GUIDEBOOK FOR QUALITY ASSURANCE/QUALITY CONTROL PROCEDURES FOR SUBMISSION OF DATA FOR THE LAND DISPOSAL RESTRICTIONS PROGRAM

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1. INTRODUCTION

The Agency is publishing this handbook to explain how to generate data which characterizes the performance of hazardous waste treatment systems in terms of the composition of treated hazardous waste streams plus treatment system operation and design. If acceptable, these data will be the basis of treatment standards for newly listed wastes or for future EPA changes to currently existing treatment standards set during the First, Second, and Third Thirds rules, dating back to August 1988.

EPA has now developed a QA/QC Methodology Background Document (QMBD) for the hazardous waste treatment community to use as a guidance document in developing hazardous waste treatability data. This handbook summarizes the major substantive requirements of the QMBD. The QMBD will include the contents of this handbook plus a longer discussion of the regulatory issues involved in this undertaking and also a detailed presentation of the calculations used to develop numerical treatment standards.

1.1 Purpose and Availability

EPA announces here that the QMBD and this auxiliary handbook replace and make obsolete the 1987 Generic Quality Assurance Project Plan for Land Disposal Restrictions Program ("BDAT"), known as the "Generic" or the "Brown Book" which has been disseminated in two editions since March 1987 throughout the hazardous waste treatment and regulatory community. The QMBD incorporates all the substantive analytical and documentation provisions of the 1987 Generic in its Section II, which is intended to serve as a guide to generating acceptable data. Section I of the QMBD is an introductory overview of the BDAT program and its place in the Land Disposal Restrictions effort under HSWA; it attempts to put EPA's QA/QC specifications into historical regulatory context. Section III explains the procedures EPA intends to follow in deriving concentration-based treatment standards from acceptable data collected in response to this notice. Section IV explains how EPA developed existing treatment standards. The QMBD will be available through NTIS in the same way that waste code-specific background documents were available with each of the Thirds final and proposed rules. Extra copies of this handbook are available from the Treatment Technology Section.

The QA/QC Methodology Background Document and this auxiliary handbook deal exclusively with criteria for data to be submitted as part of setting new or revised treatment standards as part of 40 CFR 268 Land Disposal Restriction standards. Compliance sampling, analysis, and monitoring issues fall outside the scope of this handbook; they are the concern of

the Office of Solid Waste's Permits and State Programs Division and State and Regional permitting offices.

1.2 EPA Goals in Soliciting Data

Although EPA is confident that existing standards reflect adequate treatment and can be verified with currently available analytical instrumentation, EPA believes that the time has come to reevaluate all existing treatability data together with legitimate concerns about analytical verification of these standards in compliance and enforcement operations. Improvements are possible in any undertaking and EPA believes there exists a growing body of treatability data in the regulated community which were not available to the Agency in the Thirds rulemaking and which may support the development of standards better meeting the Agency's goals yet more easily verifiable by the regulated community.

The Agency's long-range goal of ensuring the destruction or immobilization of toxic constituents in hazardous waste treatment standards remains. Two criteria simultaneously drive this goal. The first criterion is that the hazardous waste treatment industry should be able to achieve the standards with existing treatment processes and be able to verify achieving these standards with existing analytical chemical laboratory capabilities. The second criterion is that the standards must reflect the lowest post-treatment concentrations in the waste treatment residual achievable with existing treatment processes; i.e., the maximum possible extent of treatability.

EPA believes that treatability data from ongoing waste treatment carried out in compliance with existing Land Ban standards and backed up by documentation of the analytical methods used to generate these data, is a particularly promising source of data on which to set standards. Therefore, EPA has undertaken in this handbook the responsibility to spell out unambiguously and explicitly what level of treatability data quality and documentation must accompany data submitted by industry and other interested parties so that these data are of such quality to be consistent with the data EPA has used in the three Thirds rulemaking.

Because concentration-based standards ensure that hazardous constituents have been reduced to a certain low level, EPA prefers them to methods of treatment as standards. Concentration-based standards also encourage innovation in the waste treatment industry because they provide a powerful incentive to meet these targets as economically as possible via introduction of superior new technologies or improvements to existing technologies. The value of methods of treatment as standards in reducing contaminant concentrations depends entirely on treatment process conditions, which cannot be monitored as well as contaminant concentrations themselves in treatment residuals.

The reason for methods of treatment as standards is to ensure treatment of a waste analytically problematic enough that treatment to a concentration-based standard could not be verified; a method bypasses delays in the treatment-disposal sequence caused by controversy over the validity of questionable analytical data or the reliability of the analytical procedure or any other source of dispute between regulators and treaters over whether a given waste can be land disposed.

EPA maintains the prerogative of making the final decision on whether a waste stream is too analytically complex to regulate with numerical standards. Data suggesting a waste poses significant analytical problems precluding consistent quantification of constituents of concern will be scrutinized rigorously for evidence that these problems are inevitable in analyzing the waste stream in question.

2. QUALITY ASSURANCE PROJECT PLAN FOR LAND DISPOSAL RESTRICTIONS PROGRAM

Under the Land Disposal Restrictions Program, a document entitled *Generic Quality Assurance Project Plan for Land Disposal Restrictions Program ("BDAT")* was developed and published in March 1987 (EPA/530-SW-87-011). A "Project Plan" describes the QA/QC activities in any single EPA data collection program such as developing Land Disposal Restrictions. The purpose of this document is to serve as the update to that project plan and to provide additional clarification and guidance for collection of treatment test data for the LDR Program by EPA and by others such as industry or research organizations.

2.1 Overview of QA Concepts and Procedures Involved in Generating Data for Land Disposal Restrictions Standards

EPA is soliciting data on treatability of a variety of hazardous wastes as discussed in the May 30, 1991 Federal Register and subsequent notices. Although EPA will examine any waste treatment data submitted, data generated and presented according to the requirements of this Project Plan are less likely to be rejected for use in developing treatment standards because of data quality problems.

Quality assurance/quality control (QA/QC) is the body of administrative and technical procedures used to generate analytical chemical data which both accurately reflect the compositions of the waste streams involved and also include a subset of data verifying the validity of the results plus data characterizing the performance of the treatment system.

QA/QC requirements can be expressed in two different contexts: substantively as those procedures a laboratory must carry out to generate acceptable data or conceptually as data quality indicators (or objectives) which represent important factors to consider in planning for or evaluating data quality. The QMBD discusses conceptual QA/QC requirements at length: the rest of this handbook will focus on substantive QA/QC requirements such as laboratory procedures and documentation requirements.

The major substantive QA/QC requirements for generating data, which must be discussed in the SAP and the OER, are the following:

Sample Handling

- Documentation of basis for selecting sample point.
- Documentation that SW-846 sample preservation procedures were followed.
- Documentation that chain of custody procedures were followed.

Sample Analysis

- Instrument calibration: documentation of instrument calibration procedures.
- Availability of calibration reagents.
- Blanks: results of analysis of field, laboratory, and trip blanks, clearly labeled.
- Matrix spike duplicates: results of matrix spike duplicate analyses performed on one sample from every set of samples from a single sampling point of one of every twenty samples.
- Detection limits: verified detection limits of 1 ppm in treatment residual matrices or documentation of attempts to reach these detection limits.
- Clear designation of analytical results on raw and untreated waste samples, including documentation of quantitative results of all method-specific QC procedures for each sample whose results are reported.

Data Reporting Format

- Documents for reporting results in a standard format: Sampling and Analysis Plan (SAP) and Onsite Engineering Report (OER).

QA/QC requirements for reporting on treatment technology operating conditions are to be developed on a treatment test by treatment test basis and defined in the SAP. The operating conditions and design parameters to report for each treatment process being tested depend, of course, on the type of technology and the various engineering refinements the system being

tested exhibits. EPA welcomes opportunities to evaluate draft SAPs or OERs from a commenter wishing to submit treatability data so a potential commenter's concerns about developing the appropriate format for treatment system design and operation data can be readily resolved once the commenter initiates contact with EPA by requesting review at any preliminary level of SAP or OER development.

One element of analytical QA/QC assuming a new role in the post-Thirds BDAT program is the analytical detection limit. As of the publication of the First Update to the Third Edition of SW-846, the definition of the detection limit in SW-846 is changing from the 1986 Third Edition (Zero Update) definition: this definition is becoming more quantitatively rigorous. Detection limits are important because they are frequently the basis of numerical standards and thus the definition of the detection limit can profoundly affect the magnitude of the standard.

The 1986 Third Edition (Zero Update) definition in Chapter One, which sets baseline QA/QC requirements for all SW-846 procedures, is that the method detection limit (MDL) is three times the standard deviation of the average noise level divided by the slope of the calibration line generated with solutions of known quantities of the analyte in question. The 1991 First Update to the Third Edition defines the method detection limit (MDL) as the product of the standard deviation (from at least three analyses of a matrix spiked with the analyte of interest at a level believed to be near the detection level) and the t-statistic (one-sided, 99 percent level of probability, chosen as a function of the number of analyses).

For both the 1986 and the 1991 versions, some of the methods themselves have more rigorous detection limits definitions which are spelled out in the QA/QC heading of the method chapter itself; the First Update changes to the Chapter One global QA/QC requirements for all SW-846 methods do not invalidate any of the method-specific requirements but rather they make the chapter-specific QA/QC requirements more uniform among each other by bringing them up to a higher degree of rigor.

An acceptable data package will generally consist of two documents: the Sampling and Analysis Plan (SAP) and the On-Site Engineering Report (OER). The exception is the case where the data have already been generated; in this case the organization submitting the data will do well to study the contents of a "good" SAP as presented in Section 2.2 but their data must be arranged into the OER format presented in Section 2.3.

The Sampling and Analysis Plan (SAP) describes how the raw and treated waste will be sampled, preserved, shipped, and analyzed. It includes a table assigning a unique code to each sample, duplicate, and blank, a description and justification of each sampling point, the preservations, spikes, replicates, and analyses to be performed plus provisions for documenting

the chain of custody and for assembling documentation of these sampling and analytical procedures as they are actually performed.

The Onsite Engineering Report (OER) is the summary of the results of these samplings and analyses and is essentially documentation both tabular and narrative of how the activities planned in the SAP were carried out in reality. Listing and discussing deviations from the SAP which occurred in the course of these activities is an important part of the OER.

2.1.1 Data Quality Objectives

The overall objective for the BDAT Program's sampling and analysis efforts is to produce well-documented data of known quality that can be used to determine the best demonstrated available technologies for the various listed wastes and to develop BDAT treatment standards for these wastes.

The treatment data, i.e., data resulting from treatment tests, consist of the results of analytical tests of the composition of the untreated wastes and the treatment residuals. The treatment data, which are the concentrations of hazardous constituents, can then be used to evaluate the performance of the technology on the listed hazardous waste.

The constituents to be quantified in the BDAT Program investigations are presented in Table 2-1. This list is updated periodically as additional information is obtained on the analytical procedures used to measure the hazardous constituents listed in Appendix VIII. The untreated wastes and treatment residual should be screened for most of the BDAT constituents to determine which constituents are present or were formed, which constituents were treated (or were formed during treatment), and which constituents should be regulated.

