



U.S. Army  
Environmental  
Center

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# *RI/FS SAMPLING AND ANALYSIS PLAN*

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**FORT McCLELLAN, ALABAMA  
TASK ORDER 005  
Contract Number DAAA15-91-D-0017**

*Final Report*

*FINAL*

**Volume IV: QA/QC APPENDICES**

*Prepared for:*

**U.S. Army Environmental Center  
Installation Restoration Division  
Aberdeen Proving Ground, Maryland 21010-5401**

**April 6, 1994**

**SAMPLING AND ANALYSIS PLAN  
FOR  
FORT McCLELLAN RI/FS  
ANNISTON, ALABAMA**

*Volume IV: Quality Assurance Appendices*

**FINAL**

**Submitted to:**

**U.S. Army Environmental Center  
Installation Restoration Division  
SFIM-AEC-IRB  
Aberdeen Proving Ground, Maryland 21010-5401**

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**USATHAMA Contract DAAA15-91-D-0017  
Task Order 5**

**SAIC Project No. 01-0827-03-6520-007**

**April 6, 1994**

**APPENDIX C**  
**ANALYTICAL METHODS**  
**FOR SOIL AND WATER ANALYSES**

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**PART I**

**NONPERFORMANCE DEMONSTRATED METHODS  
FOR SOIL AND WATER ANALYSES**

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### Appendix C Part I. Nonperformance Demonstrated Methods for Water Analysis

Method No.	Analyte Code	Analyte Name	Reporting Limit ( $\mu\text{g/L}$ )
SW7041*	Sb	Antimony	3.0
SW6010	Cd	Cadmium	4.0
SW7841	Tl	Thallium	1.0
SW8010	$\text{C}_2\text{H}_3\text{Cl}$	Vinyl Chloride	1.0
SW8310	BAANTR	Benzo(a)anthracene	0.1
	BAPYR	Benzo(a)pyrene	0.2
	BBFANT	Benzo(b)fluoranthene	0.2
	BKFANT	Benzo(k)fluoranthene	0.2
	CHRY	Chrysene	0.2
	DBAHA	Dibenzo(a,h)anthracene	0.3
	ICDPYR	Indeno(1,2,3-cd)pyrene	0.4
SW8040	PCP	Pentachlorophenol	1

\* SW7041 also will be used for soil samples requiring antimony samples.

**PART II**

**USAEC PERFORMANCE DEMONSTRATED METHODS  
FOR SOIL AND WATER ANALYSES**

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**Appendix C. USAEC Performance Demonstrated Methods for Water Analyses  
DataChem Laboratories, Ft. McClellan, Anniston, Alabama**

Method No.	Analyte Code	Analyte Name	Certified Reporting Limit	Upper Limit
<b>METALS/WATER/GFAA (<math>\mu\text{g/L}</math>)</b>				
AX8	AS	Arsenic	2.350	121.00
SD18	PB	Lead	4.470	200.0
SD25	SE	Selenium	2.530	200.0
<b>METALS/WATER/CVAA (<math>\mu\text{g/L}</math>)</b>				
CC8	HG	Mercury	0.100	2.00
<b>METALS/WATER/ICP (<math>\mu\text{g/L}</math>)</b>				
SS12	AG	Silver	10.00	2000.0
SS12	AL	Aluminum	112.00	125000.0
SS12	B	Boron	230.00	10000.0
SS12	BA	Barium	2.82	12000.0
SS12	BE	Beryllium	1.12	1000.0
SS12	CA	Calcium	105.00	200000.0
SS12	CO	Cobalt	25.00	10000.0
SS12	CR	Chromium	16.80	1000.0
SS12	CU	Copper	18.80	10000.0
SS12	FE	Iron	77.50	500000.0
SS12	K	Potassium	1240.00	250000.0
SS12	MG	Magnesium	135.00	250000.0
SS12	MN	Manganese	9.67	10000.0
SS12	MO	Molybdenum	52.70	10000.0
SS12	NA	Sodium	279.00	50000.0
SS12	NI	Nickel	32.10	20000.0
SS12	SN	Tin	59.90	20000.0
SS12	TE	Tellurium	118.00	20000.0
SS12	V	Vanadium	27.60	10000.0
SS12	ZN	Zinc	18.00	10000.0
<b>VOLATILES/WATER/GCMS (<math>\mu\text{g/L}</math>)</b>				
UM21	111TCE	1,1,1-Trichloroethane	1.00	100.0
UM21	112TCE	1,1,2-Trichloroethane	1.00	100.0
UM21	11DCE	1,1-Dichloroethene	1.00	150.0
UM21	11DCLE	1,1-Dichloroethane	1.00	150.0
UM21	12DCD4	1,2-Dichloroethane-d <sub>4</sub> -S	1.00	150.0
UM21	12DCE	1,2-Dichloroethene	5.00	150.0
UM21	12DCLE	1,2-Dichloroethane	1.00	150.0
UM21	12DCLP	1,2-Dichloropropane	1.00	150.0
UM21	13DCLB	1,3-Dichlorobenzene	1.00	150.0
UM21	13DCP	1,3-Dichloropropane	4.80	100.0
UM21	13DMB	1,3-Dimethylbenzene	1.00	150.0

**Appendix C. USAEC Performance Demonstrated Methods for Water Analyses  
DataChem Laboratories, Ft. McClellan, Anniston, Alabama (continued)**

Method No.	Analyte Code	Analyte Name	Certified Reporting Limit	Upper Limit
UM21	2CLEVE	2-Chloroethylvinyl Ether	3.50	100.0
UM21	ACET	Acetone	8.00	100.0
UM21	ACRYLO	Acrylonitrile	8.40	150.0
UM21	BRDCLM	Bromodichloromethane	1.00	100.0
UM21	C2H5CL	Chloroethane	8.00	150.0
UM21	C6H6	Benzene	1.00	150.0
UM21	CCL3F	Trichlorofluoromethane	1.00	100.0
UM21	CCL4	Carbon Tetrachloride	1.00	100.0
UM21	CDCL2	Methylene Chloride-d <sub>2</sub> -S	9.70	150.0
UM21	CH2CL2	Methylene Chloride	1.00	150.0
UM21	CH3BR	Bromomethane	14.00	150.0
UM21	CH3CL	Chloromethane	1.20	100.0
UM21	CHBR3	Bromoform	11.00	100.0
UM21	CHCL3	Chloroform	1.00	150.0
UM21	CLC6H5	Chlorobenzene	1.00	150.0
UM21	DBRCLM	Dibromochloromethane	1.00	100.0
UM21	DCLB	1,2-Dichlorobenzene	2.00	300.0
UM21	ETBD10	Ethylbenzene-d <sub>10</sub> -S	1.00	150.0
UM21	ETC6H5	Ethylbenzene	1.00	150.0
UM21	MEC6D8	Toluene-d <sub>8</sub> -S	1.00	150.0
UM21	MEC6H5	Toluene	1.00	150.0
UM21	MEK	Methylethylketone	10.00	100.0
UM21	MIBK	Methylisobutylketone	1.40	100.0
UM21	TCLEA	1,1,2,2-Tetrachloroethane	1.50	150.0
UM21	TCLEE	Tetrachloroethene	1.00	150.0
UM21	TRCLE	Trichloroethene	1.00	150.0
UM21	XYLEN	1,2-Dimethylbenzene	2.00	300.0
<b>SEMIVOLATILES/WATER/GCMS (µg/L)</b>				
UM25	123TCB	1,2,3-Trichlorobenzene ✓	5.80	200.0
UM25	124TCB	1,2,4-Trichlorobenzene ✓	2.40	200.0
UM25	12DCLB	1,2-Dichlorobenzene ✓	1.20	100.0
UM25	12DPH	1,2-Diphenylhydrazine ✓	13.00	100.0
UM25	13DBD4	1,3-Dichlorobenzene-d <sub>4</sub> -S ✓	14.00	300.0
UM25	13DCLB	1,3-Dichlorobenzene ✓	3.40	100.0
UM25	14DCLB	1,4-Dichlorobenzene ✓	1.50	100.0
UM25	236TCP	2,3,6-Trichlorophenol ✓	1.70	100.0
UM25	245TCP	2,4,5-Trichlorophenol ✓	2.80	300.0
UM25	246TBP	2,4,6-Tribromophenol ✓	20.00	300.0
UM25	246TCP	2,4,6-Trichlorophenol ✓	3.60	300.0
UM25	24DCLP	2,4-Dichlorophenol ✓	8.40	300.0
UM25	24DMPN	2,4-Dimethylphenol ✓	4.40	300.0
UM25	24DNP	2,4-Dinitrophenol ✓	176.00	300.0

**Appendix C. USAEC Performance Demonstrated Methods for Water Analyses  
DataChem Laboratories, Ft. McClellan, Anniston, Alabama (continued)**

Method No.	Analyte Code	Analyte Name	Certified Reporting Limit	Upper Limit
UM25	24DNT	2,4-Dinitrotoluene ✓	5.80	100.0
UM25	26DNA	2,6-Dinitroaniline ✓	8.80	100.0
UM25	26DNT	2,6-Dinitrotoluene ✓	6.70	100.0
UM25	2CLP	2-Chlorophenol ✓	2.80	100.0
UM25	2CLPD4	2-Chlorophenol-d <sub>4</sub> -S ✓	47.00	200.0
UM25	2CNAP	2-Chloronaphthalene ✓	2.60	100.0
UM25	2FBP	2-Fluorobiphenyl ✓	17.00	300.0
UM25	2FP	2-Fluorophenol ✓	22.00	100.0
UM25	2MNAP	2-Methylnaphthalene ✓	1.30	300.0
UM25	2MP	2-Methyl Phenol ✓	3.60	200.0
UM25	2NP	2-Nitrophenol ✓	8.20	300.0
UM25	33DCBD	3,3'-Dichlorobenzidine ✓	5.00	300.0
UM25	35DNA	3,5-Dinitro-aniline ✓	21.00	200.0
UM25	3NANIL	3-Nitroaniline ✓	15.00	200.0
UM25	3NT	3-Nitrotoluene ✓	2.90	200.0
UM25	4BRPPE	4-Bromophenyl Phenyl Ether ✓	22.00	300.0
UM25	4CL3C	4-Chloro-3-methylphenol ✓	8.50	300.0
UM25	4CLPPE	4-Chlorophenyl Phenyl Ether ✓	23.00	300.0
UM25	4MP	4-Methyl Phenol ✓	2.80	200.0
UM25	4NP	4-Nitrophenol ✓	96.00	600.0
UM25	ANAPNE	Acenaphthene ✓	5.80	100.0
UM25	ANAPYL	Acenaphthylene ✓	5.10	200.0
UM25	ANTRC	Anthracene ✓	5.20	100.0
UM25	B2CEXM	bis(2-Chloroethoxy) Methane ✓	6.80	300.0
UM25	B2CIPE	bis(2-Chloroisopropyl) Ether ✓	5.00	300.0
UM25	B2CLEE	bis(2-Chloroethyl)ether ✓	0.68	100.0
UM25	B2EHP	bis(2-Ethylhexyl)phthalate ✓	7.70	200.0
UM25	BBZP	Butyl Benzyl Phthalate ✓	28.00	200.0
UM25	BGHIPY	Benzo(g,h,i)perylene ✓	15.00	100.0
UM25	BRMCIL	Bromacil ✓	2.90	100.0
UM25	BZALC	Benzyl Alcohol ✓	4.00	200.0
UM25	CL3P	Trichlorophenols ✓	2.80	300.0
UM25	CL6ET	Hexachloroethane ✓	8.30	100.0
UM25	CPMS	p-Chlorophenylmethyl Sulfide ✓	10.00	300.0
UM25	CPMSO	p-Chlorophenylmethyl Sulfoxide ✓	15.00	300.0
UM25	CPMSO2	p-Chlorophenylmethyl Sulfone ✓	5.30	300.0
UM25	DBCP	Dibromochloropropane ✓	12.00	300.0
UM25	DBZFUR	Dibenzofuran ✓	5.10	300.0
UM25	DCPD	Dicyclopentadiene ✓	5.50	300.0
UM25	DDVP	Vapona ✓	8.50	300.0
UM25	DEP	Diethyl Phthalate ✓	5.90	200.0
UM25	DEPD4	Diethyl Phthalate-d <sub>4</sub> -S ✓	8.70	300.0

