the rerun LCS is still noncompliant, an NCR may be required. CA is required to identify and correct the cause of the nonconformance. Associated samples may require reanalysis, depending on the degree of the indicated bias and the magnitudes of analyte concentrations in the samples. If a noncompliant LCS is associated with any sample data reported to the SPO, an NCR must be initiated.

13.3.3 Laboratory Blanks (LBs)

LBs are analyzed at a minimum frequency of one per analytical batch. The LBs must undergo all sample preparation procedures (i.e., methanol or water extraction) performed on the associated field samples. LBs are acceptable if analyte concentrations are less than three times the Table 13-1 MDLs for all target analytes. If the LB is not acceptable, an NCR may be required. CA is required to identify and correct the cause of the nonconformance. Associated samples may require reanalysis, depending on whether the source of contamination can be identified, the degree of the indicated bias and the magnitudes of analyte concentrations in the samples. If a noncompliant LB is associated with any sample data reported to the SPO, an NCR must be initiated.

13.3.4 Matrix Spikes (MSs) and Matrix Spike Duplicates (MSDs)

Duplicate matrix spikes on individual field samples are performed at the minimum frequency of one pair (MS plus MSD) per analysis batch. For GC/MS analysis, the MS and MSD must contain at least five of the purgeable VOC target analytes listed in Table 13-1. The MS and MSD for GC/FID analysis must contain all of the nonhalogenated VOC target analytes listed in Table 13-1.

Matrix spike and matrix spike duplicate results are acceptable if the Table 13-1 criteria for accuracy and precision are met, i.e., if percent recoveries (for both MS and MSD) meet the accuracy criteria and the RPDs between the MS and MSD results meet the precision criteria.

If MSs and MSDs do not meet the precision and accuracy criteria, the noncompliance is documented on checklists and in the data report narrative. NCRs are not initiated for noncompliant MS and MSD results because these results are highly dependent upon individual sample matrices, and specific corrective actions cannot be identified.

13.3.5 Surrogate Compounds

Surrogate compounds are added to each field sample and laboratory QC sample. The choice of surrogates is site-specific. Surrogates used by ACL are identified in ACMM 9261. If surrogate percent recoveries do not meet the criteria specified in Table 13-2, the noncompliance is documented in checklists and the data report narrative. NCRs are not initiated for noncompliant surrogate recoveries because these results are highly dependent upon individual sample matrices, and specific corrective actions cannot be identified.

13.3.6 Blind Audit Samples

ACL participates in the RCRA solid PDP as specified by program requirements (see Section 2.7) on a nominal semiannual basis. PDP samples are analyzed and reported using the same methods and handling procedures as are used on field samples.

13.4 Instrument Testing, Inspection, and Maintenance Requirements

13.4.1 GC/MS Instrument Requirements

ACL uses ion trap GC/MSs to analyze for purgeable VOCs in support of TWCP. Ion trap instruments are more sensitive than traditional quadrupole mass spectrometers, and thus are better suited to analysis of radioactively-contaminated samples because smaller sample aliquots can be analyzed. The GC/MSs are operated in full scan mode to allow the detection and quantitation of all target analytes listed in Table 13-1, and identification of nontarget compounds. The GC/MS is equipped with a purge and trap (P&T) sample introduction system.

Before the analysis of any samples, the GC/MS system must meet 4-bromofluorobenzene (BFB) tuning criteria specified in Table 13-3. The tuning criteria specified in Table 13-3 must be met at the beginning of each 12 hours of operation and before the analysis of any standard or samples by analyzing 50 ng of BFB.

13.4.2 GC Instrument Requirements

The GCs used for nonhalogenated VOC analysis of TWCP samples meet configured with duel FIDs and are equipped with two dissimilar megabore capillary columns.

13.5 Instrument Calibration and Frequency

The GC/MS and GC calibration requirements are summarized in Table 13-4. Detailed instructions for calibrating the GC, GC/MS, and related equipment are provided in specific ACMM methods. ACL maintains instrument use logs from which calibration sequences and frequencies may be reconstructed.

13.5.1 GC/MS Calibration

GC/MS Initial Calibration. After instrument performance criteria (i.e., BFB tune criteria) have been satisfied, a multipoint internal standard calibration is performed. The multipoint calibration consists of a minimum of five analytical standards that define the calibration range of the instrument for the purgeable target analytes. One of the standards must be at concentrations less than the PRQLs

Table 13-3. 4-bromofluorobenzene key ions and abundance criteria.

Mass	Intensity required (relative abundance)[a]
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	Base peak, 100% relative abundance
96	5 to 9% of mass 95
173	< 2% of mass 174
174	> 50% of mass 95
175	5 to 9% of mass 174
176	> 95%, but < 101% of mass 174
177	5 to 9% of mass 176

---

a. SW-846 Method 8260A.

Table 13-4. GC/MS and GC/FID calibration requirements for total VOC analyses.

Technique	Procedure	Frequency of Procedure	Acceptance criteria
GC/MS	BFB tune	Every 12 hours	Table 13-3 criteria met
	Five-point initial calibration	Initially and as needed	RRF %RSD for CCCs ≤ 30; RRF for SPCCs ≥ 0.30[a]; per analyte, average RRF is used if %RSD ≤15; linear or quadratic regression equation is generated if %RSD > 15
	Continuing calibration	Every 12 hours	RRF or concentration %D for CCCs ≤ 20; RRF for SPCCs ≥ 0.30[a]; RT for internal standards must be ± 30 seconds from the last CCAL; ISA count must be > 50% and < 200% of the area counts from the last daily calibration check; surrogate compound %R must meet Table 13-2 criteria.
GC/FID	Three-point initial calibration	Initially and as needed	Correlation coefficient r ≥ 0.93 (calibration curves) or %RSD for RFs < 35 for all analytes
	Continuing calibration	Every 12 hours	RF or measured concentration %D for all analytes ≤ 15 of ICAL; RTs within ICAL RT windows.

---

a. Bromoform ≥ 0.25.

of the purgeable target analytes (see Table 13-1). Calibration check compounds (CCCs) and system performance check compounds (SPCCs) used are that are common to Table 13-1 and SW-846 Method 8260A.

Relative response factors (RRFs) are generated for each specified target analyte. For the initial five-point calibration to be valid, the %RSD for the RRFs of each CCC must be less than or equal to 30% and the average RRF for each SPCC must be greater than or equal to 0.300 (0.250 for bromoform). Average RRFs for each analyte are used for quantitation if the %RSD is less than or equal to 15%. If the average RRF %RSD for any analyte is greater than 15%, then a linear or quadratic regression equation is used for quantitation of that analyte.

ACMM 9261 specifies calibration procedures for the GC/MS analysis method. A valid ICAL must exist before any samples analyses are performed. A new ICAL is required if there is a change in the instrument that may affect the analytical results or if indicated as a CA.

GC/MS Continuing Calibration. The initial GC/MS calibration (i.e., the ICAL) is verified using a continuing calibration (CCAL) standard. The CCAL standard must contain all target analytes at concentrations near the midpoint of the calibration range. The CCAL standard is analyzed at the beginning of each 12 hours of operation and after an acceptable BFB tune. For the CCAL to be valid, it must meet all of the daily calibration criteria for surrogate compound recovery, SPCCs, CCCs, internal standard area count criteria and RTs, as specified in Table 13-4, per SW-846 Method 8260A.

If the CCAL does not satisfy the calibration requirement, the CCAL standard may be remade and rerun once to eliminate CCAL standard preparation as the source of error. If the rerun CCAL still does not meet criteria, a new five-point ICAL must be generated. Sample analysis cannot proceed until the GC/MS system has satisfied the calibration requirements.

13.5.2 GC Calibration

GC Initial Calibration. A multipoint external standard calibration is performed using a minimum of three standards. The calibration range must not exceed the linear range of the instrument for the nonhalogenated VOC target analytes. One of the standards must be at concentrations less than the PRQLs for nonhalogenated target analytes (see Table 13-1). ACMM 9441 describes the procedures used to calibrate the GC and to determine RT windows.

ACL uses a linear regression to construct the calibration plot. The correlation coefficient for the regression must be ≥ 0.93 for the ICAL to be valid. A valid ICAL must exist before any samples are analyzed. A new ICAL is required if there is a change in instrument conditions that may affect the analytical results or if indicated as a CA.

GC Continuing Calibration. At the beginning of each 12 hours of operation, and prior to sample analysis, a continuing calibration verification (CCV) standard is analyzed. The CCV standard must contain all analytes in concentrations that are near the midpoint in the calibration range. For the CCV to be valid, the %R of all analytes in the CCV must be between 85% and 115% and the RTs must be within the ICAL RT windows. If the CCV does not meet these requirements, the standard may be remade and rerun once to eliminate CCV standard preparation as a source of error. If the rerun CCV does not meet criteria, a new ICAL curve must be generated. Sample analysis cannot proceed until the GC system has satisfied calibration and RT requirements.

13.6 Data Management

Data management includes requirements for data reduction, data validation, and reporting. All of the data management requirements defined in Section 3 of this QAPjP, as well as the specific procedures described below, apply to total VOC analysis data. Specific equations and sample calculations are detailed in the associated ACL ACMM methods.

13.6.1 Data Reduction

All results for field samples are reported in units of mg/kg on a weight/wet-weight basis and are limited to two significant figures. All trip blank results are reported in units of µg/ℓ and are limited to two significant figures. All calculations (raw data reduction and QC results) are performed prior to rounding.

Target compound concentrations are not blank corrected. Blanks are treated and reported in the same manner as other samples.

Library searches of the NIST mass spectral database are performed for the identification of unknown peaks with a total ion current area greater than 10% of the nearest (retention time) internal standard. Compounds identified by forward library searching are reported as TICs. Concentrations for TICs are calculated assuming a relative response factor equal to one using the nearest internal standard.

13.6.2 Data Validation

All total VOC data are reviewed by an independent technical reviewer prior to report generation. Review checklists are used to document the independent technical review process. For GC/MS (purgeable VOCs) data review, an example checklist is provided in Figure 13-1. An example of the independent technical review checklists for GC (nonhalogenated VOCs) data is provided in Figure 13-2.

13.6.3 Data Reporting

GC/MS VOC Reporting Requirements. Data reports for GC/MS total VOC data consist of a cover page and five sections:

- **Cover page**—The cover page includes the laboratory name, the data report number, the report date, the report table of contents, and release authorization signatures. An example cover page is provided in Figure 13-3.

- **Section 1: Sample Identification Table**—This section includes the cross-reference between field and laboratory sample identification numbers (example provided in Figure 13-4).

- **Section 2: Sample Custody Documents**—This section includes copies of the field COC form(s) that accompanied the samples to the laboratory (see example provided in Figures 6-1).

- **Section 3: Analysis Results**—This section includes the analytical batch narrative, containing information pertinent to program-level review, and the analysis data sheets (see example in Figure 13-5) for each sample included in the data report. A separate analysis data sheet is provided for each sample, and contains the following information: laboratory name, program name, data report number, analytical batch number, sampling batch

TWCP GCMS VOC INDEPENDENT DATA REVIEW CHECKLIST

Analytical Batch:	Method: 430.4		Form Revision: 0	
Run # and Analysis Date	Analyst	Independent Reviewer	Date of Review	OK for Release?
1:				Yes or No
2:				Yes or No
3:				Yes or No
4:				Yes or No
5:				Yes or No

Instructions: Complete one checklist per Analysis Batch. If more than five instrument runs were needed to complete the batch analysis, use a second checklist. Enter appropriate response for each question. Each "No" response requires explanation. A "No" response to a question in **Bold type** may require initiation of an NCR.

REQUIREMENT	Run #1		Run #2		Run #3		Run #4		Run #5		COMMENTS
	YES	NO	YES	NO	YES	NO	YES	NO	YES	NO	
1. INITIAL CALIBRATION (ICAL)											
a. **Does the BFB tune meet the TWCP QAPP requirements [Table 13-3]?**											
b. **Are the relative response factor %RSDs for CCCs < 30?**											
c. **Are the response factors RSDs for SPCCs >0.30 (> 0.25 for Bromoform)?**											
d. **Was an average RRF used for %RSDs which were ≤ 15, and a regression equation generated if the %RSD was >15?**											
e. Is the ICAL raw data signed by the analyst and included in the file, or referenced, as appropriate for the report?											
2. BFB TUNE											
a. **Was a BFB tune performed each day prior to analysis of samples?**											
b. Is the raw data signed by the analyst and present in the file for each BFB tune?											
c. **Do all BFB tunes meet the TWCP QAPP requirements [Table 13-3]?**											
3. CONTINUING CALIBRATION (CCAL)											
a. **Was a CCAL performed each day after the BFB tune and prior to analysis of samples?**											
b. Is the raw data signed by the analyst and present in the file for each CCAL?											
c. **Is the response factor or concentration %D for the CCCs ≤ 20 for all analytes?**											
d. **Are the response factors for SPCCs >0.30 (> 0.25 for Bromoform)?**											
e. Are the retention times of the internal standards within 30 seconds of those in the last CCAL or ICAL?											
f. **Are the internal standard areas within 50 to 200% of those in the last CCAL or ICAL?**											
g. Do the surrogate recoveries meet the TWCP QAPP requirements [Table 13-2]?											
4. BLANKS											
a. **Was at least one laboratory blank extracted and analyzed with the analytical batch?**											
b. **Was a method blank analyzed daily prior to analysis of samples?**											

Page 1 of 2

Figure 13-1. Example of independent technical review checklist for total purgeable VOC analysis.

TWCP GCMS VOC INDEPENDENT DATA REVIEW FOR BATCH # ____________

REQUIREMENT	Run #1		Run #2		Run #3		Run #4		Run #5		COMMENTS
	YES	NO	YES	NO	YES	NO	YES	NO	YES	NO	
b. Do the recoveries for all analytes in each LCS fall between 80% and 120% [Table 13-2]?											
5. LABORATORY CONTROL SAMPLES (LCSs)											
a. Was at least one unique LCS analyzed with the analytical batch?											
b. Doe the recoveries for all analytes in each LCS fall between 80% and 120% [Table 13-2]?											
6. DUPLICATE SAMPLE											
a. Was at least one field sample from the analytical batch analyzed in duplicate, or as a matrix spike duplicate?											
b. Do the RPDs meet the TWCP QAPP precision requirements [Table 13-2]?											
7.MATRIX SPIKE SAMPLE											
a. Was at least one field sample from the analytical batch analyzed as a matrix spike?											
b. Do the %Rs for all MS and MSDs meet the TWCP QAPP requirements [Table 13-2]?											
8. SAMPLES (INCLUDING LCSs, BLANKS, DUPLICATES AND SPIKES)											
a. Are the raw data signed by the analyst and present in the file for all samples listed on the Analysis Request Form, and for all associated LCSs, blanks, duplicates, and spikes?											
b. Are the internal standard areas within 50 to 200% of those in the associated CCAL?											
c. Were all samples analyzed within 12 hours of the daily BFB tune?											
d. In your opinion, do the sample spectra for positive hits match the reference spectra?											
e. Was a TIC search conducted on each sample, blank and LCS in the batch?											
f. Were the TICs reviewed, initialed, and dated?											
g. Were all samples having analytes detected in amounts exceeding the calibration range, reanalyzed on a dilution or at a lower volume?											
h. Were all samples and any associated reanalyses or dilutions analyzed within 14 days of sampling if analyzed directly, or if extracted, prepared within 14 days and analyzed within 40 days?											

Additional Comments:

Figure 13-1. (continued).

TWCP GC 440.2 INDEPENDENT DATA REVIEW CHECKLIST

Analytical Batch:	Method 440.2: NH-VOCs by GC	Form Revision: 0

Run # and Analysis Date	Analyst	Independent Reviewer	Date of Review	OK for Release?
1:				Yes or No
2:				Yes or No
3:				Yes or No

Instructions: Complete one checklist per Analysis Batch. If more than 3 instrument runs were needed to complete the batch analysis, use a second checklist. Enter appropriate response for each question. Each "No" response requires explanation. A "No" response to a question in **Bold type** may require initiation of an NCR.

REQUIREMENT	Run #1		Run #2		Run #3		COMMENTS
	YES	NO	YES	NO	YES	NO	
1. INITIAL CALIBRATION (ICAL)							
a. **Is there an ICAL associated with all sample analyses in the analytical batch?**							
b. **Are the correlation coefficient(s) > 0.93 [Table 13-3]?**							
2. CONTINUING CALIBRATION VERIFICATION (CCV)							
a. **Was a CCV analyzed each day prior to analysis of samples?**							
b. Is the raw data signed by the analyst and present in the file for each CCV?							
c. **Is the measured concentration percent difference (%D) for each analyte <15% [Table 13-3]?**							
d. Are the retention times within the most recently established window?							
e. Are Analysis Data Sheets (Form 1), Analyte Identification Sheets (Form 12), and CCV Forms (Form 7) present for all CCVs associated with the run?							
3. LABORATORY BLANKS							
a. **Was at least one laboratory blank extracted and analyzed with the analytical batch?**							
b. **Are all laboratory blank results less than 3 times the MDL for all analytes [Table 13-2]?**							
c. Are Analysis Data Sheets (Form 1), Analyte Identification Sheets (Form 12), and Blank Forms (Form 4A and 4B) present for all lab blanks associated with the run?							
4. LABORATORY CONTROL SAMPLES (LCSs)							
a. **Was at least one unique LCS analyzed with the analysis batch?**							
b. **Do the recoveries for all analytes in each LCS fall between 80% and 120% [Table 13-2]?**							
c. Are Analysis Data Sheets (Form 1), Analyte Identification Sheets (Form12) and LCS Analysis Forms (Form 9) present for all LCSs analyzed with the run?							

Rev. 6/96

Page 1 of 2

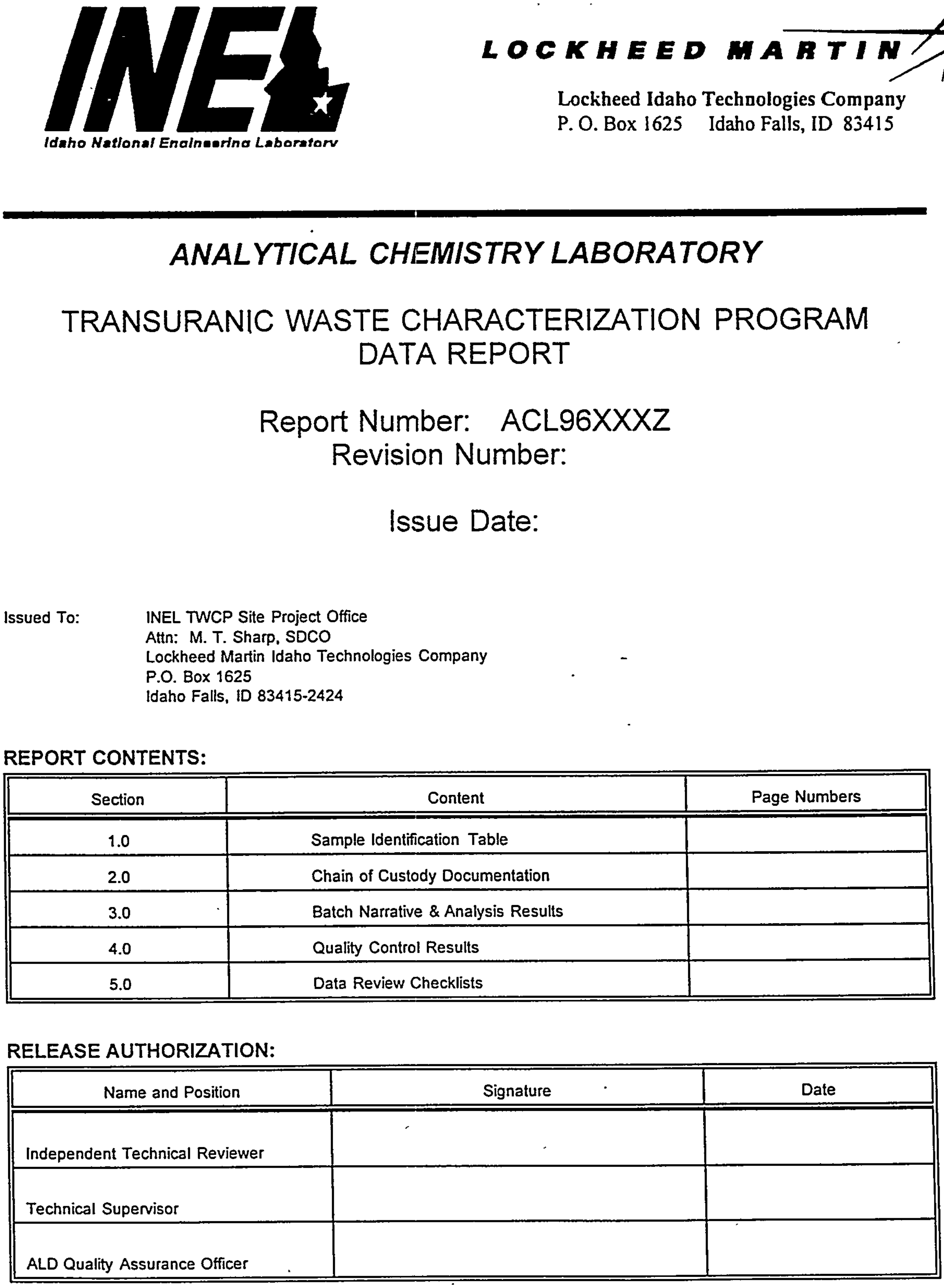
Figure 13-2. Example of independent technical review checklist for total nonhalogenated VOC analysis.

TWCP GC 440.1 INDEPENDENT DATA REVIEW CHECKLIST

Analysis Batch # ______________________ (cont'd)

REQUIREMENT	Run #1		Run #2		Run #3		COMMENTS
	YES	NO	YES	NO	YES	NO	
5. DUPLICATE SAMPLE							
a. Was at least one field sample from the analytical batch analyzed in duplicate, or as a matrix spike duplicate?							
b Do the RPDs for all analytes meet the TWCP QAPP requirements [Table 13-2]?							
c. Are Analysis Data Sheets (Form 1), Analyte Identification Sheets (Form 12), and/or Laboratory Duplicate Forms (Form 11), Matrix Spike Duplicate Forms (Form 3) present for all LCS and/or sample duplicates analyzed with the run?							
6. MATRIX SPIKE SAMPLE							
a. Was at least one field sample from the analytical batch analyzed as a matrix spike?							
b Do the %Rs for all MS and MSDs meet the TWCP QAPP requirements [Table 13-2]?							
6. SAMPLES (INCLUDING LCSs, BLANKS, DUPLICATES, AND SPIKES)							
a. Are the raw data signed and dated by the analyst and present in the file for all samples listed on the Analysis Request From, and for all associated LCSs, blanks, duplicates, and spikes?							
b. Were all samples analyzed within 12 hours of the daily CCV?							
c Are all reported hits within the RT windows of the column used for quantitation?							
d Are all reported hits quantitated from the quant column, and confirmed by similar chromatography and quantitation from a confirmation column?							
e Are all analytes showing discrepancies between columns 1 and 2 (interferences) quantitated from the column with the least interference?							
f Are analyte retention time windows checked for multiple peaks falling within the RT windows?							
g. Have peak baselines been checked for correct integration?							
h. Were the appropriate quantitation routines used for each column?							
i. Are all chromatograms consistent with their associated External Standard Report?							
j Are peak areas proportionally consistent with those of the CCV?							
k. Were all samples having analytes detected in amounts exceeding the calibration range reanalyzed on a dilution?							
l. Were all samples and any associated dilutions or reanalyses analyzed within 14 days of sampling?							
7. Reporting Forms							
a. Are all forms completely and correctly filled out (e.g., reported concentrations match External Standard Reports, Form 1 values correctly rounded, batch number is correct, sample IDs consistent with Analysis Request Form, file IDs and analysis date/time consistent with raw data)?							
b Is the correct quantitation column identified on all Form 5s?							
c Does a note regarding interferences appear on Form 1s for all samples having analytes with questionable confirmation or quantitation?							
d Are all values less than the MDL reported as the MDL adjusted for dilution and flagged with a "U" qualifier?							
e. Are all values ≥ MDL and < PRQL (dilution corrected) reported with a "J" qualifier?							

Figure 13-2. (continued).