The data quality for analytical measurements of the BDAT list constituents in raw waste and in treated waste residuals are primarily assessed by means of the following indicators: analytical method detection limits, precision, and accuracy. These indicators are particularly important in BDAT investigations and special QA/QC documentation requirements apply. The additional indicators assessed are completeness, representativeness, and comparability. Each of these indicators is discussed in detail below.

(1) Detection limits. Matrix detection limits should be calculated for the untreated wastes and each treatment residual sample following the procedures given in *Test Methods for Evaluating Solid Waste (SW-846), Third Edition (USEPA 1986)*, where applicable. If samples

Table 2-1 BDAT Constituent List

Constituent	CAS no.	BDAT reference no.
Volatile organics		
Acetone	67-64-1	222
Acetonitrile	75-05-8	1
Acrolein	107-02-8	2
Acrylonitrile	107-13-1	3
Benzene	71-43-2	4
Bromodichloromethane	75-27-4	5
Bromomethane	74-83-9	6
n-Butyl alcohol	71-36-3	223
Carbon tetrachloride	56-23-5	7
Carbon disulfide	75-15-0	8
Chlorobenzene	108-90-7	9
2-Chloro-1,3-butadiene*	126-99-8	10
Chlorodibromomethane	124-48-1	11
Chloroethane	75-00-3	12
2-Chloroethyl vinyl ether	110-75-8	13
Chloroform	67-66-3	14
Chloromethane	74-87-3	15
3-Chloropropene	107-05-1	16
1,2-Dibromo-3-chloropropane	96-12-8	17
1,2-Dibromoethane	106-93-4	18
Dibromomethane	74-95-3	19
*trans-1,4-Dichloro-2-butene	110-57-6	20
Dichlorodifluoromethane	75-71-8	21
1,1-Dichloroethane	75-34-3	22
1,2-Dichloroethane	107-06-2	23
1,1-Dichloroethylene	75-35-4	24
trans-1,2-Dichloroethene	156-60-5	25
1,2-Dichloropropane	78-87-5	26
trans-1,3-Dichloropropene	10061-02-6	27
cis-1,3-Dichloropropene	10061-01-5	28
1,4-Dioxane	123-91-1	29
(Deleted-2-ethoxyethanol)	110-80-5	224
Ethyl acetate	141-78-6	225
Ethyl benzene	100-41-4	226
Ethyl cyanide	107-12-0	30

Table 2-1 (Continued)

Constituent	CAS no.	BDAT reference no.
Volatile Organics (continued)		
Ethyl ether	60-29-7	227
Ethyl methacrylate	97-63-2	31
Ethylene oxide	75-21-8	214
Iodomethane	74-88-4	32
Isobutyl alcohol	78-83-1	33
Methanol*	67-56-1	228
Methyl ethyl ketone	78-93-3	34
Methyl isobutyl ketone	108-10-1	229
Methyl methacrylate	80-62-6	35
Methacrylonitrile	126-98-7	37
Methylene chloride	75-09-2	38
(Deleted-2-Nitropropane)	79-46-9	230
Pyridine	110-86-1	39
1,1,1,2-Tetrachloroethane	630-26-6	40
1,1,2,2-Tetrachloroethane	79-34-6	41
Tetrachloroethene	127-18-4	42
Toluene	108-88-3	43
Tribromomethane (Bromoform)	75-25-2	44
1,1,1-Trichloroethane	71-55-6	45
1,1,2-Trichloroethane	79-00-5	46
Trichloroethene	79-01-6	47
Trichloromonofluoromethane	75-69-4	48
1,2,3-Trichloropropane	96-18-4	49
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	231
Vinyl chloride	75-01-4	50
1,2-Xylene	97-47-6	215
1,3-Xylene	108-38-3	216
1,4-Xylene	106-44-5	217
Semivolatile Organics		
Acenapthalene	208-96-8	51
Acenaphthene	83-32-9	52
Acetophenone	96-86-2	53
Acrylamide*	79-06-1	233

Table 2-1 (Continued)

Constituent	CAS no.	BDAT reference no.
Semivolatile Organics (continued)		
2-Acetylaminofluorene	53-96-3	54
4-Aminobiphenyl	92-67-1	55
Aniline	62-53-3	56
Anthracene	120-12-7	57
Aramite*	140-57-8	58
Benz(a)anthracene	56-55-3	59
Benzal chloride*	98-87-3	218
Benzenethiol*	108-98-5	60
(Deleted-Benzidine)	92-87-5	61
Benzo(a)pyrene	50-32-8	62
Benzo(b)fluoranthene	205-99-2	63
Benzo(ghi)perylene	191-24-2	64
Benzo(k)fluoranthene	207-08-9	65
p-Benzoquinone*	106-51-4	66
Bis(2-chloroethyoxy)methane	111-91-1	67
Bis(2-chloroethyl)ether	111-44-4	68
Bis(2-chloroisopropyl)ether	39638-32-9	69
Bis(2-ethylhexyl)phthalate	117-81-7	70
4-Bromophenyl phenyl ether	101-55-3	71
Butyl benzyl phthalate	85-68-7	72
2-sec-Butyl-4,6-dinitrophenol	88-85-7	73
p-Chloroaniline	106-47-8	74
Chlorobenzilate*	510-15-6	75
p-Chloro-m-cresol	59-50-7	76
2-Chloronapthalene	91-58-7	77
2-Chlorophenol	95-57-8	78
(Deleted-3-chloropropionitrile)	542-76-7	79
Chrysene	218-01-9	80
o-Cresol	95-48-7	81
p-Cresol	106-44-5	82
Cyclohexanone*	108-94-1	232
Dibenz(a,h)anthracene	53-70-3	83
Dibenzo(a,e)pyrene*	192-65-4	84
(Deleted-Dibenzo(a,i)pyrene)	189-55-9	85
m-Dichlorobenzene	541-73-1	86

Table 2-1 (Continued)

Constituent	CAS no.	BDAT reference no.
Semivolatile Organics (continued)		
o-Dichlorobenzene	95-50-1	87
p-Dichlorobenzene	106-46-7	88
3,3'-Dichlorobenzidine*	91-94-1	89
cis-1,4-Dichloro-2-butene*	1476-11-5	234
2,4-Dichlorophenol	120-83-2	90
2,6-Dichlorophenol	87-65-0	91
Diethyl phthalate	84-66-2	92
3,3'-Dimethoxybenzidine*	119-90-4	93
p-Dimethylaminoazobenzene*	60-11-7	94
3,3'-Dimethylbenzidine*	119-93-7	95
2,4-Dimethylphenol	105-67-9	96
Dimethyl phthalate	131-11-3	97
Di-n-butyl phthalate	84-74-2	98
1,4-Dinitrobenzene	100-25-4	99
4,6-Dinitro-o-cresol	534-52-1	100
2,4-Dinitrophenol	51-28-5	101
2,4-Dinitrotoluene	121-14-2	102
2,6-Dinitrotoluene	606-20-2	103
Di-n-octyl phthalate	117-84-0	104
Di-n-propylnitrosamine	621-67-7	105
Diphenylamine	122-39-4	106
Diphenylnitrosamine	86-30-6	219
1,2-Diphenylhydrazine	122-66-7	107
Fluoranthene	206-44-0	108
Fluorene	86-73-7	109
Hexachlorobenzene	118-74-1	110
Hexachlorobutadine	87-68-3	111
Hexachlorocyclopentadiene*	77-47-4	112
Hexachloroethane	67-72-1	113
Hexachlorphene*	70-30-4	114
Hexachloropropene	1888-71-7	115
Indeno(1,2,3-cd)pyrene	193-39-5	116
Isosafrole	120-58-1	117
Methapyrilene	91-80-5	118
3-Methylcholanthrene	56-49-5	119

Table 2-1 (Continued)

Constituent	CAS no.	BDAT reference no.
Semivolatile Organics (continued)		
4,4'-Methylenebis(2-chloroaniline)	101-14-4	120
Methyl methanesulfonate	66-27-3	36
Naphthalene	91-20-3	121
1,4-Naphthoquinone*	130-15-4	122
1-Napthylamine*	134-32-7	123
2-Napthylamine*	91-59-8	124
p-Nitroaniline	100-01-6	125
Nitrobenzene	98-95-3	126
4-Nitrophenol	100-02-7	127
N-Nitrosodi-n-butylamine	924-16-3	128
N-Nitrosodiethylamine	55-18-5	129
*N-Nitrosodimethylamine	62-75-9	130
N-Nitrosomethylethylamine	10595-95-6	131
N-Nitrosomorpholine	59-98-2	132
N-Nitrosopiperidine	100-75-4	133
N-Nitrosopyrrolidine	930-55-2	134
5-Nitro-o-toluidine	99-65-8	135
Pentachlorobenzene	608-93-5	136
Pentachloroethane*	76-01-7	137
Pentachloronitrobenzene	82-68-8	138
Pentachlorophenol	87-86-5	139
Phenacetin	62-44-2	140
Phenanthrene	85-01-8	141
Phenol	108-95-2	142
Phthalic anhydride*	85-44-9	220
(Deleted-2-Picoline)	109-06-8	143
Pronamide	23950-58-5	144
Pyrene	129-00-0	145
Resorcinol*	108-46-3	146
Safrole	94-59-7	147
1,2,4,5-Tetrachlorobenzene	95-94-3	148
2,3,4,6-Tetrachlorophenol	58-90-2	149
1,2,4-Trichlorobenzene	120-82-1	150
2,4,5-Trichlorophenol	95-95-4	151
2,4,6-Trichlorophenol	88-06-2	152

Table 2-1 (Continued)

Constituent	CAS no.	BDAT reference no.
Semivolatile Organics (continued)		
Tris(2,3-dibromopropyl) phosphate*	126-72-7	153
Metals		
Antimony	7440-36-0	154
Arsenic	7440-38-2	155
Barium	7440-39-3	156
Beryllium	7440-41-7	157
Cadmium	7440-43-9	158
Chromium (total)	7440-47-3	159
Chromium (hexavalent)	--	221
Copper	7440-50-8	160
Lead	7439-92-1	161
Mercury	7439-97-6	162
Nickel	7440-02-0	163
Selenium	7782-49-2	164
Silver	7440-22-4	165
Thallium	7440-28-0	166
Vanadium	7440-62-2	167
Zinc	7440-66-6	168
Inorganics Other Than Metals		
Cyanide	57-12-5	169
Fluoride	16964-48-8	170
Sulfide	8496-25-8	171
Organochlorine Pesticides		
Aldrin	309-00-2	172
alpha-BHC	319-84-6	173
beta-BHC	319-85-7	174
delta-BHC	319-86-6	175
gamma-BHC	58-89-9	176
Chlordane	57-74-9	177

Table 2-1 (Continued)

