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DATA QUALITY ASSESSMENT

LOS ALAMOS NATIONAL LABORATORY

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TC/TO

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APPENDIX A -- SUMMARY OF POSITIVE RESULTS
APPENDIX B -- CLP FORM I's

1.0 INTRODUCTION

Metcalf & Eddy (M&E) has performed a quality assurance review of the analytical data obtained for the oversight soil boring samples collected for MDA L at the Los Alamos National Laboratory (LANL) site. Sixteen soil samples, plus two blind field duplicate samples, two field blanks, and three trip blanks were submitted to Accutest, Inc. in Dayton, New Jersey. These samples were analyzed for volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs), Pesticides/PCBs, RCRA metals, and cyanide.

The volatile organic compounds, semivolatile organic compounds and pesticides/PCBs were analyzed in accordance with the methods contained in the *Contract Laboratory Program Statement of Work for Organic Analysis*, OLMO1.8, August 1991, modified to include the non-Target Compound List (TCL) compounds in the initial and continuing calibrations. Inorganic analytes were analyzed in accordance with the Contract Laboratory Program Statement of Work for Inorganic Analyses, ILMO2.0. September 1991.

The organic and inorganic analytical data for the LANL oversight samples have been reviewed and evaluated in accordance with *National Functional Guidelines for Organic Data Review*, December 1990 and *Laboratory Data Validation Functional Guidelines for Evaluating Inorganics Analyses*, July 1988. This included holding times, instrument tuning and calibration, method and field blank contamination, recoveries of surrogate spikes and matrix spike/matrix spike duplicate (MS/MSD) pairs, precision as determined from field duplicate results, internal standard performance (organics only), laboratory control samples (metals only), and serial dilution results (metals analyzed by ICP only). The Appendix IX constituents to which the EPA validation guidelines do not strictly apply (non-Target Compound List constituents) were evaluated according to the same criteria as were the Target Compound List parameters.

2.0 VOLATILE ORGANIC COMPOUNDS

Sixteen soil samples, plus two blind field duplicate samples were collected by Metcalf & Edy, Inc. (M&E) and submitted to Accutest, Inc. for analysis of the 40 CFR Part 264 Appendix IX volatile organic compounds by the *Contract Laboratory Program Statement of Work for Organic Analysis*, OLMO1.8, August 1991, modified to include the non-TCL compounds. In addition, three trip blanks and two field blanks were submitted for Appendix IX, VOC analyses.

2.1 TECHNICAL HOLDING TIMES

All samples were analyzed within the required 14-day holding time.

2.2 GC/MS INSTRUMENT PERFORMANCE CHECKS

The bromofluorobenzene tuning criteria were satisfied for each 12-hour shift during which the project samples and associated calibration standards and method blanks were analyzed.

2.3 INITIAL CALIBRATION

Calibration data are evaluated in two sections identified by the groups of samples analyzed on each of two instruments. The percent relative standard deviations (%RSD) and mean relative response factors (RRF) satisfied acceptance criteria for all compounds except as noted below.

2.3.1 Initial Calibration of 11/23/93 20:26 Matrix (soil)

The %RSDs for chloroethane, acetone, acrolein, vinyl acetate, and pentachloroethane were >30.0%. The quantitation limits for these compounds are qualified as estimated, UJ. No positive results

were observed for chloroethane, acetone, acrolein, vinyl acetate, and pentachloroethane. Thus, no further action was taken.

The mean RRF for acrolein, iso-butyl alcohol, and vinyl acetate was <0.05. No positive results were observed for these compounds. In accordance with the *Functional Guidelines* the non-detect data for these three compounds are unusable and, thus, qualified as"R".

2.3.2 Initial Calibration of 11/24/93 00:05 Matrix (water)

The %RSDs for chloroethane, acetone, 2-butanone, 2-hexanone, 1,1,2,2-tetrachloroethane, methacrylonitrile, acrolein, trichlorofluoromethane, iso-butyl alcohol, acetonitrile, 1,4-dioxane, trans-1,4-dichloro-2-butene, and pentachloroethane were >30.0%. The quantitation limits for these compounds are qualified as estimated, UJ. No positive results were observed for these compounds. Thus, no further action was taken.