**Appendix C. USAEC Performance Demonstrated Methods for Water Analyses  
DataChem Laboratories, Ft. McClellan, Anniston, Alabama (continued)**

Method No.	Analyte Code	Analyte Name	Certified Reporting Limit	Upper Limit
UM25	DIMP	Diisopropyl methylphosphonate	21.00	200.0
UM25	DITH	Dithiane	3.30	100.0
UM25	DMMP	Dimethyl methylphosphonate	130.00	200.0
UM25	DMP	Dimethyl Phthalate	2.20	200.0
UM25	DNBP	di-N-Butyl Phthalate	33.00	300.0
UM25	DNOP	di-N-Octyl Phthalate	1.50	100.0
UM25	DNOPD4	di-N-Octylphthalate-d <sub>4</sub>	13.00	300.0
UM25	FANT	Fluoranthene	24.00	300.0
UM25	FLRENE	Fluorene	9.20	100.0
UM25	HCBD	Hexachlorobutadiene	8.70	200.0
UM25	ISOPHR	Isophorone	2.40	200.0
UM25	MIREX	Mirex	24.00	200.0
UM25	MLTHN	Malathion	21.00	300.0
UM25	NAP	Naphthalene	0.50	100.0
UM25	NB	Nitrobenzene	3.70	200.0
UM25	NBD5	Nitrobenzene-d <sub>5</sub>	26.00	200.0
UM25	NNDMEA	N-Nitrosodimethylamine	9.70	100.0
UM25	NNDNPA	N-Nitroso-di-N-propylamine	6.80	100.0
UM25	NNDPA	N-Nitrosodiphenylamine	3.70	200.0
UM25	OXAT	1,4-Oxathiane	27.00	300.0
UM25	PHANTR	Phenanthrene	9.90	100.0
UM25	PHEND6	Phenol-d <sub>6</sub>	34.00	100.0
UM25	PHENOL	Phenol	2.20	300.0
UM25	PRTHN	Parathion	37.00	300.0
UM25	PYR	Pyrene	17.00	100.0
UM25	SUPONA	Supona	19.00	200.0
UM25	TRPD14	Terphenyl-d <sub>14</sub>	35.00	300.0
<b>PESTICIDES/WATER/GCEC (µg/L)</b>				
UH20	ABHC	alpha-BHC	0.0025	0.5000
UH20	AENSLF	Endosulfan I	0.0025	0.5000
UH20	ALDRN	Aldrin	0.0074	0.5000
UH20	BBHC	beta-BHC	0.0099	0.5000
UH20	BENSLF	Endosulfan II	0.0077	0.5000
UH20	CLDAN	Chlordane	0.0312	4.0000
UH20	DBHC	delta-BHC	0.0034	0.5000
UH20	DLDRN	Dieldrin	0.0074	0.5000
UH20	ENDRN	Endrin	0.0176	0.5000
UH20	ENDRNA	Endrin Aldehyde	0.0504	2.5000
UH20	HPCL	Heptachlor	0.0025	0.2500
UH20	HPCLE	Heptachlor Epoxide	0.0063	0.1840
UH20	ISODR	Isodrin	0.0025	0.5000
UH20	LIN	Lindane (gamma-BHC)	0.0025	0.5000
UH20	MEXCLR	Methoxychlor	0.0750	5.0000

**Appendix C. USAEC Performance Demonstrated Methods for Water Analyses  
DataChem Laboratories, Ft. McClellan, Anniston, Alabama (continued)**

Method No.	Analyte Code	Analyte Name	Certified Reporting Limit	Upper Limit
UH20	ENDRNA	Endrin Aldehyde	0.0504	2.5000
UH20	HPCL	Heptachlor	0.0025	0.2500
UH20	HPCLE	Heptachlor Epoxide	0.0063	0.1840
UH20	ISODR	Isodrin	0.0025	0.5000
UH20	LIN	Lindane (gamma-BHC)	0.0025	0.5000
UH20	MEXCLR	Methoxychlor	0.0750	5.0000
UH20	PCB016	PCB-1016	0.3850	10.0000
UH20	PCB260	PCB-1260	0.1760	10.0000
UH20	PPDDD	4,4'-DDD	0.0081	0.5720
UH20	PPDDE	4,4'-DDE	0.0039	0.5370
UH20	PPDDT	4,4'-DDT	0.0025	0.2500
UH20	TXPHEN	Toxaphene	1.6400	50.0000
<b>EXPLOSIVES/WATER/HPLC (<math>\mu\text{g/L}</math>)</b>				
UW25	135TNB	1,3,5-Trinitrobenzene	0.2100	20.00
UW25	13DNB	1,3-Dinitrobenzene	0.4580	20.00
UW25	246TNT	2,4,6-Trinitrotoluene	0.4260	40.00
UW25	24DNT	2,4-Dinitrotoluene	0.3970	20.00
UW25	26DNT	2,6-Dinitrotoluene	0.6000	40.00
UW25	HMX	Cyclotetramethylenetetranitramine	0.5330	16.00
UW25	NB	Nitrobenzene	0.6820	40.00
UW25	RDX	Hexahydro-1,3,5-trinitro-1,3,5-triazine	0.4160	40.00
UW25	TETRYL	N-methyl-N,2,4,6-tetranitroaniline	0.6310	40.00
<b>NG and PETN/WATER/HPLC (<math>\mu\text{g/L}</math>)</b>				
UW27	NG	Nitroglycerine	1.4900	100.0000
UW27	PETN	Pentaerythritol tetranitrate	2.0000	200.0000

**Appendix C. USAEC Performance Demonstrated Methods for Water Analyses  
Environmental Science and Engineering,  
Ft. McClellan, Anniston, Alabama**

Method No.	Analyte Code	Analyte Name	Certified Reporting Limit	Upper Limit
<b>ORGANOSULFURS/WATER/GCFP (<math>\mu\text{g/L}</math>)</b>				
UL04	BTZ	Benzothiazole	2.1100	42.2000
UL04	CPMS	<i>p</i> -Chlorophenylmethyl sulfide	1.2600	25.3000
UL04	CPMSO	<i>p</i> -Chlorophenylmethyl sulfoxide	4.2300	106.0000
UL04	CPMSO2	<i>p</i> -Chlorophenylmethyl sulfone	4.7200	106.0000
UL04	DITH	Dithiane	1.1100	22.2000
UL04	DMDS	Dimethyl disulfide	1.1400	22.8000
UL04	OXAT	1,4-Oxathiane	1.9800	39.5000
<b>ORGANICS/WATER/IC (<math>\mu\text{g/L}</math>)</b>				
UT03	CLC2A	Chloroacetic acid	25.0000	1500.0000
UT03	FC2A	Fluoroacetic acid	25.0000	1500.0000
UT03	IMPA	Isopropyl methylphosphonate	25.0000	1500.0000
UT03	MPA	Methylphosphonic acid	50.0000	1500.0000
<b>ORGANOSULFURS/WATER/HPLC (<math>\mu\text{g/L}</math>)</b>				
UW22	TDGCL	Thiodiglycol	48.8000	4880.0000
UW22	TDGCLA	Thiodiglycolic acid	52.7000	1780.0000
<b>ORGANOPHOSPHOR/WATER/GCFP (<math>\mu\text{g/L}</math>)</b>				
T8	DIMP	Diisopropyl methylphosphonate	10.5000	210.0000
T8	DMMP	Dimethyl methylphosphonate	15.2000	305.0000

**Appendix C. USAEC Performance Demonstrated Methods for Soil Analyses  
DataChem Laboratories, Ft. McClellan, Anniston, Alabama**

Method No.	Analyte Code	Analyte Name	Certified Reporting Limit	Upper Limit
<b>METALS/SOIL/CVAA (<math>\mu\text{g/g}</math>)</b>				
Y9	HG	Mercury	0.050	1.00
<b>METALS/SOIL/GFAA (<math>\mu\text{g/g}</math>)</b>				
B9	AS	Arsenic	2.500	50.00
JD20	SE	Selenium	0.4490	20.00
JD21	PB	Lead	0.4670	20.00
<b>METALS/SOIL/ICP (<math>\mu\text{g/g}</math>)</b>				
JS12	AG	Silver	0.803	200.0
JS12	AL	Aluminum	11.200	50000.0
JS12	B	Boron	6.640	1000.0
JS12	BA	Barium	3.290	1000.0
JS12	BE	Beryllium	0.427	1000.0
JS12	CA	Calcium	25.300	50000.0
JS12	CD	Cadmium	1.200	1250.0
JS12	CO	Cobalt	2.500	1000.0
JS12	CR	Chromium	1.040	1000.0
JS12	CU	Copper	2.840	1000.0
JS12	FE	Iron	6.660	50000.0
JS12	K	Potassium	131.000	50000.0
JS12	MG	Magnesium	10.100	50000.0
JS12	MN	Manganese	9.870	1000.0
JS12	MO	Molybdenum	14.300	1000.0
JS12	NA	Sodium	38.700	50000.0
JS12	NI	Nickel	2.740	2000.0
JS12	SN	Tin	7.430	2000.0
JS12	TE	Tellurium	14.900	2000.0
JS12	TL	Thallium	34.300	5000.0
JS12	V	Vanadium	1.410	1000.0
JS12	ZN	Zinc	2.340	1000.0
<b>VOLATILES/SOIL/GCMS (<math>\mu\text{g/g}</math>)</b>				
LM23	111TCE	1,1,1-Trichloroethane	0.200	10.00
LM23	112TCE	1,1,2-Trichloroethane	0.330	10.00
LM23	11DCE	1,1-Dichloroethene	0.270	10.00
LM23	11DCLE	1,1-Dichloroethane	0.490	10.00
LM23	12DCD4	1,2-Dichloroethane-d <sub>4</sub> -S	0.500	10.00
LM23	12DCE	1,2-Dichloroethene	0.320	10.00
LM23	12DCLE	1,2-Dichloroethane	0.320	10.00
LM23	12DCLP	1,2-Dichloropropane	0.530	10.00
LM23	13DCLB	1,3-Dichlorobenzene	0.140	10.00
LM23	13DCP	1,3-Dichloropropane	0.200	10.00
LM23	13DMB	1,3-Dimethylbenzene	0.230	10.00