Constituent	CAS no.	BDAT reference no.
Organochlorine Pesticides (continued)		
p,p'-DDD	72-54-8	178
o,p'-DDD	53-19-0	235
p,p'-DDE	72-55-9	179
o,p'-DDE	3424-82-6	236
p,p'-DDT	50-29-3	180
o,p'-DDT	789-02-6	237
Dieldrin	60-57-1	181
Endosulfan I	939-98-8	182
Endosulfan II	33213-6-5	183
Endosulfan sulfate	1031-07-8	238
Endrin	72-20-8	184
Endrin aldehyde	7421-93-4	185
Heptachlor	76-44-8	186
Heptachlor epoxide	1024-57-3	187
Isodrin	465-73-6	188
Kepone	143-50-0	189
Methoxychlor	72-43-5	190
Toxaphene	8001-35-2	191
Phenoxyacetic Acid Herbicides		
2,4-Dichlorophenoxyacetic acid	94-75-7	192
Silvex	93-72-1	193
2,4,5-Trichlorophenoxyacetic acid	93-76-5	194
Organophosphorous Insecticides		
Disulfton	298-04-4	195
Famphur	52-85-7	196
Methyl parathion	298-00-0	197
Parathion	56-38-2	198
Phorate	298-02-2	199

Table 2-1 (Continued)

Constituent	CAS no.	BDAT reference no.
PCBs		
Aroclor 1016	12674-11-2	200
Aroclor 1221	11104-28-2	201
Aroclor 1232	11141-16-5	202
Aroclor 1242	53469-21-9	203
Aroclor 1248	12672-29-6	204
Aroclor 1254	11097-69-1	205
Aroclor 1260	11096-82-5	206
Dioxins and Furans		
Hexachlorodibenzo-p-dioxins	--	207
Hexachlorodibenzofurans	--	208
Pentachlorodibenzo-p-dioxins	--	209
Pentachlorodibenzofurans	--	210
Tetrachlorodibenzo-p-dioxins	--	211
Tetrachlorodibenzofurans	--	212
2,3,7,8-Tetrachlorodibenzo-p-dioxin	1746-01-6	213

*Because of the analytical problems associated with these constituents, their analysis should be undertaken only if they are suspected to be present in the matrix of interest. For EPA projects, approval for analyzing the specific constituents should be obtained from the EPA Project Manager and the designated QA Officer.

are diluted, the matrix detection limit should be calculated as the detection limit for the particular matrix times the dilution factor.

For the constituents of interest, the detection limit should be at a maximum 1 ppm in the matrix to be analyzed. For multicomponent target analysis such as PCDDs and PCDFs, the detection limit should be reported in terms of a single isomer. The laboratory should try to achieve the lowest detection limit possible for all constituents of interest. Figure 2-1 provides a decision tree diagram of the steps that the laboratory must take if a 1-ppm or lower detection limit cannot be achieved for all constituents.

For EPA tests, if a detection limit of 1 ppm or lower cannot be obtained based on the amount of sample that will be used for sample extraction, digestion, or other sample preparation step, the laboratory is to stop work and immediately contact the Contractor Project Manager or his/her designee. At this time, the laboratory should make recommendations on how to proceed with the analysis, including recommendations on any additional cleanup methods that could be used to eliminate the interference or matrix problems that are preventing the laboratory from achieving this data quality objective. The Contractor Project Manager must then immediately notify the EPA Project Manager or his/her designee of the problem. The EPA Project Manager will then evaluate the recommendations and determine whether (1) the laboratory should proceed even though a 1-ppm or lower detection limit cannot be achieved, (2) the laboratory should implement the additional cleanup techniques to achieve better detection limits, or (3) the work should be discontinued since the expected detection limits are not adequate to evaluate treatment performance. Note that the laboratory must obtain approval for exceeding the 1-ppm detection limit requirement if it has determined by a review of historical data or by a screening technique that to achieve better analytical results the amount of sample to be extracted or digested should be reduced from the sample quantity recommended for samples with low constituent concentrations.

If sufficient sample is extracted or digested such that a detection limit of 1 ppm or lower is expected to be achieved for the constituents of interest in the sample, but some constituents are present at concentrations greater than the linear range of the calibration curve, then the laboratory is authorized to quantify the diluted sample results following each method's procedures without first notifying the Contractor Project Manager that a 1-ppm detection limit may not be achieved for all constituents in that sample. The laboratory, however, must then notify the Contractor Project Manager and EPA Project Manager that the concentration levels of some constituents were high, impacting the detection limits of other constituents. The laboratory should make recommendations on additional sample cleanup techniques that may be used to achieve better detection limits for these other constituents.

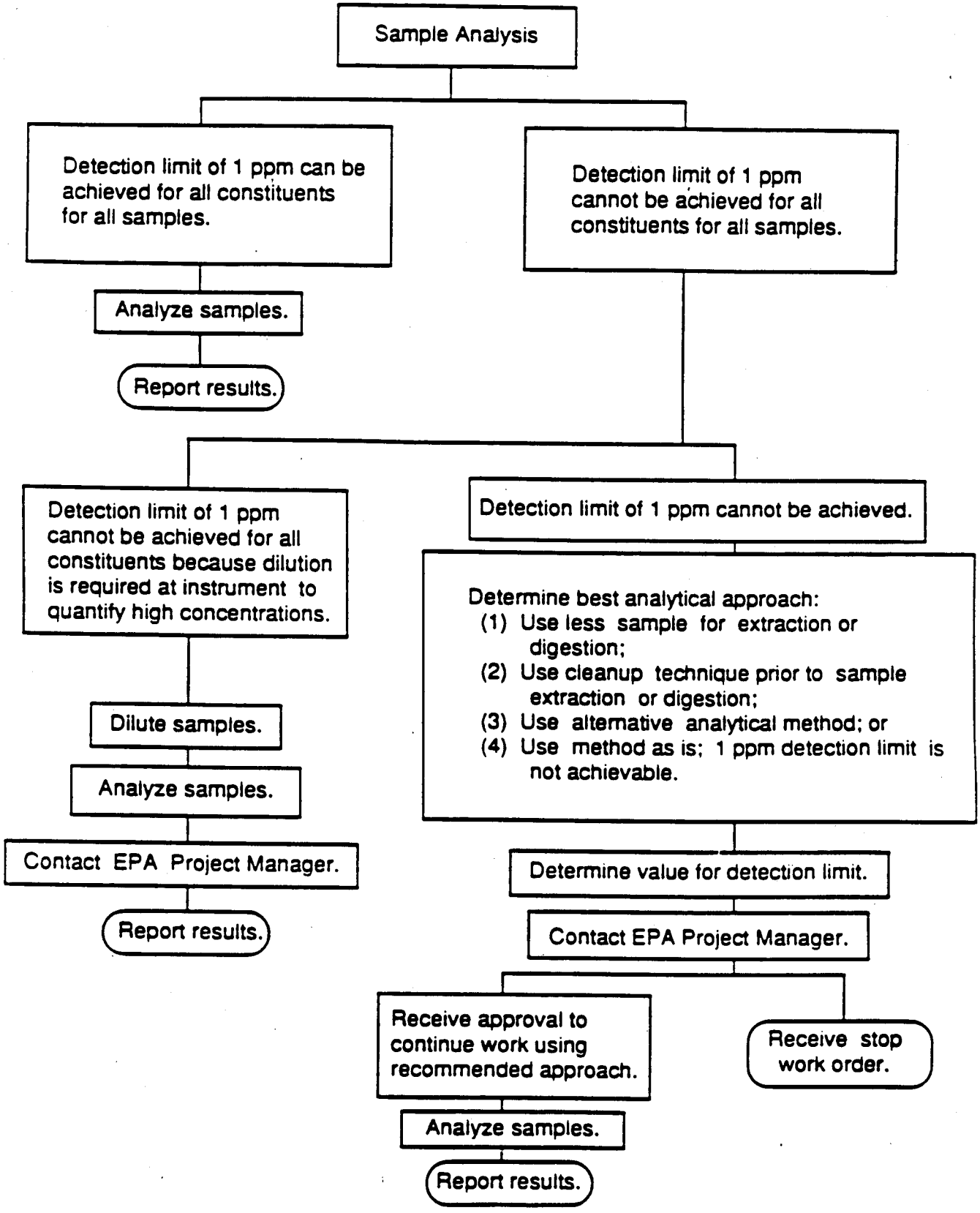

Figure 2-1. Decision Tree Diagram for Achieving Detection Limit

The matrix detection limit is to be calculated following the procedures given in each analytical method. The method detection limit should be calculated following the procedures given in the revised Section 1 of SW-846. The method detection limit is calculated using the following equation:

$$\text{Method Detection Limit} = 6.9s$$

where s = the standard deviation calculated from three replicates.

(2) Precision and accuracy. Precision is defined in terms of relative percent difference of the matrix spike and the matrix spike duplicate, where applicable. The site-specific sampling and analysis plan (SAP) for each treatment test should specify the samples designated for this analysis.

Precision will be calculated using the following equation for relative percent difference:

$$RPD\ (\%) = \frac{(C_1 - C_2)\ x\ 100}{[(C_1 + C_2)/2]}$$

where:

RPD = relative percent difference,
C_1 = the larger of the two values for matrix spike duplicates or laboratory duplicates, and
C_2 = The smaller of the two values for matrix spike duplicates or laboratory duplicates.

Although EPA is not yet specifying acceptable limits for precision, a RPD result should be reported in the data packages received from the laboratory and in the ensuing onsite engineering reports.

Percent recovery of laboratory matrix spikes is the quantitative measure of accuracy. For the treatment test analysis, a matrix spike and a matrix spike duplicate will be completed, at a minimum, on one sample of each type of treatment residual.

The spike constituents should be determined on a site-specific basis for each sampling activity and should be presented in the SAP together with the code numbers for each sample to

be taken. Spiking should be completed at the laboratory prior to extraction or digestion of the sample. (If less than 1 liter of sample is required for the matrix spike and matrix spike duplicate, then one sample container will be filled in the field and the laboratory will take the sample aliquots for the matrix spike and the matrix spike duplicate from the same container. If more than 1 liter of sample is required, then multiple sample containers are required and the matrix spike and matrix spike duplicate will be taken from different containers.) The spike concentration levels should be within 5 times the initial concentration level prior to spiking or at 5 times the expected matrix detection limit for constituents expected to be at the nondetect level. If the sample was not spiked within these ranges, the impact on the quality of the data should be assessed and the EPA Project Manager should be notified. If necessary, the samples may be respiked and reanalyzed.

When the March 1987 generic quality assurance project plan was published, no limits for accuracy were specified. Subsequently, it was determined that the recoveries for the matrix spike and matrix spike duplicate should be between 20 and 200 percent. If recoveries are less than 20 percent, the EPA Project Manager must be notified. The EPA Project Manager will determine whether any additional work is required to achieve spike recoveries of at least 20 percent. If recoveries are greater than 200 percent, the data must be flagged; review on a case-by-case basis will determine whether the results are usable.

The following equation should be used to calculate recoveries:

$$\textit{Percent Recovery (\%)} = \frac{(C_i - C_o)}{C_t} \times 100$$

where:

C_i = concentration of spiked aliquot,
C_o = concentration of unspiked aliquot, and
C_t = concentration of spike added.

(3) Completeness. Completeness is defined as the number of activities initiated that are actually finished. For this project, the first activity is acquiring the samples and the final activity is reporting the analytical data. The degree of completeness is the number of samples for which acceptable analytical data are generated divided by the total number of samples collected times 100. The QA objective for completeness in the CSD sampling and analysis efforts is 100 percent. If the completeness is less than 100 percent, documentation must be

provided to explain why the QA objective was not met in terms of sample handling, analysis, and documentation and to describe the impact on the project of these failures to achieve 100 percent completeness.