INEL
Idaho National Engineering Laboratory

LOCKHEED MARTIN
Lockheed Idaho Technologies Company
P. O. Box 1625 Idaho Falls, ID 83415

ANALYTICAL CHEMISTRY LABORATORY

TRANSURANIC WASTE CHARACTERIZATION PROGRAM DATA REPORT

Report Number: ACL96XXXZ
Revision Number:

Issue Date:

Issued To:
INEL TWCP Site Project Office
Attn: M. T. Sharp, SDCO
Lockheed Martin Idaho Technologies Company
P.O. Box 1625
Idaho Falls, ID 83415-2424

REPORT CONTENTS:

Section	Content	Page Numbers
1.0	Sample Identification Table	
2.0	Chain of Custody Documentation	
3.0	Batch Narrative & Analysis Results	
4.0	Quality Control Results	
5.0	Data Review Checklists	

RELEASE AUTHORIZATION:

Name and Position	Signature	Date
Independent Technical Reviewer		
Technical Supervisor		
ALD Quality Assurance Officer		

Figure 13-3. Example of ACL data report cover page

ANALYTICAL CHEMISTRY LABORATORY

TRANSURANIC WASTE CHARACTERIZATION PROGRAM SAMPLE IDENTIFICATION TABLE

DATA REPORT NUMBER: ACL96XXXO

FIELD SAMPLE ID	LAB SAMPLE ID	ANALYTICAL BATCH#	VOC	NH-VOC	SVOC	METALS	NOT USED
ID000001101V1	6ZZ01	9601011	✓				
ID000001101V2	6ZZ01	9601011		✓			
ID000001101V3	6ZZ01MS	9601011	✓				
ID000001101V4	6ZZ01MSD	9601011	✓				
ID000001102V1	6ZZ02	9601011	✓				
ID000001102V2	6ZZ02	9601011		✓			
ID000001102V3	6ZZ02MS	9601011		✓			
ID000001102V4	6ZZ02MSD	9601011		✓			
ID00000110CM1	6ZZ03	9601011			✓		
ID000002101V1	6ZZ56	9601011	✓				
ID000002101V2	6ZZ56	9601011		✓			
ID000002101V3	6ZZ56	N/A					✓
ID000002101V4	6ZZ56	N/A					✓
ID000002102V1	6ZZ57	9601011	✓				
ID000002102V2	6ZZ57	9601011		✓			
ID000002102V3	6ZZ57	N/A					✓
ID000002102V4	6ZZ57	N/A					✓
ID00000210CM1	6ZZ58	9601011			✓		

Figure 13-4. Example of sample identification table

TOTAL VOCS ANALYSIS DATA SHEET

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES COMPANY
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Sampling Batch No: ____________ Analytical Batch No: ____________

Field Sample ID: ____________ Lab Sample ID: ____________

Date Sampled: ____________ Date Received: ____________

Date Extracted: ____________ Date Analyzed: ____________

Data Report No: ____________

Method Number: ____________

COMPOUND	CONCENTRATION (mg/kg)	Q
Benzene		
Bromoform		
Carbon disulfide		
Carbon tetrachloride		
Chlorobenzene		
Chloroform		
1,2-Dichloroethane		
1,1-Dichloroethylene		
Ethylbenzene		
Methylene chloride		
1,1,2,2-Tetrachloroethane		
Tetrachloroethylene		
Toluene		
1,1,1-Trichloroethane		
1,1,2-Trichloroethane		
Trichloroethylene		
Trichlorofluoromethane		
1,1,2-Trichloro-1,2,2-trifluoroethane		
Vinyl chloride		
m-xylene and p-xylene		
o-xylene		

FORM I VOC Rev 6/96

Figure 13-5. Example of total purgeable VOC analysis data sheet.

number, field sample ID, laboratory sample ID, date sampled, date received at the laboratory, date extracted (if applicable), date analyzed, method number, listing of program analytes, and analytical results in mg/kg. Data qualifying flags are used as follows:

- B: Analyte detected in associated LB
- E: Reported analyte concentration exceeds the calibration range
- D: Reported analyte concentration is from a secondary dilution or reduced analysis aliquot of the sample
- J: Analyte concentration is <PRQL but ≥MDL
- U: Analyte was undetected (reported as sample-specific MDL)
- Z: Estimated concentration; one or more QC sample results are outside the acceptance criteria
- N: Indicates presumptive evidence of a compound, based on a mass spectral library search (TICs only).

TICs are reported on forms equivalent to those used for target analytes (see example in Figure 13-6). For TICs, estimated concentrations are reported along with RTs. Reported concentrations for identified TICs are always qualified with a J flag to indicate that the reported value has high analytical uncertainty. Estimated concentrations for TICs labeled as "unknowns" are reported by assuming an RRF of 1 relative to the nearest internal standard.

- **Section 4: Batch Related QC Samples**—This section contains forms reporting results of LCSs (see example in Figure 13-7), matrix spike and matrix spike duplicate recoveries and RPDs (see example in Figure 13-8) and surrogate spike recoveries (see example in Figure 13-9).

- **Section 5: Data Review Checklists**—This section includes the TWCP Sample Receiving & Custody Review Checklist (see example in Figure 6-2), the ACMM Method 9261 Independent Data Review Checklist (see example in Figure 13-1), and the Quality Assurance Data Review Checklist for ACMM 9261 (see example in Figure 3-1). Copies of applicable NCRs are also included in this section, as necessary.

The following items are retained in ACL files, but are not included in the data reports sent to the SPO:

- **Data package** filed by data report number, with all raw data, including sample preparation logs, standard preparation logs, original instrument readouts for all tunes, CCALs, samples and QC samples, calculation records, results of all associated QC samples and measurements [LBs, method (instrument) blanks, CCALs, LCSs, MSs, MSDs, surrogates,

TOTAL VOCS ANALYSIS DATA SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES COMPANY

ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Sampling Batch No: ____________ Analytical Batch No: ____________

Field Sample ID: ____________ Lab Sample ID: ____________

Date Sampled: ____________ Date Received: ____________

Date Extracted: ____________ Date Analyzed: ____________

Number of TICs found ____________ Data Report No: ____________

Method Number: ____________

Tentatively Identified VOCs		CONCENTRATION (mg/kg)	Q	Retention Time (minutes)
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				

FORM I- TIC VOC Rev 6/96

Figure 13-6. Example of total purgeable VOC TIC Analysis Data Sheet.

TOTAL VOCS ANALYSIS
LABORATORY CONTROL SAMPLE FORM

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES COMPANY
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Data Report No: ______________

Analytical Batch No: ______________

Lab Sample ID: ______________

Lab File ID: ______________

Date Analyzed: ______________

Method Number: ______________

COMPOUND	MEASURED CONCENTRATION (µg/l)	KNOWN CONCENTRATION (µg/l)	RECOVERY (%)
Carbon tetrachloride			
Chlorobenzene			
Chloroform			
1,2-Dichloroethane			
Ethylbenzene			
Tetrachloroethylene			
Toluene			
1,1,1-Trichloroethane			
m-xylene and p-xylene			
o-xylene			

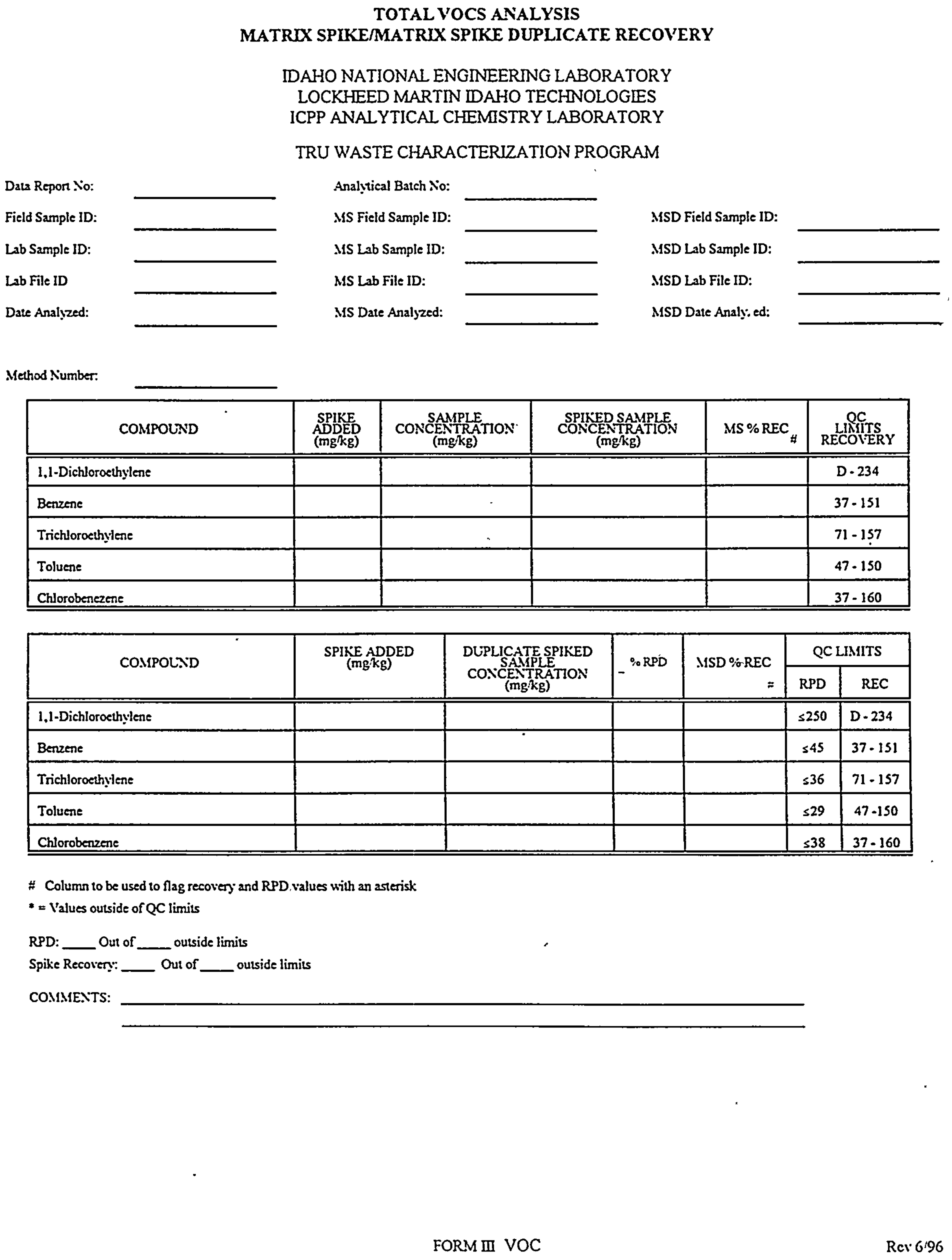
NA = Not Applicable

QC LIMITS
80-120 %

FORM IX VOC

Rev. 6/96

Figure 13-7. Example of LCS reporting form for total purgeable VOC analysis.



TOTAL VOCS ANALYSIS
MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Data Report No:	______	Analytical Batch No:	______		
Field Sample ID:	______	MS Field Sample ID:	______	MSD Field Sample ID:	______
Lab Sample ID:	______	MS Lab Sample ID:	______	MSD Lab Sample ID:	______
Lab File ID	______	MS Lab File ID:	______	MSD Lab File ID:	______
Date Analyzed:	______	MS Date Analyzed:	______	MSD Date Analyzed:	______

Method Number: ______

COMPOUND	SPIKE ADDED (mg/kg)	SAMPLE CONCENTRATION (mg/kg)	SPIKED SAMPLE CONCENTRATION (mg/kg)	MS % REC #	QC LIMITS RECOVERY
1,1-Dichloroethylene					D - 234
Benzene					37 - 151
Trichloroethylene					71 - 157
Toluene					47 - 150
Chlorobenezene					37 - 160

COMPOUND	SPIKE ADDED (mg/kg)	DUPLICATE SPIKED SAMPLE CONCENTRATION (mg/kg)	% RPD	MSD % REC #	QC LIMITS	
					RPD	REC
1,1-Dichloroethylene					≤250	D - 234
Benzene					≤45	37 - 151
Trichloroethylene					≤36	71 - 157
Toluene					≤29	47 -150
Chlorobenzene					≤38	37 - 160

\# Column to be used to flag recovery and RPD values with an asterisk
* = Values outside of QC limits

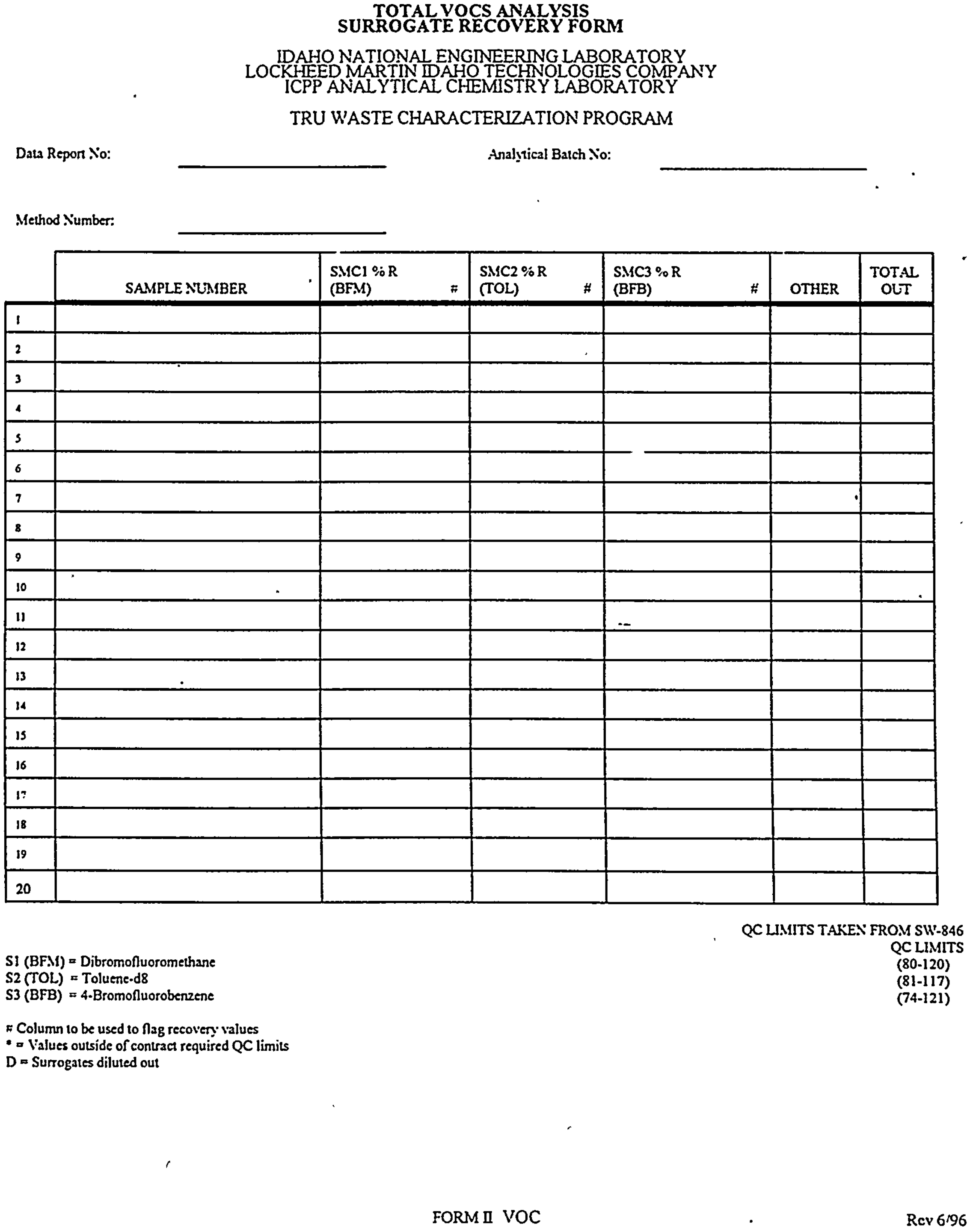
RPD: _____ Out of _____ outside limits
Spike Recovery: _____ Out of _____ outside limits

COMMENTS: ______________________________

FORM III VOC

Rev 6/96

Figure 13-8. Example of matrix spike/matrix spike duplicate reporting form for total purgeable VOC analysis.



TOTAL VOCS ANALYSIS
SURROGATE RECOVERY FORM

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES COMPANY
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Data Report No: ____________ Analytical Batch No: ____________

Method Number: ____________

	SAMPLE NUMBER	SMC1 % R (BFM) #	SMC2 % R (TOL) #	SMC3 % R (BFB) #	OTHER	TOTAL OUT
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						

QC LIMITS TAKEN FROM SW-846

	QC LIMITS
S1 (BFM) = Dibromofluoromethane	(80-120)
S2 (TOL) = Toluene-d8	(81-117)
S3 (BFB) = 4-Bromofluorobenzene	(74-121)

Column to be used to flag recovery values
* = Values outside of contract required QC limits
D = Surrogates diluted out

FORM II VOC Rev 6/96

Figure 13-9. Example of surrogate spike reporting form for total purgeable VOC analysis.

and internal standard areas], and reference to the associated ICAL. Examples of forms providing blank results, LB summaries, method blank summaries, BFB tune results and internal standard area summaries are given in Figures 13-10, 13-11, 13-12, 13-13, and Figure 13-14, respectively. Examples of forms reporting results of CCALs are provided in Figures 13-15 and 13-16.

- **ACMM 9261 Calibration Records**, filed under Calibration Records according to method, instrument, and ICAL number, which include standard preparation logs for all calibration standards (source reference), raw data for the ICAL and associated BFB tune, method identification, calibration date and time, %RSD calculations, and report forms providing results of the ICAL (see example in Figure 13-17).

- **MDL records**, filed by MDL determination date and instrument identification, which include all raw data and calculations for MDL determinations, along with the MDL reporting form (see example in Figure 13-18).

- **Original COC forms**, filed in the data package.

GC VOC Reporting Requirements. Data reports for GC total VOC data consist of a cover page and five sections:

- **Cover page**—The cover page includes the laboratory name, the data report number, the report date, the report table of contents, and release authorization signatures. The cover page is similar to the example provided for GC/MS total VOCs analysis in Figure 13-3.

- **Section 1: Sample Identification Table**—This section includes the cross reference between field and laboratory sample identification numbers (example provided in Figure 13-4).

- **Section 2: Sample Custody Documents**—This section includes copies of the field COC form(s) that accompanied the samples to the laboratory (see example provided in Figure 6-1).

- **Section 3: Analysis Results**—This section includes the analytical batch narrative, which contains information pertinent to program-level review, and the analysis data sheets (see example in Figure 13-19) for each sample included in the data report. A separate analysis data sheet is provided for each sample, and contains the following information: laboratory name, program name, data report number, analytical batch number, sampling batch number, field sample ID, laboratory sample ID, date sampled, date received at the laboratory, date extracted, date analyzed, method number, listing of program analytes, and analytical results in mg/kg. Data qualifying flags are used as follows:

    - B: Analyte detected in associated LB

    - E: Reported analyte concentration exceeds the calibration range

TOTAL VOCS ANALYSIS
BLANK FORM

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES COMPANY
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Data Report No: ____________ Analytical Batch No: ____________
Lab Sample ID: ____________ Lab File ID: ____________
Date Extracted: ____________ Date Analyzed: ____________

Method Number: ____________

COMPOUND	CONCENTRATION (mg/kg)	Q
Benzene		
Bromoform		
Carbon disulfide		
Carbon tetrachloride		
Chlorobenzene		
Chloroform		
1,2-Dichloroethane		
1,1-Dichloroethylene		
Ethylbenzene		
Methylene chloride		
1,1,2,2-Tetrachloroethane		
Tetrachloroethylene		
Toluene		
1,1,1-Trichloroethane		
1,1,2-Trichloroethane		
Trichloroethylene		
Trichlorofluoromethane		
1,1,2-Trichloro-1,2,2-trifluoroethane		
Vinyl chloride		
m-xylene and p-xylene		
o-xylene		

Acceptance Limits: < 3 X MDLs

FORM IVA VOC

Rev 6/96

Figure 13-10. Example of blank results reporting form for total purgeable VOC analysis.

TOTAL VOCS ANALYSIS
LABORATORY BLANK SUMMARY

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES COMPANY
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

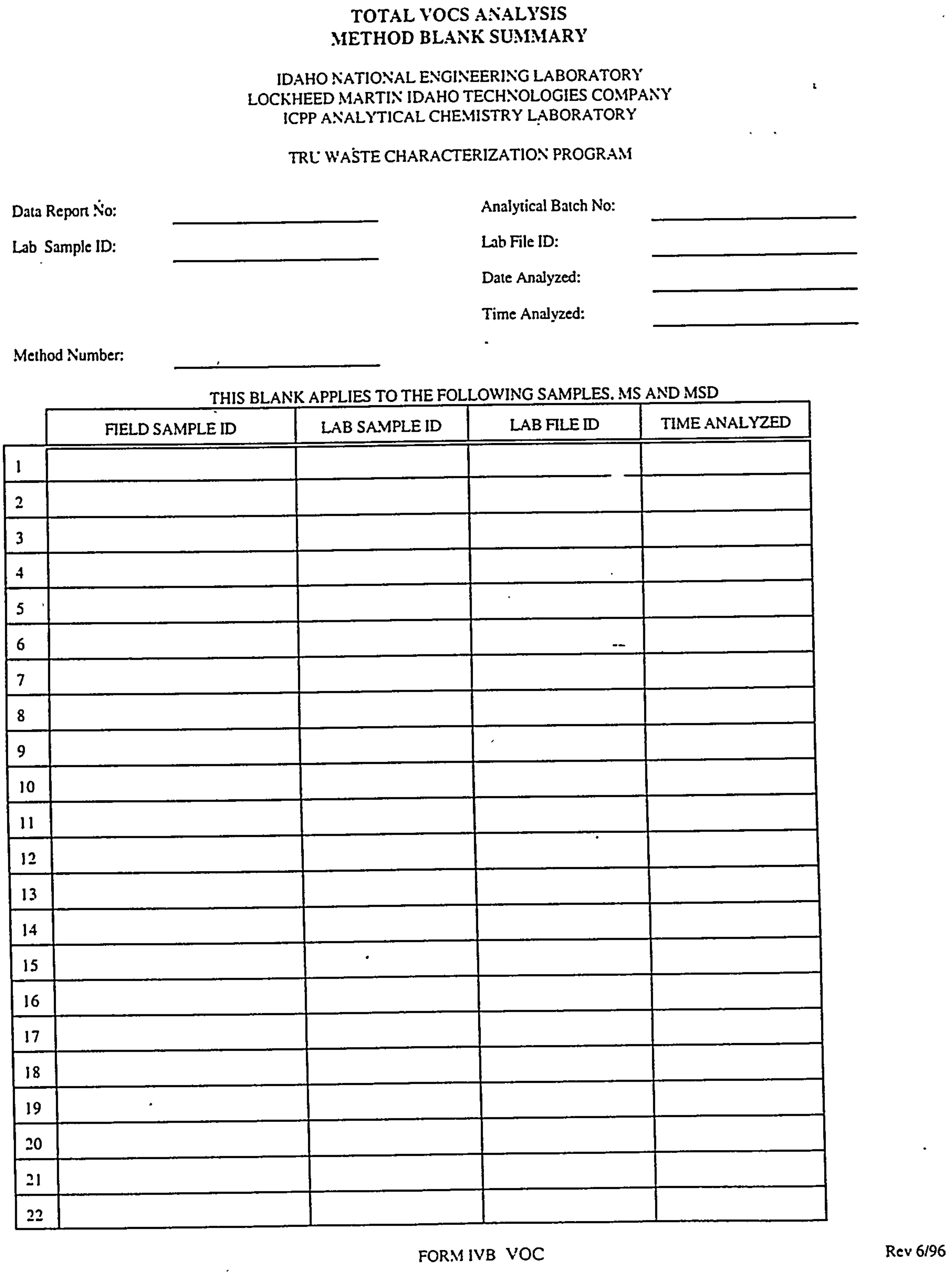
Data Report No: ____________________
Lab Sample ID: ____________________
Date Extracted: ____________________
Method Number: ____________________

Analytical Batch No: ____________________
Lab File ID: ____________________
Date Analyzed: ____________________
Time Analyzed: ____________________

THIS BLANK APPLIES TO THE FOLLOWING SAMPLES, MS AND MSD

	FIELD SAMPLE ID	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED	TIME ANALYZED
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					

Figure 13-11. Example of LB summary reporting form for total purgeable VOC analysis.