**Appendix C. USAEC Performance Demonstrated Methods for Soil Analyses  
DataChem Laboratories, Ft. McClellan, Anniston, Alabama (continued)**

Method No.	Analyte Code	Analyte Name	Certified Reporting Limit	Upper Limit
LM23	2CLEVE	2-Chloroethylvinyl Ether	0.500	10.00
LM23	ACET	Acetone	3.300	10.00
LM23	ACRYLO	Acrylonitrile	2.000	10.00
LM23	BRDCLM	Bromodichloromethane	0.200	10.00
LM23	C2H3CL	Vinyl Chloride	1.800	10.00
LM23	C2H5CL	Chloroethane	0.640	10.00
LM23	C6H6	Benzene	0.100	10.00
LM23	CCL3F	Trichlorofluoromethane	0.230	10.00
LM23	CCL4	Carbon Tetrachloride	0.310	10.00
LM23	CDCL2	Methylene Chloride-d <sub>2</sub> -S	2.400	10.00
LM23	CH2CL2	Methylene Chloride	4.400	10.00
LM23	CH3BR	Bromomethane	0.260	10.00
LM23	CH3CL	Chloromethane	0.960	10.00
LM23	CHBR3	Bromoform	0.200	10.00
LM23	CHCL3	Chloroform	0.240	10.00
LM23	CLC6H5	Chlorobenzene	0.100	10.00
LM23	DBRCLM	Dibromochloromethane	0.250	10.00
LM23	DCLB	1,2-Dichlorobenzene	0.200	20.00
LM23	ETBD10	Ethylbenzene-d <sub>10</sub> -S	0.100	10.00
LM23	ETC6H5	Ethylbenzene	0.190	10.00
LM23	MEC6D8	Toluene-d <sub>8</sub> -S	0.100	10.00
LM23	MEC6H5	Toluene	0.100	10.00
LM23	MEK	Methylethylketone	4.300	10.00
LM23	MIBK	Methylisobutylketone	0.630	10.00
LM23	TCLEA	1,1,2,2-Tetrachloroethane	0.200	10.00
LM23	TCLEE	Tetrachloroethene	0.160	10.00
LM23	TRCLE	Trichloroethene	0.230	10.00
LM23	XYLEN	1,2-Dimethylbenzene	0.780	20.00
<b>SEMIVOLATILES/SOIL/GCMS (µg/g)</b>				
LM25	123TCB	1,2,3-Trichlorobenzene	0.0320	6.20
LM25	124TCB	1,2,4-Trichlorobenzene	0.2200	6.20
LM25	12DCLB	1,2-Dichlorobenzene	0.0420	12.00
LM25	12DPH	1,2-Diphenylhydrazine	0.5200	6.20
LM25	13DBD4	1,3-Dichlorobenzene-d <sub>4</sub> -S	0.0500	12.00
LM25	13DCLB	1,3-Dichlorobenzene	0.0420	12.00
LM25	14DCLB	1,4-Dichlorobenzene	0.0340	12.00
LM25	236TCP	2,3,6-Trichlorophenol	0.6200	12.00
LM25	245TCP	2,4,5-Trichlorophenol	0.4900	12.00
LM25	246TBP	2,4,6-Tribromophenol	0.5200	6.20
LM25	246TCP	2,4,6-Trichlorophenol	0.0610	12.00
LM25	24DCLP	2,4-Dichlorophenol	0.0650	12.00
LM25	24DMPN	2,4-Dimethylphenol	3.0000	6.20
LM25	24DNP	2,4-Dinitrophenol	4.7000	12.00

**Appendix C. USAEC Performance Demonstrated Methods for Soil Analyses  
DataChem Laboratories, Ft. McClellan, Anniston, Alabama (continued)**

Method No.	Analyte Code	Analyte Name	Certified Reporting Limit	Upper Limit
LM25	24DNT	2,4-Dinitrotoluene	1.4000	6.20
LM25	26DNA	2,6-Dinitroaniline	0.5700	6.20
LM25	26DNT	2,6-Dinitrotoluene	0.3200	6.20
LM25	2CLP	2-Chlorophenol	0.0550	12.00
LM25	2CLPD4	2-Chlorophenol-d <sub>4</sub> -S	0.3500	6.20
LM25	2CNAP	2-Chloronaphthalene	0.2400	12.00
LM25	2FBP	2-Fluorobiphenyl	0.0570	6.20
LM25	2FP	2-Fluorophenol	0.1500	12.00
LM25	2MNAP	2-Methylnaphthalene	0.0320	12.00
LM25	2MP	2-Methyl Phenol	0.0980	6.20
LM25	2NP	2-Nitrophenol	1.1000	6.20
LM25	33DCBD	3,3'-Dichlorobenzidine	1.6000	25.00
LM25	35DNA	3,5-Dinitro-aniline	1.6000	6.20
LM25	3NANIL	3-Nitroaniline	3.0000	12.00
LM25	3NT	3-Nitrotoluene	0.3400	6.20
LM25	46DN2C	4,6-Dinitro-2-cresol	0.8000	6.20
LM25	4BRPPE	4-Bromophenyl Phenyl Ether	0.0410	6.20
LM25	4CL3C	4-Chloro-3-methylphenol	0.9300	12.00
LM25	4CLPPE	4-Chlorophenyl Phenyl Ether	0.1700	6.20
LM25	4MP	4-Methyl Phenol	0.2400	12.00
LM25	4NP	4-Nitrophenol	3.3000	12.00
LM25	ANAPNE	Acenaphthene	0.0410	12.00
LM25	ANAPYL	Acenaphthylene	0.0330	12.00
LM25	ANTRC	Anthracene	0.7100	6.20
LM25	B2CEXM	bis(2-Chloroethoxy) Methane	0.1900	6.20
LM25	B2CIPE	bis(2-Chloroisopropyl) Ether	0.4400	12.00
LM25	B2CLEE	bis(2-Chloroethyl)ether	0.3600	12.00
LM25	B2EHP	bis(2-Ethylhexyl)phthalate	0.4800	6.20
LM25	BAANTR	Benzo(a)anthracene	0.0410	12.00
LM25	BAPYR	Benzo(a)pyrene	1.2000	6.20
LM25	BBFANT	Benzo(b)fluoranthene	0.3100	12.00
LM25	BBZP	Butyl Benzyl Phthalate	1.8000	6.20
LM25	BGHIPY	Benzo(g,h,i)perylene	0.1800	12.00
LM25	BKFANT	Benzo(k)fluoranthene	0.1300	12.00
LM25	BZALC	Benzyl Alcohol	0.0320	1.30
LM25	CHRY	Chrysene	0.0320	12.00
LM25	CL6BZ	Hexachlorobenzene	0.0800	12.00
LM25	CL6CP	Hexachlorocyclopentadiene	0.5200	12.00
LM25	CL6ET	Hexachloroethane	1.8000	12.00
LM25	CPMS	p-Chlorophenylmethyl Sulfide	0.0970	6.20
LM25	CPMSO	p-Chlorophenylmethyl Sulfoxide	0.3200	3.10
LM25	CPMSO2	p-Chlorophenylmethyl Sulfone	0.0660	12.00
LM25	DBAHA	Dibenzo(a,h)anthracene	0.3100	3.10

**Appendix C. USAEC Performance Demonstrated Methods for Soil Analyses  
DataChem Laboratories, Ft. McClellan, Anniston, Alabama (continued)**

Method No.	Analyte Code	Analyte Name	Certified Reporting Limit	Upper Limit
LM25	DBCP	Dibromochloropropane	0.0710	6.20
LM25	DBZFUR	Dibenzofuran	0.0380	6.20
LM25	DCPD	Dicyclopentadiene	0.5700	12.00
LM25	DDVP	Vapona	0.0680	12.00
LM25	DEP	Diethyl Phthalate	0.2400	6.20
LM25	DEPD4	Diethyl Phthalate-d <sub>4</sub> -S	0.0600	12.00
LM25	DITH	Dithiane	0.0650	12.00
LM25	DMP	Dimethyl Phthalate	0.0630	12.00
LM25	DNBP	di-N-Butyl Phthalate	1.3000	6.20
LM25	DNOP	di-N-Octyl Phthalate	0.2300	3.10
LM25	DNOPD4	di-N-Octylphthalate-d <sub>4</sub>	0.0650	12.00
LM25	ESFSO4	Endosulfan Sulfate	1.2000	6.20
LM25	FANT	Fluoranthene	0.0320	6.20
LM25	FLRENE	Fluorene	0.0650	12.00
LM25	HCBD	Hexachlorobutadiene	0.9700	6.20
LM25	ICDPYR	Indeno(1,2,3-cd)pyrene	2.4000	12.00
LM25	ISOPHR	Isophorone	0.3900	6.20
LM25	MIREX	Mirex	0.1400	12.00
LM25	MLTHN	Malathion	0.1800	3.10
LM25	NAP	Naphthalene	0.7400	6.20
LM25	NB	Nitrobenzene	1.8000	12.00
LM25	NBD5	Nitrobenzene-d <sub>5</sub>	0.2200	6.20
LM25	NNDMEA	N-Nitrosodimethylamine	0.4600	12.00
LM25	NNDNPA	N-Nitroso-di-N-propylamine	1.1000	12.00
LM25	NNDPA	N-Nitrosodiphenylamine	0.2900	12.00
LM25	OXAT	1,4-Oxathiane	0.0750	12.00
LM25	PCP	Pentachlorophenol	0.7600	6.20
LM25	PHANTR	Phenanthrene	0.0320	12.00
LM25	PHEND6	Phenol-d <sub>6</sub>	0.0690	12.00
LM25	PHENOL	Phenol	0.0520	12.00
LM25	PRTHN	Parathion	1.7000	12.00
LM25	PYR	Pyrene	0.0830	6.20
LM25	SUPONA	Supona	0.9200	12.00
LM25	TRPD14	Terphenyl-d <sub>14</sub>	0.1300	6.20
<b>PESTICIDES/SOIL/GCEC (µg/g)</b>				
LH17	ABHC	alpha-BHC	0.0028	0.1000
LH17	AENSLF	Endosulfan I	0.0010	0.0906
LH17	ALDRN	Aldrin	0.0014	0.0860
LH17	BBHC	beta-BHC	0.0077	0.1000
LH17	BENSLF	Endosulfan II	0.0007	0.0705
LH17	CLDAN	Chlordane	0.0684	0.8000
LH17	DBHC	delta-BHC	0.0085	0.0500
LH17	DLDRN	Dieldrin	0.0016	0.1000
LH17	ENDRN	Endrin	0.0065	0.0500
LH17	HPCL	Heptachlor	0.0022	0.1000