(4) Representativeness. For this project, representativeness is addressed through selection of appropriate sampling locations and procedures. For the treatment tests, the goal is to obtain samples representative of the untreated matrix and treatment residuals such that the performance of the treatment could be evaluated. One way this can be accomplished is by obtaining matched in and out sample pairs (or sets) of the untreated matrix and treatment residuals. (Note that residence times must be taken into account.)

(5) Comparability. For this project, comparability for each treatment test will be addressed through use of the same analytical procedures to analyze the samples. The analytical data should be reported in the same units for each test.

2.1.2 Project Organization

The EPA Program Manager will have the overall quality assurance (QA) responsibility for all sampling and analysis data collected for the BDAT program. All sampling and analysis plans (SAPs) must be approved by the EPA Program QA Coordinator or his designees. Figure 2-2 presents a general organization chart. A test-specific chart organization should be prepared for each SAP. Responsibilities of the various positions are described below.

EPA Project Manager:	Overall responsibility for all sampling and analysis data and for ensuring data compliance with the program's data quality objective.
EPA QA Officer:	Responsible for ensuring data compliance with the program's data quality objectives, approving site-specific SAPs and onsite engineering reports, and conducting audits, if necessary.
Contractor Program Manager:	Responsible for all work performed by the contractor.
Contractor Project Manager:	Responsible for budgets and scheduling; project technical oversight and coordination; and project staff--principal engineers, sampling staff, and laboratory staff.

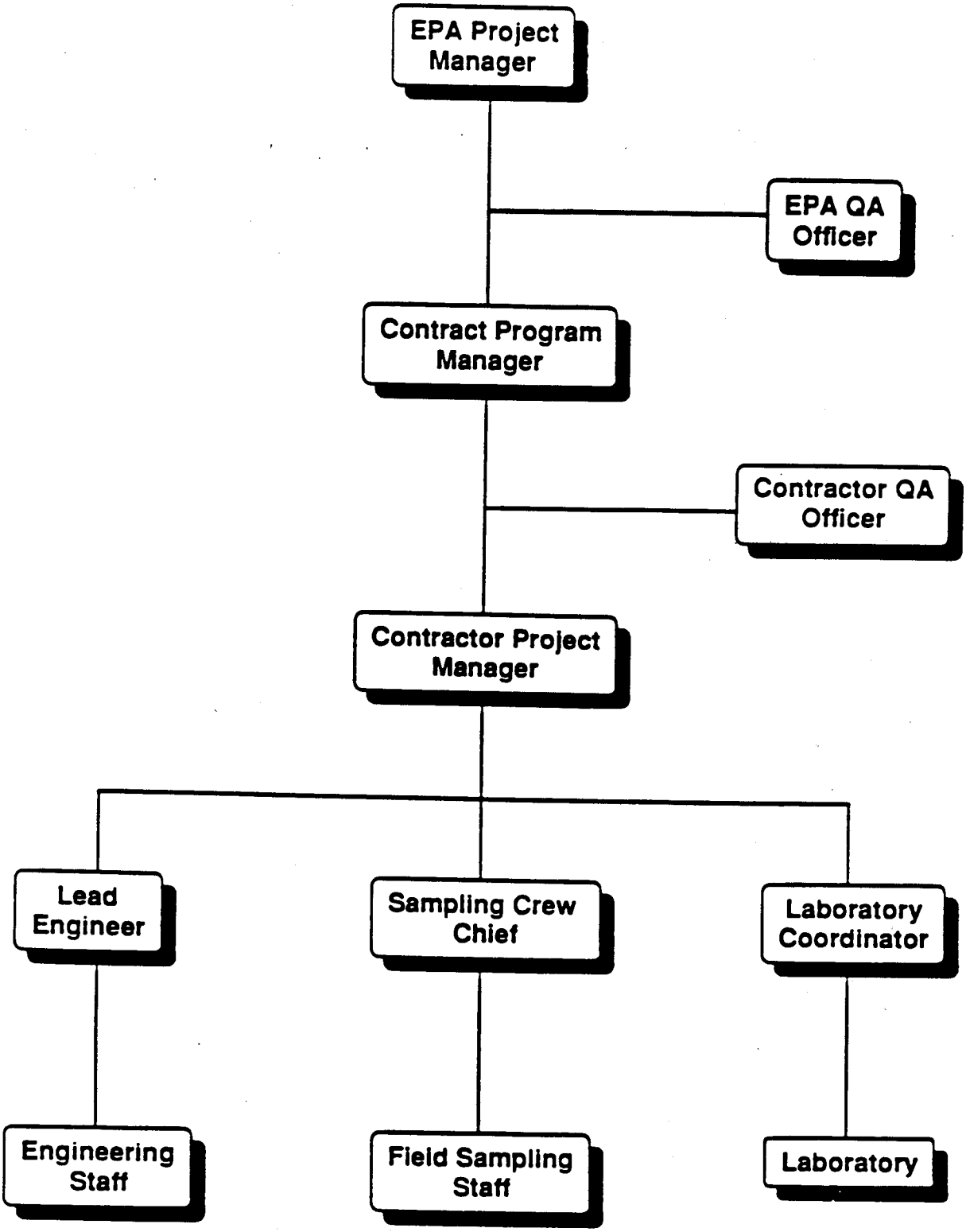

Figure 2-2. Project Organization

Contractor QA Officer:	Responsible for ensuring that the sampling and analysis data meet the project's data quality objectives and reviewing all data management activities.
Principal Engineer:	Responsible for obtaining background information on the waste to be treated and on the applicable treatment technologies, scheduling the treatment tests, and preparing the site-specific SAPs and onsite engineering reports.
Sampling Crew Chief:	Responsible for ensuring that all samples and data required by the site-specific SAP are collected in accordance with the project's QAPjP, ensuring that the field staff members have adequate training, and ensuring onsite compliance with the appropriate health and safety requirements.
Laboratory Coordinator:	Responsible for scheduling the analytical work and for ensuring compliance with the analytical requirements of the QAPjP and SAP.

2.1.3 Collection Plan for Field Samples and Design and Operating Parameters

The site-specific sampling and analysis plan must contain the following information characterizing the waste being treated and the treated residual. Note that these bulleted items are appropriate section headings.

- Sampling point descriptions. Describe the sampling points and provide the justification for selection of these sampling points. Identify all sampling points on the schematic diagram for the waste treatment system.

- Sample collection method. All samples should be collected as grab samples. Describe sample collection procedures for each sample location.

- Sampling Scheduling. Frequency of sample collection will vary depending on the treatment system. Specify the frequency of sample collection at each sampling location in the SAP and should be selected to best characterize the variability in (1) the waste stream, (2) the treatment process, and (3) the analytical results.

- Constituents to be analyzed. For all sampling points, specify which of the compounds shown in Table 2-1 (BDAT Constituents List) will be analyzed. All analyses should be performed using SW-846 (Third Edition). Deviations from this list of compounds should be justified. (For example, if one sample of the untreated waste is analyzed and the data show that particular compounds are not present, then further analysis of these compounds may not be required for the other samples from the plant.) Table 2-2 provides an example table that can be used to summarize planned analysis and quality control samples.

- Total composition and TCLP extracts. For the treated residuals, analysis will be completed on both the total composition sample for organics and inorganics and the TCLP extracts for inorganics only. For all other samples collected, analysis will be completed only for total composition. (It should be noted that in the March 1987 generic quality assurance project plan, TCLP analysis was required for both organic and inorganic constituents in the treated residuals since at the time it was not determined whether the treatment standards were to be developed using total composition or TCLP data. Subsequently, EPA decided to use total composition data to develop the treatment standards for organics.)

- Sample containerization and preservation. Follow the procedures for sample containerization and preservation presented in SW-846 (Third Edition, Table 2-16). The specific types of containers and the required sample preservation should be specified in the SAP. All sampling vessels and containers will be cleaned prior to the sample collection. The procedures used should be specified in the site-specific SAP. Table 2-3 provides examples of sample containers, sizes, holding times, and preservation requirements.

- Design and operating data collection. To evaluate the treatment design and operation, the SAP must contain (1) all design and operating data to be collected, the method of collecting these data, and the reason for collecting these data; (2) the specific frequency for collecting the operating data; and (3) identified locations for collecting operating data on the treatment system schematic.

Sampling procedures, locations, and frequencies must be documented in the site-specific SAP. Sampling times for the untreated and treated samples must take into account the residence time of the treatment system. The untreated and treated samples should be corresponding matched pairs so that waste characteristics can be evaluated. Any deviations from obtaining matched pairs must be documented in the SAP and must be approved by the EPA Project Manager. If possible, six sets of untreated and treated samples should be collected. However,

Table 2-2 Example Summary of Planned Analyses and Quality Control Samples

Analytical procedure	Number of samples collected		
	Characterization sample	Untreated waste	Treatment residual
Semivolatiles			
Primary samples	1	6	6
Matrix spikes[a]	1	1	1
Matrix spike duplicates[a]	0	1	1
Field sampling blank	1	0	1
Equipment blank	0	0	1
Metals			
Primary samples	1	6	6
Matrix spikes[a]	1	1	1
Matrix spike duplicates[a]	0	1	1
Field sampling blank	1	1	1
Equipment blank	1	1	1

[a]Analyses of the matrix spike and matrix spike duplicate samples are to be completed for the third set of matched samples collected for the untreated soil and the treatment residuals. Note that sufficient sample aliquot amounts must be collected for this set of samples to complete these analyses.

Table 2-3 Example of Sample Containers, Sizes, Holding Times, and Preservation Requirements

Parameter	Container	Sample size	Holding time	Preservation[a]
Wastewaters				
Total metals	P,G	1 one-liter jar	6 months (except mercury at 28 days)	pH <2 with HNO_3
TCLP (metals only)[b]	P,G	1 one-liter jar	6 months (except mercury at 28 days)	Cool ≤4°C
pH	-	-	Immediately	-
Chloride)	P,G	1 500-ml jar	28 days	Cool ≤4°C
Sulfate)			28 days	
Total solids)			7 days	
Total organic carbons	G	2 40-ml VOA vials	28 days	pH <2 with H_2SO_4, cool ≤4°C
Volatile organics	G	2 40-ml VOA vials	14 day	Cool ≤4°C
Semivolatile organics[c]	G	2 one-liter jars	7 days to extraction 40 days to analysis	Cool ≤4°C
Dioxins and furans[c]	G	2 one-liter jars	30 days to extraction 45 days to analysis from collection	Cool ≤4°C
Solids				
Total metals)	P,G	1 500-ml wide-mouth jar	6 months (except mercury at 28 days	Cool ≤4°C
)			6 months to TCLP extraction,	
TCLP (metals only)[b])			6 months to analysis (except mercury at 28 days and 28 days, respectively)	
Chloride)	G	1 250-ml jar	28 days	Cool ≤4°C
Sulfate)				
Total organic carbon)				
Volatile organics	G	1 120-ml jar	14 days	Cool ≤4°C
Semivolatile organics[c]	G	1 250-ml jar	14 days to extraction 40 days to analysis	Cool ≤4°C

Table 2-3 (Continued)

Parameter	Container	Sample size	Holding time	Preservation[a]
Dioxins and furans[c]	G	1 120-ml jar	30 days to extraction, 45 days to analysis from collection	Cool ≤4°C
Sludges				
Total metals) TCLP (metals only)[b])	P,G	2 one-liter wide-mouth jars	6 months (except mercury at 28 days)	Cool ≤4°C
Chloride) Sulfate) Total organic carbon) Total solids)	G	1 500-ml wide-mouth jar	28 days 28 days 28 days 7 days	Cool ≤4°C
Volatile organics	G	2 40-ml VOA vials	14 days	Cool ≤4°C
Semivolatile organics[b]	G	2 one-liter wide-mouth jars	14 days to extraction, 40 days to analysis	Cool ≤4°C
Dioxins and furans[c]	G	2 one-liter wide-mouth jars	30 days to extraction 45 days to analysis from collection	Cool ≤4°C

Footnotes:

P - Plastic
G - Glass

[a]Field samples will be packed on ice for shipment. Upon receipt at the laboratory, the samples will be stored at ≤4°C.