TOTAL VOCS ANALYSIS
METHOD BLANK SUMMARY

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES COMPANY
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Data Report No: ____________

Lab Sample ID: ____________

Analytical Batch No: ____________

Lab File ID: ____________

Date Analyzed: ____________

Time Analyzed: ____________

Method Number: ____________

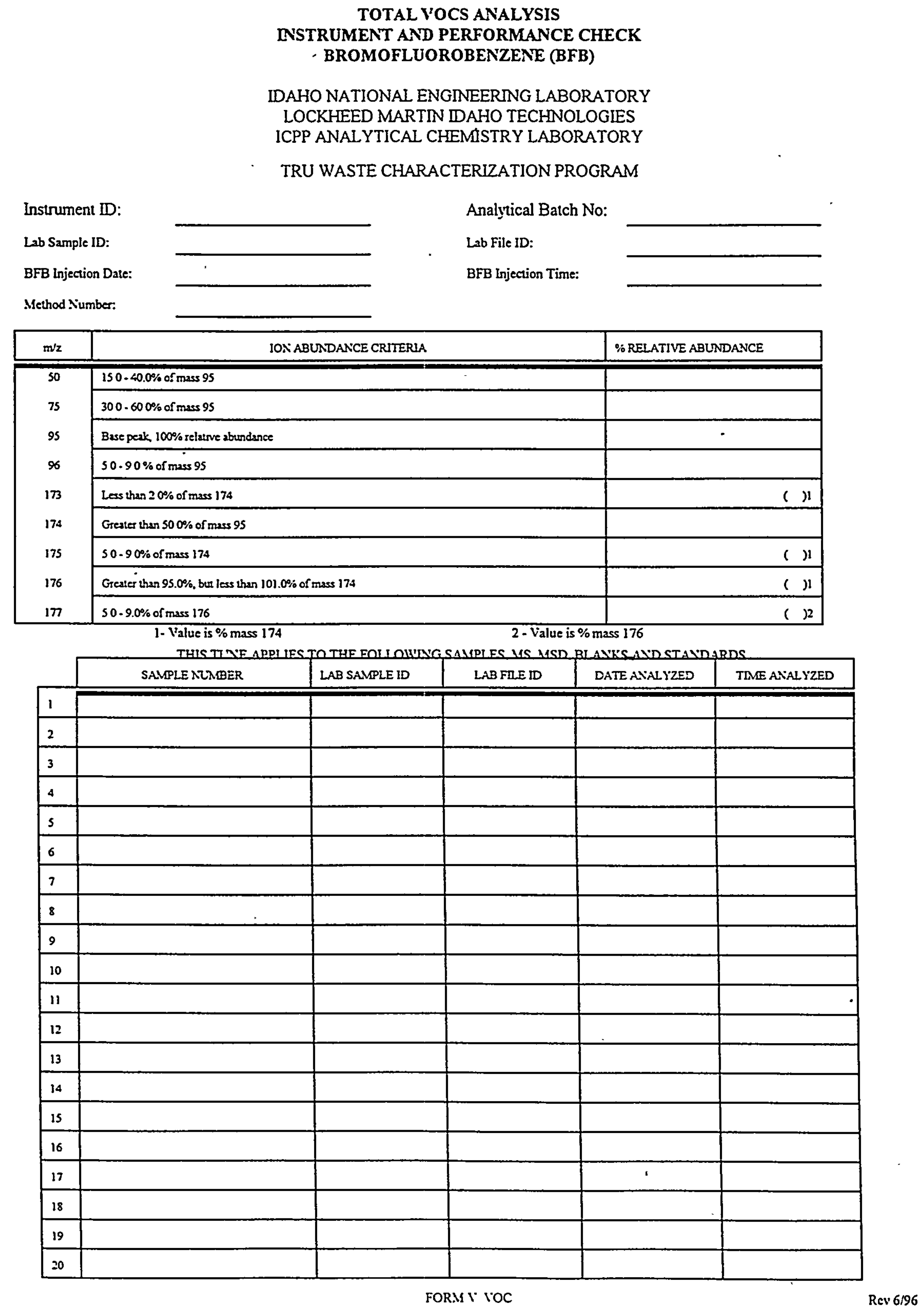
THIS BLANK APPLIES TO THE FOLLOWING SAMPLES, MS AND MSD

	FIELD SAMPLE ID	LAB SAMPLE ID	LAB FILE ID	TIME ANALYZED
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				

FORM IVB VOC

Rev 6/96

Figure 13-12. Example of method blank summary form for total purgeable VOC analysis.



TOTAL VOCS ANALYSIS
INSTRUMENT AND PERFORMANCE CHECK
BROMOFLUOROBENZENE (BFB)

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Instrument ID: ____________ Analytical Batch No: ____________

Lab Sample ID: ____________ Lab File ID: ____________

BFB Injection Date: ____________ BFB Injection Time: ____________

Method Number: ____________

m/z	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE
50	15.0 - 40.0% of mass 95	
75	30.0 - 60.0% of mass 95	
95	Base peak, 100% relative abundance	
96	5.0 - 9.0 % of mass 95	
173	Less than 2.0% of mass 174	( )1
174	Greater than 50.0% of mass 95	
175	5.0 - 9.0% of mass 174	( )1
176	Greater than 95.0%, but less than 101.0% of mass 174	( )1
177	5.0 - 9.0% of mass 176	( )2

1- Value is % mass 174 2 - Value is % mass 176

THIS TUNE APPLIES TO THE FOLLOWING SAMPLES, MS, MSD, BLANKS AND STANDARDS.

	SAMPLE NUMBER	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED	TIME ANALYZED
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					

FORM V VOC Rev 6/96

Figure 13-13. Example of BFB tune reporting form for total purgeable VOC analysis.

TOTAL VOCS ANALYSIS
INTERNAL STANDARD AREA AND RT SUMMARY

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES COMPANY
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Analytical Batch No: ____________

CCAL Sample ID: ____________

CCAL Lab File ID: ____________

Date Analyzed: ____________

Method Number: ____________

		IS1 (BCM) AREA #	RT	IS2 (DFB) AREA #	RT	IS3 (CBZ) AREA #	RT
	12 HOUR STD						
	UPPER LIMIT						
	LOWER LIMIT						
	SAMPLE ID						
01							
02							
03							
04							
05							
06							
07							
08							
09							
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							

IS1 (BCM) = Bromochloromethane
IS2 (DFB) = 1,4 - Difluorobenzene
IS3 (CBZ) = Chlorobenzene-d5

UPPER LIMIT = - 100% of internal standard area.

LOWER LIMIT = - 50 % of internal standard area.

RT Upper Limit = - 30 seconds of internal standard RT
Column used to flag internal standard area values

RT Lower Limit = - 30 seconds of internal standard RT
* = Values outside of QC limits

FORM VIIIA VOC

Rev 6/96

Figure 13-14. Example of internal standard area reporting form for total purgeable VOC analysis.

TOTAL VOCS ANALYSIS
CONTINUING CALIBRATION CHECK

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES COMPANY
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Instrument ID: ____________ Analytical Batch No: ____________

Initial Calibration Date: ____________ Calibration Date: ____________

Initial Calibration Time: ____________ Calibration Time: ____________

Lab Sample ID: ____________ Lab File ID: ____________

Method Number: ____________

COMPOUND	$\overline{RRF}$	RRF _____	% D
Benzene			
Bromoform **			
Carbon disulfide			
Carbon tetrachloride			
Chlorobenzene **			
Chloroform *			
1,2-Dichloroethane			
1,1-Dichloroethylene *			
Ethylbenzene *			
Methylene chloride			
1,1,2,2-Tetrachloroethane **			
Tetrachloroethylene			
Toluene *			
1,1,1-Trichloroethane			
1,1,2-Trichloroethane			
Trichloroethylene			
Trichlorofluoromethane			
1,1,2-Trichloro-1,2,2-trifluoroethane			
Vinyl chloride *			
m-xylene and p-xylene			
o-xylene			

CCC- Calibration Check Compounds (*)
% D ≤ 20

SPCC- System Performance Check Compounds (**)
RRF ≥ 0.30 (≥ 0.25 for Bromoform)

FORM VII VOC Rev 6/96

Figure 13-15. Example of CCAL reporting form for total purgeable VOC analysis.

TOTAL VOCS ANALYSIS
CONTINUING CALIBRATION CHECK
INTERNAL STANDARD AREA AND RT SUMMARY

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES COMPANY
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Analytical Batch No: ____________

ICAL/CCAL Sample ID: ____________ Daily CCAL Sample ID: ____________

ICAL/CCAL Lab File ID: ____________ Daily CCAL Lab File ID: ____________

Date Analyzed: ____________ Date Analyzed: ____________

Method Number: ____________

		IS1 (BCM) AREA #	RT	IS2 (DFB) AREA #	RT	IS3 (CBZ) AREA #	RT
	12 HOUR STD						
Prev	UPPER LIMIT						
CCAL	LOWER LIMIT						
Continuing Calibration Check							

IS1 (BCM) = Bromochloromethane
IS2 (DFB) = 1,4 - Difluorobenzene
IS3 (CBZ) = Chlorobenzene-d5

UPPER LIMIT = + 100% of internal standard area.

LOWER LIMIT = - 50 % of internal standard area.

RT Upper Limit = + 30 seconds of internal standard RT
Column used to flag internal standard area values

RT Lower Limit = - 30 seconds of internal standard RT
* = Values outside of QC limits

* Last CCAL or ICAL whichever is most recent

FORM VIIIB VOC

Rev 6/96

Figure 13-16. Example of CCAL Internal Standard Area/RT reporting form for total purgeable VOC analysis.

TOTAL VOCS ANALYSIS
INITIAL CALIBRATION DATA

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Instrument ID: ____________________ Calibration Date: ____________________

LAB FILE ID: RRF ___ = ______	RRF ___ = ______ RRF ___ = ______			RRF ___ = ______ RRF ___ = ______			
COMPOUND	RF ___	RF ___	RF ___	RF ___	RF ___	$\overline{\text{RRF}}$	%RSD #
Benzene							
Bromoform **							
Carbon disulfide							
Carbon tetrachloride							
Chlorobenzene **							
Chloroform *							
1,2-Dichloroethane							
1,1-Dichloroethylene *							
Ethylbenzene *							
Methylene chloride							
1,1,2,2-Tetrachloroethane **							
Tetrachloroethylene							
Toluene *							
1,1,1,-Trichloroethane							
1,1,2-Trichloroethane							
Trichloroethylene							
Trichlorofluoromethane							
1,1,2-Trichloro-1,2,2-trifluoroethane							
Vinyl chloride *							
m-xylene and p-xylene							
o-xylene							

Column used to flag % RSD values
% RSD ≤ 15%
RRF ≥ 0.30 (≥ 0.25 for Bromoform)

# Column used to flag modeled compounds
M = Modeled compound. A model report will be attached

FORM VI VOC

Rev 6/96

Figure 13-17. Example of ICAL reporting form for total purgeable VOC analysis.

TOTAL VOCS ANALYSIS
MDL REPORTING FORM

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES COMPANY
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Instrument ID: ______________________

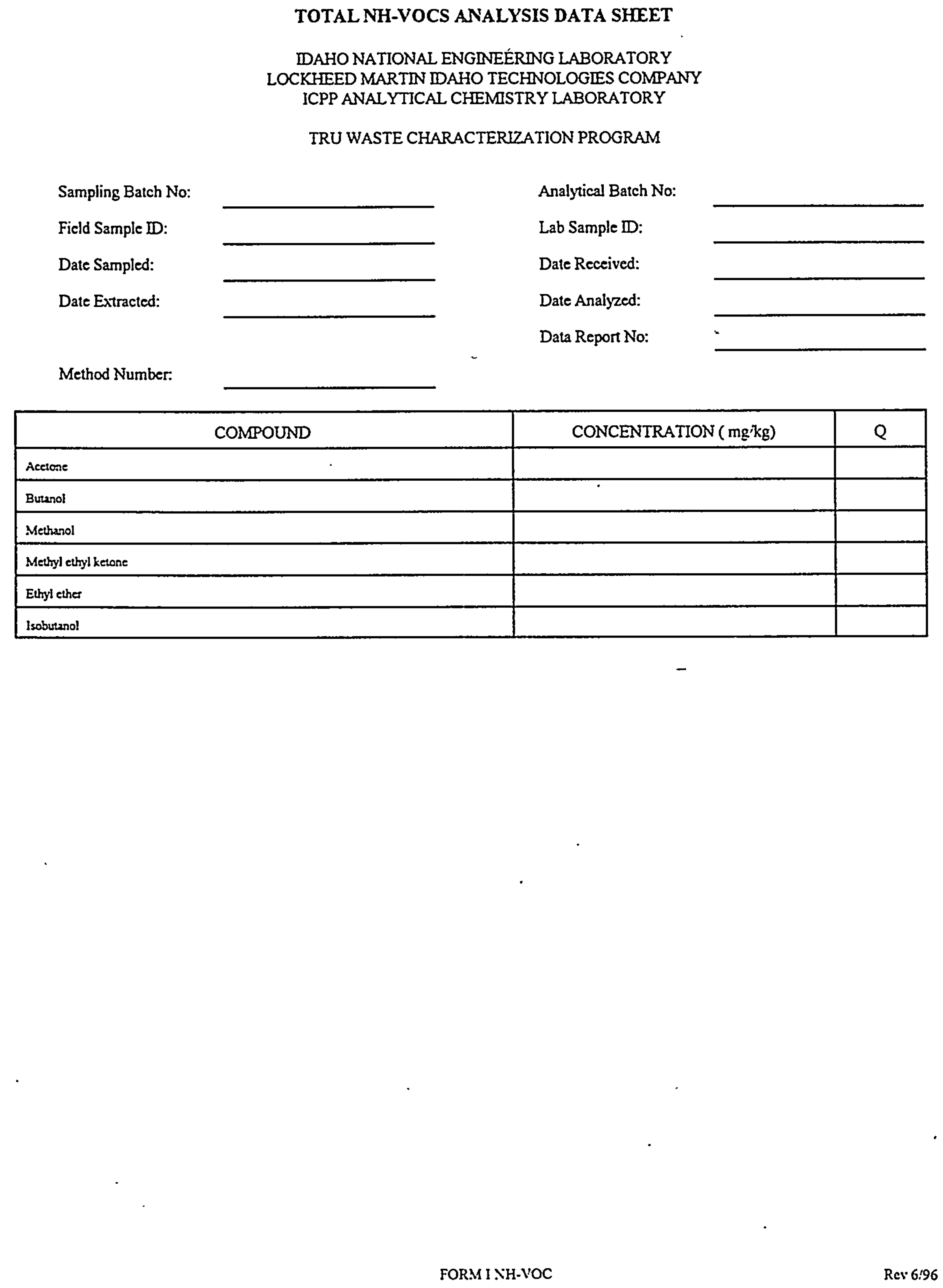
MDL Determination Dates: __________ __________

Method Number: ______________________

COMPOUND	MDL (mg/kg)*	Program Required MDL (mg/kg)	PRQL (mg/kg)
Benzene		1.0	10
Bromoform		1.0	10
Carbon disulfide		1.0	10
Carbon tetrachloride		1.0	10
Chlorobenzene		1.0	10
Chloroform		1.0	10
1,2-Dichloroethane		1.0	10
1,1-Dichloroethylene		1.0	10
Ethylbenzene		1.0	10
Methylene chloride		1.0	10
1,1,2,2-Tetrachloroethane		1.0	10
Tetrachloroethylene		1.0	10
Toluene		1.0	10
1,1,1-Trichloroethane		1.0	10
1,1,2-Trichloroethane		1.0	10
Trichloroethylene		1.0	10
Trichlorofluoromethane		1.0	10
1,1,2-Trichloro-1,2,2-trifluoroethane		1.0	10
Vinyl chloride		1.0	4.0
m-xylene and p-xylene		1.0	10
o-xylene		1.0	10

* Assumes 2.0 g sample extracted in 10 mL methanol
Purge: __ μL injected into __ mL water

Figure 13-18. Example of MDL reporting form for total purgeable VOC analysis.



TOTAL NH-VOCS ANALYSIS DATA SHEET

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES COMPANY
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Sampling Batch No: ______________

Analytical Batch No: ______________

Field Sample ID: ______________

Lab Sample ID: ______________

Date Sampled: ______________

Date Received: ______________

Date Extracted: ______________

Date Analyzed: ______________

Data Report No: ______________

Method Number: ______________

COMPOUND	CONCENTRATION (mg/kg)	Q
Acetone		
Butanol		
Methanol		
Methyl ethyl ketone		
Ethyl ether		
Isobutanol		

FORM I NH-VOC

Rev 6/96

Figure 13-19. Example of total nonhalogenated VOC analysis data sheet.

- D: Reported analyte concentration is from a secondary dilution of the sample
- J: Analyte concentration is <PRQL but ≥MDL
- U: Analyte was undetected (reported as sample-specific MDL).
- Z: Estimated concentration; one or more QC sample results are outside the acceptance criteria.

- **Section 4: Batch Related QC Samples**—This section contains forms reporting results of LCSs (see example in Figure 13-20) and matrix spike/matrix spike duplicate recoveries and RPDs (see example in Figure 13-21).

- **Section 5: Data Review Checklists**—This section includes the TWCP Sample Receiving & Custody Review Checklist (see example in Figure 6-2), the ACMM 9441 Independent Data Review Checklist (see example in Figure 13-2), and the Quality Assurance Data Review Checklist for ACMM 9441 (see example in Figure 3-2). Copies of applicable NCRs are also included in this section, if necessary.

The following items are retained in ACL files, but are not included in the data reports sent to the SPO:

- **Data package** filed by data report number, with all raw data, including sample preparation logs, standard preparation logs, original instrument external standard reports for all CCVs, samples and QC samples, calculation records, results of all associated QC samples (LBs, CCVs, LCSs, MS, MSDs), and reference to the associated ICAL. Examples of forms providing LB, CCV results, and analyte identification confirmation are provided in Figures 13-22, 13-23, and Figure 13-24, respectively.

- ACMM 9441, filed under Calibration Records according to ICAL number, which include standard preparation logs for all calibration standards and verification standards (source reference), raw data for the ICAL, method identification, calibration date and time, linear regressions for each analyte, and report forms providing results of the ICAL (see example in Figure 13-25).

- **MDL records**, filed by MDL determination date, which include all raw data and calculations for MDL determinations, along with the MDL reporting form (see example in Figure 13-26).

- **RT window records**, filed by RT window determination date, which include all raw data and calculations for RT window determination.

- **Original COC forms**, filed in the data package.

TOTAL NH-VOCS ANALYSIS
LABORATORY CONTROL SAMPLE FORM

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES COMPANY
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Data Report No: ____________________

Analytical Batch No: ____________________

Lab Sample ID: ____________________

Lab File ID: ____________________

Date Analyzed: ____________________

Method Number: ____________________

COMPOUND	MEASURED CONCENTRATION (ng/mL)	KNOWN CONCENTRATION (ng/mL)	RECOVERY (%)
Acetone			
Butanol			
Methanol			
Methyl ethyl ketone			
Ethyl ether			
Isobutanol			

QC LIMITS
80-120 %

NA = Not Applicable

FORM IX NH-VOC

Rev. 6/96

Figure 13-20. Example of LCS reporting form for total nonhalogenated VOC analysis.

TOTAL NH-VOCS ANALYSIS
MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Data Report No: ____________ Analytical Batch No: ____________

Field Sample ID: ____________ MS Field Sample ID: ____________ MSD Field Sample ID: ____________

Lab Sample ID: ____________ MS Lab Sample ID: ____________ MSD Lab Sample ID: ____________

Lab File ID ____________ MS Lab File ID: ____________ MSD Lab File ID: ____________

Date Analyzed: ____________ MS Date Analyzed: ____________ MSD Date Analyzed: ____________

Method Number: ____________

COMPOUND	SPIKE ADDED (mg/kg)	SAMPLE CONCENTRATION (mg/kg)	SPIKED SAMPLE CONCENTRATION (mg/kg)	MS % REC #	QC LIMITS RECOVERY
Acetone					60-150
Butanol					60-150
Methanol					60-150
Methyl ethyl ketone					60-150
Ethyl ether					60-150
Isobutanol					60-150

COMPOUND	SPIKE ADDED (mg/kg)	DUPLICATE SPIKED SAMPLE CONCENTRATION (mg/kg)	% RPD	MSD % REC #	QC LIMITS	
					RPD	REC
Acetone					≤50	60-150
Butanol					≤50	60-150
Methanol					≤50	60-150
Methyl ethyl ketone					≤50	60-150
Ethyl ether					≤50	60-150
Isobutanol					≤50	60-150

Column to be used to flag recovery and RPD values with an asterisk

* = Values outside of QC limits

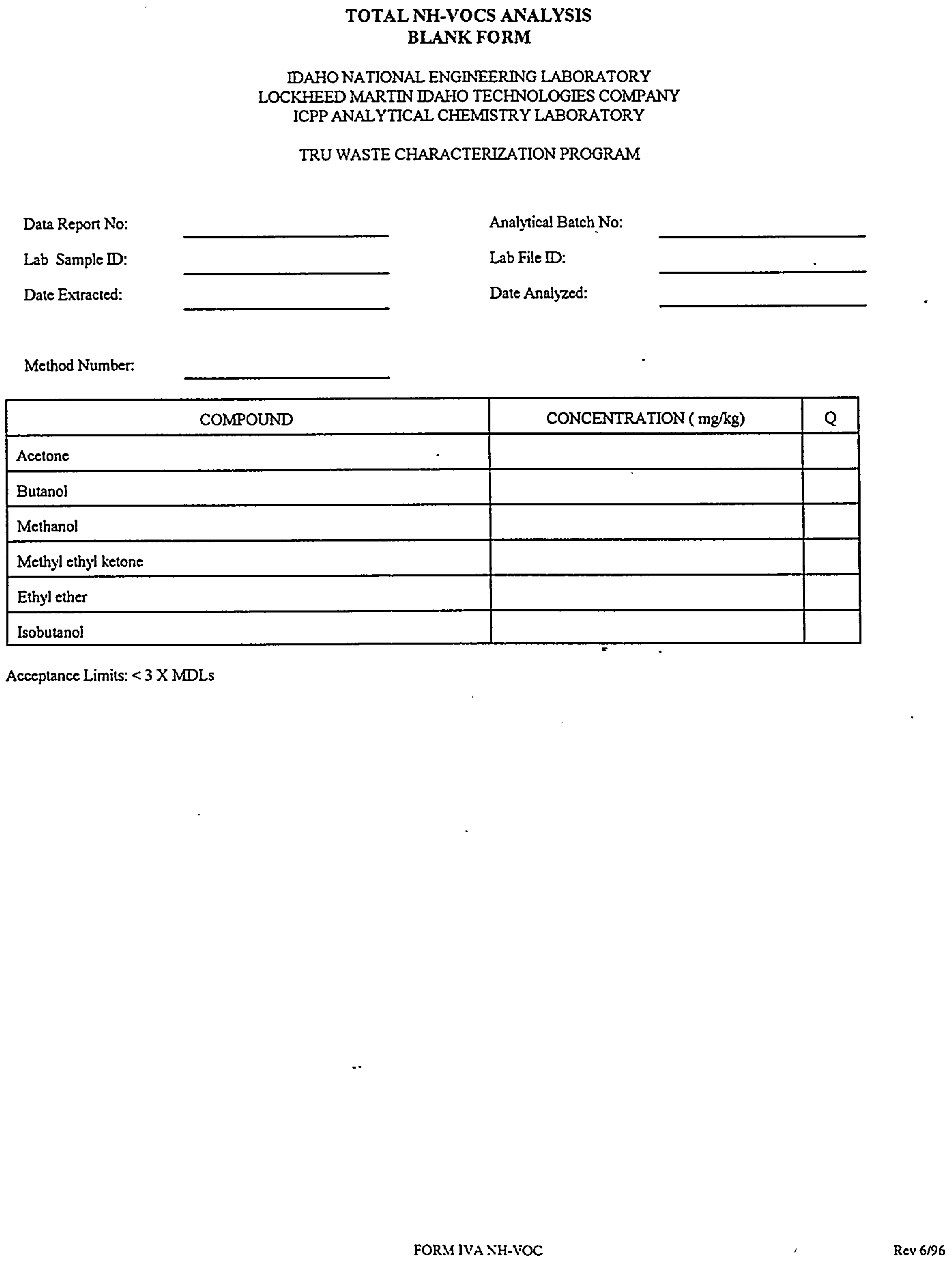
RPD ____ Out of ____ outside limits

Spike Recovery ____ Out of ____ outside limits

COMMENTS ____

FORM III NH-VOC Rev 6/96

Figure 13-21. Example of matrix spike/matrix spike duplicate reporting form for total nonhalogenated VOC analysis.



TOTAL NH-VOCS ANALYSIS
BLANK FORM

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES COMPANY
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Data Report No: ____________________
Analytical Batch No: ____________________
Lab Sample ID: ____________________
Lab File ID: ____________________
Date Extracted: ____________________
Date Analyzed: ____________________

Method Number: ____________________

COMPOUND	CONCENTRATION (mg/kg)	Q
Acetone		
Butanol		
Methanol		
Methyl ethyl ketone		
Ethyl ether		
Isobutanol		

Acceptance Limits: < 3 X MDLs

FORM IVA NH-VOC

Rev 6/96

Figure 13-22. Example of LB reporting form for total nonhalogenated VOC analysis.