**Appendix C. USAEC Performance Demonstrated Methods for Soil Analyses  
DataChem Laboratories, Ft. McClellan, Anniston, Alabama (continued)**

Method No.	Analyte Code	Analyte Name	Certified Reporting Limit	Upper Limit
LH17	HPCLE	Heptachlor Epoxide	0.0013	0.1000
LH17	ISODR	Isodrin	0.0030	0.1000
LH17	LIN	Lindane (gamma-BHC)	0.0010	0.1000
LH17	MEXCLR	Methoxychlor	0.0359	1.0000
LH17	PCB016	PCB-1016	0.1000	2.0000
LH17	PCB260	PCB-1260	0.0479	2.0000
LH17	PPDDD	4,4'-DDD	0.0027	0.1000
LH17	PPDDE	4,4'-DDE	0.0027	0.1000
LH17	PPDDT	4,4'-DDT	0.0035	0.1000
LH17	TXPHEN	Toxaphene	0.2260	10.0000
<b>EXPLOSIVES/SOIL/HPLC (<math>\mu\text{g/g}</math>)</b>				
LW23	135TNB	1,3,5-Trinitrobenzene	0.9220	250.0000
LW23	13DNB	1,3-Dinitrobenzene	0.5040	250.0000
LW23	246TNT	2,4,6-Trinitrotoluene	2.0000	500.0000
LW23	24DNT	2,4-Dinitrotoluene	2.5000	250.0000
LW23	26DNT	2,6-Dinitrotoluene	2.0000	500.0000
LW23	HMX	Cyclotetramethylenetetranitramine	2.0000	500.0000
LW23	NB	Nitrobenzene	1.1400	500.0000
LW23	RDX	Hexahydro-1,3,5-trinitro-1,3,5-triazine	1.2800	500.0000
LW23	TETRYL	N-Methyl-N,2,4,6-tetranitroaniline	2.1100	500.0000
<b>NG and PETN/SOIL/HPLC (<math>\mu\text{g/g}</math>)</b>				
LW27	NG	Nitroglycerine	0.5100	50.0000
LW27	PETN	Pentaerythritol tetranitrate	1.0000	100.0000

**Appendix C. USAEC Performance Demonstrated Methods for Soil Analyses  
Environmental Science and Engineering,  
Ft. McClellan, Anniston, Alabama**

<b>Method No.</b>	<b>Analyte Code</b>	<b>Analyte Name</b>	<b>Certified Reporting Limit</b>	<b>Upper Limit</b>
<b><i>ORGANOSULFURS/SOIL/GCFP (µg/g)</i></b>				
LL03	BTZ	Benzothiazole	1.0800	13.2000
LL03	CPMS	p-Chlorophenylmethyl sulfide	1.0800	21.6000
LL03	CPMSO	p-Chlorophenylmethyl sulfoxide	2.2500	45.0000
LL03	CPMSO2	p-Chlorophenylmethyl sulfone	2.3700	47.4000
LL03	DITH	Dithiane	1.4700	11.4000
LL03	DMDS	Dimethyl disulfide	0.6920	13.8000
LL03	OXAT	1,4-Oxathiane	0.8560	17.1000
<b><i>IMPA/FC2A/SOIL (µg/g)</i></b>				
LT03	CLC2A	Chloroacetic acid	0.5000	20.0000
LT03	IMPA	Isopropyl methylphosphonate	0.5000	20.0000
LT03	MPA	Methylphosphonic acid	0.5000	20.0000
<b><i>AGENTPRODS/SOIL/HPLC (µg/g)</i></b>				
LW18	CLC2A	Chloroacetic acid	18.0000	302.0000
LW18	TDGCL	Thiodiglycol	3.9400	102.0000
<b><i>ORGANOPHOSPHOR/SOIL/GCFP (µg/g)</i></b>				
TT9	DIMP	Diisopropyl methylphosphonate	0.1140	4.5700
TT9	DMMP	Dimethyl methylphosphonate	0.1330	4.1800

**APPENDIX D**

**DATA CHEM LABORATORIES  
QUALITY ASSURANCE PROGRAM PLAN  
FOR  
U.S. ARMY ENVIRONMENTAL CENTER**

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QUALITY ASSURANCE PROGRAM PLAN  
for  
U.S. ARMY ENVIRONMENTAL CENTER  
REVISION CONTROL LOG

**RECORD OF MAJOR REVISIONS**

Revision No.	Revision Date	Affected Section Page		Description of Change
5	7/21/93	ALL	ALL	Document brought under Document Control.

**RECORD OF MINOR REVISIONS**

Revision No.	Revision Date	Affected Section Page		Description of Change
5	7/21/93	4.3.3	9	Updated from ASTM Type I grade water to ASTM Type II grade water.
5	8/12/93	ALL	ALL	Updated Agency references from USATHAMA to USAEC.

**QUALITY ASSURANCE  
PROGRAM PLAN**

**for  
U.S. ARMY ENVIRONMENTAL AGENCY**

**September 26, 1991**

**Laboratory Analysis  
of Environmental Samples**

**DCL Document QA-3/87**

**DataChem Laboratories**  
960 West LeVoy Drive  
Salt Lake City, Utah 84123

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**1.0**  
**DOCUMENT IDENTIFICATION**

Document Title: Quality Assurance Program Plan  
for the U.S Army Environmental Center

Document Control Number: QA-3/87

Organization: DataChem Laboratories (DCL)  
960 W. LeVoy Dr.  
Salt Lake City, Utah 84123

Director: James H. Nelson, Ph.D.  
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## 2.0 INTRODUCTION

This document is the DCL Quality Assurance/Quality Control Plan, prepared in compliance with the requirements of the U.S. Army Environmental Center (USAEC, formerly USATHAMA) with analytical laboratory services in support of the implementation of various installation restoration programs. This plan adheres to, and is an implementation of, the USATHAMA QA Program, January 1990, First Edition.

DCL is committed, in strictly following this plan, to provide to USAEC analytical data that are of a quality that may be used in litigation. All deviations from this plan or the USATHAMA QA Program will be submitted to USAEC for approval prior to implementation in the laboratory. Such deviations will be properly and fully documented.

DCL has conducted analyses for USAEC since 1984 under the 1982 USATHAMA QA Program, the Second Edition (March 1987) of the 1985 USATHAMA QA Program, and the January 1990 USATHAMA QA Program, First Edition.

## 3.0 ORGANIZATION AND RESPONSIBILITIES

### 3.1 Introduction

Ultimate responsibility for the conduct of all projects, and approval for the implementation of all programs at DCL resides with the Laboratory Director, Dr. James H. Nelson. Functional responsibility for the analytical work is delegated to the Project Manager, Mr. David W. Gayer; to the Analytical Task Managers, Mr. A. Brent Torgensen, and Mr. Richard Wade; and to the Quality Assurance Coordinator, Mr. Ronald H. Marsden.

### 3.2 Laboratory Director

The Laboratory Director is responsible to assure that DCL resources are adequately allocated to the project and that sufficient staffing and equipment are provided. He oversees and supports the Quality Assurance Coordinator.

### 3.3 Project Manager

The Project Manager has the responsibility of communication with the USAEC Program Contract Officer and oversees and supports the Analytical Task Managers in development, implementation, and operation of the analytical program organization. He is directly responsible for the interpretation of the provisions of the contract for DCL. The Project Manager is also responsible to assure that QA/QC recommendations and corrective actions are implemented.

The Project Manager is authorized to conduct official discussions with the Program Contract Officer concerning the original contractual agreement and delivery orders, and any subsequent modifications to the contractual agreement and/or delivery orders. Laboratory personnel matters are decided in concert with the Analytical Task Manager and appropriate Section Managers.

### 3.4 Analytical Task Manager

The Analytical Task Manager has the responsibility of implementing the USATHAMA 1990 QA Plan, and for coordinating the sample analysis flow in the laboratory. This will be achieved through the following:

1. Assuring the provision of sufficient equipment, laboratory space, resources, personnel, and quality reagents and materials to properly conduct the required analyses;
2. Supporting the Quality Assurance Coordinator;

3. Submitting documented analytical methods and laboratory certification data to the USAEC Project Officer prior to the analysis of field samples;
4. Ensuring that all provisions of the approved Project Quality Control Plan are fully implemented in the laboratory;
5. Ensuring the implementation of corrective action for any QA/QC deficiencies.

The Analytical Task Manager has the authority to suspend analytical work for quality control problems and to implement corrective actions recommended by the Quality Assurance Coordinator. He also has authority to accept or reject increases in the delivery rate of samples, within the bounds set by the contract. He confers with section managers and the Project Manager on personnel matters when they impact on the project.

### 3.5 Quality Assurance Coordinator

The Quality Assurance Coordinator (QAC) has the responsibility of establishing, overseeing, and auditing specific procedures for documenting, controlling, and validating analytical data quality. This is accomplished, in part, through the following:

1. Monitoring the QA and QC activities of the laboratory to ensure conformance with authorized policies, procedures, and good laboratory practices, and recommending improvements as necessary;
2. Informing the Project Manager and/or the Analytical Task Manager of noncompliance with the approved QA Program;
3. Requesting standard analytical reference materials from USAEC;
4. Ensuring that all records, logs, standard operating procedures, project plans and analytical results are maintained in a retrievable fashion;
5. Ensuring that standard operating procedures and project QA/QC plans are distributed to all appropriate laboratory personnel;
6. In consultation with the analysts and the Analytical Task Manager, establishing appropriate analytical lot size, including the correct QC samples;
7. Establishing the correct procedures and criteria for evaluating whether analytical performance is acceptable and in-control;
8. Ensuring that samples are received and logged properly, including lot sizing, introduction of required QC samples, and numbering of field samples and control samples;
9. Reviewing all laboratory data before those data are released, verifying that data were collected properly under an in-control analytical system;

10. Ensuring that the DCL quality control chemist, or appropriate analysts, are properly preparing QC samples;
11. Maintaining quality control charts, ensuring timely distribution of such charts, documenting corrective actions, and ensuring that analysts implement and document corrective actions as they become necessary;
12. Ensuring that sample logs, instrumentation logs, and all QC documents are properly maintained, including frequency of entries;
13. Discussing control chart results with the Analytical Task Manager and submitting updated, current charts to the USAEC Project Officer on a weekly basis, or as required by USAEC;
14. Maintaining an awareness of the entire laboratory operation to detect conditions which might jeopardize controls of the various analytical systems;
15. As directed by USAEC, auditing sampling documentation and procedures to ensure proper labeling, handling, transportation, and storage.