The mean RRF for acrolein, iso-butyl alcohol, and 1,4-dioxane was <0.05. No positive results were observed for these compounds. In accordance with the *Functional Guidelines* the non-detect data for these three compounds for all samples were rejected, and thus qualified as "R".

2.4 CONTINUING CALIBRATION

Continuing calibration data are evaluated in four sections identified by the date of the continuing calibration. The percent differences (%D) and mean RRFs satisfied acceptance criteria for all compounds except as noted below.

2.4.1 Continuing Calibration of 11/23/93 09:33

The %D was >25% for iso-butyl alcohol. In addition, the continuing calibration RRF for iso-butyl alcohol, 1,4-dioxane,

and acrolein are <0.05. In accordance with the *Functional Guidelines* results for these compounds from samples S06, S10, S11, and S12 are unusable, and therefore qualified as R.

2.4.2 Continuing Calibration of 11/26/93 11:32

The %Ds were >25% for carbon disulfide, 2-butanone, 4-methyl-2-pentanone, 2-hexanone, methacrylonitrile, acrolein, acrylonitrile, propionitrile, iso-butyl alcohol, acetonitrile, 1,4-dioxane, methyl methacrylate, 1,2-dibromo-3-chloropropane, and pentachloroethane. Quantitation limits for these compounds are qualified as estimated. In addition, the continuing calibration RRF for acrolein, propionitrile, iso-butyl alcohol, and 1,4-dioxane are <0.05. In accordance with the *Functional Guidelines*, results for these compounds from samples S13, S14, S15, S16, S17, S08, S09 and S11 RE are unusable, and therefore qualified as R.

2.4.3 Continuing Calibration of 11/29/93 10:42

The %Ds were >25% for 2-butanone, 2-hexanone, propionitrile, acrolein, iso-butyl alcohol, acetonitrile, vinyl acetate, 1,4-dioxane, 1,2-dibromo-3-chloropropane, trans-1,4-dichloro-2-butene, and pentachloroethane. Quantitation limits for these compounds are qualified as estimated. In addition, the continuing calibration RRF for acrolein, propionitrile, iso-butyl alcohol, 1,4-dioxane, and trans-1,4-dichloro-2-butene are <0.05. In accordance with the *Functional Guidelines*, results for these compounds from samples S18, S15 DL, S16 DL, S17 DL, and S18 DL are unusable, and therefore qualified as R.

2.4.4 Continuing Calibration of 11/30/93 9:49

The %Ds were >25% for chloroethane, 4-methyl-2-pentanone, methacrylonitrile, acrolein, propionitrile, chloroprene, acetonitrile, 1,4-dioxane, dibromomethane, and trans-1,4-

dichloro-2-butene. Quantitation limits for these compounds are qualified as estimated. In addition, the continuing calibration RRF for acrolein, propionitrile, iso-butyl alcohol, 1,4-dioxane, and trans-1,4-dichloro-2-butene are <0.05. In accordance with the *Functional Guidelines*, results for these compounds from sample S14 DL are unusable, and therefore qualified as R.

2.5 BLANKS

2.5.1 Method Blanks

The six method blanks associated with the samples did not contain measurable quantities of Appendix IX volatile organic compounds. Therefore, no qualification of volatile organic compound data for the investigative samples was performed based on method blanks.

2.5.2 Field Blanks

The two field blanks associated with the samples did not contain measurable quantities of Appendix IX volatile organic compounds. Therefore, no qualification of volatile organic compound data for the investigative samples was performed based on the field blank.

2.5.3 Trip Blanks

The three trip blanks which accompanied the samples from Los Alamos to the laboratory did not contain measurable quantities of Appendix IX volatile organic compounds. Therefore, no qualification of volatile organic compound data for the investigative samples was performed based on the trip blanks.