[b]If TCLP extracts are to be analyzed for organics, holding times are as follows: volatiles, 14 days to TCLP extraction and 14 days to analysis (28 days total); semivolatiles, 7 days to TCLP extraction, 7 days to preparative extraction, and 40 days to analysis (54 days total).

[c]For samples requiring QA analyses (MS and MSD), collect twice the amount.

Note: Sample containers must be filled to ensure that adequate sample is available for analysis.

the final selection of the number of sampling sets needed to evaluate the treatment system must be approved by the EPA Project Manager and presented in the treatment test SAP.

2.1.4 Sample Custody and Transport

Field chain of custody must be maintained for all samples collected for the LDR Program. Documentation of all field activities is required to provide backup for any deviations from the SAP. All samples collected should be labeled and identified using a multi-part label; an example of a three-part label is shown in Figure 2-3. The labels have a preprinted number that becomes the field sample number. One portion will be completed and affixed to the sample bottle; another portion will be entered into the field notebook with pertinent information entered alongside the label. At a minimum, all replicate volumes for a particular sample/parameter should have the same field sample number assigned to them.

Sample custody seals (see Figure 2-4) will be placed around all shipping container lids to detect unauthorized tampering with samples following collection and prior to the time of analysis. (This includes any untreated waste or treatment residuals that are being shipped for the purpose of being used in a treatment test.) The seal must be attached in such a way that it is necessary to break it in order to open the container. Seals must be affixed at the time of packaging by the sampling crew chief or his/her designee. The seal should include the signature of the sampling crew chief and the date.

Sample custody will begin at the time of sample collection by placing the sample in an ice chest, or other appropriate container, in the possession of the sampling crew chief or his designee. The chain of custody record form (see Figure 2-5) should be filled out immediately and signed by the sampling crew chief or his/her designee. The chain of custody record must be filled out completely and accurately since this form provides documentation for what was collected in the field and the analysis to be completed in the laboratory. The chain of custody record form should include the following information:

- Project name/code
- Site/facility name
- Sample location
- Sample type or matrix
- Sample date and time
- Signature of sampling crew chief or his/her designee
- Analysis required

Any additional pertinent remarks concerning the samples, e.g., sample preservative used, should also be included.

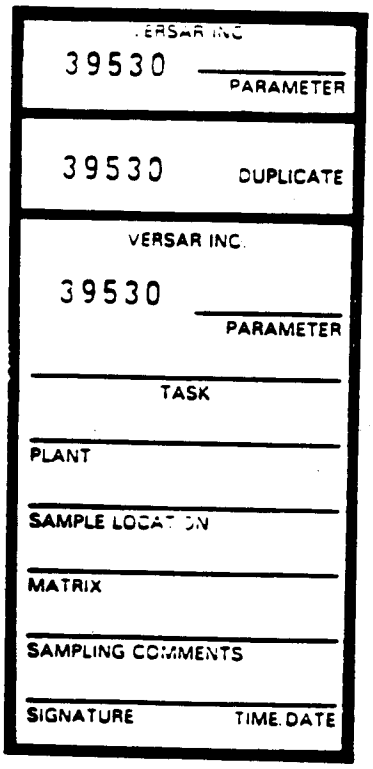

Figure 2-3. Example of Three-Part Label

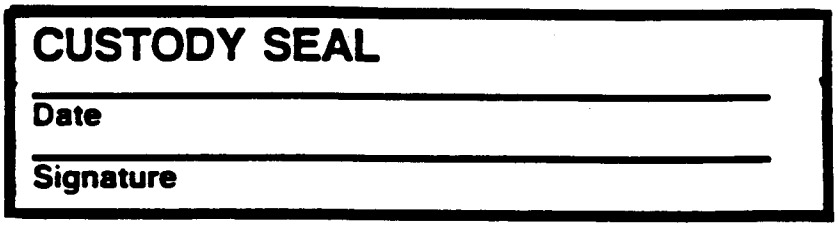

Figure 2-4. Example of Custody Seal

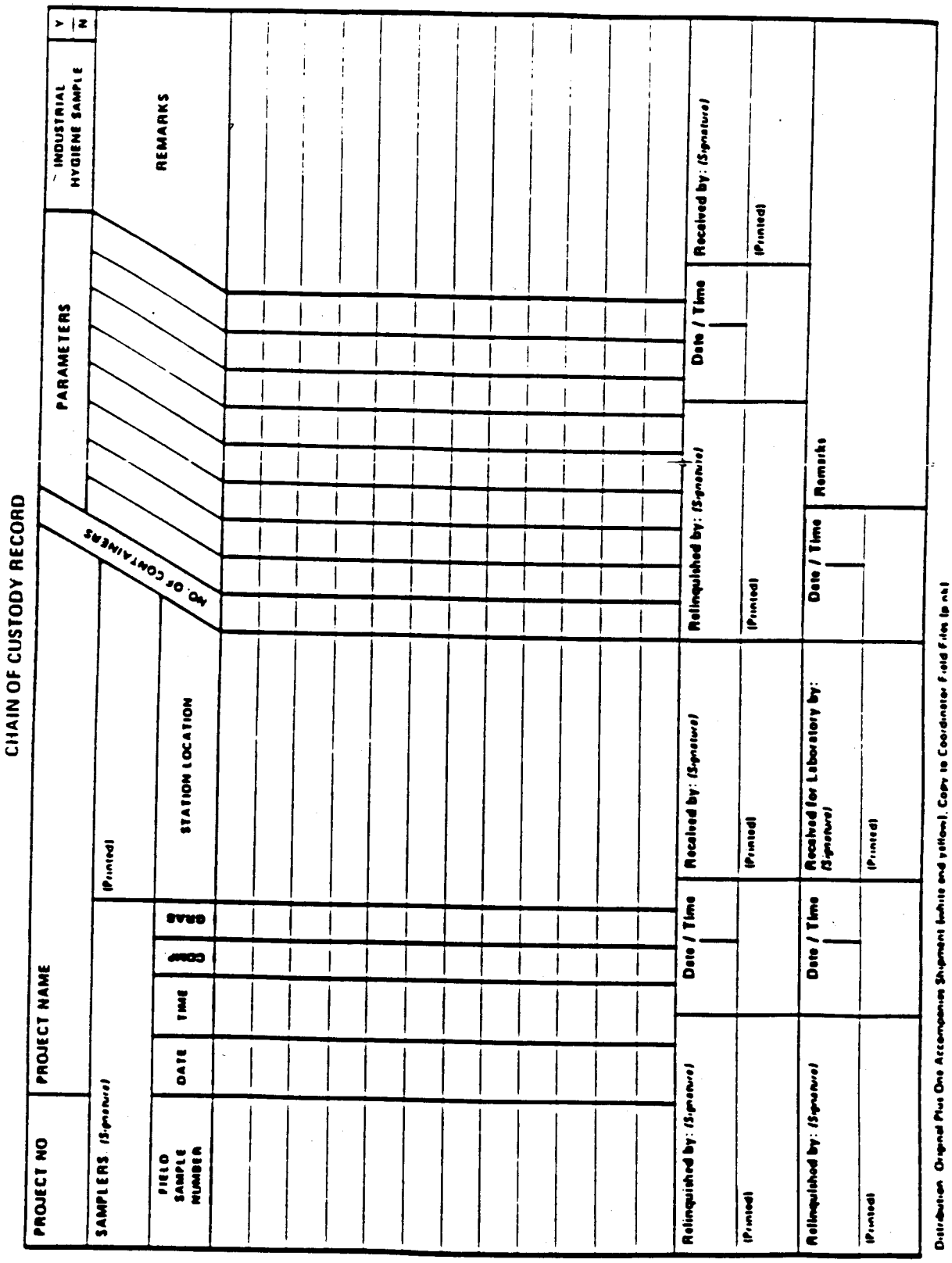

Figure 2-5. Example of Chain of Custody Record

Upon completion of the form, the sampling crew chief or his designee will sign, date, enter the time, and confirm completeness of all descriptive information contained on the chain of custody record. Each individual who subsequently assumes responsibility for the sample will sign the chain of custody record and indicate the reason for assuming custody. The field chain of custody record will terminate upon laboratory receipt of samples. The field sample custodian should retain a copy of the chain of custody record for the program files.

Samples must be packaged and labeled for shipment in compliance with current U.S. Department of Transportation (DOT) and International Air Transport Association (IATA) dangerous goods regulations. Any additional requirements stipulated by the overnight carrier must be followed. The packaging and labeling requirements should be documented in the site-specific SAP. In addition to the complete mailing address, each ice chest must be clearly marked with "this end up" arrows on all four sides, a label on each side of the container indicating the proper shipping description of the samples, and the originator's address.

A metal or plastic ice chest should be used as the outside shipping container for hazardous waste samples, unless otherwise specified by the shipping regulations. The outside container must be able to withstand a 4-foot drop on solid concrete in the position most likely to cause damage. Each ice chest should be lined with two 6-mil thick plastic bags. Styrofoam or bubble wrap will be used to absorb shock. When sample containers are placed in an ice chest for shipment, all samples from a single sampling location, except for replicate field samples, if collected, will be kept together as a set unless the SAP specifies otherwise. Replicate samples will be packaged and shipped in a separate ice chest. Since the replicate sample containers are collected only to ensure that a sufficient sample quantity is available should a problem occur during sample transport, the chain of custody forms should have these samples marked as "hold for analysis." When more than one set can fit into an ice chest, one of the sets will be placed in a separate plastic bag to prevent cross-contamination if breakage should occur. VOA vials will be packaged inside a plastic "ziplock" bag. Styrofoam or bubble wrap can be used to prevent bottle breakage. The outside of the VOA package will be labeled with the appropriate sample identification number. VOA vials should be shipped with appropriate sample sets from a given sample location.

After sample containers are sufficiently packaged, the 6-mil thick plastic bags should be sealed around the samples by twisting the top and securely taping the bag closed to prevent leakage. The custody seal will be placed around the neck of the bag. When preservation requirements dictate, ice will be placed between the inner and outer plastic bags, with the latter taped shut.

Chain of custody records and any other shipping/sample documentation accompanying the shipment will be enclosed in a waterproof plastic bag and taped to the underside of the ice chest lid.