The Quality Assurance Coordinator has the authority to:

1. Approve all analytical reports;
2. Reject analytical data which does not meet applicable quality control criteria;
3. Require re-performance of sample analyses which are determined to be out-of-control;
4. Evaluate data and determine apparent long-term trends which may require corrective action;
5. Suspend analytical work, when necessary, to assure corrective actions are taken and that an analysis is again in control.

The Quality Assurance Coordinator also attends and participates in conferences for discussion of quality control and quality assurance problems and procedures.

## 4.0 CERTIFICATION

### 4.1 Laboratory Certification

DCL, as a laboratory, rather than as individual analysts, certifies as proficient in conducting analyses for USAEC. Each member of the organization has the education and training necessary to enable that individual to perform assigned functions. A personnel training file is maintained for each individual. Each individual updates the training file as necessary.

Management personnel have earned a Baccalaureate degree from an accredited college or university.

Analytical Chemists have earned a Baccalaureate Degree in Science or related fields from an accredited college or university.

Technical Staff have applicable training, including on the job training, and/or experience in related fields.

### 4.2 Analytical Methods

Analytical methods used for the analysis of environmental samples are described in a set of written instructions completely defining the procedure to be followed to process a sample and obtain an analytical result. An analytical method describes, as a minimum, the analytes for which it is valid, the matrix type, sample preparation, reagent and standards preparation, instrument calibration, and computations used to evaluate the analytical results. Standards and quality control sample requirements are also defined.

Analytical methods are either supplied by USAEC or, with approval, developed by DCL. The documentation for proposed methods development includes:

1. The submission of documentation to USAEC.
2. A statement of the problem.
3. A description of the technical approach to include specific details on procedures, solvents, instrumentation, etc.
4. An estimate of resources required (to include labor hours, funds and schedule).

When the testing of the analytical procedures has been successfully completed, the method is documented in the standardized USATHAMA method format. The format for documentation of all analytical methods is provided in Table 1. The format for data analysis is established by USAEC-provided statistical analysis computer software. Updates to the software are implemented upon receipt.

**Table 1.**  
**FORMAT FOR DOCUMENTATION OF METHOD CERTIFICATION**

- I. Summary**
  - A. Analytes
  - B. Matrix
  - C. General Method
  
- II. Application**
  - A. Tested Concentration Range
  - B. Sensitivity
  - C. Reporting Limit
  - D. Interferences
  - E. Analysis Rate
  - F. Safety Information
  
- III. Glassware and Chemicals**
  - A. Glassware/Hardware
  - B. Instrumentation
  - C. Analytes
  - D. Reagents and SARMS
  
- IV. Calibration**
  - A. Initial Calibration
  - B. Daily Calibration
  
- V. Certification Testing**
  
- VI. Sample Handling and Storage**
  - A. Sampling Procedure
  - B. Containers
  - C. Storage Conditions
  - D. Holding Time Limits
  - E. Solution Verification
  
- VII. Procedure**
  - A. Separations
  - B. Chemical Reactions
  - C. Instrumental Analysis
  - D. Confirmational Analysis
  
- VIII. Calculations**
  
- IX. Daily Quality Control**
  - A. Control Samples
  - B. Control Charts
  
- X. References**
  
- XI. Data**

The analytical method, once certified, is followed for all USATHAMA analyses. Instrumental conditions are optimized within the limits specified by method and documented by the analyst. Any deviation, other than the optimization of instrumental conditions, is pre-approved by USAEC before implementation.

All copies of USATHAMA-certified methods are individually numbered. Each distributed method copy must be signed for and dated. A comprehensive list of all distributed methods is kept by the Quality Assurance Coordinator.

#### 4.3 Method Certification

Before field samples may be analyzed by the laboratory, the methods of analysis must be certified. Certification for selected methods, accomplished under other USAEC contracts, may be determined by USAEC to be acceptable for the work performed under this contract for identical analytes and matrices. If analytes are required for a particular certified method in addition to those which have already been certified, the additional analytes are appended to the current certified method by following full certification procedures for the additional analytes. The current certified method standards, concentrations and analytical conditions are used to certify the additional compounds.

Some methods, including calibration of test and measurement equipment, do not require certification, due to either the nature of the measurement or the intended use of the data. When such methods are part of a project, USAEC will not provide a standardized method. However, laboratories must submit sufficient information in test plans, work plans, and project QC plans to describe exactly the procedures to be used. A copy of a proposed method must be submitted to the USAEC Chemistry Branch before it is used on any project.

The following methods do not require certification by the USAEC Chemistry Branch: temperature, conductivity, pH, oil and grease, hardness, asbestos, alkalinity (carbonate/bicarbonate/hydroxide), total organic carbon, biochemical oxygen demand, chemical oxygen demand, total dissolved solids, total suspended solids, total solids, total petroleum hydrocarbons, salinity, and acidity.

##### 4.3.1 Written Method

A draft of the analytical method proposed for certification is submitted to USAEC for approval with the precertification performance data package.

##### 4.3.2 Standards

Standard Analytical Reference Materials (SARMs), provided by USAEC, are used in all method certification analyses. DCL obtains suitable, certified Reference Materials from the EPA or other commercial sources for analytes for which USAEC is not able to provide SARMs. Standard water and standard soil are used by DCL for all USAEC analyses done.

#### 4.3.3 Standard Water

Standard water samples are prepared by adding a known quantity of target analyte to a known volume of water. The volume of water is specific in the method being performed. All target analytes for the method are added. ASTM Type II grade water is used for all analyses. The method and reagents used to prepare spiking solutions are specified in the standardized methods.

#### 4.3.4 Standard Soil

Standard soil samples are prepared by adding a known quantity of target analyte to a known weight of selectively blended standard soil as provided by the Chemistry Branch of USAEC.

#### 4.3.5 Precertification Calibration

Before initiating method certification, precertification calibration is performed. DCL holds discussions with USAEC delineating anticipated environmental concentrations. The concentration range tested includes the Target Reporting Limit (TRL). Additional concentrations of calibration standards may be included for expanding the range of certification. Duplicate analyses are performed on all of the calibration standards.

The certified check standards are obtained from a source other than USAEC, whenever possible. In the absence of suitable commercially prepared mixtures, the DCL Quality Control Chemist prepares appropriate mixtures from certified pure stock reagents. The mixtures contain the analyte(s) of interest at concentrations near the high end of the certification range.

The calibration standard data is tabulated and graphed for analysis of Lack of Fit (LOF) and Zero Intercept (ZI), then submitted to USAEC for evaluation. The check standard results are required to fall within the acceptability limits defined by the originator.

#### 4.3.6 Certification

Certified methods meet the following conditions: The Target Reporting Limit (TRL) and the range of certification are selected in consultation with USAEC. A pre-certification analysis is performed and reported to USAEC, with a copy of the analytical method. Upon approval from USAEC, a Class 1, Class 1A, Class 1B, or Class 2 certification process is initiated. See Table 2.

Data derived from certification is processed using USAEC supplied software, and submitted to USAEC for evaluation. The method Certified Reporting Limit (CRL) and certified range are determined from this data evaluation.

Methods certified under previous editions of the USATHAMA Quality Assurance Program and determined by USAEC to be valid for current work do not require recertification.

All certification data are properly maintained in archive files.

#### 4.3.7 Method Modifications and Control

Any modifications, additions, or deletions proposed to any certified USATHAMA method must be submitted to USAEC for approval before such a change is made. Following approval, the revised method (with changes plainly noted) shall be distributed to appropriate laboratory personnel as described in DCL SOP-GLP-002, and the old method collected for retirement.

#### 4.4 Analyst Training

An analyst certifying a new method is qualified to perform that method during routine field sample analysis. An analyst who is required to perform on a procedure which has already been certified is required to satisfactorily analyze an appropriate set of quality control samples to demonstrate ability to perform the method. The demonstration sample data must pass current quality control criteria. Successful certification performance is reflected by an addition to the analyst's training file.

The analyst prepares all data records and a data package, as required for field sample analysis data. The data and the data package must be approved by Quality Assurance. The data and data package are maintained in archives.

**Table 2.**  
**NUMBERS AND CONCENTRATIONS OF CALIBRATION STANDARDS**  
**(LINEAR AND ZERO-INTERCEPT)**

PRECERTIFICATION - CLASS 1

Minimum Testing Range (MTR): 12 Standards + 1 Check Standard (SC)  
Blank, \*0.5, 1, 2, 5, & \*10 TRL (Duplicate) + CS  
MTR + 1 Order of Magnitude Extension: 18 Standards + 1 Check Standard (CS)  
Blank, \*0.5, 1, 2, 5, 10, 20, 50, & \*100 TRL (Duplicate) + CS  
MTR + 2 Orders of Magnitude Extension: 24 Standards + 1 Check Standard (CS)  
Blank, \*0.5, 1, 2, 5, 10, 20, 50, 100, 200, 500, & \*1000 TRL (Duplicate) + CS

PRECERTIFICATION - CLASS 1A

Minimum Testing Range (MTR): 8 Standards  
Blank, \*0.5, 2, & \*10 TRL (Duplicate)  
MTR + 1 Order of Magnitude Extension: 12 Standards  
Blank, \*0.5, 2, 10, 50, & \*200 TRL (Duplicate)  
MTR + 2 Orders of Magnitude Extension: 16 Standards  
Blank, \*0.5, 2, 10, 50, 200, 500, & \*2000 TRL (Duplicate)

PRECERTIFICATION - CLASS 1B

Minimum Testing Range (MTR): 8 Standards + 1 Check Standard (CS)  
Blank, \*0.5, 2, & \*10 TRL (Duplicate) + CS  
MTR + 1 Order of Magnitude Extension: 12 Standards + 1 Check Standard (CS)  
Blank, \*0.5, 2, 10, 50, & \*200 TRL (Duplicate) + CS  
MTR + 2 Orders of Magnitude Extension: 16 Standards + 1 Check Standard (CS)  
Blank, \*0.5, 2, 10, 50, 200, 500, & \*2000 TRL (Duplicate) + CS

PRECERTIFICATION - CLASS 2

(Not Required)

INITIAL CALIBRATION - CLASS 1

Minimum Testing Range (MTR): 7 Standards + 1 Check Standard (CS)  
Blank, \*0.5, 1, 2, 5, \*10, & \*10 TRL + CS  
MTR + 1 Order of Magnitude Extension: 10 Standards + 1 Check Standard  
Blank, \*0.5, 1, 2, 5, 10, 20, 50, \*100, & \*100 TRL + CS  
MTR + 2 Orders of Magnitude Extension: 13 Standards + 1 Check Standard  
Blank, \*0.5, 1, 2, 5, 10, 20, 50, 100, 200, 500, \*1000, & \*1000 TRL + CS

\* 10 percent to 25 percent Range Extension

**Table 2  
(Continued)**

INITIAL CALIBRATION - CLASS 1A

Minimum Testing Range (MTR): 5 Standards  
Blank, \*0.5, 2, \*10, & \*10 TRL  
MTR + 1 Order of Magnitude Extension: 7 Standards  
Blank, \*0.5, 2, 10, 50, \*200, & \*200 TRL  
MTR + 2 Orders of Magnitude Extension: 9 Standards  
Blank, \*0.5, 2, 10, 50, 200, 500, \*2000, & \*2000 TRL