2.6 SURROGATE SPIKES

All three surrogate spike recoveries from the samples were within the acceptance criteria with the exception 1,2-dichloroethane-d4 in samples S11, S11 RE, and S17. Since the percent surrogate

recovery for each sample was greater than the upper acceptance limit, results for non-detected compounds were not qualified. Positive results were qualified as estimated, J.

2.7 MATRIX SPIKES/MATRIX SPIKE DUPLICATES

All matrix spike percent recoveries were within the acceptance criteria. All relative percent differences for matrix spike/matrix spike duplicate pairs were within the acceptance criteria, with the exception of 1,1-dichloroethene of sample S14 DL. No action was taken to the non-detect result for this sample.

2.8 FIELD DUPLICATES

Two blind field duplicate samples were submitted to the laboratory (S10 duplicate of S9, and S17 duplicate of S16). Precision as determined from these field duplicate sample analyses was acceptable. However, acetone detected in samples S16 (91 ug/kg) and S17 (Duplicate of S16, 150 ug/kg) was qualified as estimated, J.

2.9 INTERNAL STANDARDS

One internal standard area count (1,4-difluorobenzene) was below the lower acceptable limit during the analyses of samples S14 MS, S14 MSD, and S14 DL. Positive results using 1,4-difluorobenzene were qualified as estimated, J. Non-detected compounds were qualified as UJ. The retention times of all internal standards were within the acceptable limits.

2.10 OVERALL ASSESSMENT OF DATA -- VOLATILE ORGANIC COMPOUNDS

All volatile organic compound data are acceptable for use except for the results for acrolein, propionitrile, iso-butyl alcohol, 1,4-dioxane, and trans-1,4-dichloro-2-butene which are qualified

as unusable, R (see section 3). Quantitation limits for several compounds are qualified as estimated as noted in sections 1,3, and 4.

3.0 SEMIVOLATILE ORGANIC COMPOUNDS

Eighteen soil samples and two aqueous field blanks were collected by Metcalf & Eddy, Inc. (M&E) and submitted to Accutest, Inc. for analysis of the semivolatile organic compounds in Appendix IX, 40 CFR Part 264.

3.1. TECHNICAL HOLDING TIMES

All samples were extracted within the specified holding times of 14 days for soils and seven days for waters. All sample extracts were analyzed within the specified holding time of 40 days from extraction.

3.2 GC/MS INSTRUMENT PERFORMANCE CHECK

The instrument performance check solution, decafluorotriphenylphosphine (DFTPP) was analyzed at the beginning of each 12-hour period during which samples or standards were analyzed. The DFTPP ion abundances monitored as part of the instrument performance check each day satisfied the ion abundance acceptance criteria.

3.3 INITIAL CALIBRATION

The initial calibration was performed on 12/12/93 and included standards analyzed at five different concentrations from 10 to 80 ppb. All relative response factors (RRF) and average RRFs were evaluated to determine if they were greater than or equal to 0.05. Linearity was acceptable if the percent relative standard deviation (%RSD) of the RRFs for the five concentrations of each standard analyzed was less than or equal to 30%. Linearity and

response were acceptable for all compounds except those listed below.

Compound	Unacceptable RRF or %RSD
P-Phenylenediamine	RRF (80 ppb) = 0.034
P-Phenylenediamine	%RSD = 45.7
4-Nitroquinoline-1-oxide	RRF (10 ppb) = 0.040
4-Nitroquinoline-1-oxide	RRF (40 ppb) = 0.046
4-Nitroquinoline-1-oxide	RRF (60 ppb) = 0.034
4-Nitroquinoline-1-oxide	RRF (80 ppb) = 0.025
4-Nitroquinoline-1-oxide	RRF (mean) = 0.039
SYM-Trinitrobenzene	RRF (10 ppb) = 0.046
Aramite	RRF (25 ppb) = 0.043
Aramite	RRF (40 ppb) = 0.038
Aramite	RRF (60 ppb) = 0.032
Aramite	RRF (80 ppb) = 0.027
Aramite	RRF (mean) = 0.038
3,3'-Dimethylbenzidine	RRF (25 ppb) = 0.037
3,3'-Dimethylbenzidine	RRF (40 ppb) = 0.038
3,3'-Dimethylbenzidine	RRF (60 ppb) = 0.039
3,3'-Dimethylbenzidine	RRF (80 ppb) = 0.039
3,3'-Dimethylbenzidine	RRF (mean) = 0.045
3,3'-Dimethylbenzidine	%RSD = 75.7
Hexachlorophene	RRF (25 ppb) = 0.001
Hexachlorophene	RRF (40 ppb) = 0.002
Hexachlorophene	RRF (60 ppb) = 0.002
Hexachlorophene	RRF (80 ppb) = 0.002
Hexachlorophene	RRF (mean) = 0.002
Dimethoate	%RSD = 31.5
Methabyrilene	RRF (60 ppb) = 0.049
Methabyrilene	RRF (80 ppb) = 0.039
Methabyrilene	%RSD = 84.2