Each ice chest prepared for shipment will be securely taped shut. Custody seals will be affixed across the joint between the top and bottom (both in front and in back) of each ice chest prepared for shipment.

The actual transportation mode should be selected based on holding times for individual analytes. All samples should be either delivered by the sampling crew or shipped via a commercial overnight carrier.

Upon receipt of the samples in the laboratory, the ice chests will be checked for intact custody seals. The samples will then be unpackaged and the information on the accompanying chain of custody records examined. If the samples shipped match those described on the chain of custody record, the laboratory sample custodian will sign the form and assume responsibility for the samples. If problems are noted with the sample shipment, the laboratory custodian will sign the form and record problems in the "Remarks" box. The appropriate Project Manager (for EPA projects, the contractor and EPA Project Managers) should be notified of any problems.

All samples will then be logged into a sample logbook and/or computerized information system. The following information will be documented:

- Date and time of sample receipt,
- Project number,
- Field sample number,
- Laboratory sample number (assigned during log-in procedure),
- Sample matrix,
- Sample parameters,
- Storage location, and
- Log-in person's initials.

All information relevant to the samples will be secured at the end of each business day. All samples will be stored in a designated sample storage refrigerator, access to which will be limited to laboratory employees.

2.1.5 Selection of Analytical Methods

Analytical methods will be selected, whenever possible, from EPA/OSW-approved methods, most of which appear in *Test Methods for Evaluating Solid Waste* (SW-846), Third

Edition (USEPA 1986). Exceptions to the requirement will be allowed for cases in which the EPA/OSW-approved methods are not appropriate for the preparation or analysis of a specific sample matrix or are not available for a particular constituent or other parameter of interest.

References to be used for selecting alternatives to the approved methods include the following:

1. *Methods for the Chemical Analysis of Water and Wastes* (MCAWW), EPA 600/4-79-020 (USEPA 1983);

2. Other available EPA methods, e.g., methods described in the Statement of Work (SOW) for EPA's Contract Laboratory Program (CLP);

3. *Standard Methods for the Examination of Water and Wastewater* (SM), 16th Edition (American Public Health Association, American Water Works Association, and Water Pollution Control Federation 1985); and

4. Methods published annually by the American Society of Testing and Materials (ASTM).

If appropriate methods to analyze specific waste matrices or to analyze specific other parameters for waste characterization are not available in the aforementioned references, then a literature search may be completed to obtain an appropriate method to complete the analysis.

All SAPs should specify the exact analytical methods to be used for the samples collected during the treatment test. Since the SAPs are site-specific, they should include any cleanup or preparation steps that may be required to analyze the samples. Table 2-4 presents recommended SW-846 methods and other methods that may be used to analyze BDAT constituents and waste characteristics affecting performance.

Whether an EPA-approved or other method is used for the constituent parameter of interest, the laboratory must provide documentation concerning the methods used and any modifications or deviations required to analyze the various samples. If feasible, the laboratory should obtain approval from the EPA Project Manager or his/her designee for method modifications or deviations prior to implementation. This information must be included in the onsite engineering report completed for the treatment test.

Table 2-4 Recommended Analytical Methods

Parameter	Preparation method[a]	Analysis method[a]
Solids		
BDAT list constituents:		
Volatile organics	5030	8240
Methanol	5040	8015
Semivolatile organics	3540/3550	8270
TCLP for organics	1311 followed by methods for organics in wastewaters	Follow methods for organics in wastewaters
Metals, total		
ICP metals	3050	6010
Arsenic	3050	7060
Chromium (hexavalent)	TCLP-51 FR 40643	7197
Lead	3050	7421
Mercury	7471	7471
Selenium	3050	7740
Thallium	3050	7841
Metals, TCLP	1311 followed by:	
ICP metals	3010	6010
Arsenic	7060	7060
Chromium (hexavalent)	7197	7197
Lead	3020	7421
Mercury	7470	7470
Selenium	7740	7740
Thallium	3020	7841
Cyanides	9012	9012
Fluorides	MCAWW 340.2	MCAWW 340.2
Sulfides	9030	9030
Organochlorine pesticides	8080	8080
Phenoxyacetic acid herbicides	8150	8150
Organophosphorous insecticides	8140	8140

Table 2-4 (continued)

Parameter	Preparation method[a]	Analysis method[a]
PCBs	8080	8080
Dioxins and furans	8280	8280
Other parameters:		
Ash content	N/A	ASTM D3174
Ash fusibility	N/A	ASTM E953
Chloride	N/A	9250
Corrosivity	N/A	1110
Heating value	N/A	ASTM D2015
Moisture content	N/A	ASTM D2216
Oil and grease	N/A	9071
pH	N/A	9045
Sulfate	N/A	9036
Sulfur content	N/A	ASTM D4239
Total halogens	N/A	ASTM D808
Total organic carbon (TOC)	N/A	Lloyd Kahn
Total organic halides	N/A	9020
Wastewaters		
BDAT list parameters:		
Volatile organics	8240	8240
Semivolatile organics	3510/3520	8270
Metals		
ICP metals	3010	6010
Arsenic	7060	7060
Chromium (hexavalent)	7197	7197
Lead	3020	7421
Mercury	7471	7471
Selenium	7740	7740
Thallium	3020	7841
Cyanides	9012	9012
Fluorides	N/A	MCAWW 340.2
Sulfides	N/A	9030
Organochlorine pesticides	8080	8080
Phenoxyacetic acid herbicides	8150	8150
Organophosphorous insecticides	8140	8140
PCBs	8080	8080

Table 2-4 (continued)

Parameter	Preparation method[a]	Analysis method[a]
Dioxins and furans	8280	8280
Other parameters		
Acidity	N/A	MCAWW 305.1
Alkalinity	N/A	MCAWW 310.1
Bromide	N/A	MCAWW 320.1
Chemical oxygen demand (COD)	N/A	MCAWW 410.1-.4
Chloride	N/A	9250-52
Color	N/A	MCAWW 110.1-.3
Conductance	N/A	MCAWW 120.1
Corrosivity	N/A	1110
Hardness, total	N/A	MCAWW 130.1-.2
Heat value	N/A	ASTM E711
Iodide	N/A	MCAWW 345.1
Nitrogen		
Ammonia	N/A	MCAWW 350.1-.3
Kjeldahl, total	N/A	MCAWW 351.1-.4
Nitrate	N/A	MCAWW 352.1
Nitrate-nitrite	N/A	MCAWW 353.1-.3
Nitrite	N/A	MCAWW 354.1]
Oil and grease	N/A	9070
pH	N/A	MCAWW 365.1-.4
Solids		
Filterable, gravimetric	N/A	MCAWW 160.1
Nonfilterable, gravimetric	N/A	MCAWW 160.2
Total, gravimetric	N/A	MCAWW 160.3
Volatile gravimetric	N/A	MCAWW 160.4
Settleable matter	N/A	MCAWW 160.5
Sulfate	N/A	9035/9036/9038
Total organic carbon (TOC)	N/A	9060
Total organic halides (TOX)	N/A	9020/9022
Viscosity	N/A	ASTM D445

[a]All methods are SW-846 methods unless otherwise specified.
N/A - Not Applicable

2.1.6 Quality Assurance/Quality Control Procedures

The overall effectiveness of a quality control program depends on operating in the field and laboratory in accordance with a program that systematically ensures the precision and accuracy of analyses by detecting errors and preventing their recurrence or measuring the degree of error inherent in the methods applied.

Most of the analytical methods to be used give guidelines for number and frequency of replicates, matrix spikes, and calibration standards. The matrix spikes, replicates, calibration standards, etc., are analyzed in the same way as the field samples and are interspersed with the field samples. The analytical results are used to document the validity and control of data.

- Spikes: A matrix spike and matrix spike duplicate analysis should be performed on at least one sample of each treatment residual taken during a treatment test. The SAPs should specify which samples are to be spiked and identify the spiking components. Samples should be spiked with constituents of interest expected to be present in the waste. The matrix spike and matrix spike duplicate should meet the requirements for precision and accuracy as specified in Section 2.1.1.

- Laboratory duplicate analysis: One laboratory duplicate analysis of the spiked sample extract should be performed for each group of the treated residual samples taken from the same sampling point. The laboratory duplicate analysis should also be completed on the TCLP extract. Analytical results of the duplicate injection must be within ±20 percent of each other for values greater than 200 ppb. For values less than or equal to 200 ppb, analytical results for the duplicate injection should be within ±100 percent of each other. (The precision results of the matrix spike and matrix spike duplicate can be substituted for the laboratory duplicate analysis.) If these criteria are not met, the data should be flagged and should be reviewed on a case-by-case basis to determine their usability.

- Surrogates: For GC/MS and GC methods, surrogates (i.e., chemically inert compounds not expected to occur in an environmental sample) will be spiked into each sample to provide matrix recovery values. Surrogates should be used if specified in the analytical method. (Because of limited experience in analyzing each of the waste matrices, precision and accuracy requirements are not being specified.)

- Calibration standards: Calibration standards will be prepared in accordance with the specifications provided in the methods. Calibration standards will be analyzed at a frequency specified in the methods. Reagent grade compounds that conform to the

current specifications of the Committee on Analytical Reagents of the American Chemical Society should be used if possible.

- QC check standards: For the metal analytes, a QC check standard will be analyzed with each batch of samples. This standard is prepared by spiking laboratory pure water with a stock solution of the analyte that was obtained from a source independent of the source used to obtain standards for the calibration curve.

- Calibration check samples: For GC/MS analysis, calibration check samples should be prepared and analyzed as specified in the appropriate methods.

- Method blank: A minimum of one method blank will be prepared per set of samples of similar matrix collected during the same sampling episode or a set of 20 samples of similar matrix, whichever is smaller. In cases where the concentration detected in any of the compounds detected in the blank is 10 percent or greater than the concentration detected in any of the samples in the batch, the laboratory must take corrective actions, as specified in Section 2.1.8.

- Internal standards: Internal standards should be used where feasible to monitor for the consistency of GC/MS response factors and relative response times. The internal standards projected to be used are specified in the methods, e.g., SW-846 Methods 8240 and 8270. If the internal standards are not specified in the analytical method, they should be specified in the site-specific SAP.

- System performance check compounds: For GC/MS analysis, system performance check samples should be prepared and analyzed as specified in the appropriate methods (e.g., SW-846 Methods 8240 and 8270).

- Laboratory pure water: Laboratory water should be prepared by particulate filtration, carbon filtration, reverse osmosis, and deionization, or by an equivalent procedure.

Quality control checks to be taken during field activities will include calibration of any field monitoring equipment as well as collection of the blanks discussed below.

- One trip blank that is not opened in the field should be collected to check for sample contamination originating from sample transport, shipping, or site conditions. The parameters for analysis should be specified in the SAP.

- Equipment blanks should be taken as needed. Collection and frequency must be specified in the SAP. To prepare an equipment blank, laboratory pure water or solvents are brought to the field in a sealed container and then opened in the field. The contents are poured over or through the sample collection device and then collected in the sample container. The parameters for analysis will be specified in the SAP. If contamination in the equipment blank is detected, the effect of the contamination on the samples collected should be presented in the onsite engineering report for the treatment system.