TOTAL NH-VOCS ANALYSIS
CONTINUING CALIBRATION VERIFICATION

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES COMPANY
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Instrument ID: ____________ Analytical Batch No: ____________

Initial Calibration Date: ____________ Calibration Date: ____________

Initial Calibration Time: ____________ Calibration Time: ____________

Lab Sample ID: ____________ Lab File ID: ____________

Method Number: ____________

COMPOUND	Known Value (μg/mL)	Measured Value (μg/mL)	% D	RT Window		RT
				From	To	
Acetone						
Butanol						
Methanol						
Methyl ethyl ketone						
Ethyl ether						
Isobutanol						

% D ≤ 20

Figure 13-23. Example of CCV reporting form for total nonhalogenated VOC analysis.

TOTAL NH-VOCS ANALYSIS
ANALYTE IDENTFICATION FORM

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Data Report No: ____________
Analytical Batch No: ____________
Field Sample ID: ____________
Lab Sample ID: ____________

Method Number: ____________
Instrument ID: ____________

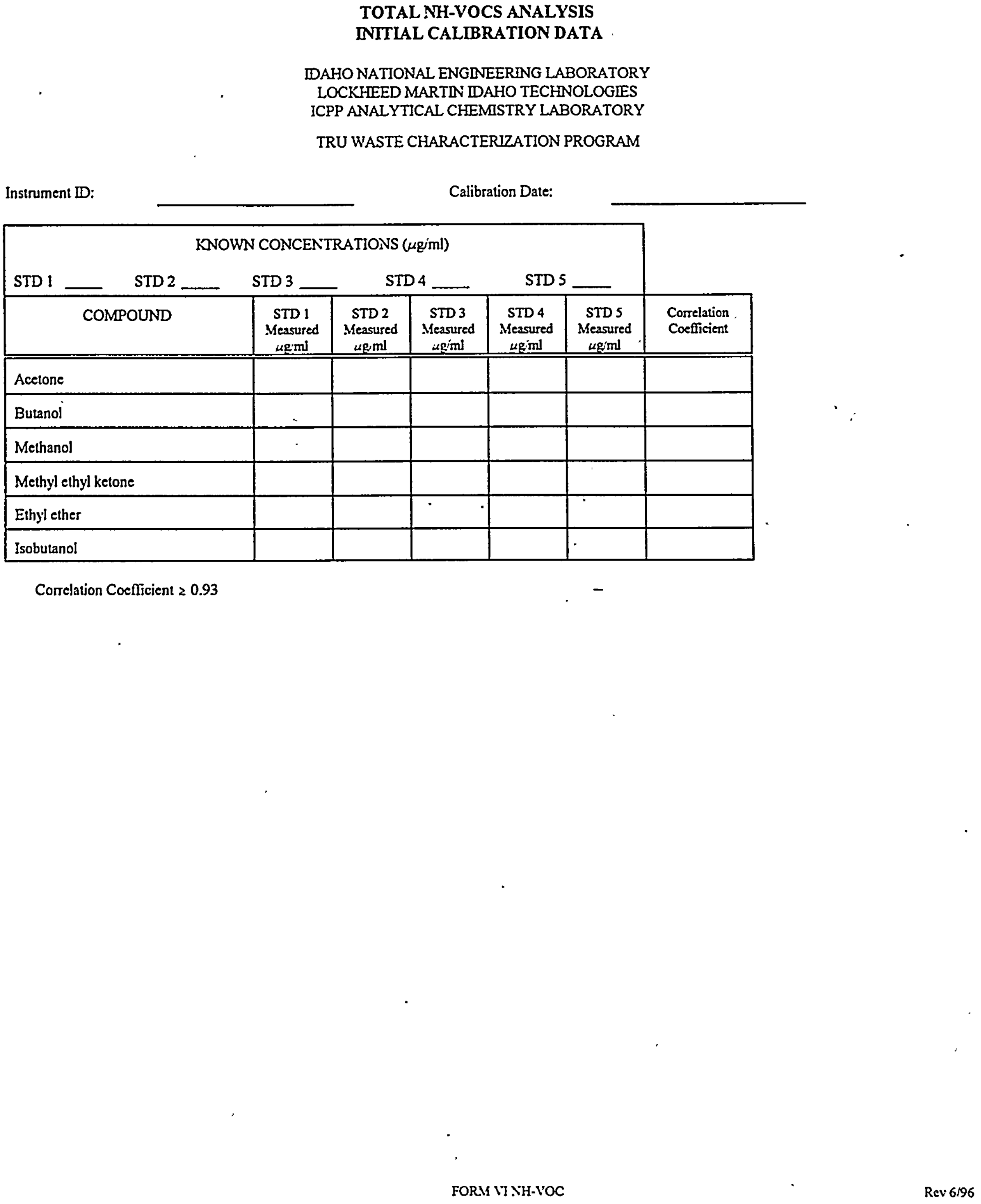
Colunm 1 ____________
Column 2 ____________
Lab File ID: ____________
Lab File ID: ____________
Date Analyzed: ____________
Date Analyzed: ____________
Time Analyzed: ____________
Time Analyzed: ____________

ANALYTE	COL	QUANT	RETENTION TIME	RT WINDOW		CONCENTRATION	
				FROM	TO	mg/kg	Q
Acetone	1						
	2						
Butanol	1						
	2						
Methanol	1						
	2						
Methyl ethyl ketone	1						
	2						
Ethyl ether	1						
	2						
Isobutanol	1						
	2						

FORM X11 NH-VOC

Rev 6 96

Figure 13-24. Example of analyte identification form for total nonhalogenated VOC analysis.



TOTAL NH-VOCS ANALYSIS
INITIAL CALIBRATION DATA

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Instrument ID: ____________________ Calibration Date: ____________________

KNOWN CONCENTRATIONS (μg/ml)

STD 1 ____ STD 2 ____ STD 3 ____ STD 4 ____ STD 5 ____

COMPOUND	STD 1 Measured μg/ml	STD 2 Measured μg/ml	STD 3 Measured μg/ml	STD 4 Measured μg/ml	STD 5 Measured μg/ml	Correlation Coefficient
Acetone						
Butanol						
Methanol						
Methyl ethyl ketone						
Ethyl ether						
Isobutanol						

Correlation Coefficient ≥ 0.93

FORM VI NH-VOC Rev 6/96

Figure 13-25. Example of ICAL reporting form for total nonhalogenated VOC analysis.

TOTAL NH-VOCS ANALYSIS
INITIAL CALIBRATION DATA

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Instrument ID: ______________________ Calibration Date: ______________________

KNOWN CONCENTRATIONS (μg/ml)

STD 1 ____ STD 2 ____ STD 3 ____ STD 4 ____ STD 5 ____

COMPOUND	STD 1 Measured μg/ml	STD 2 Measured μg/ml	STD 3 Measured μg/ml	STD 4 Measured μg/ml	STD 5 Measured μg/ml	Correlation Coefficient
Acetone						
Butanol						
Methanol						
Methyl ethyl ketone						
Ethyl ether						
Isobutanol						

Correlation Coefficient ≥ 0.93

FORM VI NH-VOC Rev 6/96

Figure 13-25. Example of ICAL reporting form for total nonhalogenated VOC analysis.

14. TOTAL SEMIVOLATILE ORGANIC COMPOUND ANALYSIS

This section identifies the required QA elements for the analysis of total SVOCs in samples of homogenous solids and soil/gravel.

14.1 Quality Assurance Objectives

The program DQOs for total SVOC analysis are listed in Section 1.5 of this document. The QA objectives specified in Table 14-1 were developed in order to ensure the generation of total SVOC data that meet the program DQOs. Key data quality indicators for laboratory measurements are defined below and the methods to qualitatively and quantitatively assess these indicators are discussed in Section 3 of this QAPjP.

14.1.1 Precision

Precision is assessed by analyzing laboratory matrix spike duplicates (MSDs), replicate analyses of laboratory control samples (LCSs) and PDP blind audit samples. Results from these measurements are compared to the criteria listed in Table 14-1. These QC measurements are used to demonstrate acceptable method performance and to trigger CA when specification limits are exceeded.

14.1.2 Accuracy

Accuracy as %R is assessed for the laboratory operations by analyzing LCSs, matrix spikes (MSs), surrogate compounds, and PDP blind audit samples. Results from these measurements are compared to the criteria listed in Table 14-1. These QC measurements are used to demonstrate acceptable method performance and to trigger CA when specification limits are exceeded.

14.1.3 Method Detection Limits

MDLs are expressed in mg/kg for SVOCs. MDLs must be determined every six months, and must be less than or equal to the program-specified maximum values listed in Table 14-1. Detailed procedures for MDL determination, which are based on the method described by Glaser et al. (1981), are included in the appropriate ACMM methods.

14.1.4 Program Required Quantitation Limits

The capability to quantitate analytes at or below the PRQL concentrations listed in Table 14-1 is demonstrated by semiannual checks of MDL compliance and by setting the concentration of at least one calibration standard below the PRQL for each analyte.

14.1.5 Completeness

Laboratory analysis completeness is expressed as the number of samples analyzed with valid results (see completeness definitions) as a percent of the total number of samples submitted for

Table 14-1. Total semivolatile organic compounds target analyte list and QA objectives.

Compound	CAS Number	Precision[a] (%RDS or RPD)	Accuracy[a] (%R)	MDL (mg/kg)	PRQL (mg/kg)	Completeness (%)
2-Methylphenol	95-48-7	≤50	60–150	5	40	90
3-Methylphenol[b]	108-39-4	≤50	60–150	5	40	90
4-Methylphenol[b]	106-44-5	≤50	60–150	5	40	90
1,4-Dichlorobenzene	106-46-7	≤86	20–124	5	40	90
ortho-Dichlorobenzene	95-50-1	≤64	32–129	5	40	90
2,4-Dinitrophenol	51-28-5	≤119	D-172[c]	5	40	90
2,4-Dinitrotoluene	121-14-2	≤46	39–139	0.3	2.6	90
Hexachlorobenzene	118-74-1	≤319	D-152[c]	0.3	2.6	90
Hexachloroethane	67-72-1	≤44	70–113	5	40	90
Nitrobenzene	98-95-3	≤72	35–180	5	40	90
Pentachlorophenol	87-86-5	≤128	14–176	5	40	90
Pyridine	110-86-1	≤50	60–150	5	40	90

---

a. Criteria apply to PRQL concentrations.

b. These individual isomers are not separated during analysis and are quantitated and reported as the 3-methylphenol/4-methylphenol combination.

c. Detected; result must be greater than zero.

MDL = method detection limit (maximum permissible value).
PRQL = program required quantitation limit; calculated from the TC level for nitrobenzene assuming a 100 g sample, 2 L of extraction fluid, and 100 percent analyte extraction.

analysis. Ninety percent of the total number of samples submitted for analysis must result in valid analytical data.

14.1.6 Comparability

The comparability of ACL data sets to those generated by different sites is achieved through the use of standardized methods and traceable standards, and by participation in the PDP.

14.1.7 Representativeness

Representativeness of total SVOC analyses is achieved by use of standardized sample handling protocols (e.g., storage, aliquotting) and analysis of laboratory blanks to maintain sample integrity.

14.2 Methods Requirements

ACL uses ultrasonic extraction and gas chromatograph mass spectrometry (GC/MS) methods to perform analyses for total SVOCs. These ACMM methods, listed below, are based on EPA SW-846 and TWCP Methods Manual protocols:

- ACMM 9271, *Determination of Semivolatile Organic Compounds in TRU Waste Characterization Samples*: A gas chromatographic/mass spectrometric technique using capillary columns, derived from EPA SW-846 Method 8270B and TWCP Method 430.6.
- ACMM 9501, *Sample Preparation of TRU Waste Characterization Samples for Organic Analysis*: Extraction procedures for organic analysis, including: methanol and aqueous extractions for purgeable and nonhalogenated VOCs (see Section 13); and an ultrasonic extraction technique using methylene chloride for SVOCs, derived from EPA SW-846 Method 3550A.

Detailed operating procedures for these methods, including calibration requirements, sample analysis, and data reduction, are specified in the referenced ACMM methods.

14.2.1 Criteria for Standards

Primary liquid standards may be purchased from the best available source for the analytes listed in Table 14-1. Commercially-purchased primary standards must be certified by the manufacturer, and their concentrations must be traceable to NIST, EPA, or other nationally-recognized standards. ACL ACMM methods , SOP R.1.20, *Analytical Chemistry Quality Control Program*, and ACLP 1.01, *Preparation of Quality Control Reagents and Standards*, specify detailed preparation and documentation requirements for the preparation of all SVOC standards in the laboratory.

14.2.2 Criteria for GC/MS Qualitative and Quantitative Analysis

To be qualitatively identified as an analyte by GC/MS, a sample component must elute within a RRT window of ±0.06 RRT units and have a mass spectrum that corresponds to the analyte mass spectrum. RRT windows are calculated from the individual analyte RTs in the continuing calibration (CCAL) standard.

ACL uses internal standards for quantitating analyte concentrations. All instrument tuning and calibration criteria specified in Section 14.4 and 14.5 must be met before performing qualitative analysis of samples. Quantitation of target analytes is based on the integrated abundance from the EICP of the primary ion. All analytes must be quantitated within the calibration range; multiple dilutions may be required when analyte concentrations exceed the calibration range.

Nontarget compounds are reported as TICs. For samples containing TICs with total ion current peaks greater than 10% of the nearest (retention time) internal standard, a search of the NIST mass spectral library is performed on the 20 TICs greatest in area count to identify the compound(s). Quantitation of a TIC assumes that the compound's calibration response factor is equal to that of the nearest internal standard. Therefore, TIC concentrations have higher associated analytical uncertainty than do target analyte concentrations.

14.3 Quality Control

To ensure that data of known and documented quality are generated, the QC criteria specified in this section must be met for all total SVOC analyses. The ALD QAO is responsible for monitoring and documenting procedure performance, including the analysis of laboratory control samples, laboratory blanks, matrix spikes and matrix spike duplicates. The ALD QAO, the organic section supervisor, and the organic analysis laboratory TL are responsible for implementing CAs when acceptable procedure performance, as specified in this section, is not met.

Specific QC samples and frequencies are summarized in Table 14-2. Analytical QC samples are associated with field samples through the use of analytical batches (see Section 6.3).

14.3.1 Method Performance Samples

Before the analysis of any samples for TWCP, acceptable method performance must be demonstrated for each method to be used. This demonstration consists of determination of MDLs (see Section 14.1.3), and analysis of method performance samples (MPSs). Initially, seven replicate MPSs (standards containing known concentrations of all analytes) must be analyzed to demonstrate that the criteria specified for precision and accuracy listed in Table 14-1 can be met. If the seven replicates do not meet the criteria, then seven more replicates must be analyzed until the initial procedure performance demonstration criteria are met.

Continuing acceptable procedure performance is demonstrated semiannually by analyzing four replicate MPSs. If the precision and accuracy criteria specified in Table 14-1 are not met for the four replicates, four additional replicates must be analyzed until the criteria in Table 14-1 are met.

14.3.2 Laboratory Control Samples (LCSs)

LCSs are analyzed at a minimum frequency of one per analysis batch. A single LCS may not be shared between two separate analysis batches. LCSs are prepared from commercially purchased primary standards that are independent (i.e., different manufacturer) from those used for instrument calibration. LCSs are made in methylene chloride, and at a minimum must contain 1,4-dichlorobenzene, 1,4-dinitrotoluene, hexachloroethane and nitrobenzene. LCSs are not carried through the sample preparation procedure performed on field samples because the solid extraction procedure is incompatible with the liquid LCS.

LCS results are acceptable if the recoveries for all analytes are between 80 and 120% (i.e., 80% $\leq$ %R $\leq$ 120%). If LCS results do not meet this criteria, the LCS may be reanalyzed once; if the results of the rerun LCS meet specifications, sample analysis may continue. If the rerun LCS is still

Table 14-2. Summary of laboratory QC samples and frequencies for total SVOC analysis.

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
Method performance samples	Seven (7) samples initially and four (4) semiannually	Meet Table 14-1 QA objectives	Repeat until acceptable
Laboratory blanks	One (1) per analytical batch	Analyte concentrations <3 × MDLs	See Section 14.3.3
Laboratory control samples	One (1) per analytical batch	80% ≤ %R ≤ 120%	See Section 14.3.2
Matrix spikes	One (1) per analytical batch	Meet Table 14-1 %Rs	See Section 14.3.4
Matrix spike duplicates	One (1) per analytical batch	Meet Table 14-1 %Rs and RPDs	See Section 14.3.4
Surrogate compounds	Each analytical sample	Average %R from a minimum of 30 samples from a given matrix ± 3 standard deviations	See Section 14.3.5
Blind audit samples	Samples and frequency controlled by the Solid PDP Plan	Specified in the Solid PDP Plan	Specified in the Solid PDP Plan

noncompliant, an NCR may be required. CA is required to identify and correct the cause of the nonconformance. Associated samples may require reextractions and reanalysis, depending on the degree of the indicated bias and the magnitudes of analyte concentrations in the samples. If a noncompliant LCS is associated with any sample data reported to the SPO, an NCR must be initiated.

14.3.3 Laboratory Blanks (LBs)

LBs are analyzed at a minimum frequency of one per analytical batch. The LBs must undergo all sample preparation procedures (i.e., sonication) performed on the associated field samples. LBs are acceptable if analyte concentrations are less than three times the Table 14-1 MDLs for all target analytes. If the LB is not acceptable, an NCR may be required. CA is required to identify and correct the cause of the nonconformance. Associated samples may require reanalysis, depending on whether the source of contamination can be identified, the degree of the indicated bias and the magnitudes of analyte concentrations in the samples. If a noncompliant LB is associated with any sample data reported to the SPO, an NCR must be initiated.

14.3.4 Matrix Spikes (MSs) and Matrix Spike Duplicates (MDSs)

Duplicate matrix spikes on individual field samples are performed at the minimum frequency of one pair (MS plus MSD) per analysis batch. MS and MSDs must contain at least 3 of the VOC target analytes listed in Table 14-1.

Matrix spike and matrix spike duplicate results are acceptable if the Table 14-1 criteria for accuracy and precision are met, i.e., if percent recoveries (for both MS and MSD) meet the accuracy criteria and the RPDs between MS and MSD results meet the precision criteria.

If MSs and MSDs do not meet the precision and accuracy criteria, the noncompliance is documented on checklists and in the data report narrative. NCRs are not initiated for noncompliant MS and MSD results because these results are highly dependent upon individual sample matrices, and specific corrective actions cannot be identified.

14.3.5 Surrogate Compounds

Surrogate compounds are added to each field sample and laboratory QC sample. The choice of surrogates is site-specific. Surrogates used by ACL are identified in ACMM 9271. If surrogate percent recoveries do not meet the criteria specified in Table 14-2, the noncompliance is documented in checklists and the data report narrative. NCRs are not initiated for noncompliant surrogate recoveries because these results are highly dependent upon individual sample matrices, and specific corrective actions cannot be identified.

14.3.6 Blind Audit Samples

ACL participates in the RCRA solid PDP as specified by program requirements (see Section 2.7) on a nominal semiannual basis. PDP samples are analyzed and reported using the same methods and handling procedures as are used on field samples.

14.4 Instrument Testing, Inspection, and Maintenance Requirements

ACL uses ion trap GC/MSs to analyze for SVOCs in support of TWCP. Ion trap instruments are more sensitive that traditional quadrupole mass spectrometers, and thus are better suited to analysis of radioactively-contaminated samples because smaller sample aliquots can be analyzed. The GC/MSs are operated in full scan mode to allow the detection and quantitation of all target analytes listed in Table 14-1, and identification of nontarget compounds.

Before the analysis of any samples, the GC/MS system must meet decafluorotriphenylphosphine (DFTPP) tuning criteria specified in Table 14-3. The tuning criteria specified in Table 14-3 must be met at the beginning of each 12 hours of operation and before the analysis of any standard or samples by analyzing 5 ng of DFTPP. Note that due to the higher sensitivity of ion trap GC/MSs, the amount of DFTPP used for tuning is less than that specified in EPA Method 8270B and TWCP Method 430.6.

Table 14-3. DFTPP key ions and abundance criteria.

Mass	Ion abundance criteria[a]
51	30 to 60% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	40 to 60% of mass 198
197	< 1% of mass 198
198	Base peak, 100% relative abundance
199	5 to 9% of mass 198
275	10 to 30% of mass 198
365	> 1% of mass 198
441	Present but <. mass 443
442	> 40% of mass 198
443	17 to 23% of mass 442

---

a. SW-846 Method 8270B.

14.5 Instrument Calibration and Frequency

The GC/MS calibration requirements are summarized in Table 14-4. Detailed instructions for calibrating the GC/MS and related equipment are provided in specific ACMM procedures. ACL maintains instrument use logs from which calibration sequences and frequencies may be reconstructed.

14.5.1 GC/MS Calibration

GC/MS Initial Calibration. After instrument performance criteria (i.e., DFTPP tune criteria) have been satisfied, a multipoint internal standard calibration is performed. The multipoint calibration consists of a minimum of five analytical standards that define the calibration range of the instrument for the analytes of interest. One of the standards must be at concentrations less than the PRQLs of the target analytes (see Table 14-1). CCCs and SPCCs used are those that are common to Table 14-1 and SW-846 Method 8270B.

RRFs are generated for each specified target analyte. For the initial five-point calibration to be valid, the %RSD for the RRFs of each CCC must be less than or equal to 30% and the average RRF for each SPCC must be greater than or equal to 0.05. Average RRFs for each analyte are used for quantitation if the %RSD is less than or equal to 15%. If the average RRF %RSD for any analyte is greater than 15%, then a linear or quadratic regression equation is used for quantitation of that analyte.

Table 14-4. GC/MS calibration requirements for total SVOC analysis.

Technique	Procedure	Frequency of Procedure	Acceptance criteria
GCMS	BFB tune	Every 12 hours	Table 14-3 criteria met
	Five-point initial calibration	Initially and as needed	RRF %RSD for CCCs ≤ 30; RRF for SPCCs ≥ 0.05; per analyte, average RRF used if %RSD ≤15; linear or quadratic regression equation generated if %RSD > 15
	Continuing calibration	Every 12 hours	%D ≤ 20 for all target analytes; RRF for SPCCs ≥0.05; RT for internal standards must be ± 30 seconds from last CCAL; ISA count must be > 50% and < 200% of ISA count from CCAL; surrogate compound %R must meet Table 14-2 criteria

ACMM 9271 specifies calibration procedures for the GC/MS analysis method. A valid ICAL must exist before any samples analyses are performed. A new ICAL is required if there is a change in the instrument that may affect the analytical results or if indicated as a CA.

GC/MS Continuing Calibration. The initial GC/MS calibration (i.e., the ICAL) is verified using a continuing calibration (CCAL) standard. The CCAL standard must contain all target analytes at concentrations near the midpoint of the calibration range. The CCAL standard is analyzed at the beginning of each 12 hours of operation and after an acceptable DFTPP tune. For the CCAL to be valid, it must meet all of the daily calibration criteria for surrogate compound recovery, SPCCs, CCCs, internal standard area count criteria and RTs, as specified in Table 14-4, per SW-846 Method 8270B. Additionally, the RRF %Ds must be ≤ 20 and the RRFs must be ≥0.05 for all target analytes.

If the CCAL does not satisfy the calibration requirement, the CCAL standard may be remade and rerun once to eliminate CCAL standard preparation as the source of error. If the rerun CCAL still does not meet criteria, a new five-point ICAL must be generated. Sample analysis cannot proceed until the GC/MS system has satisfied the calibration requirements.

14.6 Data Management

Data management includes requirements for data reduction, data validation, and reporting. All of the data management requirements defined in Section 3 of this QAPjP, as well as the specific procedures described below, apply to total SVOC analysis data. Specific equations and sample calculations are detailed in the associated ACL ACMM methods.

14.6.1 Data Reduction

All results for field samples are reported in units of mg/kg on a weight/wet-weight basis and are limited to two significant figures. All calculations (raw data reduction and QC results) are performed prior to rounding.

Target compound concentrations are not blank corrected. Blanks are treated and reported in the same manner as other samples.

Library searches of the NIST mass spectral database are performed for the identification of unknown peaks with a total ion current area greater than 10% of the nearest (retention time) internal standard. Compounds identified by forward library searching are reported as TICs. Concentrations for TICs are calculated assuming a relative response factor equal to one using the nearest internal standard.

14.6.2 Data Validation

All total SVOC data are reviewed by an independent technical reviewer prior to report generation. Review checklists are used to document the independent technical review process; an example checklist is provided in Figure 14-1.