Analytical results are qualified as unusable "R" in all samples for the following compounds due to their unacceptably low RRFs: 4-Nitroquinoline-1-oxide; Aramite; Hexachlorophene. Data for SYM-Trinitrobenzene have not been qualified because the mean RRF is greater than 0.05, only one RRF is less than 0.05, and it is only slightly less (0.046). Also, the %RSD is less than 30%.

The %RSDs were recalculated for three compounds whose 10 ppb through 60 ppb standards had RRFs equal to or greater than 0.05. %RSDs of the RRFs for these four-point calibration curves were still greater than 30% and all quantitation limits for P-Phenylenediamine, 3,3'-Dimethylbenzidine, and Methabyrilene are qualified as estimated "UJ" in all samples. The %RSD recalculated for the four RRFs for the 10 ppb through 60 ppb standards for Dimethoate is 22.5%. No Dimethoate data have been qualified.

3.4. CONTINUING CALIBRATION

Continuing calibrations (CC) were performed on 12/12/93 at 23:13, 12/13/93 at 12:32, 12/14/93 at 2:04, and 12/14/93 at 13:51. RRFs for the 25 ppb standard for each compound were evaluated to determine if they were greater than or equal to 0.05 and if they had a percent difference (%D) relative to the mean RRF of the initial calibration of less than 25%. Results were acceptable except for those compounds listed below. The samples affected are also listed with the appropriate data qualifiers.

DATE AND TIME OF CC: 12/12/93: 23:13

SAMPLES QUALIFIED: S01, S02, S03, S04, S05, SO3MS, S03MSD

Compound	**Unacceptable RRF or %D**	**Data Qualifier**
Hexachlorophene	RRF = 0.001	R
4-Nitroquinoline-1-oxide	RRF = 0.033	R
Aramite	RRF = 0.043	R

3,3'-Dimethylbenzidine	RRF = 0.037	R

DATE AND TIME OF CC: 12/13/93: 12:32
SAMPLES QUALIFIED: S06, S07, S08, S09, S10, S11

Compound	**Unacceptable RRF or %D**	**Data Qualifier**
4-Nitroquinoline-1-oxide	RRF = 0.045	R
Aramite	RRF = 0.047	R
3,3'-Dimethylbenzidine	RRF = 0.040	R
Hexachlorophene	RRF = 0.002	R
P-Phenylenediamine	%D = 36.4	UJ
Methabyrilene	%D = 55.3	UJ

DATE AND TIME OF CC: 12/14/93: 2:04
SAMPLES QUALIFIED: S13, S14, S15, S16, S17

Compound	**Unacceptable RRF or %D**	**Data Qualifier**
4-Nitroquinoline-1-oxide	RRF = 0.031	R
Aramite	RRF = 0.047	R
3,3'-Dimethylbenzidine	RRF = 0.039	R
Hexachlorophene	RRF = 0	R
Methabyrilene	%D = 34.5	UJ

DATE AND TIME OF CC: 12/14/93: 13:51
SAMPLES QUALIFIED: S12, S18

Compound	**Unacceptable RRF or %D**	**Data Qualifier**
4-Nitroquinoline-1-oxide	RRF = 0.047	R
Aramite	RRF = 0.045	R
3,3'-Dimethylbenzidine	RRF = 0.038	R
Hexachlorophene	RRF = 0.002	R
P-Phenylenediamine	%D = 72.8	UJ
Dimethoate	%D = 30.1	UJ
Methabyrilene	%D = 67.0	UJ

3.5 BLANKS

Laboratory method blank and field blank results were evaluated. A method blank was extracted with each group of samples extracted. A gel permeation chromatography blank was also prepared, extracted, and analyzed. No target compound was detected in any blank except for 1 ug/L bis (2-ethylhexyl) phthalate in one field blank.