- If samples are to be collected for analysis of volatile organic compounds, a volatile organic blank should be collected once a day. This blank consists of laboratory pure water taken to the field and poured into a sample container in the are where the treatment system is located. The volatile organic blank should be analyzed for the volatile compounds specified in the SAP. If volatile organic compounds are measured in this blank, the effect of the contamination on the samples collected should be presented in the onsite engineering report for the treatment system.

2.1.7 Quality Assurance Performance and System Audits

Field activities of each contractor should be audited at least once by a representative designated by EPA to ensure that required equipment and procedures for sample collection, preservation, shipping, handling, laboratory, and documentation were used. In lieu of a third party auditor, the field activities could be evaluated by the EPA Project Manager.

For most treatment test studies (and on at least one conducted by each contractor) for the scheduled Thirds waste codes, the EPA Project Manager was present. He could observe that the procedures for sample collection, preservation, shipping, handling, and documentation (e.g., field notebooks and chain of custody) were performed in accordance with the site-specific sampling and analysis plans. Performance samples for organics and/or metals were completed by the laboratory quarterly. The results of the performance samples indicated that the laboratory could complete the analysis for the BDAT constituents satisfactorily. A formal system audit of the laboratory was not conducted; however, the laboratory was audited for other EPA projects during the period that samples were analyzed for the various treatment tests.

2.1.8 Corrective Actions

Data generated as part of the analytical quality control program were received by the QA Officer and the project's lead engineer to ensure the absence of systematic bias or trends. Corrective actions were taken upon identification of any problems with the project that affected the product quality. If problems occurred, the cause was determined, the effect of the problem

on the project was evaluated, and a solution was developed to prevent a subsequent occurrence of the problem.

The following corrective actions were taken if the program's data quality objectives for blank contamination, duplicate injection (or analysis), or matrix spike recovery were not achieved:

1. Calculations were reviewed for mathematical or transcription error.

2. The laboratory/field documentation were reviewed to determine whether procedural errors were made.

3. Equipment and reagents were examined to determine whether there was any malfunctioning equipment or reagent contamination.

4. Instrument documentation was examined to determine whether the signal response met the acceptance criteria and whether the calibration check standards agreed with the calibration curve as specified by the analytical method to determine whether the instruments were still within calibration.

If these steps did not correct the problem, the EPA Project Manager was contacted to discuss the source of the problem and its impact on the data and to determine whether any additional corrective actions, such as reanalysis of the samples, should be taken to try to obtain data that could meet the data quality objectives.

2.1.9 Calibration Procedures

2.1.9.1 Laboratory Analyses

All instruments should be calibrated each day that analyses are performed. The calibration standards should include the constituents of concern for the project. The calibration procedures described in the appropriate analytical methods will be followed.

All calibration information should be documented. If the calibration check standard does not meet the criteria specified in the method, the instrument should be recalibrated and the samples analyzed after the last calibration check standard meeting the calibration specifications should be reanalyzed. If deviations from or modifications to these procedures are necessary, approval should be obtained from EPA prior to implementation of the deviation/modification, and documentation of these deviations/modifications and the reason for their implementation must be presented in the final analytical data report.

Calibration standards must be prepared using pure standard materials or purchased as certified solutions. If the standards are made from pure standard materials, the materials must be assayed and the purity of the standard must be known. When compound purity is assayed to be 96 percent or greater, the weight may be used without correction to calculate the concentration of the stock solution unless otherwise specified in the analytical material. Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source. The name of the manufacturer and the information regarding purity of the standard or the concentration of the stock solution, if commercially prepared, must be available upon request.

Below is an overview of the calibration procedures for the analytical instruments that may be used. The concentrations of the calibration standards for each method will be determined by the detection limit and the linear curve of the range. For example, for a three-point calibration or curve, one standard would be selected near the detection limit, one or the midpoint of the linear range, and one at the upper end of the curve.

Instrument	Procedure
Flame AA	Daily four-point calibration with blank, 1, 5, and 10 mg/l standards. Check standard and blank analysis after every 10 samples.
Furnace AA	Daily five-point calibration with blank, 5, 10, 20, and 50 μl standards. Check standard and blank analysis after every 10 samples.
ICP	Daily two-point calibration with blank and 1 mg/l standards. Interference check sample analysis every 8 hours. Check standard and blank analysis after every 10 samples.
GC	Meet chromatographic acceptance criteria (such as degradation, peak shape, sensitivity signal to noise ratio, and retention time stability). Then do three-point initial calibration with 0.2, 0.25, and 1.0 μl standards followed by daily chromatographic check and calibration check.
GC/MS	Meet MS tuning criteria followed by chromatographic acceptance criteria. Then do three-point initial calibration with 20, 50, and 100 ng/ml standards followed by daily chromatographic check and calibration check.

Instrument	Procedure
Analytical balance	Prior calibration check with class S weights in the gram and milligran range. Other checks as appropriate in expected weighing range.
HPLC	Meet chromatographic acceptance criteria (such as degradation, peak shape, sensitivity, signal to noise ratio, and retention time stability). Then do multipoint initial calibration followed by daily chromographic check and calibration check.
pH meter	Three-point calibration at pH 5, 7, and 10. Calibration check after every 10 samples.
Conductivity meter	Calibration check daily and every 20 samples.
UV spectrometer	Daily multipoint calibration. Check standard every 20 samples.
Technicon	Daily multipoint calibration. Check standard every 20 samples.
TOC	Daily single-point calibration in triplicate. Check standard every 20 samples.
TOX	Daily calibration check. Check standard every 20 samples.
IC	Daily multipoint calibration. Check standard every 20 samples.
Thermometers	Check against NBS thermometer every 6 months.
Hg analyzer	Daily four-point calibration. Check standard and blank analysis after every 10 samples.

2.1.9.2 Field Calibration

All instruments should be calibrated each day that analyses are performed in the field. The calibration standards should include the constituents of concern for the project. The calibration procedures described in the appropriate Standard Operating Procedures (SOPs) written for the field team and provided in the SAP should be followed. If the calibration check standard does not meet the criteria specified in the method, the use of the instrument will be discontinued until the unit can be recalibrated. Data collected after the last calibration check standard meeting the calibration specifications should be reanalyzed with a calibrated instrument, if possible. In addition, calibration checks should be made by the crew chief at time intervals specified in the SAP.

2.1.10 Data Reduction, Validation, and Reporting

For data to be scientifically valid, legally defensible, and comparable, valid procedures must be used to prepare those data. The following sections describe the data reduction, validation, and reporting procedures to be used for field and laboratory data.

2.1.10.1 Data Reduction

The analytical laboratory should specify its data reduction methods. Wherever possible, the initial data reduction should be computerized. This reduces the frequency of transcription errors and calculation errors. Where data reduction is not computerized, calculations should be performed in permanently bound laboratory notebooks with carbon copy pages or on preprinted data reduction pages. The data reduction for some analyses includes analysts' interpretations of the raw data and manual calculations. When this is required, the analysts' decisions will be written in ink on the raw data sheets. Any corrections to data sheets will be made by lining out inaccurate information, initialing the line-out, and adding the revised information next to the line-out.

2.1.10.2 Data Validation

Data validation begins with the analyst and continues until the data are reported. The individual analysts should verify the completion of the appropriate data forms to verify the completeness and correctness of data acquisition and reduction. The Laboratory Supervisor or the data reduction staff should review computer and manual data reduction results and should inspect laboratory notebooks and data sheets to verify data reduction correctness and completeness and to ensure close adherence to the specified analytical method procotols. Calibration and QC data should be examined by the individual analysts and the Laboratory Supervisor or the data reduction staff to verify that all instrument systems were in control and

that QA objectives for precision, accuracy, completeness, and method detection limit were met for the project.

Project data that are outside specified acceptance limits established for the data quality indicators (e.g., data points with detection limits above 1 ppm) or that are associated with QC outlier data should be flagged or otherwise identified in the laboratory's final data package.

2.1.10.3 Reporting

All reports and documentation required, including chromatograms and mass spectra, calibration records, and QC results, should be clearly labeled with the laboratory sample number and associated field sample number. A flow chart depicting the overall data handling and reporting scheme is provided in Figure 2-6.

The final data package submitted by the analytical laboratory should include a summary of the analytical results for each sample as well as all reports and documentation generated as required by the analytical methods (e.g., chromatograms, extraction notes, and chain of custody forms). Note that a full CLP type package would be an acceptable data package.

2.1.11 Preventive Maintenance

2.1.11.1 Field Preventive Maintenance

All field equipment should be maintained following procedures outlined by the manufacturer. Prior to a sampling project, the field equipment to be used should be inspected and calibrated to ensure that it is working properly. Spare parts should be available and should be taken on the sampling trip if appropriate. Following its use, it should be decontaminated using the appropriate cleaning procedures required for the project.

2.1.11.2 Laboratory Preventive Maintenance

All laboratory instrumentation will be maintained following procedures outlined by the instrument manufacturers. Instrument maintenance logbooks should be kept with each instrument and will be updated by the operator whenever either routine or nonroutine maintenance procedures are performed.

2.1.12 Quality Assurance Reports to Management

The Contractor Project Manager, in conjunction with the Contractor QA Officer, should identify critical areas of the project that will be subject to inspection. These inspections should