INITIAL CALIBRATION - CLASS 1B

Minimum Testing Range (MTR): 5 Standards + 1 Check Standard (CS)  
Blank, \*0.5, 2, \*10, & \*10 TRL + CS  
MTR + 1 Order of Magnitude Extension: 7 Standards + 1 Check Standard  
Blank, \*0.5, 2, 10, 50, \*200, & \*200 TRL + CS  
MTR + 2 Orders of Magnitude Extension: 9 Standards + 1 Check Standard  
Blank, \*0.5, 2, 10, 50, 200, 500, \*2000, & \*2000 TRL + CS

INITIAL CALIBRATION - CLASS 2

Minimum Testing Range (MTR): 6 Standards  
Blank and 1 TRL (Triplicate)

DAILY CALIBRATION - CLASS 1/CLASS 1A/ CLASS 1B

Minimum Testing Range (MTR): 2 Standards  
\*10 & \*10 TRL  
MTR + 1 Order of Magnitude Extension: 2 Standards  
\*100 & \*100 TRL  
MTR + 2 Orders of Magnitude Extension: 2 Standards  
\*1000 & \*1000 TRL

DAILY CALIBRATION - CLASS 2

Minimum Testing Range (MTR): 4 Standards  
Blank and 1 TRL (Duplicate)

**Table 2  
(Continued)**

CERTIFICATION - CLASS 1

Minimum Testing Range (MTR): 9 Initial, 6 Daily  
MTR + 1 Order of Magnitude Extension: 12 Initial, 6 Daily  
MTR + 2 Orders of Magnitude Extension: 15 Initial, 6 Daily

CERTIFICATION - CLASS 1A

Minimum Testing Range (MTR): 5 Initial  
MTR + 1 Order of Magnitude Extension: 7 Initial  
MTR + 2 Orders of Magnitude Extension: 9 Initial

CERTIFICATION - CLASS 1B

Minimum Testing Range (MTR): 6 Initial, 6 Daily  
MTR + 1 Order of Magnitude Extension: 8 Initial, 6 Daily  
MTR + 2 Orders of Magnitude Extension: 10 Initial, 6 Daily

CERTIFICATION - CLASS 2

Minimum Testing Range (MTR): 6 Initial

INITIAL FIELD SAMPLE LOT - CLASS 1

Minimum Testing Range (MTR): 9 Initial  
MTR + 1 Order of Magnitude Extension: 12 Initial  
MTR + 2 Orders of Magnitude Extension: 15 Initial

INITIAL FIELD SAMPLE LOT - CLASS 1A

Minimum Testing Range (MTR): 5 Initial  
MTR + 1 Order of Magnitude Extension: 7 Initial  
MTR + 2 Orders of Magnitude Extension: 9 Initial

**Table 2  
(Continued)**

INITIAL FIELD SAMPLE LOT - CLASS 1B

Minimum Testing Range (MTR): 6 Initial  
MTR + 1 Order of Magnitude Extension: 8 Initial  
MTR + 2 Orders of Magnitude Extension: 10 Initial

INITIAL FIELD SAMPLE LOT - CLASS 2

Minimum Testing Range (MTR): 6 Initial

ADDITIONAL FIELD SAMPLE LOT - CLASS 1/CLASS 1A/CLASS 1B

Minimum Testing Range (MTR): 2 Daily  
MTR + 1 Order of Magnitude Extension: 2 Daily  
MTR + 2 Orders of Magnitude Extension: 2 Daily

ADDITIONAL FIELD SAMPLE LOT - CLASS 2

Minimum Testing Range (MTR): 4 Daily

## 5.0 SAMPLE HANDLING AND ANALYSIS

### 5.1 Sample Management

In most instances, DCL does not perform sample collection, but receives samples from designated field crews. Samples received by DCL are received by designated sample custodians. The protocols of sample management are delineated below.

#### 5.1.1 Sample Containers

As directed by USAEC, DCL will supply sample bottles and/or shipping coolers for use in the collection of field samples. A copy of DCL's "Field Sampling Information," to be used as guidance in sampling and in the completion of chains-of-custody, is included in the initial shipment of coolers to the field sampling site. All sample containers shall be cleaned before use according to the protocols specified in Appendix C. Use of commercially cleaned bottles is acceptable provided that cleaning is performed as specified in Appendix C or meets the requirements of the EPA's Contract Laboratory Program.

Generally, for water samples, this includes: septum-sealed glass vials for volatile compounds; amber glass bottles with Teflon-lined lids for organic constituents other than volatiles; and polyethylene bottles for inorganic analytes. Exceptions are noted in the certified method. For soil and sediment samples wide-mouth amber-glass bottles shall be used. Preservatives, as delineated in the DCL USATHAMA Analyte Summary (Appendix B), are provided (as necessary) with sample containers shipped to the field, for proper addition at the site.

#### 5.1.2 Sample Receipt

Samples are received at DCL by the designated Sample Receipt Officer (SRO), or his designee. At the time of receipt of a sample shipment, the sample shipping containers are opened and the samples are inspected. A Sample Receipt Form is initiated at this time. This form includes entries for date and time of receipt, airbill number, a record of the condition of seals on the shipping container and samples, documentation present, temperature and general condition of the shipment, and correlation of sample document and sample labeling information.

Any discrepancies between the samples and the documentation, including missing, broken, or damaged samples, will be reported to USAEC or its contractor within 24 hours.

The SRO or his designee signs the field chain-of-custody record at the time that the shipping container is opened. In the case of water samples, which do not usually require splitting, the SRO or his designee opens the shipping container and completes the sample inspection form and field chain-of-custody record. Sufficient copies of the field chain-of-custody record are made to allot one copy for each analytical procedure, plus one for moisture and one as a back-up.

### 5.1.3 Sample Logging

The field chain-of-custody record is used by the Sample Receipt Coordinator (SRC) to initiate sample logging procedures. Initial logging entries include field sample number, date of receipt at DCL, analyses requested, and comments on sample condition at the time of receipt as noted on the Sample Receipt Record. These are recorded in both a computer based log and in a bound logbook. After sample lotting is completed, the USAEC sample identification number for each sample and analysis is entered into the logs.

### 5.1.4 Sample Splitting

Following initial sample inspection, the SRC splits the samples into the required number of aliquots (one for each analytical procedure, one for moisture if the sample is a soil, and a large portion for back-up). The SRC properly labels the aliquots with the field sample identification number and the method of analysis, and relinquishes custody of the sample aliquots to the SRC.

### 5.1.5 Sample Lotting and Labeling

The number of samples which can be analyzed by a given method on a single day, as determined by the rate-limiting step in the analytical scheme, is designated as a "lot". The samples in a lot are labeled with a USAEC sample identification number consisting of a three letter lot code and individual three number sample designations (e.g. AAA001, AAA002). As split sample aliquots for a particular analytical procedure are received by the SRC, they are given the next alphabetical lot designation in sequence. Samples received and split at various times are grouped together in the same lot such that sample holding times are not jeopardized. The unique sample number is written in black permanent marker on white laboratory labeling tape, which is prominently placed on each sample container.

Quality control (QC) samples are a part of every lot, and are spiked according to the specific method requirements. The QC samples are provided upon request of the analyst.

### 5.1.6 Sample Storage

Samples are stored in a location appropriate to the holding requirements of the requested analytes. Heat-sensitive, light-sensitive, radioactive, or other samples having unusual physical characteristics or requiring special handling, are properly stored and maintained.

## 5.2 Chain-of-Custody

DCL maintains chain-of-custody records for all USAEC samples received at the laboratory.

A copy of applicable field chain-of-custody records is maintained with each sample lot. In addition, each lot of samples is maintained under a separate laboratory chain-of-custody record. The chain-of-custody includes unique sample number(s), date and time, source of sample(s), analyses required, signatures of relinquishing and receiving entities, and any other pertinent information. Copies of DCL's field and in-house chains-of-custodies for USAEC projects are provided in Appendix D.

### 5.3 Sample Handling Procedures

After samples have been received, split, and lotted, those not requiring extraction procedures are transferred to a central walk-in cold storage area. They are stored in this area until they are scheduled for analysis. Samples not requiring extraction procedures are prepared for analysis, within the required holding times, by the analyst or by a technician working under the direction of the analyst. These samples are usually analyzed within hours after preparation.

Samples which require extraction, distillation, or digestion procedures are prepared for analysis by the appropriate Inorganic or Organic Sample Preparation groups after lotting procedures have been completed. Extracts or distillates are stored in refrigerators in appropriate analytical areas of the laboratory.

The samples and extracts are maintained in their designated lots and under chain-of-custody, at all times. Separate preparation logbooks are maintained by the sample preparation groups to document sample handling.

### 5.4 Toxicity Characteristic Leaching Procedure

Samples which require Toxicity Characteristic Leaching Procedure (TCLP) are split and assigned a unique three-letter lot code. Chains-of-custody for these samples are signed off in the same manner as other samples requiring a certified USATHAMA analysis. At the same time, chains-of-custody are printed (but not "initiated") for all prospective analyses to be generated from the TCLP leachate(s).

Once the original sample has been satisfactorily leached, both the chain-of-custody and any remaining original sample are transferred to Long Term Storage. The chains-of-custody for all generated leachates are now initiated by TCLP personnel. These leachates (along with their chains-of-custody) are stored and handled as any other USAEC samples which have been prepared for analysis.

The chains-of-custody for the original sample and the leachates are cross-referenced to facilitate traceability.

### 5.5 Holding Times

The holding times specified in DCL's USATHAMA Analyte Summary (Appendix B) are adhered to for all USAEC samples, extracts, distillates, and digestates.

## 5.6 Sample Analysis

### 5.6.1 Standards

Analytical standards are prepared either from Standard Analytical Reference Materials (SARMS) or Interim Reference Materials (IRM) supplied by USAEC, or from standard materials obtained by DCL from the EPA, the National Institute of Standards and Technology (NIST), or other commercial sources. Secondary standard materials may be used when SARM materials are available in only limited quantity. The secondary standards, which must be positively identified with an estimation of purity, are referenced to SARMS and periodically checked against them.

Standard materials procured from commercial sources other than USAEC, the Environmental Protection Agency (EPA), or the NIST are considered as "off-the-shelf" materials. The purity and identity of these materials is established from both analysis documentation supplied by the vendor and DCL analytical data. Materials are characterized by two independent methods whenever possible, including, but not limited to IR, GC, GC/MS, HPLC, and other inorganic techniques.

Metals are traceable to NIST, whenever possible. "Off-the-shelf" materials are characterized against EPA or NBS known standards whenever possible. All SARMS are stored in the quality control laboratory, under controlled access conditions. Generally, organic compounds are stored under refrigeration, while metals solutions are stored at room temperature.