14.6.3 Data Reporting

Data reports for total SVOC data consist of a cover page and five sections:

- **Cover page**—The cover page includes the laboratory name, the data report number, the report date, the report table of contents, and release authorization signatures. An example cover page is provided in Figure 13-3.

- **Section 1: Sample Identification Table**—This section includes the cross-reference between field and laboratory sample identification numbers (example provided in Figure 13-4).

- **Section 2: Sample Custody Documents**—This section includes copies of the field COC form(s) that accompanied the samples to the laboratory (see example provided in Figures 6-1).

- **Section 3: Analysis Results**—This section includes the analytical batch narrative, containing information pertinent to program-level review, and the analysis data sheets (see example in Figure 14-2) for each sample included in the data report. A separate analysis data sheet is provided for each sample, and contains the following information: laboratory name, program name, data report number, analytical batch number, sampling batch number, field sample ID, laboratory sample ID, date sampled, date received at the laboratory, date extracted, date analyzed, method number, listing of program analytes, and analytical results in mg/kg. Data qualifying flags are used as follows:

    - B: Analyte detected in associated LB

TWCP GCMS SVOC INDEPENDENT DATA REVIEW CHECKLIST

Analysis Batch:	Method: 430.6		Form Revision: 0	
Run # and Analysis Date	Analyst	Independent Reviewer	Date of Review	OK for Release?
1:				Yes or No
2:				Yes or No
3:				Yes or No
4:				Yes or No
5:				Yes or No

Instructions: Complete one checklist per Analysis Batch. If more than five instrument runs were needed to complete the batch analysis, use a second checklist. Enter appropriate response for each question. Each "No" response requires explanation. A "No" response to a question in Bold type may require initiation of an NCR.

REQUIREMENT	Run #1		Run #2		Run #3		Run #4		Run #5		COMMENTS
	YES	NO	YES	NO	YES	NO	YES	NO	YES	NO	
1. INITIAL CALIBRATION (ICAL)											
a. **Does the DFTPP tune meet the TWCP QAPP requirements [Table 14-3]?**											
b. **Are the relative response factor %RSDs for CCCs < 30 [Table 14-4]?**											
c. **Are the relative response factors for SPCCs > 0.05 [Table 14-4]?**											
d. **Was an average RRF used for %RSDs which were ≤ 15, and a regression equation generated if the %RSD was >15?**											
e. Is the ICAL raw data signed by the analyst and included in the file, or referenced, as appropriate for the report?											
2. DFTPP TUNE											
a. **Was a DFTPP tune performed each day prior to analysis of samples?**											
b. Is the raw data signed by the analyst and present in the file for each DFTPP tune?											
c. **Do all DFTPP tunes meet the TWCP QAPP requirements [Table 14-3]?**											
3. CONTINUING CALIBRATION (CCAL)											
a. **Was a CCAL performed each day after the DFTPP tune and prior to analysis of samples?**											
b Is the raw data signed by the analyst and present in the file for each CCAL?											
c. **Is the %D < 20 for all analytes [Table 14-4]?**											
d. **Are the response factors for SPCCs > 0.05 [Table 14-4]?**											
e. Are the retention times of the internal standards within 30 seconds of those in the last CCAL or ICAL?											
f. Are the internal standard areas within 50 to 200% of those in the last CCAL or ICAL?											
g. Do the surrogate recoveries meet the TWCP QAPP requirements [Table 14-2]?											

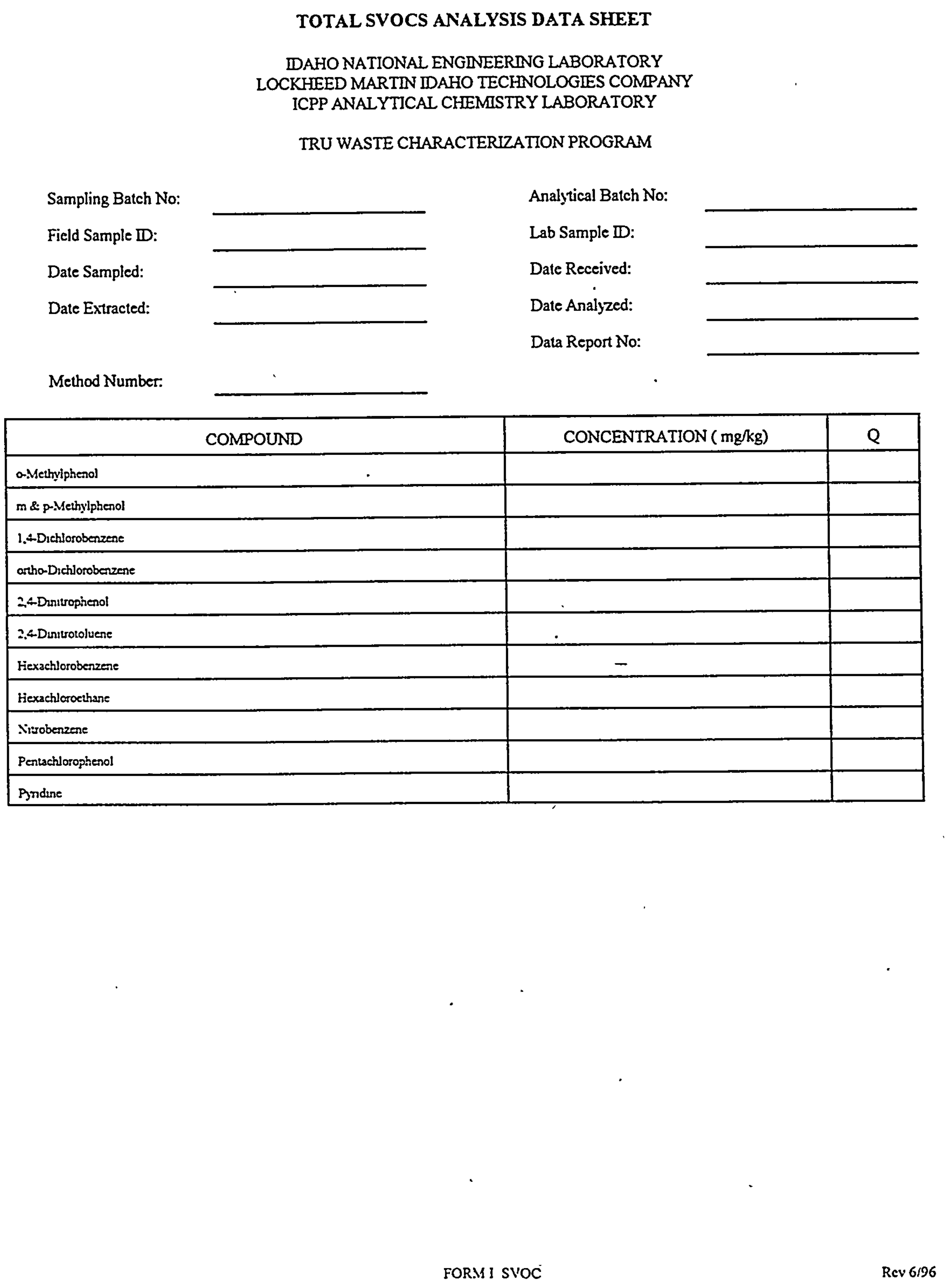
Figure 14-1. Example of independent technical review checklist for total SVOC analysis.

TWCP GCMS SVOC INDEPENDENT DATA REVIEW CHECKLIST FOR BATCH # ____________

REQUIREMENT	Run #1		Run #2		Run #3		Run #4		Run #5		COMMENTS
	YES	NO	YES	NO	YES	NO	YES	NO	YES	NO	
4. LABORATORY BLANKS											
a. Was at least one laboratory blank extracted and analyzed with the analytical batch?											
b. Are all laboratory blank results less than 3 times the MDLs for all analytes [Table 14-2]?											
5. LABORATORY CONTROL SAMPLES (LCSs)											
a. Was at least one unique LCS analyzed with the analytical batch?											
b. Do the recoveries for all analytes in each LCS fall between 80% and 120% [Table 14-2]?											
6. DUPLICATE SAMPLE											
a. Was at least one field sample from the analytical batch analyzed in duplicate, or as a matrix spike duplicate?											
b. Do the RPDs meet the TWCP QAPP precision requirements [Table 14-2]?											
7.MATRIX SPIKE SAMPLE											
a. Was at least one field sample from the analytical batch analyzed as a matrix spike?											
b. Do the %Rs for all MS and MSDs meet the TWCP QAPP requirements [Table 14-2]?											
8. SAMPLES (INCLUDING LCSs, BLANKS, DUPLICATES AND SPIKES)											
a. Are the raw data signed by the analyst and present in the file for all samples listed on the Analysis Request Form, and for all associated LCSs, blanks, duplicates, and spikes?											
b. Are the internal standard areas within 50 to 200% of those in the associated CCAL?											
c. Were all samples analyzed within 12 hours of the daily DFTPP tune?											
d. In your opinion, do the sample spectra for positive hits match the reference spectra?											
e. Was a TIC search conducted on each sample, blank and LCS in the batch?											
f. Were the TICs reviewed, initialed, and dated?											
g. Were all samples having analytes detected in amounts exceeding the calibration range, reanalyzed on a dilution or at a lower volume?											
h. Were all samples and any associated reanalyses or dilutions prepared within 14 days, and analyzed within 40 days of sampling?											

Additional Comments:

Figure 14-1. (continued).



TOTAL SVOCS ANALYSIS DATA SHEET

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES COMPANY
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Sampling Batch No: ____________

Field Sample ID: ____________

Date Sampled: ____________

Date Extracted: ____________

Analytical Batch No: ____________

Lab Sample ID: ____________

Date Received: ____________

Date Analyzed: ____________

Data Report No: ____________

Method Number: ____________

COMPOUND	CONCENTRATION (mg/kg)	Q
o-Methylphenol		
m & p-Methylphenol		
1,4-Dichlorobenzene		
ortho-Dichlorobenzene		
2,4-Dinitrophenol		
2,4-Dinitrotoluene		
Hexachlorobenzene		
Hexachloroethane		
Nitrobenzene		
Pentachlorophenol		
Pyridine		

FORM I SVOC

Rev 6/96

Figure 14-2. Example of total SVOC analysis data sheet.

- E: Reported analyte concentration exceeds the calibration range
- D: Reported analyte concentration is from a secondary dilution or reduced analysis aliquot of the sample
- J: Analyte concentration is <PRQL but ≥MDL
- U: Analyte was undetected (reported as sample-specific MDL)
- Z: Estimated concentration; one or more QC sample results are outside the acceptance criteria
- N: Indicates presumptive evidence of a compound, based on a mass spectral library search (TICs only).

TICs are reported on forms equivalent to those used for target analytes (see example in Figure 14-3). For TICs, estimated concentrations are reported along with RTs. Reported concentrations for identified TICs are always qualified with a J flag to indicate that the reported value has high analytical uncertainty. Estimated concentrations for TICs labeled as "unknowns" are reported by assuming an RRF of 1 relative to the nearest internal standard.

- **Section 4: Batch Related QC Samples**—This section contains forms reporting results of LCSs (see example in Figure 14-4), matrix spike/matrix spike duplicate recoveries and RPDs (see example in Figure 14-5) and surrogate spike recoveries (see example in Figure 14-6).
- **Section 5: Data Review Checklists**—This section includes the TWCP Sample Receiving & Custody Review Checklist (see example in Figure 6-2), the ACMM Method 9271 Independent Data Review Checklist (see example in Figure 14-1), and the Quality Assurance Data Review Checklist for ACMM 9271 (see example in Figure 3-3). Copies of applicable NCRs are also included in this section, as necessary.

The following items are retained in ACL files, but are not included in the data reports sent to the SPO:

- **Data package** filed by data report number, with all raw data, including sample preparation logs, standard preparation logs for ACMM Method 9501, original instrument readouts for all tunes, CCALs, samples and QC samples, calculation records, results of all associated QC measurements (LBs, CCALs, LCSs, MSs, MSDs, surrogates, and internal standard areas), and reference to the associated ICAL. Examples of forms providing LB results, LB summaries, DFTPP tune results, and internal standard areas are provided in Figures 14-7, 14-8, 14-9 and Figure 14-10, respectively. Examples of forms reporting CCAL results are provided in Figures 14-11 and 14-12.

TOTAL SVOCS ANALYSIS DATA SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES COMPANY

ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Sampling Batch No: ____________ Analytical Batch No: ____________

Field Sample ID: ____________ Lab Sample ID: ____________

Date Sampled: ____________ Date Received: ____________

Date Extracted: ____________ Date Analyzed: ____________

Number of TICs found ____________ Data Report No: ____________

Method Number: ____________

Tentatively Identified SVOCs		CONCENTRATION (mg/kg)	Q	Retention Time (minutes)
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				

FORM I- TIC SVOC Rev 6/96

Figure 14-3. Example of TIC analysis data sheet for total SVOC analysis.

TOTAL SVOCS ANALYSIS
LABORATORY CONTROL SAMPLE FORM

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES COMPANY
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Data Report No: ____________________

Analytical Batch No: ____________________

Lab Sample ID: ____________________

Lab File ID: ____________________

Date Analyzed: ____________________

Method Number: ____________________

COMPOUND	MEASURED CONCENTRATION (ng/µl)	KNOWN CONCENTRATION (ng/µl)	RECOVERY (%)
1,4-Dichlorobenzene			
2,4-Dinitrotoluene			
Hexachloroethane			
Nitrobenzene			

QC LIMITS
80-120 %

NA = Not Applicable

FORM IX SVOC

Rev. 6/96

Figure 14-4. Example of LCS reporting form for total SVOC analysis.

TOTAL SVOCS ANALYSIS
MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Data Report No: ____________ Analytical Batch No: ____________

Field Sample ID: ____________ MS Field Sample ID: ____________ MSD Field Sample ID: ____________

Lab Sample ID: ____________ MS Lab Sample ID: ____________ MSD Lab Sample ID: ____________

Lab File ID ____________ MS Lab File ID: ____________ MSD Lab File ID: ____________

Date Analyzed: ____________ MS Date Analyzed: ____________ MSD Date Analyzed: ____________

Method Number: ____________

COMPOUND	SPIKE ADDED (mg/kg)	SAMPLE CONCENTRATION (mg/kg)	SPIKED SAMPLE CONCENTRATION (mg/kg)	MS % REC #	QC LIMITS RECOVERY
1,4-Dichlorobenzene					20-124
2,4-Dinitrotoluene					39-139
Hexachloroethane					40-113
Nitrobenzene					35-180

COMPOUND	SPIKE ADDED (mg/kg)	DUPLICATE SPIKED SAMPLE CONCENTRATION (mg/kg)	% RPD	MSD % REC #	QC LIMITS	
					RPD	REC
1,4-Dichlorobenzene					≤86	20-124
2,4-Dinitrotoluene					≤46	39-139
Hexachloroethane					≤44	40-113
Nitrobenzene					≤72	35-180

Column to be used to flag recovery and RPD values with an asterisk

* = Values outside of QC limits

RPD: _____ Out of _____ outside limits

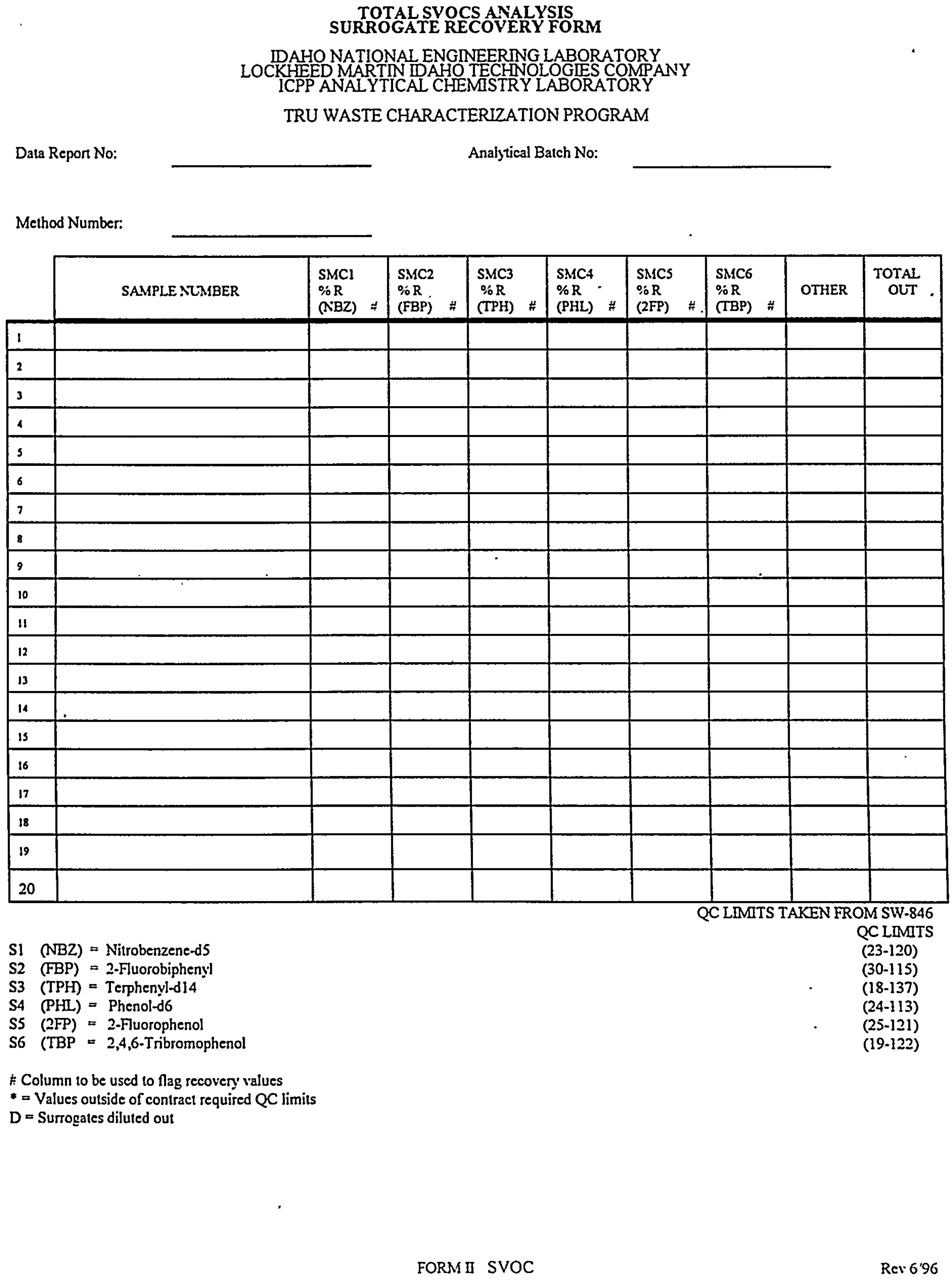
Spike Recovery: _____ Out of _____ outside limits

COMMENTS: ____

____

FORM III SVOC Rev 6/96

Figure 14-5. Example of matrix spike/matrix spike duplicate reporting form for total SVOC analysis.



TOTAL SVOCS ANALYSIS
SURROGATE RECOVERY FORM

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES COMPANY
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Data Report No: ____________ Analytical Batch No: ____________

Method Number: ____________

	SAMPLE NUMBER	SMC1 %R (NBZ) #	SMC2 %R (FBP) #	SMC3 %R (TPH) #	SMC4 %R (PHL) #	SMC5 %R (2FP) #	SMC6 %R (TBP) #	OTHER	TOTAL OUT
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									
11									
12									
13									
14									
15									
16									
17									
18									
19									
20									

QC LIMITS TAKEN FROM SW-846

	QC LIMITS
S1 (NBZ) = Nitrobenzene-d5	(23-120)
S2 (FBP) = 2-Fluorobiphenyl	(30-115)
S3 (TPH) = Terphenyl-d14	(18-137)
S4 (PHL) = Phenol-d6	(24-113)
S5 (2FP) = 2-Fluorophenol	(25-121)
S6 (TBP = 2,4,6-Tribromophenol	(19-122)

Column to be used to flag recovery values
* = Values outside of contract required QC limits
D = Surrogates diluted out

FORM II SVOC Rev 6'96

Figure 14-6. Example of surrogate spike reporting form for total SVOC analysis.

TOTAL SVOCS ANALYSIS
BLANK FORM

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES COMPANY
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Data Report No: ____________ Analytical Batch No: ____________

Lab Sample ID: ____________ Lab File ID: ____________

Date Extracted: ____________ Date Analyzed: ____________

Method Number: ____________

COMPOUND	CONCENTRATION (mg/kg)	Q
o-Methylphenol		
m & p-Methylphenol		
1,4-Dichlorobenzene		
ortho-Dichlorobenzene		
2,4-Dinitrophenol		
2,4-Dinitrotoluene		
Hexachlorobenzene		
Hexachloroethane		
Nitrobenzene		
Pentachlorophenol		
Pyridine		

Acceptance Limits: < 3 X MDLs

FORM IVA SVOC

Rev 6/96

Figure 14-7. Example of LB results reporting form for total SVOC analysis.

TOTAL SVOCS ANALYSIS
LABORATORY BLANK SUMMARY

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES COMPANY
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Data Report No: __________

Lab Sample ID: __________

Date Extracted: __________

Method Number: __________

Analytical Batch No: __________

Lab File ID: __________

Date Analyzed: __________

Time Analyzed: __________

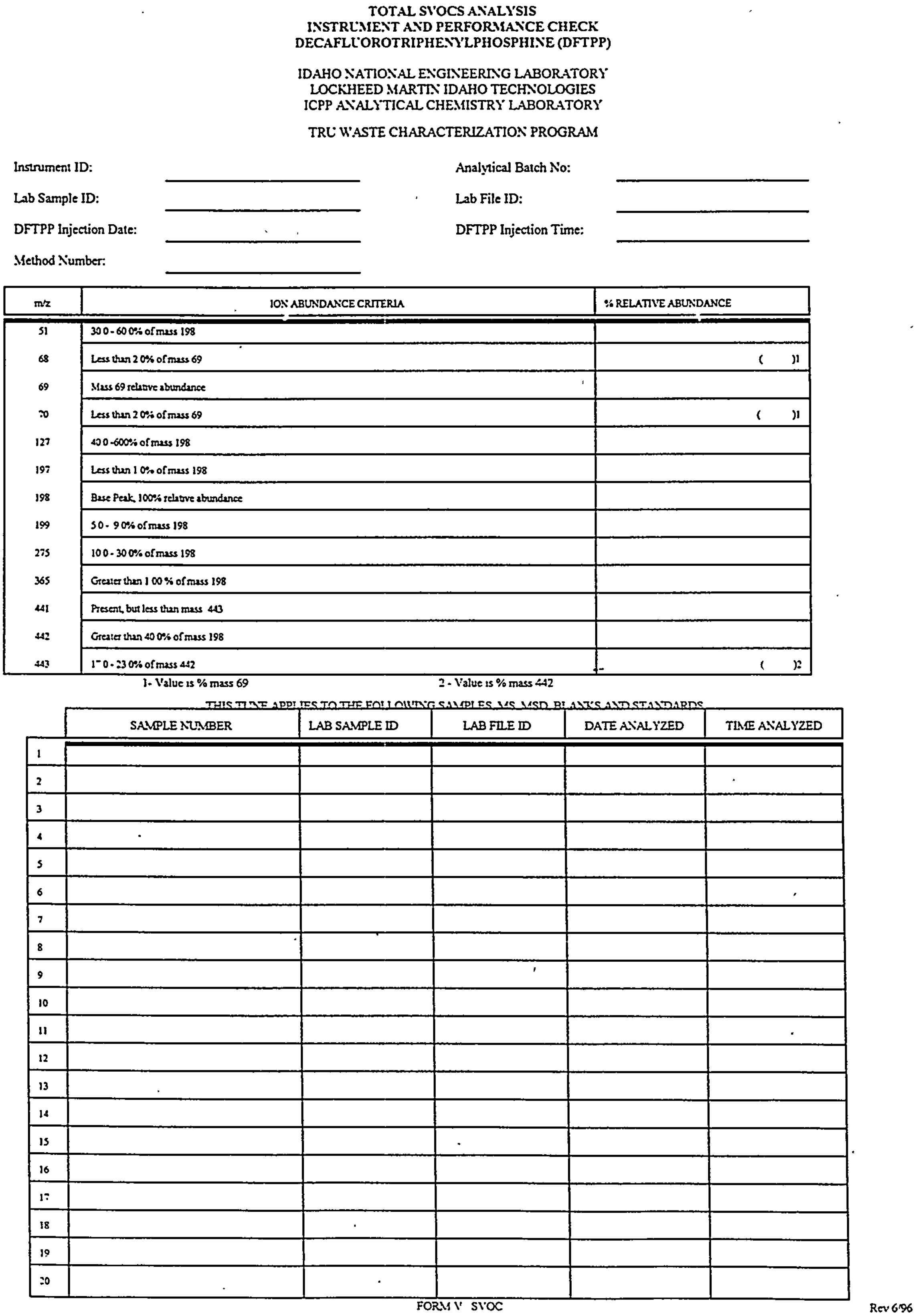
THIS BLANK APPLIES TO THE FOLLOWING SAMPLES, MS AND MSD

	FIELD SAMPLE ID	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED	TIME ANALYZED
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					

FORM IVB SVOC

Rev 6/96

Figure 14-8. Example of LB summary reporting form for total SVOC analysis.