The *Functional Guidelines* requires that sample data be qualified if the analyte in question is greater than the Contract Required Quantitation Limit (CRQL) and less than five times (ten times for common laboratory blank contaminants such as phthalates) its highest concentration in any blank associated with that sample. The analyte concentration is then reported with a "U" qualifier to indicate that it was not detected at the reported concentration. No sample data required qualification for target compounds on the basis of evaluation of positive blank contamination. Blanks did contain small estimated concentrations of tentatively identified compounds (TICs). Evaluation of TIC blank contamination is discussed in Section 3.12.

3.6 SURROGATE SPIKES

All surrogate spike recoveries were within the acceptance limits in all samples.

3.7 MATRIX SPIKE/MATRIX SPIKE DUPLICATE

Sample S03 was selected for matrix spike (MS)/matrix spike duplicate (MSD) analyses. MS and MSD recoveries were within the acceptance limits for all matrix spike compounds in all samples. The relative percent differences (RPDs) of matrix spike compounds were also within the acceptance limits for all compounds in all samples.

3.8 FIELD DUPLICATES

Samples S10 and S17 were field duplicates for S09 and S16, respectively. Analytical results were identical for all compounds in both pairs of field duplicates except for phenanthrene, which was not detected in S10, but was reported in S09 at an estimated concentration of 41J ug/kg, well below the CRQL of 330 ug/kg. No data were qualified on the basis of the field duplicate results, since the phenanthrene result reported for S09 has already been qualified.

3.9 INTERNAL STANDARDS

Internal standard area counts and retention times were acceptable for all samples and for all blanks, except for perylene-d12, which was not detected at all in the GPC blank and in one method blank. No sample data were affected or required qualification.

3.10 TARGET COMPOUND IDENTIFICATION

Target compounds were identified correctly according to the identification acceptance criteria and were detected at the correct retention times. All major peaks on the chromatograms were identified, either as target compounds, internal standards, surrogate compounds, or TICs.

3.11 COMPOUND QUANTITATION AND REPORTED CRQLs

No errors were found in sample quantitation. CRQLs were adjusted to reflect initial sample weights and percent moisture. No sample required dilution.

3.12 TENTATIVELY IDENTIFIED COMPOUNDS

For each sample, the laboratory performed a mass spectral search of the National Institute of Standards and Technology (NIST) mass spectral library. For each peak searched, the TIC was reported as the compound with the best match according to the results of the computer library search. Peaks found both in the method blanks and the associated samples were qualified by the laboratory with a "B". Matching was done on the basis of retention time only in these cases, not on the basis of the tentative identification of the compound.

TIC review included a comparison of the estimated concentrations of TICs in samples and in the associated blanks. According to the *Functional Guidelines*, TICs present at concentrations not sufficiently greater than their concentrations in the method blanks should not be reported. TIC concentrations reported by the laboratory with the "B" qualifier were never present at estimated concentrations greater than five times their estimated concentrations in the associated blanks. Therefore, all TICs in all samples that were qualified with a "B" by the laboratory should not be reported.

The remainder of the TICs reported for this group of samples consists of alkanes or compounds with poor matches with the NIST library spectra or compounds with no match at all with the library spectra, in which case the laboratory has identified the TIC only as an unknown compound. Because the individual alkane identifications for the various TICs are not accurate, TICs for each sample have been reviewed and reidentified in a more general way as either total unknowns or total alkanes. Total estimated concentrations are reported for each category for each sample as follows.