### 5.6.2 Solutions

Analytical standard working solutions are normally prepared by the analyst performing the analysis, in accordance with the protocol defined in the approved analytical method. In some analytical procedures, a designated analyst prepares the standards, while other analysts carry out the procedure.

As new or replacement standard solutions are prepared, they are validated against either the previously used standard, a commercially prepared quantitative standard, or a standard prepared by another analyst for the purpose of validation.

Although validation acceptance criteria are established for each analytical method, protocol guidelines for acceptance of a new solution is that it is found, by analysis, to be within  $\pm 5\%$  of the target value. All validations are documented either in the analyst's notebook or in a standards preparation logbook unique to USAEC and the analytical area using the standards.

### 5.6.3 Sample Preparation

Soil and water field samples are prepared for analysis according to the protocol defined in the analytical method for the specific analyte(s) being analyzed. Procedures for the preparation of mixed-matrix field samples, such as sediment, sludge, sewer, or lake-bottom samples, are discussed with USAEC on a case-by-case basis.

#### 5.6.4 Instrument Calibration

The USATHAMA QA Program delineates, in detail, the requirements for instrument calibration for precertification, full method certification, initial calibration for analysis work, and daily calibration during sample analysis. DCL has implemented these guidelines for all USAEC work, as follows. Also see Section 4.3.6 (Certification) for additional details.

Instruments are tuned, as applicable, and the required number and concentrations of standards are analyzed daily with each lot of samples. Calibration criteria are either passed or corrective action is pursued by the analyst. If daily calibration criteria are not met, then initial calibration procedures are instituted to bring the analytical system back into calibration.

#### 5.6.5 Initial Calibration

During initial calibration, a minimum of one blank and five calibration standards (Class 1) or one blank and three calibration standards (Class 1A and Class 1B) that bracket the certification testing range is analyzed singularly on one day. The concentrations of the calibration standards, in the solvent that results from all the preparation steps of the method, take into account any concentration steps that are part of the method. Concentrations in the solvent correspond to those in an environmental matrix as if the method preparation steps had been performed.

In addition to the initial calibration standards, Class 1 and 1B methods require the analysis of calibration check standards (Section 5.6.7). During a Class 1 or Class 1B initial calibration, a calibration check standard is analyzed at the completion of calibration. If the method requires what could be an initial calibration each day analysis is performed, then the calibration check standards are analyzed once a week rather than each day.

If the results of the calibration check standard are not acceptable, immediate reanalysis of the calibration check standard is required. If the results of the reanalysis still exceed the limits of acceptability, the system is considered to have failed calibration. Sample analysis is halted and will not resume until successful completion of initial calibration. Corrective actions taken to restore initial calibration are documented in the analysts' notebook.

#### 5.6.6 Daily Calibration

Calibration standards are analyzed each day to verify that instrument response has not changed from previous calibration. Each day before sample analysis, the highest concentration standard is analyzed. The response must fall within the required percentage or two standard deviations of the mean response for the same concentration, as determined from precertification, certification, and prior initial/daily calibrations. If the response fails this test, the daily standard is reanalyzed. If the response from the second analysis fails this range, initial calibration is performed before analyzing samples.

Each day after sample analyses are completed, the highest concentration standard is analyzed. If the response is not within the required percentage or two standard deviations of the mean response from precertification, certification, and prior initial/daily calibrations, the daily standard shall be reanalyzed. If the response from the second analysis fails the range, the system is considered to have failed calibration. Initial calibration is performed and all samples analyzed since the last acceptable calibration are reanalyzed.

For non-linear or non-zero-intercept calibration curves, daily calibration consists of analysis of the low, middle, and high standards at the beginning of the day. When sample analyses are completed at the end of the day, the low and high standards are analyzed. Instrument responses for each concentration determination must fall within two standard deviations of the mean response, as described previously, for the appropriate standard. For calibrations fitted by the quadratic equation, a minimum of four standards over the certified range are required and the highest level standard analyzed at the end of the day. For all other equations, one more standard than needed to meet the degrees of freedom for any lack-of-fit is required, as a minimum.

#### 5.6.7 Calibration Check Standards

Calibration check standards are required for all Class 1 and 1B methods and are analyzed during precertification and with each initial certification. The calibration check standard contains all analytes of interest for the method in question at a concentration near the upper end of the calibration range. Results of the calibration check standards shall fall within the limits of acceptability as described below:

##### CASE 1.

A certified check standard is available from the EPA or some other source with both the true value and limits of acceptability specified by the supplier. The results must fall within the limits specified by the supplier, or +/-10 percent for inorganics, +/-25 percent for organics, whichever is less.

##### CASE 2.

A certified check standard is available from the EPA or some other source with a true value specified but without limits of acceptability. The results must fall within +/-10 percent for inorganics and within +/-25 percent for organics.

##### CASE 3.

If no certified check standard is available, the contractor laboratory shall prepare a check standard using a second source of reference material. This standard shall be prepared by a different analyst than the one who prepared the calibration standard. If weighing of the material is required, a different balance should be used, if possible. The results must fall within +/-10 percent for inorganics and within +/-25 percent for organics.

##### CASE 4.

If there is only one source of reference material available, then the calibration and calibration check standards must be prepared from the same material. The standards shall be prepared by different analysts. If weighing is required, different balances should be used, if possible. The results must fall within +/-10 percent for inorganics and within +/-25 percent for organics.

For all cases listed above, after the seventh acceptable calibration check standard, the limits of acceptability are +/- two standard deviations, as determined from the first seven points.

For multi-analyte methods, the calibration check standard contains all analytes of interest. For the check standard to be deemed acceptable at least 2/3 of the analytes must meet the limits of acceptability as defined above (also see Table 3). In addition, if a single analyte falls outside the limits of acceptability for two consecutive times, then the calibration check standard is deemed unacceptable. If a calibration check standard is not acceptable, the procedures detailed above are followed.

**Table 3.**  
**MINIMUM NUMBER OF IN-CONTROL POINTS**  
**FOR MULTI-ANALYTE METHODS**

<u>Required Control Analytes Per Method</u>	<u>Required Number of Data Values Falling Between the UCL and LCL</u>
1	1
2	2
3	2
4	3
5	4
6	4
7	5
8	6
9	6
10	7
11	8
12	8
13	9
14	10
15	10
16	11
17	12
18	12
19	13
20	14
21	14
22	15
23	16
24	16
25	17

**5.6.8 Analytical Procedures**

All field samples are analyzed according to approved, laboratory certified USATHAMA analytical methods. All deviations shall be approved by USAEC prior to implementation. These deviations are also documented in the analyst's notebook.

#### 5.6.9 Second-Column Confirmation

In several GC and HPLC methods (e.g., organochlorine pesticides and explosives), the presence of compounds is routinely confirmed on a second column. The confirmation is usually performed on the basis of a Class 2 certification. Confirmation does not necessarily have to be performed within holding times, but must be accomplished within ten (10) days of sample analysis.

#### 5.7 Data Handling

Although the primary emphasis of the USATHAMA QA Program is the control of sample analysis and the handling of data, record keeping maintains its importance in the overall assessment of the production of quality of data and is used in part to document the control of sample analysis. The degree of rigor used in documenting sampling and analysis activities cannot be understated. All activities require extensive documentation and special handling protocols. All activities are to be performed under chain-of-custody procedures. Particularly in these situations, the attitude is: "If you didn't write it down, you didn't do it."

For most USAEC projects, this degree of documentation is required. For some projects, documentation in the form of an EPA CLP package is required. In any case, the records described in this Quality Assurance document shall be maintained and will be available for inspection by USAEC.

##### 5.7.1 Data Reduction

Generally, data have been collected during the analysis of samples either into computer based data files or onto hard copy sheets, which, in turn, are either machine generated or hand written. All of the data are eventually compiled in computer files. The data pertaining to analytical standards are either compared to the most recent initial calibration curve, in the case of a daily calibration, or used to generate new initial calibration curves, in accordance with those generated during pre-certification. The appropriate standard curve is used to evaluate the field sample data to determine the amount of analyte present. Finally, all of the computer generated calculations are generated as hard copy output.

##### 5.7.2 Data Validation

Initial data validation is accomplished during data collection through the use of quality control samples and calibration check standards. Errors detected through a review of these monitors by Quality Assurance during analysis are corrected during the data collection phase of the analysis. Only analytically valid data are processed further.

Following an analyst's computer-based reduction of data and production of a numerical results report, the entire assemblage of data is given to a peer analyst for review and validation. The peer analyst checks that the analytical method was followed, that there are no errors in the transcription of data, that the best-fit curve was used, and that the numerical report of data contains no calculation or transcription errors.

The data package is then reviewed by the appropriate Group Leader or Section Manager. The data report is particularly scrutinized to assure that all reported data values are in the proper range or have dilution factors, that the method has been carefully followed, that instrumentation was properly tuned or calibrated, and that the instrumental data was properly interpreted. A general review of the data package is also made to assure that all required documentation is present.

The final step in data validation is the review by Quality Assurance. The content of each data package is closely checked for errors or omissions that would negatively impact on the admissibility of the data in litigation proceedings. Corrective action is initiated and documented as outlined in section 10.0.

### 5.7.3 Data Reporting

The results for samples analyzed for USAEC projects are entered into the USAEC-provided software program (IRDMS). Data created using the IRDMS can then be electronically transmitted to USAEC Via Potomac Research Inc. (PRI), or a diskette together with hard copy printouts can be submitted.

Data is entered on a coding form by the analyst, which is verified by the peer checker and, group leader/section manager. QA personnel review data for obvious errors. These data are encoded onto a diskette, checked through two USAEC software routines, then printed out and verified by visual inspection by a Data Entry Specialist. Verified analytical results are then submitted to USAEC. DCL retains a copy diskette of all data submitted.

All information pertaining to the analysis of a lot of samples is collected into a data package at the completion of analysis. The contents of data packages varies with methods of analysis. The package is reviewed by Quality Assurance to eliminate technical errors that might affect the litigation quality of the data. The reported data is also reviewed by Data Entry for completeness before release.

All data packages are archived at DCL until a task or delivery order at a particular installation is complete. At that time, all pertinent documentation filed in appropriately-labeled boxes is delivered either to USAEC directly, or to the prime contractor responsible for final review of the data packages. In the second case, the prime contractor is responsible for the delivery of DCL data boxes to USAEC.

## 6.0 ANALYTICAL SYSTEM CONTROLS

### 6.1 Sample Control

As discussed in the section of this QA Plan on Sample Management, DCL is not generally responsible for the collection of samples from sites in the field. However, DCL efforts in sample control may extend into field sample collection. As directed by USAEC or the prime contractor, DCL provides proper sample collection bottles, sample preservatives, labeling material, sample shipping containers (coolers), and technical assistance to field sample collection crews. DCL also works in concert with USAEC or the prime contractor on sample shipping and receiving.

Samples received at DCL are under the control of Sample Receipt personnel from receipt at the lab to acceptance by an analyst for extraction or preparation. Samples are not released for processing until all documentation is completed and the samples are properly lotted and labeled. Holding times are closely monitored by the analysts, Sample Receipt and laboratory management.