TOTAL SVOCS ANALYSIS
INSTRUMENT AND PERFORMANCE CHECK
DECAFLUOROTRIPHENYLPHOSPHINE (DFTPP)

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Instrument ID: ____________ Analytical Batch No: ____________

Lab Sample ID: ____________ Lab File ID: ____________

DFTPP Injection Date: ____________ DFTPP Injection Time: ____________

Method Number: ____________

m/z	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE
51	30.0 - 60.0% of mass 198	
68	Less than 2.0% of mass 69	( )1
69	Mass 69 relative abundance	
70	Less than 2.0% of mass 69	( )1
127	40.0 -60.0% of mass 198	
197	Less than 1.0% of mass 198	
198	Base Peak, 100% relative abundance	
199	5.0 - 9.0% of mass 198	
275	10.0 - 30.0% of mass 198	
365	Greater than 1.00 % of mass 198	
441	Present, but less than mass 443	
442	Greater than 40.0% of mass 198	
443	17.0 - 23.0% of mass 442	( )2

1- Value is % mass 69 2 - Value is % mass 442

THIS TUNE APPLIES TO THE FOLLOWING SAMPLES, MS, MSD, BLANKS AND STANDARDS

	SAMPLE NUMBER	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED	TIME ANALYZED
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					

FORM V SVOC

Rev 6/96

Figure 14-9. Example of DFTPP tune reporting form for total SVOC analysis.

TOTAL SVOCS ANALYSIS
INTERNAL STANDARD AREA AND RT SUMMARY

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES COMPANY
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Analytical Batch No: ____________

CCAL Sample ID: ____________

CCAL Lab File ID: ____________

Date Analyzed: ____________

Method Number: ____________

		IS1 (DCB) AREA #	RT	IS2 (NPT) AREA #	RT	IS3 (ANT) AREA #	RT
	12 HOUR STD						
	UPPER LIMIT						
	LOWER LIMIT						
	SAMPLE ID						
01							
02							
03							
04							
05							
06							
07							
08							
09							
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							

IS1 (DCB) = 1,4-Dichlorobenzene-d4
IS2 (NPT) = Naphthalene-d8
IS3 (ANT) = Acenaphthene-d10

UPPER LIMIT = - 100% of internal standard area

LOWER LIMIT = - 50 % of internal standard area

RT Upper Limit = – 30 seconds of internal standard RT
Column used to flag internal standard area values

RT Lower Limit = - 30 seconds of internal standard RT
* = Values outside of QC limits

FORM VIIIA SVOC-1

Rev 6 96

Figure 14-10. Example of internal standard area reporting form for total SVOC analysis.

TOTAL SVOCS ANALYSIS
INTERNAL STANDARD AREA AND RT SUMMARY

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES COMPANY
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Analytical Batch No: ____________

CCAL Sample ID: ____________

CCAL Lab File ID: ____________

Date Analyzed: ____________

Method Number: ____________

		IS4 (PHN) AREA #	RT	IS5 (CRY) AREA #	RT	IS6 (PRY) AREA #	RT
	12 HOUR STD						
	UPPER LIMIT						
	LOWER LIMIT						
	SAMPLE ID						
01							
02							
03							
04							
05							
06							
07							
08							
09							
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							

IS4 (PHN) = Phenathrene-d10
IS5 (CRY) = Chrysene-d12
IS6 (PRY) = Perylene-d12

UPPER LIMIT = + 100% of internal standard area

LOWER LIMIT = - 50 % of internal standard area.

RT Upper Limit = + 30 seconds of internal standard RT
= Column used to flag internal standard area values

RT Lower Limit = - 30 seconds of internal standard RT
* = Values outside of QC limits

FORM VIIIA SVOC-2

Rev 6/96

Figure 14-10. (continued).

TOTAL SVOCS ANALYSIS
CONTINUING CALIBRATION CHECK

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES COMPANY
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Instrument ID: ______________

Initial Calibration Date(s): ______________

Initial Calibration Time(s): ______________

Lab Sample ID: ______________

Analytical Batch No: ______________

Calibration Date: ______________

Calibration Time: ______________

Lab File ID: ______________

Method Number: ______________

COMPOUND	$\overline{\text{RRF}}$	RRF _____	%D
o-Methylphenol			
m & p-Methylphenol			
1,4-Dichlorobenzene *			
ortho-Dichlorobenzene			
2,4-Dinitrophenol **			
2,4-Dinitrotoluene			
Hexachlorobenzene			
Hexachloroethane			
Nitrobenzene			
Pentachlorophenol *			
Pyridine			

CCC- Calibration Check Compounds (*)
%D ≤ 20 %

SPCC- System Performance Check Compounds (**)
RRF ≥ 0.05

Figure 14-11. Example of CCAL reporting form for total SVOC analysis.

TOTAL SVOCS ANALYSIS
CONTINUING CALIBRATION CHECK
INTERNAL STANDARD AREA AND RT SUMMARY

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES COMPANY
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Analytical Batch No: ________________

ICAL/CCAL Sample ID: ________________

Daily CCAL Sample ID: ________________

ICAL/CCAL Lab File ID: ________________

Daily CCAL Lab File ID: ________________

Date Analyzed: ________________

Date Analyzed: ________________

Method Number: ________________

		IS1 (DCB) AREA	#	RT	IS2 (NPT) AREA	#	RT	IS3 (ANT) AREA	#	RT
Prev CCAL	12 HOUR STD									
	UPPER LIMIT									
	LOWER LIMIT									
Continuing Calibration Check										

IS1 (DCB) = 1,4-Dichlorobenzene-d4
IS2 (NPT) = Naphthalene-d8
IS3 (ANT) = Acenaphthene-d10

RT Upper Limit = – 30 seconds of internal standard RT
Column used to flag internal standard area values

[a] Last CCAL or ICAL whichever is most recent

UPPER LIMIT = + 100% of internal standard area.

LOWER LIMIT = - 50 % of internal standard area.

RT Lower Limit = - 30 seconds of internal standard RT
* = Values outside of QC limits

FORM VIIIB SVOC-1

Rev 6/96

Figure 14-12. Example of CCAL Internal Standard Area/RT reporting form for total SVOC analysis.

TOTAL SVOCS ANALYSIS
CONTINUING CALIBRATION CHECK
INTERNAL STANDARD AREA AND RT SUMMARY

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES COMPANY
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Analytical Batch No: ______

ICAL/CCAL Sample ID: ______
Daily CCAL Sample ID: ______

ICAL/CCAL Lab File ID: ______
Daily CCAL Lab File ID: ______

Date Analyzed: ______
Date Analyzed: ______

Method Number: ______

		IS4 (PHN) AREA	#	RT	IS5 (CRY) AREA	#	RT	IS6 (PRY) AREA	#	RT
	12 HOUR STD									
Prev	UPPER LIMIT									
CCAL	LOWER LIMIT									
Continuing Calibration Check										

IS4 (PHN) = Phenathrene-d10
IS5 (CRY) = Chrysene-d12
IS6 (ANT) = Perylene-d12

UPPER LIMIT = + 100% of internal standard area.

LOWER LIMIT = - 50 % of internal standard area.

RT Upper Limit = + 30 seconds of internal standard RT
Column used to flag internal standard area values

RT Lower Limit = - 30 seconds of internal standard RT
* = Values outside of QC limits

[a] Last CCAL or ICAL whichever is most recent

FORM VIIIB SVOC-2

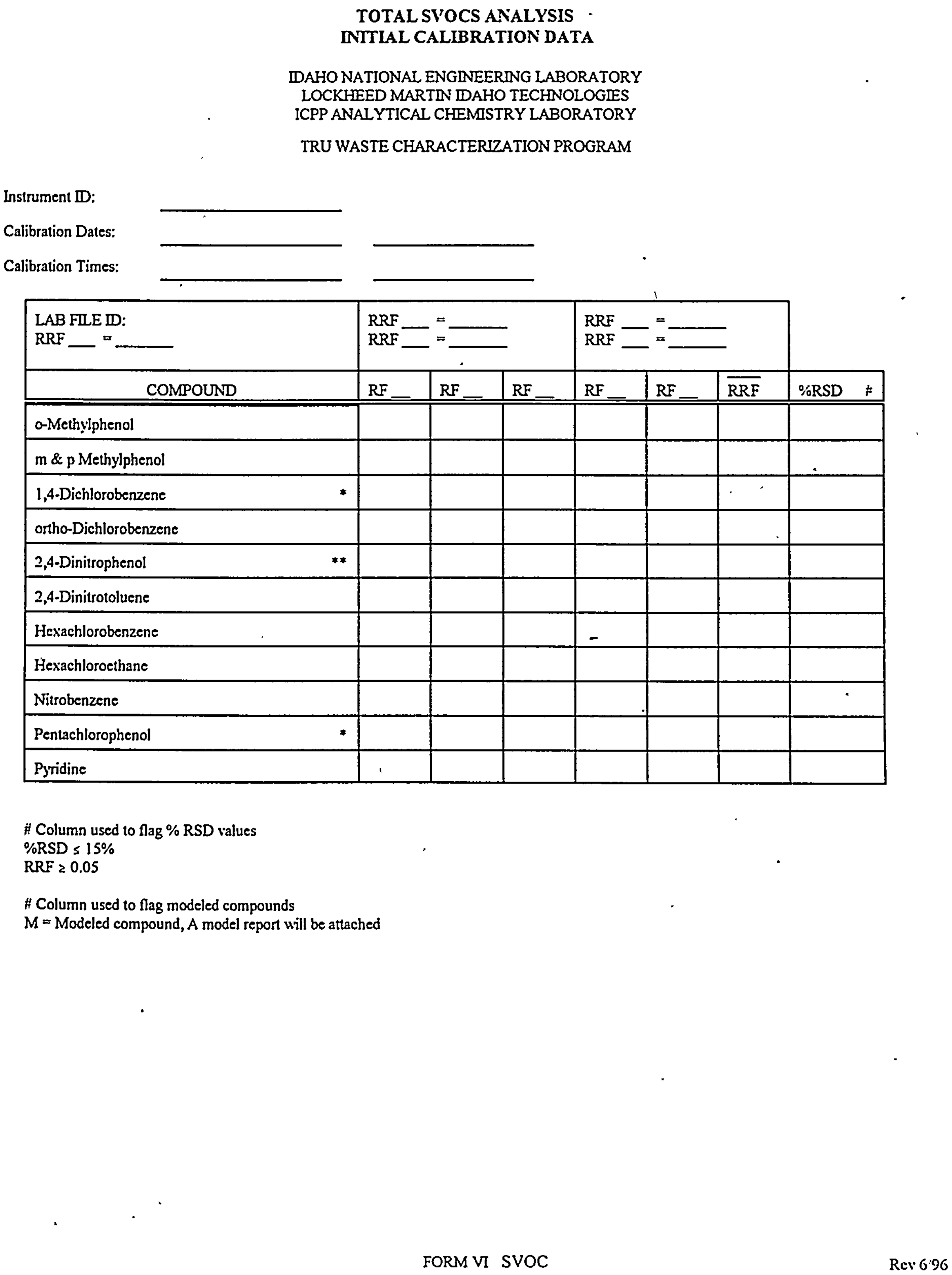
Rev 6/96

Figure 14-12. (continued).

- **ACMM 9271 Calibration Records**, filed under Calibration Records according to method, instrument, and ICAL number, which include standard preparation logs for all calibration standards (source reference), raw data for the ICAL and associated BFB tune, method identification, calibration date and time, %RSD calculations, and report forms providing results of the ICAL (see example in Figure 14-13).

- **MDL records**, filed by MDL determination date and instrument identification, which include all raw data and calculations for MDL determinations, along with the MDL reporting form (see example in Figure 14-14).

- **Original COC forms**, filed in the data package.



TOTAL SVOCS ANALYSIS
INITIAL CALIBRATION DATA

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Instrument ID: ______________

Calibration Dates: ______________ ______________

Calibration Times: ______________ ______________

LAB FILE ID: RRF ___ = ______	RRF ___ = ______ RRF ___ = ______			RRF ___ = ______ RRF ___ = ______			
COMPOUND	RF __	RF __	RF __	RF __	RF __	$\overline{RRF}$	%RSD #
o-Methylphenol							
m & p Methylphenol							
1,4-Dichlorobenzene *							
ortho-Dichlorobenzene							
2,4-Dinitrophenol **							
2,4-Dinitrotoluene							
Hexachlorobenzene							
Hexachloroethane							
Nitrobenzene							
Pentachlorophenol *							
Pyridine							

Column used to flag % RSD values
%RSD ≤ 15%
RRF ≥ 0.05

# Column used to flag modeled compounds
M = Modeled compound, A model report will be attached

FORM VI SVOC

Rev 6/96

Figure 14-13. Example of ICAL reporting form for total SVOC analysis.

TOTAL SVOCS ANALYSIS
MDL REPORTING FORM

IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES COMPANY
ICPP ANALYTICAL CHEMISTRY LABORATORY

TRU WASTE CHARACTERIZATION PROGRAM

Instrument ID: ______________________

MDL Determination Dates: __________ __________

Method Number: ______________________

COMPOUND	MDL (mg/kg)	Program Required MDL (mg/kg)	PRQL (mg/kg)
o-Methylphenol		5	40
m& p-Methylphenol		5	40
1,4-Dichlorobenzene		5	40
ortho-Dichlorobenzene		5	40
2,4-Dinitrophenol		5	40
2,4-Dinitrotoluene		0.3	2.6
Hexachlorobenzene		0.3	2.6
Hexachloroethane		5	40
Nitrobenzene		5	40
Pentachlorophenol		5	40
Pyridine		5	40

FORM X SVOC

Rev 6/96

Figure 14-14. Example of MDL reporting form for total SVOC analysis.

15. TOTAL METALS ANALYSIS

This section identifies the required QA elements for the analysis of total metals in samples of homogenous solids and soil/gravel. Total metals are those solubilized by hot acid leaching (e.g., SW-846 Method 3051).

15.1 Quality Assurance Objectives

The program DQOs for total metals analysis are listed in Section 1.5 of this document. The QA objectives specified in Table 15-1 were developed in order to ensure the generation of total metals data that meet the program DQOs. Key data quality indicators for laboratory measurements are defined below and the methods to qualitatively and quantitatively assess these indicators are discussed in Section 3 of this QAPjP.

15.1.1 Precision

Precision is assessed by analyzing laboratory matrix spike duplicates (MSDs), replicate analyses of laboratory control samples (LCSs) and PDP blind audit samples. Results from these measurements are compared to the criteria listed in Table 15-1. These QC measurements are used to demonstrate acceptable method performance and to trigger CA when specification limits are exceeded.

15.1.2 Accuracy

Accuracy as %R is assessed for the laboratory operations by analyzing LCSs, matrix spikes (MSs), and PDP blind audit samples. Results from these measurements are compared to the criteria listed in Table 15-1. These QC measurements are used to demonstrate acceptable method performance and to trigger CA when specification limits are exceeded.

15.1.3 Instrument Detection Limits

Instrument detection limits (IDLs) are expressed in $\mu g/\ell$ for metals. Maximum permissible values for IDLs are the program required detection limits (PRDLs) listed in Table 15-1. IDLs must be determined at least every six months, and must be less than or equal to the PRDLs for the method used to quantitate a specific analyte. For high concentration samples, an exception to the above requirement is made in cases where the sample concentration exceeds five times the IDL of the instrument being used. In these cases, the analyte concentration may be reported even though the IDL may exceed the PRDL. Detailed procedures for IDL determination, which are based those of the EPA Contract Laboratory Program (CLP) (EPA, 1994b) and similar to the MDL determination method described by Glaser et al. (1981), are included in the appropriate ACMM methods.

15.1.4 Program Required Quantitation Limits

The capability to quantitate analytes at or below the PRQL concentrations listed in Table 15-1 is demonstrated by semiannual checks of IDL compliance and by setting the concentration of at least one calibration standard below the solution-equivalent of the mg/kg PRQL (assuming the same dilution factor as is used for sample preparation) for each analyte.

Table 15-1. Total metals target analyte list and QA objectives.

Analyte	CAS Number	Precision[a] (%RSD or RPD)	Accuracy[a] (%R)	PRDL[b] (ug/L)	PRQL[b] (mg/kg)	Completeness (%)
Antimony	7440-36-0	≤30	80–120	100	100	90
Arsenic	7440-38-2	≤30	80–120	100	100	90
Barium	7440-39-3	≤30	80–120	2000	2000	90
Beryllium	7440-41-7	≤30	80–120	100	100	90
Cadmium	7440-43-9	≤30	80–120	20	20	90
Chromium	7440-47-3	≤30	80–120	100	100	90
Lead	7439-92-1	≤30	80–120	100	100	90
Mercury	7439-97-6	≤30	80–120	4.0	4.0	90
Nickel	7440-02-0	≤30	80–120	100	100	90
Selenium	7782-49-2	≤30	80–120	20	20	90
Silver	7440-22-4	≤30	80–120	100	100	90
Thallium	7440-28-0	≤30	80–120	100	100	90
Vanadium	7440-62-2	≤30	80–120	100	100	90
Zinc	7440-66-6	≤30	80–120	100	100	90

---

a. ≤ 30% specification limit applies when sample and duplicate (or MS and MSD) concentrations are ≥ 10 x IDL for ICP-AES and AA techniques. If less than these limits, the absolute difference between the two values shall be less than or equal to the PRDL.

b. Applies to LCSs. If a solid LCS that has established statistical control limits is used, then the established control limits for that material should be used for accuracy requirements.

c. PRDL set such that it is a factor of 10 below the PRQL for 100% solid samples, assuming a 100X dilution during digestion.

PRDL = Program required detection limit (i.e., maximum permissible value for IDL)
PRQL = Program required quantitation limit

15.1.5 Completeness

Laboratory analysis completeness is expressed as the number of samples analyzed with valid results (see completeness definition) as a percent of the total number of samples submitted for analysis. Ninety percent of the total number of samples submitted for analysis must result in valid analytical data.

15.1.6 Comparability

The comparability of ACL data sets to those generated by different sites is achieved through the use of standardized methods and traceable standards, and by participation in the PDP.

15.1.7 Representativeness

Representativeness of total metals analyses is achieved by use of standardized sample handling protocols (e.g., storage, aliquotting) and analysis of laboratory blanks to maintain sample integrity.

15.2 Methods Requirements

ACL uses acid digestion followed by several spectrochemical analyses to determine total metals in TWCP solid samples. These ACMM methods, listed below, are based on EPA SW-846 and TWCP Methods Manual protocols:

- ACMM 8909, *Microwave Assisted Digestion of Homogeneous Solids and Soil/Gravel*: A microwave digestion technique utilizing aqua regia, derived from EPA SW-846 Method 3051A, and TWCP Method 610.1.
- ACMM 2900, *Determination of Trace Metals in Environmental Samples by ICP Emission Spectrometry*: A determinative method using inductively coupled plasma atomic emission spectrometry (ICP-AES) to determine total metals concentrations in digested samples, based by EPA SW-846 Method 6010A and TWCP Method 640.1
- ACMM 2350, *Determination of Arsenic and Selenium in Environmental Samples Using Graphite Furnace Atomic Absorption Spectrometry*: A determinative method using atomic absorption spectrometry with electrothermal atomization to determine total metals concentrations in digested samples; also called graphite furnace atomic absorption (GFAA), based on EPA SW-846 7000 Series methods and TWCP Method 650.2.
- ACMM 7802, *Determination of Mercury by Cold-Vapor Fluorescence Spectrophotometry*: A determinative method using cold-vapor atomic fluorescence spectrometry (CVAF) to determine total mercury concentrations in digested samples, based on EPA SW-846 Method 7471A and TWCP Method 650.3.
- ACMM 7801, *Determination of Mercury by Cold-Vapor Atomic Absorption Spectrophotometry*: A determinative method using cold-vapor atomic absorption spectrometry (CVAA) to determine total mercury concentrations in digested samples, based on EPA SW-846 Method 7471 and TWCP Method 650.3.
- ACMM 8969, *Determination of Percent Solids*: A procedure used to determine percent solids, based on EPA CLP protocol.

All metals except mercury are determined digestion method ACMM 8909 followed by analyses using ACMM 2900 (ICP-AES). Method 2350 (GFAA) is used for selenium if acceptable IDLs cannot be achieved by ICP-AES. Sample preparation for mercury determination is performed using an acid/potassium permanganate digestion; instructions for sample preparation are included in ACMM 7802. ACMM 7802 (CVAF) is routinely used for mercury determination; ACMM 7801 (CVAA) is used only in case of CVAF instrument failure. Percent solids is determined on all samples at the time of sample digestion. Detailed operating procedures for these methods, including calibration requirements, sample analysis, and data reduction, are specified in the referenced ACMM methods.

15.2.1 Criteria for Standards

Primary standards may be purchased from the best available source for the analytes listed in Table 15-1. Commercially-purchased primary standards must be certified by the manufacturer, and their concentrations must be traceable to NIST, EPA, or other nationally-recognized standards. Commercially prepared stock solutions must not be used beyond their manufacturer-specified shelf-life. ACMM methods, SOP R.1.20, *Analytical Chemistry Quality Control Program*, ACLP 1.01, *Preparation of Quality Control Reagents and Standards*, and ACLP 2.05, *Control, Distribution and Use of Spectrochemical Standards*, specify detailed preparation and documentation requirements for the preparation of all metals standards in the laboratory. These procedures comply with the frequency, source, and preparation requirements of Section 15.2 of the TWCP QAPP.

15.2.2 Criteria for ICP-AES Qualitative and Quantitative Analysis

All samples must be digested using ACMM Method 8909 prior to instrument analysis. The cited analytical methods specify reagent purity, instrument operating conditions, background correction procedures, and interference detection and evaluation. A minimum of two replicate emission measurements are made for each standard and sample, with the average emission values used for quantitation. All analytes are quantitated within the calibration range of the ICP-AES for determinative analysis. Multiple dilutions of sample digestates are analyzed as necessary to meet this requirement.

Interelement interference correction factors (IECFs) are determined at least annually for each ICP-AES used to support TWCP. Concentrations of interfering elements (typically aluminum, calcium, magnesium, iron and uranium) are quantitated in each sample. Interelement correction factors are applied manually after data generation for samples having interfering element concentrations sufficient to cause an interference effect of a magnitude exceeding 5 times the instrument IDL.

15.2.3 Criteria for GFAA Qualitative and Quantitative Analysis

All samples must be digested using ACMM Method 8909 prior to instrument analysis. The cited analytical methods specify reagent purity, instrument operating conditions, and background correction procedures. Two replicate furnace injections are made for each standard and sample, with the average absorbance value used for quantitation. All analytes are quantitated within the calibration range of the GFAA for determinative analysis. Multiple dilutions of sample digestates are analyzed as necessary to meet this requirement.

The choice of matrix modifier is documented in ACMM 2350. Smith-Heiftje background correction is used on the GFAA for all sample analyses.

15.2.4 Criteria for Mercury (CVAF & CVAA) Qualitative and Quantitative Analysis

All samples must be digested using ACMM Method 7802 (acid/permanganate digestion) prior to instrument analysis. The cited analytical methods specify reagent purity, instrument operating conditions, and background correction procedures. Mercury determination is routinely performed by atomic fluorescence, which is more sensitive than atomic absorption. A single 60-second integration using the flow-through system is used for CVAF quantitation. Background correction is not used on the CVAF system. All analytes are quantitated within the calibration range of the CVAF for determinative analysis. Multiple dilutions of sample digestates are analyzed as necessary to meet this requirement.

If the CVAA contingency method, ACMM 7801, is used, the sample preparation procedures in ACMM 7802 are followed. A minimum of two replicate absorption measurements are made for each standard and sample, and the average absorbance value used for quantitation. Background correction is used as necessary to compensate for non-analyte absorbance at the quantitation wavelength. All analytes are quantitated within the calibration range of the CVAA for determinative analysis. Multiple dilutions of sample digestates are analyzed as necessary to meet this requirement.

15.3 Quality Control

To ensure that data of known and documented quality are generated, the QC criteria specified in this section must be met for all total metals analyses. The ALD QAO is responsible for monitoring and documenting procedure performance, including the analysis of laboratory control samples, laboratory blanks, matrix spikes and matrix spike duplicates. The ALD QAO, the inorganic chemistry section supervisor, the spectrochemistry TL and the special analysis TL are responsible for implementing CAs when acceptable procedure performance, as specified in this section, is not met.