Sample	Total Unknowns, estimated ug/kg	Total Hydrocarbons, estimated ug/kg
S01	4740	1600
S02	1350	380
S03	480	0
S04	1000	1300
S05	1100	960
S06	2400	3600
S07	1200	2000
S08	610	0
S09	1300	2200
S10	420	290
S11	880	1600
S12	410	0
S13	0	0
S14	280	290
S15	120	610
S16	260	0
S17	260	380
S18	940	280

3.13 OVERALL ASSESSMENT OF DATA

Data for all TCL compounds in all samples are usable as reported by the laboratory with no further qualifications. Data for all non-TCL compounds in all samples are usable as reported by the laboratory with the exceptions noted above in Sections 3.3 and 3.4. Data for four compounds with very poor responses are qualified as unusable in all samples: Aramite; 4-Nitroquinoline-1-oxide; 3,3'-Dimethylbenzidine; and Hexachlorophene. Data for TICs have been regrouped as total unknown compounds or total alkanes due to the uncertainty of the identifications, as discussed in Section 3.12.

4.0 PESTICIDES/PCBs

Sixteen soil samples, plus two blind field duplicate samples and two field blanks were collected by Metcalf & Edy, Inc. (M&E) and submitted to Accutest, Inc. for analysis of pesticides/PCBs by the *Contract Laboratory Program Statement of Work for Organic Analysis*, OLMO1.8, August 1991.

4.1 TECHNICAL HOLDING TIME

All soil samples were extracted within the recommended fourteen day holding time, and were analyzed within 40 days after extraction, with the exception of the reanalyses of samples S16 RE, S17 RE, and S18 RE. No positive results were detected in the original analyses or in these reanalyses. The quantitation limits for these three samples are qualified as UJ. The water samples were extracted within seven days of collection and analyzed within fourty days.

4.2 INSTRUMENT PERFORMANCE

Instrument performance was acceptable. The DDT retention time was greater than 12 minutes. All standards were within the established retention time windows. The percent breakdown for 4,4'-DDT and endrin did not exceed twenty percent.

4.3 CALIBRATION

Both the initial and continuing calibrations were satisfactory. The percent Relative Standard Deviation (RSD) of calibration factors for aldrin, endrin, and DDT, did not exceed ten percent. All standards were analyzed within the 72-hour sequence. The Continuing Calibration Factor for each standard was within fifteen percent of the standard at the beginning of the analytical sequence.

4.4. BLANKS

No pesticides or PCBs were detected in the method blanks or field blanks. Therefore, no qualification of data was required due to blanks.

4.5. SURROGATE RECOVERY

The surrogate spike percent recoveries for all samples including method blanks were within acceptance criteria with the following exceptions. The decachlorobiphenyl (DCB) percent recovery for B01 was low on the primary column and within acceptance criteria on the secondary column. No action was taken to the data. The tetrachloro-m-xylene (TCX) recoveries for samples S13, S14, and S15 were low. The quantitation limits for these samples were qualified as estimated, UJ. Both the TCX and DCB percent recoveries for samples S16, S17, and S18 were near zero. These data are unusable and thus, qualified as R. Samples, S16, S17, and S18 were reextracted and reanalyzed outside of the holding time. The surrogate percent recoveries were within criteria in the reanalyzed samples.

4.6 MATRIX SPIKE/MATRIX SPIKE DUPLICATE

Precision and accuracy as determined by MS/MSD analyses were acceptable, with the exception of gamma-BHC (lindane) in sample S03. The non-detect result for lindane in sample S03 is qualified as estimated, UJ. Six RPDs were outside of QC limits. However, no further action was taken to the data based on MS/MSD analyses.

4.7 FIELD DUPLICATES

Two blind field duplicate samples were submitted to the laboratory (S10 duplicate of S9, and S17 duplicate of S16).

Precision as determined from these field duplicate sample analyses was acceptable.

4.8 COMPOUND IDENTIFICATION AND QUANTITATION LIMITS

Compound identification was acceptable. No positive results were observed in the investigative samples. Compound quantitation and Contract Required Quantitation Limits (CRQLs) are accurate, and have been adjusted to reflect dry weight factors. Dilutions were not necessary, since positive results were not observed.