DCL Project Managers communicate regularly with USAEC and/or other involved prime contractors to alleviate sample shipping, holding time, and analysis difficulties.

### 6.2 Document Control

Document control is primarily the responsibility of Quality Assurance. Sample documents generated in the field during sample collection and shipping are maintained in QA files. Laboratory chain-of-custody records, sample receipt and tracking records, data reporting forms and analysis data packages, and corrective action records are maintained by Quality Assurance. On a schedule determined by contract requirements, QA also archives or otherwise controls all bound notebooks and logbooks containing data pertinent to USAEC work.

### 6.3 Quality Control Samples

Quality control chemists within the Quality Assurance Section of DCL prepare most of the quality control samples required during sample analysis. These samples are prepared from USAEC-supplied SARM and IRM stocks, and other reference materials. Other reference materials include EPA, and NIST standard materials, and "off-the-shelf" materials. "Off-the-shelf" materials are analyzed by DCL, with positive identification and estimate of purity, with EPA standard reference materials, where possible, using at least two different methods.

Quality control stock and dilute working solutions are prepared and maintained separately from those used by analysts as standards. Exceptions to this procedure are made only when primary stock material is in very short supply, or when the primary solution is unstable. In these cases, the same primary solution is used to prepare separate dilute working solutions. Samples are prepared in accordance with parameters defined in each analytical method. These parameters include the control analytes, the concentration levels at which the analytes should be spiked, control sample matrix, spike equilibration time, and procedures for preparation of the sample for analysis.

Quality control samples which are not regularly prepared by the quality control chemists include surrogate spiking solutions and spiked samples required in the GC/MS methods for volatile and semi-volatile organic compounds. These surrogate preparations are handled by the GC/MS Group and the Extraction Group, respectively.

Quality control samples are included in every lot of USAEC samples, as required in the USATHAMA QA Program and specified in each certified analytical method. The control samples are processed through the entire analytical method and quantitated on the same calibration curve as the field samples. The results for the quality control samples are evaluated first by the analyst, and then by Quality Assurance, to determine their acceptability.

Calibration check standards are prepared by someone other than the person preparing the standards. Calibration check standards are analyzed at the time of an initial calibration, or once per week when routine initial calibrations replace daily calibrations. The analysis results must meet the criteria established by their originator.

#### 6.4 Control Charts

For Class 1, Class 1A, and Class 1B certified methods, control charts are used to monitor the variations in the precision and accuracy of routine analyses and to detect trends in these variations. The construction of a control chart requires initial data to establish the mean and range of measurements. The QC control charts are constructed from data representing performance of the complete analytical method. Data used in control charts is not adjusted for accuracy. Control charts are not used with Class 2 certified methods.

Control charts include the analyte, method number, DCL laboratory code of UB, spike concentration, and chart title. All data presented on a control chart are also presented in tabular form. The following charts may be selected from the USAEC-supplied computer control chart program:

1. Single-Day X-Bar Control Chart (High Spike Conc.)
2. Single-Day Range Control Chart (High Spike Conc.)
3. Three-Day X-Bar Control Chart (Low Spike Conc.)
4. Three-Day Range Control Chart (Low Spike Conc.)

In addition, the following information is also included on each control chart:

- Three-letter lot designation for each point, shown on the x-axis;
- Percent recovery (for X-bar control charts), or range (for R control charts) along the y-axis;
- Upper control limit (UCL);
- Upper warning limit (UWL);
- Mean;
- Lower warning limit (LWL), on X-bar charts; and
- Lower control limit (LCL), on X-bar charts.

For some analytes specified by USAEC, warning limits on X-bar charts are deleted and replaced by modified control limits based upon data quality specifications.

#### 6.4.1 Control Chart Plotting: Single-Day

The initial control chart is prepared using the four days of certification data closest to the spiking concentration used during analysis. The average (X-bar), average range (R), and control limits for both are updated after each in-control lot for the first 20 lots. Limits established after lot 20 are used for the next 20 lots. Control charts are updated after each 20 lots thereafter, using the most recent 40 points. In interpreting the control charts developed for the initial lots (1-20), the limits established from the previous lots are used to control the current lot.

When modified limits are established, data for samples are accepted if the control data fall between the modified limits. If modified limits have not been established, data for samples are accepted, based upon the recoveries established during certification and the current performance of the method. In updating the control charts, the new data must be combined with the individual values of previous average percent recoveries and not the mean of all previous data. Only lots evaluated as in-control are applicable to the 20 and 40 lot requirements for establishing and updating control chart limits. Out-of-control or outlier points are plotted; however, such lots are not utilized in lot number requirements or control chart calculations.

All recoveries are plotted, whether or not the lot is in-control. Plotted points represent averaged instrument measurements and not the individual measurement values. Each individual recovery measurement value is tested as an outlier using Dixon's Test at the 98% confidence level. If the datum is not classified as an outlier by the test, the point is included in updating the control chart limits. If the datum is classified as an outlier, it is not used in updating the control chart limits. Range data are not subject to outlier testing.

After the first 20 in-control sample lots, control limits are recalculated using only in-control data points. The control limits are then drawn backward to encompass all previous points. Any points falling outside the control limits (UCL or LCL) are dropped from the calculations (but left on the charts) and the control limits recalculated using only points between those limits. This practice of dropping points and recalculating limits is performed only once, at the initialization of stable limits. Charts are then updated with newly calculated control limits and all points plotted.

#### 6.4.2 Three-Point Moving Average

Analytical data for analytes prepared in the single low concentration QC sample are plotted and evaluated on a three-day-moving-average control chart. Data for the surrogates spiked in a standard matrix and used in GC/MS analyses are also charted on a three-day-moving-average control chart. Plotting criteria for the three-point moving average control charts are similar to those described above (Section 6.4.1) for single-day control charts. Data for analytes prepared in duplicate QC samples at high concentrations are plotted and evaluated on single-day control charts.

Computer generated control charts maintained by Quality Assurance are updated and printed weekly, while analysts plot data points by hand as sample lots are analyzed. This allows for both computer maintenance and evaluation of a large data base with software calculation of control limits, and immediate daily surveillance of analytical trends.

#### 6.5 Out-of-Control Conditions

Results of the analysis of quality control samples are reported to QA within 48 hours of completion through the analyst's submission of a Preliminary QC Report.

The analyst quantifies each analyte in the method blank and spiked QC sample each day of analysis. Processing of additional lots will not occur until the results of the previous lots have been calculated, plotted on control charts as required, and the entire analytical method shown to be in control.

An indication of an out-of-control situation may include: A value outside the control limits or classified as outlier by statistical test; A series of seven successive points on the same side of the mean; A series of five successive points going in the same direction; A cyclical pattern of control values, or; Two consecutive points between the UWL and UCL or the LWL and LCL.

If the points for at least two-thirds of the control analytes for a multi-analyte method are classified as in-control, the method is in control and environmental sample data may be reported. A method may be deemed out-of-control even if greater than or equal to 2/3 of the control analytes meet control criteria. Of the remaining control analytes (less than 1/3 possible out-of-control), if one analyte has two consecutive out-of-control points, as defined above, the method is deemed out-of-control. If data points for fewer than 2/3 of the control analytes are classified as in control, the method is considered to be out-of-control and all work on that method must cease immediately. No data for environmental samples in that lot may be reported.

In all cases, investigation by the analyst and the Quality Assurance Coordinator is required to determine the cause of the condition and to decide on appropriate corrective action. The pertinent details of the situation and the corrective action taken are fully documented in a Corrective Action Report (CAR). (See also section 10.0.) Field sample data effected by the situation are evaluated and reanalyzed as necessary.

When a method is determined to be out of control, the analysis of field samples by that method is suspended. Corrective action must be documented and the method must be demonstrated to be in control before analysis of field samples is reinstated. Analytical control is demonstrated through the acceptable analysis of an appropriate set of QA samples.

## 7.0 PREVENTATIVE MAINTENANCE

All analytical instrumentation used at DCL is maintained to provide consistent, high-quality performance. Most instruments are maintained by the manufacturer, under contract. Instrument service records and maintenance calibrations are maintained by the appropriate section and in a logbook unique for each instrument.

The primary objective of the instrument maintenance program is to assure the quality of the analytical data generated by the instrument. While there are analytical systems which require absolute calibration, such as balances, the majority of analytical systems used by DCL for the analysis of USAEC samples are calibrated at the time of use by the analyst. This is accomplished through generation of a chemical calibration curve, based upon instrument response versus analyte concentration. This curve is used to evaluate field sample data through instrument responses.

Major instrument systems which are calibrated on an "as used" basis are maintained under either an "on call" or a preventative maintenance contract with the manufacturer. Preventative maintenance is scheduled in each instrument contract. When an instrument cannot perform to specifications and DCL technicians cannot return it to specification, a contracted repair service (usually the manufacturer) is called.

Instrument systems which must maintain an absolute calibration, such as analytical balances, are serviced under contract with the manufacturer, usually on an annual basis. Balances are also checked, on at least a weekly basis, for accuracy by Quality Assurance, using NIST-traceable weights. Temperatures of freezers, refrigerators, and walk-in coolers are recorded every working day by QA. When temperatures are noted outside the acceptable range, appropriate personnel are notified for correction. Ovens are calibrated and their temperatures maintained regularly by the appropriate section personnel.

## 8.0 RECORDKEEPING

### 8.1 Laboratory Notebooks

Bound, sequentially-numbered laboratory notebooks with pre-numbered pages are utilized by all analysts for analytical recordkeeping. Notebooks are generally issued to and used by an individual analyst. Any loose sheets of data which must be included in a notebook are securely taped into the notebook and signed and dated across the edges, halfway on the inserted sheet and halfway on the notebook page. Each data page is signed and dated by the analyst entering data on that page, as well as reviewed, signed, and dated by a witness. All entries are required to be in black ink. Corrections are made by a single strikeout, which is dated and initialed.

### 8.2 Logbooks

#### 8.2.1 General

Individual logbook entries are signed and dated by the analyst or technician making the entry. These notebooks include, for example, instrument use and maintenance/calibration logs, pH logs, sample moisture determination logs, and sample receipt logs.

Recordkeeping for sample receipt is discussed under the Sample Management Section 5.1.

#### 8.2.2 Standards

A bound logbook is maintained for all analytical reference materials used for USAEC work. The record includes the date of receipt, preparer, source, purity, composition, storage requirements, and expiration date, if applicable. Characterization data for purchased reference material is also included.

The preparation of working standards from reference materials is recorded in a bound logbook. This logbook may be of general use by several analysts for USAEC standards preparation, or an individual analyst's notebook, as for preparation of standards used for a single analytical run associated with a single lot of samples.

#### 8.2.3 Instrument

Instrument maintenance records and, where applicable, instrument tuning and calibration data, are maintained in instrument specific logbooks. Actual analytical conditions pertaining to an individual lot analysis are recorded in the analyst's notebook, along with other pertinent analytical information.