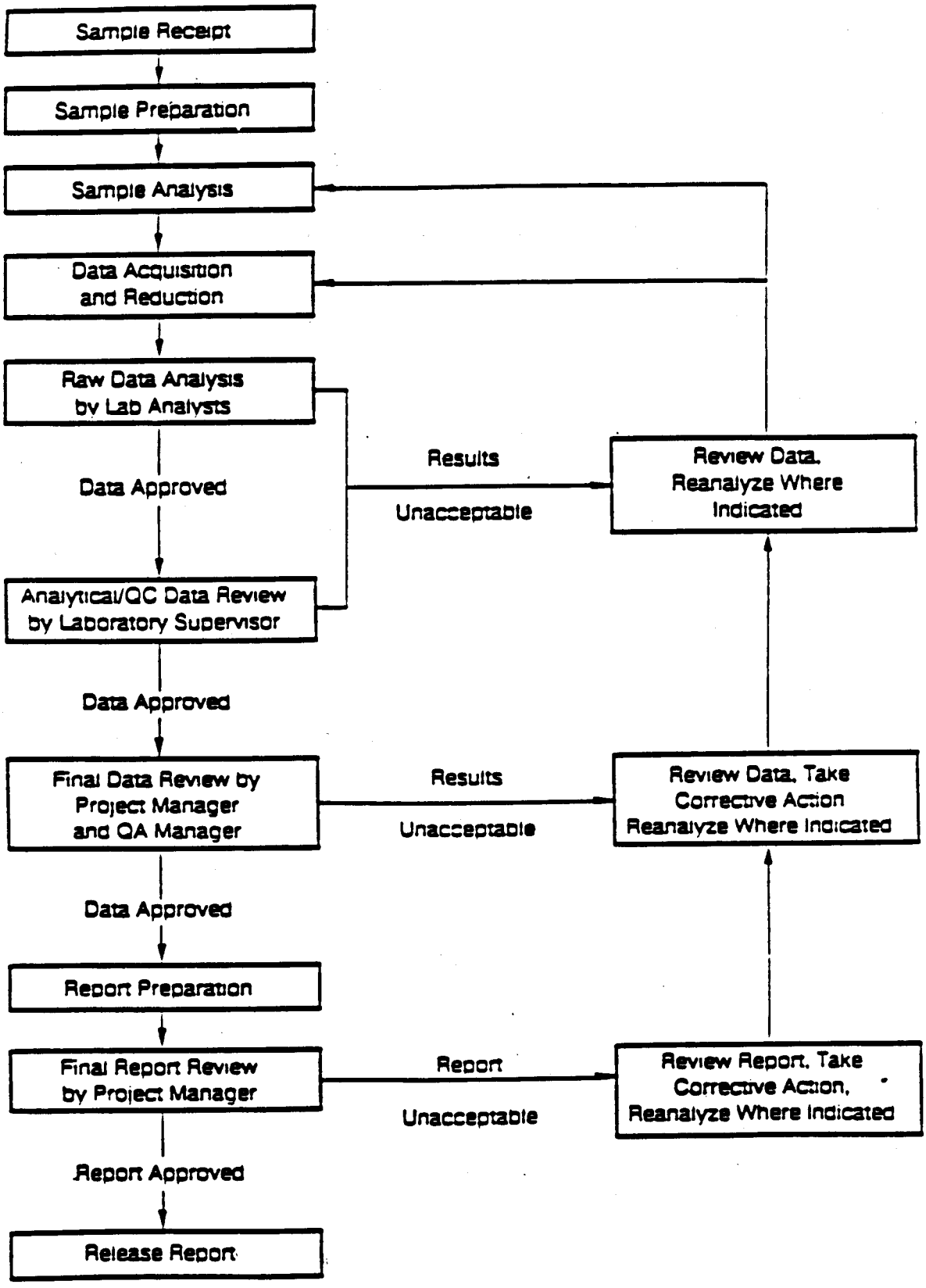

Figure 2-6. Data Reduction, Validation, and Reporting Scheme

be performed by qualified staff members who are not performing or supervising the activity. The areas inspected may include the following:

- Staff qualifications
- Equipment maintenance records
- Equipment calibration records
- Protocol adherence
- Documentation practices
- Sample traceability and control
- Data traceability and document control
- Recordkeeping practices
- Review and validation practices
- Computation practices
- QC data and practices
- QC compliance

2.2 Sampling and Analysis Plan

The following format presents prospective sampling and analysis activities in a rational and identifiable manner. "Organization" is presented here as a shorthand for the name of the industrial facility, corporation, consortium, or other entity intending to submit this data.

- Title Page

- Approval Page: Names, organizational addresses, and titles of the individuals serving as Project Manager and Quality Assurance Officer in generating this data.

- Introductory Page: Table of contents, list of tables, and list of figures.

- Section One: Introduction.

  1.1 Short description of the Organization's participation in generating data for the Land Disposal Restrictions program.

  1.2 Discussion of the objective of this treatment test in terms of the waste being treated and the technology being evaluated.

  1.3 Introductory description of the waste being treated, summarizing available analytical and other test results already performed. Tables can go into an appendix.

1.4 Names, phone numbers, and addresses of the Project Manager, Analytical Laboratory Manager, and Quality Assurance Officer with responsibility in this project.

1.5 Description of the treatment system under evaluation.

1.6 Outline and schedule of the major sampling and analysis events as anticipated.

- Section Two: Project Organization.

2.1 Organizational Chart like Figure 2-2.

2.2 Addresses and phone numbers of key individuals.

2.3 Summaries of key individuals' responsibilities.

- Section Three: Waste and Treatment System Description.

3.1 Qualitative discussion of waste: process generating it, regulatory history, previous management practices, and discussion of results of earlier analytical investigations of this waste.

3.2 Summary of existing data characterizing the waste in tabular form.

3.3 Qualitative discussion of treatment system: how it works, whether it is an established or innovative technology, whether the system is part of the generating plant's existing on-site waste management system or an off-site system or a mobile unit, dimensions and capacities of process units, and key design and operating parameters.

- Section Four: Sampling and Analysis Activities.

4.1 Table of each sample, blank, and duplicate to be taken, each numbered with a unique alphanumeric code indicating whether it is a field or equipment blank, raw or treated residual, single sample, or one of a duplicate-sample pair and indicating to what category of residual it belongs (i.e., scrubber water vs. ash for incinerator residuals) to be explained in the footnotes to this table. This table should state at which point each sample will be taken.

4.2 Schedule for sampling visit, accounting for collection, preservation, and transport of each numbered sample, duplicate or blank by identification code.

4.3 Description of proposed sampling procedure for each coded sample plus the number of samples to be collected at each site.

4.4 List of the analytes and parameters to be analyzed in each sample, the sample preparation (digestion, extraction, cleanup, etc.), and analytical methods to be used for each sample, all presented with unique sample code.

4.5 Narrative discussion of why these analytes and not others were selected from the BDAT list.

4.6 Specifications for sample aliquot size, preservation, and acceptable holding times.

- Section Five: Site-Specific QA/QC Procedures.

5.1 Description of field QA/QC activities including calibration of field monitoring equipment, preparing sampling, trip, and field blanks, ensuring that appropriate duplicates are taken and decontamination and disposal of field sampling equipment occurs.

5.2 Specify the sample aliquots upon which matrix spike analyses are to be completed and specify the spike constituents and their concentration levels.

5.3 Specify the number of trip, field, or equipment blanks to be collected and the procedures to be used. Also, specify the analyses/methods to be performed on the blanks, noting that in most cases the blanks may be marked "Hold for Analysis." Also specify procedures to be performed with reagent blanks.

5.4 List the surrogate determinations to be performed for organic analyses; if methods other than 8240 or 8270 are being used, described a surrogate use procedure similar to 8240 or 8270s to be employed.

5.5 List the QC check standards to be run for metals analyses.

5.6 List provisions for documenting all method-specific internal standards for GC and GC/MS procedures.

- Section Six: Sample Custody and Transport.

  6.1 Description of sample custody procedures and for transporting waste from generation facility to treatment facility if planned.

  6.2 Relevant information on sample packing and shipment: Shipping category for samples and any transported waste, DOT regulations and the carrier's requirements for these materials, carrier name and address of the local shipping station, address of the laboratory to which the samples will be sent, and name and phone number of the designated contact at this laboratory.

- Section Seven: Health and Safety.

  7.1 Summary of health and safety procedures to be followed on site during sampling and treatment operations. Use the facility's existing health and safety plan if one is available.

- Section Eight: References.

2.3 On-Site Engineering Report

The following format assembles the results from sampling and analysis activities in a rational and identifiable discussion of the performance of the treatment system in terms of its measured design and operating parameters and the concentration of contaminants in the raw and treated waste streams.

- Title Page

- Approval Page: Names, organizational addresses, and titles of the individuals serving as Project Manager and Quality Assurance Officer in generating these data.

- Introductory Page: Table of contents, list of tables, and list of figures.

- Section One: Introduction.

  1.1 Short description of the Organization's participation in generating data for the Land Disposal Restrictions program.

  1.2 Discussion of the goals of this treatment test, in terms of the waste being treated and the technology being evaluated, and how these goals were achieved.

1.3 Preliminary discussion of significant deviations from the SAP.

1.4 Brief introduction of the sections of the OER to follow.

1.5 Table summarizing the test site and personnel: Name and address of treatment site, site contact names with addresses and telephone numbers, treatment test dates, names, titles, and addresses of EPA personnel involved in on-site activities, names, titles, and addresses of those responsible for preparing the OER and the name, address, and phone number of the laboratory coordinator.

- Section Two: Waste Being Treated.

2.1 Qualitative discussion of waste: process generating it, regulatory history, previous management and disposal problems unique to this waste, existing management practices, and discussion of results of earlier analytical investigations of this waste.

2.2 Summary of data taken previous to this test characterizing the waste in tabular form.

2.3 Summary of analytical results on untreated waste samples in tabular form.

- Section Three: Treatment System Being Evaluated.

3.1 Qualitative discussion of treatment system: how it worked, whether it is an established or innovative technology, whether the system was part of the generating plant's existing on-site waste management system or an off-site system or a mobile unit, dimensions and capacities of process units, and key design and operating parameters.

3.2 Tabular summary of design and operating parameters measured during the test.

3.3 Process diagram of treatment system showing key units associated with design and operating parameters and sampling points.

- Section Four: Sampling and Analysis Activities and Results.

4.1 Summary schedule of treatment test events and activities.

4.2 Deviations from planned sampling and analysis operations.

4.3 Tabular summary of all analytical results, each referenced by sample code number and including the analytical method used.

NOTE: Report on all items listed in Section four of the SAP, explicitly referencing it whenever appropriate.

- Section Five: QA/QC Measures Taken.

5.1 Tabulate collection, sample preparation, and analysis dates (for preparation and analyses) and the procedures for each uniquely coded sample.

5.2 List of the BDAT List constituents analyzed for in each sample for the raw waste and the treated waste residuals plus the analytical method used for each constituent.

5.3 Narrative summary of analytical problems, deviations from SW-846, alternatives or equivalent to SW-846, and options chosen among SW-846 alternatives.

5.4 Tabulation and explanation of any detection limits exceeding 1 ppm for BDAT List constituents.

5.6 Data Quality Indicators for sample (include each sample's unique code):

- Precision and accuracy data for the treatment test sample analytical results: spiking data (matrix and matrix spike duplicates).

- Results of blanks (field, trip, laboratory, preparation, etc.) analysis.

- Instrument and matrix detection limits, together with analytical method involved.

5.7 Instrument and Procedure Verification:

- Results of surrogate determinations performed for organic analyses.

- Results of QC check standards to be run for metals analyses.

- Results of all method-specific internal standards for GC and GC/MS procedures.

1.3 Preliminary discussion of significant deviations from the SAP.

1.4 Brief introduction of the sections of the OER to follow.

1.5 Table summarizing the test site and personnel: Name and address of treatment site, site contact names with addresses and telephone numbers, treatment test dates, names, titles, and addresses of EPA personnel involved in on-site activities, names, titles, and addresses of those responsible for preparing the OER and the name, address, and phone number of the laboratory coordinator.

- Section Two: Waste Being Treated.

2.1 Qualitative discussion of waste: process generating it, regulatory history, previous management and disposal problems unique to this waste, existing management practices, and discussion of results of earlier analytical investigations of this waste.

2.2 Summary of data taken previous to this test characterizing the waste in tabular form.

2.3 Summary of analytical results on untreated waste samples in tabular form.

- Section Three: Treatment System Being Evaluated.

3.1 Qualitative discussion of treatment system: how it worked, whether it is an established or innovative technology, whether the system was part of the generating plant's existing on-site waste management system or an off-site system or a mobile unit, dimensions and capacities of process units, and key design and operating parameters.

3.2 Tabular summary of design and operating parameters measured during the test.

3.3 Process diagram of treatment system showing key units associated with design and operating parameters and sampling points.

- Section Four: Sampling and Analysis Activities and Results.

4.1 Summary schedule of treatment test events and activities.

4.2 Deviations from planned sampling and analysis operations.