Specific laboratory QC samples and frequencies are summarized in Table 15-2. Analytical QC samples are associated with field samples through the use of analytical batches (see Section 6.3).

15.3.1 Method Performance Samples

Before the analysis of any samples for TWCP, acceptable method performance must be demonstrated for each method to be used. This demonstration consists of determination of IDLs (see Section 15.1.3) and analysis of method performance samples (MPSs). Initially, seven replicate MPSs (standards containing known concentrations of all analytes) must be digested and analyzed to demonstrate that the criteria specified for precision and accuracy listed in Table 15-1 can be met. If the seven replicates do not meet the criteria, then seven more replicates must be analyzed until the initial procedure performance demonstration criteria are met.

Continuing acceptable procedure performance is demonstrated semiannually by analyzing four replicate MPSs. If the precision and accuracy criteria specified in Table 15-1 are not met for the four replicates, four additional replicates must be analyzed until the criteria in Table 15-1 are met.

15.3.2 Laboratory Control Samples

LCSs are analyzed at a minimum frequency of one per analysis batch. A single LCS may not be shared between two separate analysis batches. LCSs are prepared from commercially-purchased primary standards that are independent (i.e., different manufacturer) from those used for instrument calibration. The LCS used for metals analysis is a solid matrix that matches the expected sample matrix to the greatest extent possible; whenever possible, PDP samples from previous cycles are used

Table 15-2. Summary of laboratory QC samples and frequencies for total metals analysis.

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
Method performance samples	Seven (7) samples initially and four (4) semiannually	Meet Table 15-1 QA objectives	Repeat until acceptable
Laboratory blanks	One (1) per analytical batch	Analyte concentrations <3 × PRDLs	See Section 15.3.3
Laboratory control samples	One (1) per analytical batch	80% ≤ %R ≤ 120%	See Section 15.3.2
Matrix spikes	One (1) per analytical batch	80% ≤ %R ≤ 120%	See Section 15.3.4
Matrix spike duplicates	One (1) per analytical batch	80% ≤ %R ≤ 120% RPD ≤ 30	See Section 15.3.4
Blind audit samples	Samples and frequency controlled by the Solid PDP Plan	Specified in the Solid PDP Plan	Specified in the Solid PDP Plan

as the metals LCS. The LCS must contain quantifiable concentrations of all target analytes. LCSs must undergo all sample preparation procedures (i.e., digestion) performed on field samples.

LCS results are acceptable if the recoveries for all analytes are between 80 and 120% (i.e., 80% ≤ %R ≤ 120%). If LCS results do not meet this criteria, the LCS may be reanalyzed once to check for instrument error; if the results of the rerun LCS meet specifications, sample analysis may continue. If the rerun LCS is still noncompliant, the samples in the associated analytical batch must be redigested and reanalyzed for those analytes whose recoveries were noncompliant. If a noncompliant LCS is associated with any sample results reported to the SPO, an NCR must be initiated.

15.3.3 Laboratory Blanks

Laboratory blanks consist of all reagents used in the sample digestion process in equal proportions to those added to the samples. LBs are analyzed at a minimum frequency of one per analytical batch. The LBs must undergo all sample preparation procedures (i.e., digestion) performed on the associated field samples.

LBs are acceptable if analyte concentrations are less than three times the Table 15-1 PRDLs for all target analytes. If the LB is not acceptable, all samples in the analytical batch having analyte concentrations which are ≤ 10 times the blank concentration and ≥ 0.5 PRQL must be redigested and reanalyzed for those analytes which were noncompliant in the LB. If a noncompliant LB is associated with any sample data reported to the SPO, an NCR must be initiated.

15.3.4 Matrix Spikes and Matrix Spike Duplicates

Duplicate matrix spikes on individual field samples are performed at the minimum frequency of one pair (MS plus MSD) per analysis batch. MS and MSDs must contain all of the Table 15-1 target analytes at concentrations ≥ PRQL. Although PRQL concentrations are used as the spiking default, analyte concentrations in the spikes may be adjusted based on process knowledge to ensure that the spike added amount is sufficiently great compared to the indigenous sample concentration (e.g., at least ¼ the sample concentration) so that calculated spike recoveries are meaningful.

Matrix spike and matrix spike duplicate recoveries are acceptable if all analytes are recovered between 80% and 120% when the spike added amount was greater than ¼ the sample concentration. The duplicate precision between MS and MSDs is acceptable if the RPDs between matrix spike and matrix spike duplicate results (mg/kg) are ≤ 30 when both MS and MSD concentrations equal or exceed 10 times the IDL, or if the absolute difference between the two values is ≤ PRDL when one or both of the MS/MSD concentrations are less than 10 times the IDL.

If MSs and MSDs do not meet the precision and accuracy criteria, the noncompliance is documented on checklists and in the data report narrative. The implications of the noncompliance on data quality is discussed in the data report narrative. NCRs are not initiated for noncompliant MS and MSD results because these results are highly dependent upon individual sample matrices, and specific corrective actions cannot be identified.

15.3.5 Blind Audit Samples

ACL participates in the RCRA solid PDP as specified by program requirements (see Section 2.7) on a nominal semiannual basis. PDP samples are analyzed and reported using the same methods and handling procedures as are used on field samples.

15.4 Instrument Testing, Inspection, and Maintenance Requirements

ACL monitors spectrometry instrumentation performance through the use of performance check routines for each instrument. ICP-AES performance is checked prior to calibration by documenting the emission intensities ratio of specified concentrations of copper and manganese. GFAA and CVAF instrument performance is checked by monitoring the absorbance/fluorescence of the midrange calibration standard over time.

Instrument maintenance procedures are discussed in Section 4.3.

15.5 Instrument Calibration and Frequency

The spectrometer calibration requirements are summarized in Table 15-3. Detailed instructions for calibrating the ICP-AES, GFAA, CVAF and CVAA are provided in the specific ACMM procedures referenced in Section 15.1. ACL maintains instrument use logs from which calibration sequences and frequencies may be reconstructed.

Table 15-3. Summary of calibration requirements and analysis QC for total metals Analysis.

Technique	Procedure	Frequency of Procedure	Acceptance Criteria	Corrective Action
ICP-AES	Initial calibration (minimum 1 standard + blank)	Daily	ICV %R: 90–110% High Std %R: 95–105% ICB: ≤ 3 x PRDL	Correct problem and repeat initial calibration
	Continuing calibration	Every 10 samples plus beginning and end of run	CCV %R: 90–110% CCB: ≤ 3 x PRDL	Recalibrate and rerun last ten samples
	Interference correction verification	Beginning and end of run or twice per 8 hours, whichever is more frequent	ICSA: ≤ 3 x PRDL for analytes ICSAB: 80–120 %R for analytes	Correct problem and recalibrate
	Serial dilution	Once per analytical batch or per matrix within an analytical batch	If sample is > 50 x IDL, 5X dilution must be ≤ 10 %D of initial value	See Section 15.5.1
	Post-digestion spike	Once per analytical batch or per matrix within an analytical batch if serial dilution, MS or MSD does not meet acceptance criteria	75–125 %R	See Section 15.5.1
GFAA	Initial calibration (minimum 3 standards + blank)	Daily	ICV %R: 90–110% High Std %R: 95–105% ICB: ≤ 3 x PRDL r ≥ 0.995	Correct problem and repeat initial calibration
	Continuing calibration	Every 10 sample injections plus beginning and end of run	CCV %R: 80–120% CCB: ≤ 3 x PRDL	Recalibrate and rerun last 10 samples
	Serial dilution	Once per analytical batch or per matrix within an analytical batch	If sample is > 25 x IDL, 5X dilution must be ≤ 10 %D of initial value	Use MSA to quantitate samples of like matrix
GFAA	Post-digestion spike	Once per analytical batch or per matrix within an analytical batch	85–115 %R	Use MSA to quantitate samples of like matrix
CVAF & CVAA	Initial calibration (minimum of 5 standards + blank)	Daily	ICV %R: 90–110% High Std %R: 95–105% ICB: ≤ 3 x PRDL r ≥ 0.995	Correct problem and repeat initial calibration
	Continuing calibration	Every 10 samples plus beginning and end of run	CCV %R: 80–120% CCB: ≤ 3 x PRDL	Recalibrate and reanalyze last 10 samples
	Serial dilution	Once per analytical batch or per matrix within an analytical batch	If sample is > 25 x IDL, 5X dilution must be ≤ 10 %D of initial value	Use MSA to quantitate samples of like matrix
	Post-digestion spike	Once per analytical batch or per matrix within an analytical batch	85–115 %R	Use MSA to quantitate samples of like matrix

15.5.1 ICP-AES Calibration

The ICP-AES is calibrated daily according to the requirements in Table 15-3. Three standards and a blank are used to calibrate the ICP-AES. A minimum of two replicate emission measurements are made for each standard and blank, and the average emissions are used to generate the calibration regression. The initial calibration verification (ICV) standard, which is prepared from an independent source and contains all analytes at midrange concentrations, is used to verify correct preparation of the calibration standards. All other verification and check standards may be from the same source as the calibration standards. All initial calibration verifications (i.e., the ICV, ICB, and the high calibration standard analyzed as a sample) must be successfully completed before analysis of analytical samples. Analytical samples are defined as any field sample and all QC samples except for initial calibration verifications, continuing calibration verifications (CCVs) and initial and continuing calibration blank verifications (ICBs and CCBs).

If continuing calibration verification results (CCV and CCB) do not meet the acceptance criteria for any analyte, the analytical run for that analyte is terminated, the problem corrected (e.g., by recalibration) and all samples run since the last compliant calibration verification are reanalyzed.

If ICP-AES serial dilution or post-digestion spike results do not meet the acceptance criteria listed in Table 15-3, the noncompliance is documented on review checklists and in the data report narrative. The implications of the noncompliance on data quality are discussed in the narrative of the data report.

15.5.2 GFAA Calibration

The GFAA is calibrated daily according to the requirements in Table 15-3. Four standards and a blank are used to calibrate the GFAA. Two replicate injections are made for each standard and blank, and the average absorbance measurements are used to generate the calibration regression. The initial calibration verification (ICV) standard, which is prepared from an independent source and contains analyte at midrange concentration, is used to verify correct preparation of the calibration standards. All other verification and check standards may be from the same source as the calibration standards. All initial calibration verifications (i.e., the ICV, ICB, and the high calibration standard analyzed as a sample) must be successfully completed before analysis of analytical samples.

Continuing calibration verifications (CCV and CCB) are analyzed every 10 analytical sample injections (i.e., every 5 samples, dual injection). If continuing calibration verification results (CCV and CCB) do not meet the acceptance criteria for any analyte, the analytical run for that analyte is terminated, the problem corrected (e.g., by recalibration) and all samples run since the last compliant calibration verification are reanalyzed.

If GFAA serial dilution or post-digestion spike results do not meet the acceptance criteria listed in Table 15-3, samples of like matrix are quantitated using the Method of Standard Additions (MSA). The use of MSA for quantitation is discussed in the data report narrative.

15.5.3 Mercury Calibration

The CVAF is calibrated daily according to the requirements in Table 15-3. Five standards and a blank are used to calibrate the CVAF. A single 60-second integration is made for each standard and blank, and the fluorescence measurements are used to generate the calibration regression. The initial calibration verification (ICV) standard, which is prepared from an independent source and contains mercury at midrange concentration, is used to verify correct preparation of the calibration standards. All other verification and check standards may be from the same source as the calibration standards. All initial calibration verifications (i.e., the ICV, ICB, and the high calibration standard analyzed as a sample) must be successfully completed before analysis of analytical samples.

If continuing calibration verification results (CCV and CCB) do not meet the acceptance criteria for any analyte, the analytical run for that analyte is terminated, the problem corrected (e.g., by recalibration) and all samples run since the last compliant calibration verification are reanalyzed.

If CVAF serial dilution or post-digestion spike results do not meet the acceptance criteria listed in Table 15-3, samples of like matrix are quantitated using the Method of Standard Additions (MSA). The use of MSA of quantitation is discussed in the data report narrative.

With the exception of the signal measurement used, CVAA calibration follows the same protocol as outlined for CVAF. The average absorbance from a minimum of two replicate measurements for each standard and blank are used to establish the calibration regression instead of the single 60-second integration.

15.6 Data Management

Data management includes requirements for data reduction, data validation, and reporting. All of the data management requirements defined in Section 3 of this QAPjP, as well as the specific procedures described below, apply to total metals analysis data. Specific equations and sample calculations are detailed in the associated ACL ACMM procedures.

15.6.1 Data Reduction

All results for field samples are reported in units of mg/kg on a wet-weight basis and are limited to two significant figures. All calculations (raw data reduction and QC results) are performed prior to rounding.

Target analyte concentrations are not blank corrected. Blanks are treated and reported in the same manner as other samples.

15.6.2 Data Validation

All total metals data are reviewed by an independent technical reviewer prior to report generation. Review checklists are used to document the independent technical review process. Example checklists for ICP-AES, GFAA and CVAF/CVAA are provided in Figures 15-1, 15-2 and 15-3, respectively.

SPECTROCHEMISTRY ICP-AES INDEPENDENT DATA REVIEW CHECKLIST

Analytical Batch #:	Method: 2900 Metals by ICP-AES (TWCP Method 640.1)			Form Revision:0
Run # and Analysis Date	Data Generator	Independent Reviewer	Review Date	OK for Release?
1.				Yes or No
2.				Yes or No

Instructions: Complete one checklist per Analytical Batch. If more than 2 instrument runs were needed to complete the batch analysis, use a second checklist. Enter appropriate responses for each question. Each "No" response requires an explanation.

REQUIREMENT	RUN #1		RUN #2		COMMENTS
	YES	NO	YES	NO	
1. INITIAL CALIBRATION (ICAL)					
a. Was the initial calibration performed using a minimum of 1 standard and a blank?					
2. INITIAL CALIBRATION VERIFICATION					
a. Was the highest calibration standard analyzed as a sample and recovered within 95-105% for each reported analyte?					
b. Was an ICV analyzed after the initial calibration and prior to analysis of any samples?					
c. Are the recoveries for each reported analyte in the ICV between 90% and 110%?					
d. Is the ICV standard from a different source from the initial calibration standards?					
e. Are Initial Calibration Verification Forms and Linear Range Check Forms present and correct for all ICVs and high standard checks associated with the run?					
3. CONTINUING CALIBRATION VERIFICATION (CCV)					
a. Was a CCV analyzed prior to the analysis of samples?					
b. Is the percent recovery of each reported analyte between 90% and 110%?					
c. Was a compliant CCV analyzed for reported analytes at the completion of the analytical run?					
d. Were every 10 samples bracketed by a compliant CCV for reported analytes?					
e. Are Calibration Verification Forms present and correct for all CCVs associated with the run?					
4. BLANKS					
a. Was at least one laboratory blank digested and analyzed with the analytical batch?					
b. Are the laboratory blank results < 3x PRDL for all reported analytes?					
c. Was an ICB analyzed immediately after the ICV?					
d. Was a CCB analyzed immediately after each CCV?					
e. Are each of the ICB and CCB results < 3x PRDL for all reported analytes?					
f Are Blank Summary Forms completed and correct for all blanks associated with the analytical run (both Laboratory and calibration verification blanks)?					
5. LABORATORY CONTROL SAMPLES (LCS)					
a. Was at least one LCS digested and analyzed with the analysis batch?					
b. Is the LCS from a different source than used in ICAL standard preparation?					
c. Do the recoveries for all reported analytes fall between 80% and 120% for aqueous LCS, or within the manufacturer's specifications if solid LCS?					
d. Is the LCS Form completed and correct for the LCS analyzed with the analytical batch?					

Page 1 of 2

Figure 15-1. Example of independent technical review checklist for ICP-AES total metals analysis.

METHOD 2900 INDEPENDENT DATA REVIEW CHECKLIST FOR ANALYTICAL BATCH #:____________

REQUIREMENT	RUN #1		RUN #2		COMMENTS
	YES	NO	YES	NO	
6. MATRIX SPIKES AND MATRIX SPIKE DUPLICATES					
a. Were a matrix spike and a matrix spike duplicate performed on at least one field sample from the analytical batch?					
b. Is the duplicate RPD ≤ 30% for all analytes?					
c. Are the %Rs in the range of 80% to 120% for all analytes in the spike and spike duplicate?					
d. Are the Matrix Spike and the Laboratory Duplicate Forms completed and correct for all matrix spikes and matrix spike duplicates analyzed with the run?					
e. Are all noncompliant matrix spike recoveries and RPDs discussed in the batch narrative?					
7. BACKGROUND CORRECTION AND INTERFERENCE CHECK SAMPLES					
a. Was background correction used during the analysis and applied correctly?					
b. Were the interfering elements monitored during the analysis and ICFs correctly applied where necessary?					
c. Were the ICSA and ICSAB solutions analyzed within prescribed limits at both the beginning and end of the analytical run, or twice per 8 hour shift, whichever is more frequent?					
d. Are the Interference check sample results reported on the appropriate form?					
8. SAMPLES (Including Analytical Spikes and Serial Dilutions)					
a. Are all raw data signed and dated by the analyst?					
b. Were all samples having analytes detected in amounts exceeding the calibration range reanalyzed with a dilution?					
c. Was a serial dilution analysis performed in the analytical batch?					
d. For all analytes >50x IDL in the initial sample, are serial dilution results ≤ 10 %D of initial value?					
e. If the serial dilution or the matrix spike/matrix spike duplicate fail the acceptance limits, was a post-digestion spike analysis performed?					
f. If a post-digestion spike analysis was performed, was the spike recovery within the required range of 75% to 125%?					
g. Are the Post-digestion Spike and/or Serial Dilution results reported on the appropriate forms?					
h. Are any corrective actions taken during the analysis documented in the raw data?					
i. Are each of the sample results reported on a Total Metal Analysis Data Form?					
j. Was the % solid determined for each sample?					
k. Are all noncompliant QC discussed in the batch narrative?					
9. REPORTING FORMS					
a. Are all reporting forms completely and correctly filled out? Form 1 values correctly rounded to 2 significant figures? Batch Number correct? Sample IDs consistent with the Analysis Request Form?					
b. Are all values less than the IDL reported as the IDL corrected for dilution and flagged with a "U" qualifier?					
c. Are all values ≥ IDL and < 5x IDL (before dilution correction) reported with a "J" qualifier?					
d. Were "B" qualifiers used when the analyte blank concentration (laboratory or calibration verification) was ≥ 20% of the sample concentration prior to dilution correction?					
e. Was a "Z" qualifier used if one or more QC sample results are outside the acceptance criteria?					
f. Are all necessary nonconformance reports initiated?					

Figure 15-1. (continued).

SPECTROCHEMISTRY GFAA INDEPENDENT DATA REVIEW CHECKLIST

Analytical Batch #:	Method: 2350 Metals by GFAA (TWCP Method 650.2)			Form Revision:0
Run # and Analysis Date	Data Generator	Independent Reviewer	Review Date	OK for Release?
1.				Yes or No
2.				Yes or No

Instructions: Complete one checklist per Analytical Batch. If more than 2 instrument runs were needed to complete the batch analysis, use a second checklist. Enter appropriate responses for each question. Each "No" response requires an explanation.

REQUIREMENT	RUN #1		RUN #2		COMMENTS
	YES	NO	YES	NO	
1. INITIAL CALIBRATION (ICAL)					
a. Was the initial calibration performed using a minimum of 3 standards and a blank?					
b. Is the regression coefficient (r^2) ≥ 0.995?					
2. INITIAL CALIBRATION VERIFICATION					
a. Was the highest calibration standard analyzed as a sample and recovered within 95-105% for each reported analyte?					
b. Was an ICV analyzed after the initial calibration and prior to analysis of any samples?					
c. Are the recoveries for each reported analyte in the ICV between 90% and 110%?					
d. Is the ICV standard from a different source from the initial calibration standards?					
e. Are Initial Calibration Verification Forms and Linear Range Check Forms present and correct for all ICVs and high standard checks associated with the run?					
3. CONTINUING CALIBRATION VERIFICATION (CCV)					
a. Was a CCV analyzed prior to the analysis of samples?					
b. Is the percent recovery of each reported analyte between 80% and 120%?					
c. Was a compliant CCV analyzed for reported analytes at the completion of the analytical run?					
d. Were every 10 samples bracketed by a compliant CCV for reported analytes?					
e. Are Calibration Verification Forms present and correct for all CCVs associated with the run?					
4. BLANKS					
a. Was at least one laboratory blank digested and analyzed with the analytical batch?					
b. Are the laboratory blank results < 3x PRDL for all reported analytes?					
c. Was an ICB analyzed immediately after the ICV?					
d. Was a CCB analyzed immediately after each CCV?					
e. Are each of the ICB and CCB results < 3x PRDL for all reported analytes?					
f. Are Blank Summary Forms completed and correct for all blanks associated with the analytical run (both Laboratory and calibration verification blanks)?					
5. LABORATORY CONTROL SAMPLES (LCS)					
a. Was at least one LCS digested and analyzed with the analysis batch?					
b. Is the LCS from a different source than used in ICAL standard preparation?					
c. Do the recoveries for all reported analytes fall between 80% and 120% for aqueous LCS, or within the manufacturer's specifications if solid LCS?					

Page 1 of 2

Figure 15-2. Example of independent technical review checklist for GFAA total metals analysis.

METHOD 2350 INDEPENDENT DATA REVIEW CHECKLIST FOR ANALYTICAL BATCH #:____________

REQUIREMENT	RUN #1		RUN #2		COMMENTS
	YES	NO	YES	NO	
d. Is the LCS Form completed and correct for the LCS analyzed with the analytical batch?					
6. MATRIX SPIKES AND MATRIX SPIKE DUPLICATES					
a. Were a matrix spike and a matrix spike duplicate performed on at least one field sample from the analytical batch?					
b. Is the duplicate RPD ≤ 30% for all analytes?					
c. Are the % Rs in the range of 80% to 120% for all analytes in the spike and spike duplicate?					
d. Are the Matrix Spike and the Laboratory Duplicate Forms completed and correct for all matrix spikes and matrix spike duplicates analyzed with the run?					
e. Are all noncompliant matrix spike recoveries and RPDs discussed in the batch narrative?					
7. SAMPLES (Including Analytical Spikes and Serial Dilutions)					
a. Was Smith-Hieftje background correction used during the analysis and applied correctly?					
b. Are all raw data signed and dated by the analyst?					
c. Were all samples having analytes detected in amounts exceeding the calibration range reanalyzed with a dilution?					
d. Was a serial dilution analysis performed in the analytical batch?					
e. For all analytes >25 x IDL in the initial sample, are serial dilution results ≤ 10% D of the initial value?					
f. Was a post-digestion spike analysis performed in the analytical batch?					
g. Was the post-digestion spike recovery within the required range of 85% to 115%?					
h. Are the Post-digestion Spike and Serial Dilution results reported on the appropriate forms?					
i. Was MSA used for quantitation if the serial dilution or analytical spike did not meet acceptance criteria?					
j. Are any corrective actions taken during the analysis documented in the raw data?					
k. Are each of the sample results reported on a Total Metal Analysis Data Form?					
l. Was the % solid determined for each sample?					
m. re all noncompliant QC discussed in the batch narrative?					
8. REPORTING FORMS					
a. Are all reporting forms completely and correctly filled out? Form 1 values correctly rounded to 2 significant figures? Batch Number correct? Sample IDs consistent with the Analysis Request Form?					
b. Are all values less than the IDL reported as the IDL corrected for dilution and flagged with a "U" qualifier?					
c. Are all values ≥ IDL and < 5x IDL (before dilution correction) reported with a "J" qualifier?					
d. Were "B" qualifiers used when the analyte blank concentration (laboratory or calibration verification) was ≥ 20% of the sample concentration prior to dilution correction?					
e. Was a "Z" qualifier used when one or more QC sample results are outside the acceptance criteria?					
f. Are all necessary nonconformance reports initiated?					

Page 2 of 2

Figure 15-2. (continued).

SPECTROCHEMISTRY CVAF/CVAA INDEPENDENT DATA REVIEW CHECKLIST

Analytical Batch #:	Method: 7802/7801 Mercury (TWCP Method 650.3)			Form Revision: 0
Run # and Analysis Date	Data Generator	Independent Reviewer	Review Date	OK for Release?
1.				Yes or No
2.				Yes or No

Instructions: Complete one checklist per Analytical Batch. If more than 2 instrument runs were needed to complete the batch analysis, use a second checklist. Enter appropriate responses for each question. Each "No" response requires an explanation.