4.9 OVERALL ASSESSMENT OF DATA

All Pesticide/PCB data are acceptable for use with the exception of the initial analyses of samples S16, S17, and S18. These data are unusable due to near zero percent surrogate recoveries. Thus, these data are qualified as R. The three samples (S16, S17, and S18) were reanalyzed outside of holding time. Upon reanalysis the surrogate percent recoveries were within criteria, however the quantitation limits are qualified as estimated, UJ, due to the missed holding time.

5.0 INORGANIC ANALYSES

Eighteen soils and two aqueous field blanks were collected by Metcalf & Eddy, Inc. (M&E) and submitted to Accutest Laboratory for analysis of arsenic, barium, cadmium, chromium, mercury, lead, selenium, silver, and total cyanide.

5.1 TECHNICAL HOLDING TIMES

All sample analyses for metals were performed within the specified holding times of 180 days for metals other than mercury and 28 days for mercury. All samples analyzed for cyanide were

distilled and analyzed within the specified holding time of fourteen days.

5.2 CALIBRATION

The cyanide calibration curves consisted of a blank and six standards. The correlation coefficients were >0.995 and therefore acceptable. Mid-range standards were distilled and analyzed and had acceptable recoveries within 10% of the non-distilled standards. The initial calibration verification (ICV) and continuing calibration verification (CCV) standards were analyzed at the accepted frequency. The ICVs and CCVs had acceptable recoveries of 90-110%.

The mercury calibration curves consisted of a blank and four standards. The correlation coefficients were >0.995, and therefore acceptable. The ICVs and CCVs were analyzed at the required frequency and had acceptable recoveries of 90-110%.

Three metals - arsenic, lead, and selenium - were analyzed by graphite furnace atomic absorption (GFAA). The calibration curves consisted of a blank and five standards, the lowest being at the Contract Required Detection Limit (CRDL) for each analyte. The correlation coefficients for all curves were ·0.995, and were therefore acceptable. The ICV and CCV standards were analyzed at the required frequency and had acceptable recoveries of 90-110% for all analytes. No sample data for arsenic, selenium, or lead were qualified due to calibration evaluation.

The remainder of the metals - barium, cadmium, chromium, and silver - were analyzed by inductively coupled plasma emission spectroscopy (ICP). The analytical curve was established with a blank and one standard, as required. The ICV and CCV standards were analyzed at the recommended frequency and all had acceptable recoveries of 90-110% for all analytes.

The *Functional Guidelines* do not provide specific acceptance criteria for a low concentration standard (2xCRDL standard for ICP analyses, CRDL standard for GFAA analyses) recovery. M&E has applied the following USEPA Region I review criteria to the CRDL and 2XCRDL standard results. If the CRDL or the 2xCRDL standard is not within ±20% of the true value, positive results less than three times the CRDL and undetected analytes are reported as estimated (J and UJ, respectively). All CRDL and 2XCRDL standards were within 20% of their true values for all analytes. No data were qualified on the basis of this review item.

5.3 BLANKS

Initial calibration blank (ICB), continuing calibration blank (CCB), aqueous preparation blank (PBW), soil preparation blank (PBS), and field blank (FB) results were evaluated. All blank results were less than the absolute value of the instrument detection limit (IDL) except for the following:

Blank	**Analyte**	**Concentration**	
ICB	Barium	- 24.9	ug/L
CCB1	Barium	- 26.0	ug/L
CCB2	Barium	- 26.3	ug/L
CCB3	Barium	- 26.1	ug/L
CCB4	Barium	- 25.3	ug/L
CCB9	Barium	- 26.5	ug/L
CCB10	Barium	- 26.6	ug/L
PBW	Barium	- 22.0	ug/L
PBS	Barium	- 5.196	mg/kg
ICB	Lead	- 2.0	ug/L
CCB1 (12/2/93)	Lead	- 2.1	ug/L
CCB2 (12/2/93)	Lead	- 1.9	ug/L
CCB3 (12/2/93)	Lead	- 1.9	ug/L
CCB1 (12/14/93)	Lead	- 1.9	ug/L
CCB2 (12/14/93)	Lead	- 1.9	ug/L
CCB3 (12/14/93)	Lead	- 2.2	ug/L
PBS	Lead	- 0.342	mg/kg
FB (B04)	Lead	1.5	ug/L
CCB3	Silver	- 7.7	ug/L
CCB4	Silver	- 6.8	ug/L
CCB10	Silver	- 7.7	ug/L
PBW	Silver	6.99	ug/l