REQUIREMENT	RUN #1		RUN #2		COMMENTS
	YES	NO	YES	NO	
1. INITIAL CALIBRATION (ICAL)					
a. Was the initial calibration performed using a minimum of 5 standards and a blank?					
b. Is the regression coefficient $(r^2) \geq 0.995$?					
2. INITIAL CALIBRATION VERIFICATION					
a. Was the highest calibration standard analyzed as a sample and recovered within 95-105%?					
b. Was an ICV analyzed after the initial calibration and prior to analysis of any samples?					
c. Is the recovery of the ICV between 90% and 110%?					
d. Is the ICV standard from a different source from the initial calibration standards?					
e. Are Initial Calibration Verification Forms and Linear Range Check Forms present and correct for all ICVs and high standard checks associated with the run?					
3. CONTINUING CALIBRATION VERIFICATION (CCV)					
a. Was a CCV analyzed prior to the analysis of samples?					
b. Is the percent recovery for the CCV between 80% and 120%?					
c. Was a compliant CCV analyzed at the completion of the analytical run?					
d. Were every 10 samples bracketed by a compliant CCV?					
e. Are Calibration Verification Forms present and correct for all CCVs associated with the run?					
4. BLANKS					
a. Was at least one laboratory blank digested and analyzed with the analytical batch?					
b. Are the laboratory blank results < 3x PRDL?					
c. Was a ICB analyzed immediately after the ICV?					
d. Was a CCB analyzed immediately after each CCV?					
e. Are each of the ICB and CCB results < 3x PRDL?					
f. Are Blank Summary Forms completed and correct for all blanks associated with the analytical run (both Laboratory and calibration verification blanks)?					
5. LABORATORY CONTROL SAMPLES (LCS)					
a. Was at least one LCS digested and analyzed with the analysis batch?					
b. Is the LCS from a different source than used in ICAL standard preparation?					
c. Does the recovery fall between 80% and 120% for aqueous LCS, or within the manufacturer's specifications if solid LCS?					

Figure 15-3. Example of independent technical review checklist for CVAF/CVAA total metals analysis.

METHOD 7802/7801 INDEPENDENT DATA REVIEW CHECKLIST FOR ANALYTICAL BATCH#__________

REQUIREMENT	RUN #1		RUN #2		COMMENTS
	YES	NO	YES	NO	
d. Is the LCS Form completed and correct for the LCS analyzed with the analytical batch?					
6. MATRIX SPIKES AND MATRIX SPIKE DUPLICATES					
a. Were a matrix spike and a matrix spike duplicate performed on at least one field sample from the analytical batch?					
b. Is the duplicate RPD ≤ 30% ?					
c. Are the %Rs in the range of 80% to 120% for the spike and spike duplicate?					
d. Are the Matrix Spike and the Laboratory Duplicate Forms completed and correct for all matrix spikes and matrix spike duplicates analyzed with the run?					
e. Are all noncompliant matrix spike recoveries and RPDs discussed in the batch narrative?					
7. SAMPLES (Including Analytical Spikes and Serial Dilutions)					
a. Are all raw data signed and dated by the analyst?					
b. Were all samples having mercury detected in amounts exceeding the calibration range reanalyzed with a dilution?					
c. Was a serial dilution analysis performed in the analytical batch?					
d. If mercury was >25 x IDL in the initial sample, is the serial dilution result ≤ 10 %D of the initial value?					
e. Was a post-digestion spike analysis performed in the analytical batch?					
f. Was the post-digestion spike recovery within the required range of 85% to 115%?					
g. Are the Post-digestion Spike and Serial Dilution results reported on the appropriate forms?					
h. Was MSA used for quantitation if the serial dilution or analytical spike did not meet the acceptance criteria?					
i. Are any corrective actions taken during the analysis documented in the raw data?					
j. Are each of the sample results reported on a Total Metal Analysis Data Form?					
k. Was the % solid determined for each sample?					
l. Are all noncompliant QC discussed in the batch narrative?					
8. REPORTING FORMS					
a. Are all reporting forms completely and correctly filled out? Form 1 values correctly rounded to 2 significant figures? Batch Number correct? Sample IDs consistent with the Analysis Request Form?					
b. Are all values less than the IDL reported as the IDL corrected for dilution and flagged with a "U" qualifier?					
c. Are all values ≥ IDL and < 5x IDL (before dilution correction) reported with a "J" qualifier?					
d Were "B" qualifiers used when the analyte blank concentration (laboratory or calibration verification) was ≥ 20% of the sample concentration prior to dilution correction?					
e. Was a "Z" qualifier used when one or more QC sample results are outside the acceptance criteria?					
f. Are all necessary nonconformance reports initiated?					

Figure 15-3. (continued).

15.6.3 Data Reporting

Data reports for total metals data consist of a cover page and five sections:

- **Cover page**—The cover page includes the laboratory name, the data report number, the report date, the report table of contents, and release authorization signatures. An example cover page is provided in Figure 13-3.

- **Section 1: Sample Identification Table**—This section includes the cross-reference between field and laboratory sample identification numbers (example provided in Figure 13-4).

- **Section 2: Sample Custody Documents**—This section includes copies of the field COC form(s) that accompanied the samples to the laboratory (see example provided in Figures 6-1).

- **Section 3: Analysis Results**—This section includes the batch narrative, containing information pertinent to program-level review, and the analysis data sheets (see example in Figure 15-4) for each sample included in the data report. A separate analysis data sheet is provided for each sample, and contains the following information: laboratory name, program name, data report number, analytical batch number, sampling batch number, field sample ID, laboratory sample ID, date sampled, date received at the laboratory, date digested, date analyzed, percent solids, method number code, listing of program analytes, and analytical results in mg/kg. Data qualifying flags are used as follows:

  - B: Analyte blank concentration (laboratory or calibration verification) greater than or equal to 20 percent of the sample concentration prior to dilution correction

  - J: Analyte concentration is ≥ IDL but < 5 x IDL before dilution correction

  - U: Analyte was undetected (reported as sample-specific IDL, corrected for dilution)

  - Z: Estimated concentration; one or more QC sample results are outside the acceptance criteria.

  The following method number codes used on metals data reporting forms:

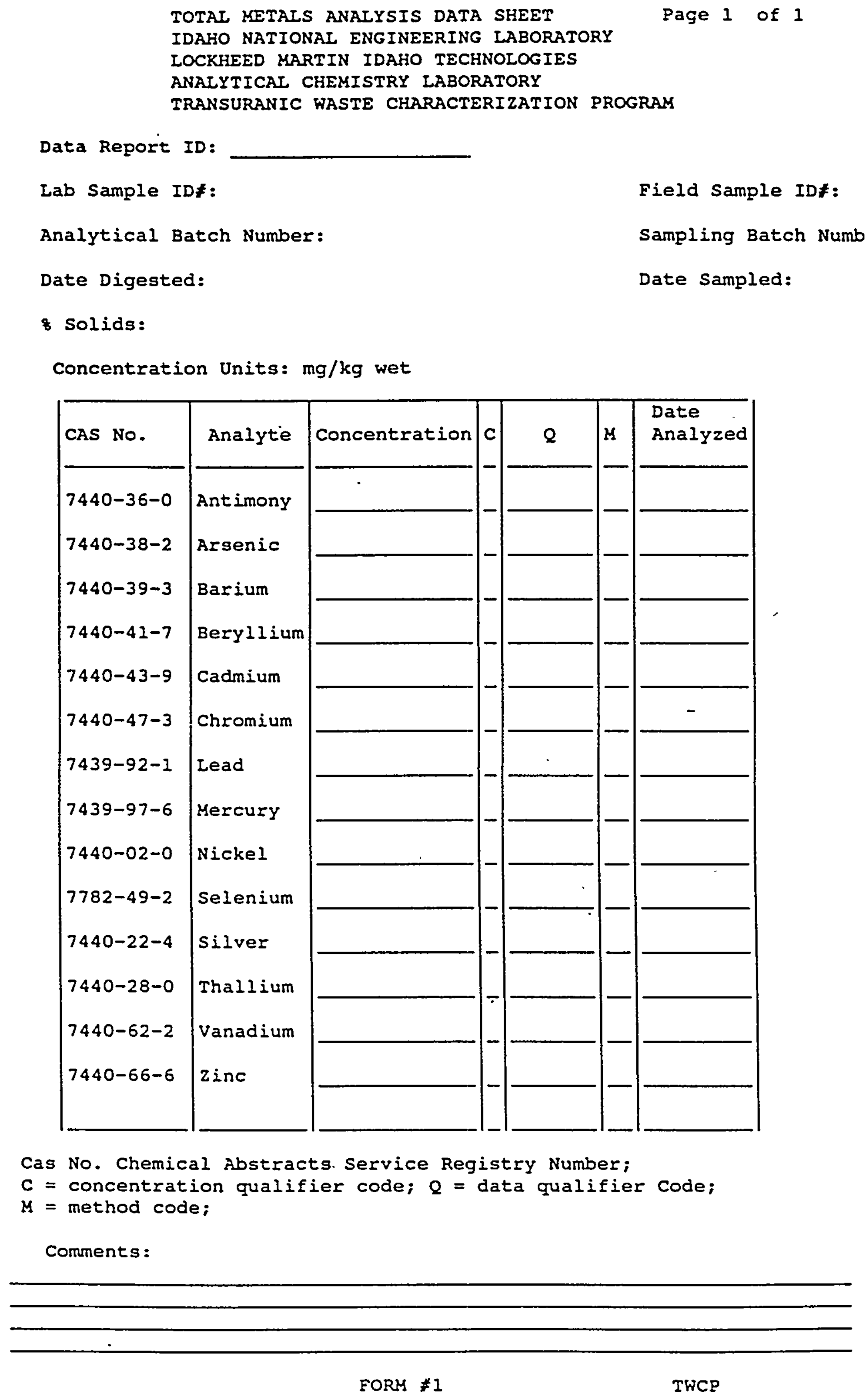
  - P: ICP-AES (ACMM 2900)

  - F: GFAA (ACMM 2350)

  - V: CVAF (ACMM 7802)

  - CV: CVAA (ACMM 7801)

- **Section 4: Batch Related QC Samples**—This section contains forms reporting results of LCSs (see example in Figure 15-5), matrix spike recoveries (see example in Figure 15-6) and matrix spike duplicate RPDs (see example in Figure 15-7).



TOTAL METALS ANALYSIS DATA SHEET Page 1 of 1
IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES
ANALYTICAL CHEMISTRY LABORATORY
TRANSURANIC WASTE CHARACTERIZATION PROGRAM

Data Report ID: ____________________

Lab Sample ID#:

Field Sample ID#:

Analytical Batch Number:

Sampling Batch Number: ___________

Date Digested:

Date Sampled:

% Solids:

Concentration Units: mg/kg wet

CAS No.	Analyte	Concentration	C	Q	M	Date Analyzed
7440-36-0	Antimony					
7440-38-2	Arsenic					
7440-39-3	Barium					
7440-41-7	Beryllium					
7440-43-9	Cadmium					
7440-47-3	Chromium					
7439-92-1	Lead					
7439-97-6	Mercury					
7440-02-0	Nickel					
7782-49-2	Selenium					
7440-22-4	Silver					
7440-28-0	Thallium					
7440-62-2	Vanadium					
7440-66-6	Zinc					

Cas No. Chemical Abstracts Service Registry Number;
C = concentration qualifier code; Q = data qualifier Code;
M = method code;

Comments:

FORM #1 TWCP

Figure 15-4. Example of Total Metals Analysis Data Sheet.

Page 1 of 1

LABORATORY CONTROL SAMPLE
IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES
ANALYTICAL CHEMISTRY LABORATORY
TRANSURANIC WASTE CHARACTERIZATION PROGRAM

Data Report ID: ____________________

Analytical Batch Number:

LCS Source:

Concentration Units: MG/KG wet weight

				CONTROL LIMITS			
Analyte	True	Found	C	Low	High	%R	M
Antimony							
Arsenic							
Barium							
Beryllium							
Cadmium							
Chromium							
Lead							
Mercury							
Nickel							
Selenium							
Silver							
Thallium							
Vanadium							
Zinc							

C = concentration qualified code; M = method code

FORM #9 TWCP

Figure 15-5. Example of LCS reporting form for total metals analysis.

Page 1 of 1

MATRIX SPIKE (MS) /MS DUPLICATE (MSD) SAMPLE
IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES
ANALYTICAL CHEMISTRY LABORATORY
TRANSURANIC WASTE CHARACTERIZATION PROGRAM

Data Report ID: ____________________

Lab Sample ID#:

Field Sample ID#:

Analytical Batch Number:

Sampling Batch Number: ___________

Concentration Units: MG/KG wet weight

Analyte	SSR	C	SR	C	SA	%R	Q	M
Antimony								
Arsenic								
Barium								
Beryllium								
Cadmium								
Chromium								
Lead								
Mercury								
Nickel								
Selenium								
Silver								
Thallium								
Vanadium								
Zinc								

SSR = spike sample result; SR = sample result; SA = spike added;
C = concentration; Q = data qualifier code; M = method code

Comments:

FORM #6

TWCP

Figure 15-6. Example of matrix spike/matrix spike duplicate recovery reporting form for total metals analysis.

Page 1 of 1

MATRIX SPIKE DUPLICATE SAMPLE
IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES
ANALYTICAL CHEMISTRY LABORATORY
TRANSURANIC WASTE CHARACTERIZATION PROGRAM

Data Report ID: ____________________

Lab Sample ID:

Field Sample ID:

Analytical Batch Number:

Sampling Batch Number: ___________

% Solids for Sample:

% Solids for Duplicate:

Concentration Units: MG/KG wet weight

Analyte	Matrix Spike Result	Matrix Spike Duplicate Result	RPD	Q	M
Antimony					
Arsenic					
Barium					
Beryllium					
Cadmium					
Chromium					
Lead					
Mercury					
Nickel					
Selenium					
Silver					
Thallium					
Vanadium					
Zinc					

RPD = relative percent difference; Q = data qualifier code;
M = method code

FORM #8

TWCP

Figure 15-7. Example of matrix spike duplicate RPD reporting form for total metals analysis.

- **Section 5: Data Review Checklists**—This section includes the TWCP Sample Receiving & Custody Review Checklist (see example in Figure 6-2), the ACMM spectrometry methods Independent Data Review Checklists (see examples in Figures 15-1, 15-2 and 15-3), and the Quality Assurance Data Review Checklist for total metals (see examples in Figures 3-4). Copies of applicable NCRs are also included in this section, as necessary.

The following items are retained in ACL files, but are not included in the data reports sent to the SPO:

- **Data package** filed by data report number, with all raw data, including sample preparation logs, percent solid determination logs, standard preparation logs, original instrument printouts for all calibrations and sample and QC analyses, calculation records, and results of all associated QC samples (ICV/CCBs, LBs, ICB, CCBs, LCSs, MSs, MSDs, ICSA, ICSAB). Examples of forms providing ICB/CCB/LB, ICV/CCV, ICSA/ICSAB, post-digestion spikes, serial dilutions, method of standard additions (MSA), and high calibration check results are provided in Figures 15-8, 15-9, 15-10, 15-11, 15-12, 15-13, and 15-14, respectively.

- **IDL records**, filed by IDL determination date and instrument identification, which include all raw data and calculations for IDL determinations, along with the IDL reporting form (see example in Figure 15-15).

- **Original COC forms**, filed in the data package.

Page 1 of 1

BLANKS
IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES
ANALYTICAL CHEMISTRY LABORATORY
TRANSURANIC WASTE CHARACTERIZATION PROGRAM

Data Report ID: ____________________

Analytical Batch Number:

Analyte	ICB (ug/L)	C	CCB (ug/L)						LB (mg/kg)	C	M
			1	C	2	C	3	C			
Antimony											
Arsenic											
Barium											
Beryllium											
Cadmium											
Chromium											
Lead											
Mercury											
Nickel											
Selenium											
Silver											
Thallium											
Vanadium											
Zinc											

ICB = initial calibration blank; CCB = continuing calibration blank;
LB = Laboratory blank; C = concentration qualifier code;
M = method code;

FORM 4 TWCP

Figure 15-8. Example of ICB/CCB/LB reporting form for total metals analysis.

INITIAL AND CONTINUING CALIBRATION VERIFICATION
IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES
ANALYTICAL CHEMISTRY LABORATORY
TRANSURANIC WASTE CHARACTERIZATION PROGRAM

Page 1 of 1

Data Report ID: ____________________

Analytical Batch Number:

Calibration Standards Source:

ICV Source:

CCV Source:

Concentration Units: UG/L

	Initial Calibration				Continuing Calibration				
Analyte	DATE	True	Found	%R	True	Found	%R(1)	Found	%R(2)
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Chromium									
Lead									
Mercury									
Nickel									
Selenium									
Silver									
Thallium									
Vanadium									
Zinc									

FORM #2 TWCP

Figure 15-9. Example of ICV/CCV reporting form for total metals analysis.

Page 1 of 1

ICP INTERFERENCE CHECK SAMPLE
IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES
ANALYTICAL CHEMISTRY LABORATORY
TRANSURANIC WASTE CHARACTERIZATION PROGRAM

Data Report ID: ____________________

Analytical Batch Number:

ICP ID Number:

ICS Source:

Concentration Units: UG/L

Analyte	True Sol. A	True Sol. AB	Initial Found Sol. A	Initial Found Sol. AB	Initial Found %R	Final Found Sol. A	Final Found Sol. AB	Final Found %R
Antimony								
Arsenic								
Barium								
Beryllium								
Cadmium								
Chromium								
Lead								
Mercury								
Nickel								
Selenium								
Silver								
Thallium								
Vanadium								
Zinc								

FORM #5 TWCP

Figure 15-10. Example of interference check reporting form for ICP-AES total metals analysis.

Page 1 of 1

POST DIGESTION SPIKE SAMPLE
IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES
ANALYTICAL CHEMISTRY LABORATORY
TRANSURANIC WASTE CHARACTERIZATION PROGRAM

Data Report ID: ____________________

Lab Sample ID#:

Field Sample ID#:

Analytical Batch Number:

Sampling Batch Number: ___________

Concentration Units: MG/KG

Analyte	SSR	C	SR	C	SA	%R	Q	M
Antimony								
Arsenic								
Barium								
Beryllium								
Cadmium								
Chromium								
Lead								
Mercury								
Nickel								
Selenium								
Silver								
Thallium								
Vanadium								
Zinc								

SSR = spiked sample result; SR = sample result;
SA = spike added; C = concentration qualifier code;
Q = data qualified code; M = method code

Comments:

FORM #7

TWCP

Figure 15-11. Example of post-digestion spike recovery reporting form for total metals analysis.

Page 1 of 1

SERIAL DILUTION SAMPLE
IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES
ANALYTICAL CHEMISTRY LABORATORY
TRANSURANIC WASTE CHARACTERIZATION PROGRAM

Data Report ID: ____________________

Lab Sample ID#:

Field Sample ID#:

Analytical Batch Number:

Sampling Batch Number: ___________

Concentration Units: UG/L

Analyte	SR	C	SDR	C	%D	Q	M
Antimony							
Arsenic							
Barium							
Beryllium							
Cadmium							
Chromium							
Lead							
Mercury							
Nickel							
Selenium							
Silver							
Thallium							
Vanadium							
Zinc							

SR = initial sample result; C = concentration qualifier code; Q = data.qualifier code; SDR = serial dilution (1:4 ratio of initial sample to diluent) sample result times 5; M = method code

FORM #10 TWCP

Figure 15-12. Example of serial dilution reporting form for total metals analysis.

Page 1 of 1

STANDARD ADDITION RESULTS
IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES
ANALYTICAL CHEMISTRY LABORATORY
TRANSURANIC WASTE CHARACTERIZATION PROGRAM

Data Report ID: ____________________

Analytical Batch Number:

Field Sample ID	An	DV	0 Add Added Conc ug/L	0 Add Inst Read.	1 ADD Added Conc ug/L	1 ADD Inst Read.	2 ADD Added Conc ug/L	2 ADD Inst Read.	3 ADD Added Conc ug/L	3 ADD Inst Read.	Final Conc. ug/L	r	M

An = analyte; DV = dilution factor; r = correlation coefficient; M = method code;

FORM #13

TWCP

Figure 15-13. Example of Method of Standard Additions reporting form for total metals analysis.

Page 1 of 1

ICP LINEAR RANGE ANALYSIS
IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES
ANALYTICAL CHEMISTRY LABORATORY
TRANSURANIC WASTE CHARACTERIZATION PROGRAM

Data Report ID: ____________________

Analytical Batch Number:

Concentration Units: UG/L

Analyte	True	FOUND	%R	M
Antimony				
Arsenic				
Barium				
Beryllium				
Cadmium				
Chromium				
Lead				
Mercury				
Nickel				
Selenium				
Silver				
Thallium				
Vanadium				
Zinc				

Linear range analysis is the highest calibration standard analyzed as a sample.

FORM 12 TWCP

Figure 15-14. Example of high standard verification check reporting form for total metals analysis.

Page 1 of 1

INSTRUMENT DETECTION LIMITS
IDAHO NATIONAL ENGINEERING LABORATORY
LOCKHEED MARTIN IDAHO TECHNOLOGIES
ANALYTICAL CHEMISTRY LABORATORY
TRANSURANIC WASTE CHARACTERIZATION PROGRAM

Data Report ID: ____________________

Analytical Batch Number:

ICP ID Number:

GFAA ID Number:

CVAA ID Number:

CVAF ID Number:

Analyte	Wave-length (nm)	BG	IDL (ug/L)	Date	M
Antimony					
Arsenic					
Barium					
Beryllium					
Cadmium					
Chromium					
Lead					
Mercury					
Nickel					
Selenium					
Silver					
Thallium					
Vanadium					
Zinc					

BG = background correction technique;
IDL = instrument detection limit; M = method code

FORM #11 TWCP

Figure 15-15. Example of IDL reporting form for total metals analysis.

REFERENCES

ANL, 1996, *CH-TRU Sludge Samples Packaging and Transportation Memorandum of Understanding Between LITCO Analytical Laboratory Department and Argonne National Laboratory-West*, ANL-W Document # W0096-0364-ES, Revision 0, May 1996.

ANSI, 1994, *General Requirements for Calibration Laboratories and Measuring and Test Equipment*, ANSI/NCSL Z540-1, 1994

ASME, 1994, *Quality Assurance Program Requirements for Nuclear Facilities*, NQA-1-1994 Edition, American Society of Mechanical Engineers.

Dean, R. B., and W. J. Dixon, 1951, "Simplified Statistics for Small Numbers of Observations," *Analytical Chemistry*, Volume 23, pp. 636–638.

DOE, 1991, "Quality Assurance," Order 5700.6C, U.S. Department of Energy, Washington, D.C., August 21, 1991.

DOE, 1995a, *Transuranic Waste Characterization Quality Assurance Program Plan*, CAO-94-1010, Revision 0, April 30, 1995, Carlsbad Area Office, U.S. Department of Energy, Carlsbad, New Mexico.

DOE, 1995b, *Records Disposition*, Order 1324.5B, U.S. Department of Energy, Washington, D.C., January 12, 1995.

DOE, 1996a, *Quality Assurance Program Document*, CA-94-1012, Revision 1, Carlsbad Area Office, U.S. Department of Energy, Carlsbad, NM, April 1996.

DOE, 1996b, *Performance Demonstration Program Plan for RCRA Constituent Analysis of Simulated Wastes*, DOE/CAO-95-1077, Revision 1, Carlsbad Area Office, U.S. Department of Energy, Carlsbad, New Mexico, March 1996.

DOE, 1996c, *Transuranic Waste Characterization Sampling and Analysis Methods Manual*, DOE/WIPP-91-043, Revision 1, April 1996, Carlsbad Area Office, U.S. Department of Energy, Carlsbad, New Mexico.

EPA, 1986, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, Third Edition, Volumes 1A and 1B, U.S. Environmental Protection Agency, Washington, D.C., November 1986.

EPA, 1991, *NEIC Policies and Procedures*, EPA-330/9-78-001-R, U.S. Environmental Protection Agency, National Enforcement Investigations Center, Denver, Colorado.

EPA, 1994a, *EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations*, EPA QA/R-5 (Draft Interim Final), U.S. Environmental Protection Agency, Washington, D.C.

EPA, 1994b, *USEPA Contract Laboratory Program Statement of Work for Inorganic Analysis, Multi-Media, Multi-Concentration*, ILM03.0, U.S. Environmental Protection Agency, Washington, D.C., 1994.

Glaser, J. A., D. L. Forest, G. D. McKee, S. A. Quave, and W. L. Budde, 1981, *Environmental Science and Technology*, Volume 15, Number 12, p. 1426.

LMITCO, 1996, *INEL Site Project Office Quality Assurance Project Plan for the TRU Waste Characterization Program*, INEL-94/0085, Revision 2, Lockheed Martin Idaho Technologies Company.