The *Functional Guidelines* requires that sample data be qualified if the analyte in question is greater than the IDL and less than five times its highest concentration in any blank associated with that sample. The analyte concentration is then reported with a "U" qualifier to indicate that it was not detected at the reported concentration. No sample data required qualification on the basis of evaluation of positive blank contamination.

The *Functional Guidelines* also requires that an evaluation be made of all sample data associated with blank results greater than the negative IDL, as the associated sample data may be negatively biased. However, the *Functional Guidelines* does not provide specific action levels and resulting qualification recommendations for such cases. M&E follows the USEPA Region I guidance, which suggests qualification of sample results that are less than three times the IDL and are associated with negative blank results whose absolute value is greater than two times the IDL. No sample data required qualification on the basis of evaluation of negative blank results. All samples analyzed in association with negative ICBs or CCBs were reanalyzed after CCBs were once again in control.

5.4 ICP INTERFERENCE CHECK SAMPLE

All ICP Interference Check Sample (ICS) results satisfied the percent recovery acceptance criteria of ±20%. Analytical results for aluminum, calcium, iron, and magnesium were not required to be reported as part of this investigation. However, their concentrations in the samples could be determined from the raw ICP data. The concentrations of these interferents in the investigative samples were in all cases less than their concentrations in the ICSs. No data were qualified or rejected on the basis of ICP ICS performance.

5.5. LABORATORY CONTROL SAMPLE

Laboratory Control Sample (LCS) results for all analytes satisfied the percent recovery acceptance criterion of 80-120% for the aqueous LCS and the EPA-established control limits for the solid LCS. No data were qualified or rejected on the basis of LCS results.

5.6 DUPLICATE SAMPLE ANALYSIS

Duplicate sample results for all analytes satisfied the applicable acceptance criterion of relative percent difference (RPD) less then 35% for soils or difference less than two times the CRDL for those results in soils less than five times the CRDL. No sample result required qualification on the basis of the duplicate analyses results.

5.7. MATRIX SPIKE SAMPLE ANALYSIS

Sample S03 was selected for matrix spike/matrix spike duplicate analyses. Matrix spike (MS) recoveries were within the 75-125% range of acceptable recoveries for all analytes.

5.8 FURNACE ATOMIC ABSORPTION QC

Precision for all GFAA analyses was acceptable since all duplicate injections were within the ±20% relative standard deviation (RSD) acceptance criterion for analyte concentrations greater than the CRDL. No data were qualified on the basis of duplicate injection precision.

Post-digestion spike (PDS) recoveries were acceptable and between 85-115% for all lead analyses. Initial PDS recoveries were slightly high for arsenic in one sample and selenium in three samples. These samples were reanalyzed and the PDS recoveries

were within the control limits. No data required qualification on the basis of PDS recovery results.

5.9 ICP SERIAL DILUTION

Serial dilution results had a percent difference of <10% for all metals. No data required qualification on the basis of the serial dilution results.

5.10 SAMPLE RESULT VERIFICATION

All ICP data were within the linear range; all other analytical results were within the calibrated ranges of their respective analyses. No transcription or calculation errors were found.

5.11 FIELD DUPLICATES

Samples S10 and S17 were field duplicates for S09 and S16, respectively. The precision for the field duplicate analyses exceeded the applicable acceptance criteria of RPD <35% for soil results greater than five times the CRDL or of two times the CRDL for soil results less than five times the CRDL for all analytes. No data required qualification on the basis of field duplicate results.

5.12 OVERALL ASSESSMENT OF DATA

All sample results in this SDG are usable as reported by the laboratory on the Form I's. No qualifications were required.