

8.3 Hard-Copy Output

Hard-copy output, (e.g., chromatograms and computer generated data evaluations) is labeled with date, time (where applicable), analytical method, sample numbers, the name or initials of the analyst generating the output, and other pertinent information. Storage of hard-copy output is with related analytical data pertaining to an individual lot analysis. All such data, comprising a complete record of an analysis, are compiled into one or more envelopes for archiving. The envelopes are properly labeled with the lot designation, method of analysis, matrix, analyst, analyst's notebook, and date of completion. When samples from multiple sites or projects are grouped together in a single lot, the data pertaining to each site are compiled (or copied) and stored separately, as directed by USAEC. All copies indicate the location of the original data.

8.4 Data Package Preparation

In general, all data should be maintained in two separate locations, the data package and the laboratory notebook(s).

Records to be contained in the data package should include, but are not limited to the following:

- Optimized instrumental conditions
- Original chromatograms, strip charts, and/or other instrument output
- Original chain-of-custody form and carrier transmittal documents
- All hardcopy GC/MS outputs
- Expanded scale blow-up of manually integrated peak(s).
- All data sheets or other pre-printed forms used by the contractor or laboratory.
- Copies of all relevant notebook pages. This should include preparation of standards, calibration, sample preparation/extraction, moisture determinations, calculations, and any other relevant comments.

Each data package should contain all information related to one lot for one installation. In cases where a lot has samples from more than one installation, then the information should be copied and placed in separate packages for each installation. In those packages which receive copies, the location of the original material should be identified.

Each data package should contain a contents and approval checklist. This should identify all materials which must be placed into the data package. This list should also list reviewer's names, dates of review, provide space for comments, notes, and corrective actions.

It is the responsibility of the contractor laboratory to review data packages for both content and correctness.

Included in the data package should be a discussion on the observations on the data contained in that data package. This discussion shall include, but not be limited to, observed matrix effects, blank results, control problems, deviations from approved SOPs, digressions from normal practices (i.e., manual integrations) and reasons thereof, etc. The impact on the usability of the data shall be discussed. Explanations on the use of the applicable flagging codes shall be provided.

A detailed SOP is currently in development at DCL.

9.0 AUDITS

DCL facilities are always available for any required audits, announced or unannounced, by USAEC representatives.

The DCL Quality Assurance Coordinator conducts internal audits of critical functions within the laboratory, including verification that record keeping procedures are adequate, verification that general good laboratory practices, analytical methods and standard operating procedures are being followed, and continual assessment of quality control sample results. A summary of such audits is available for review at the laboratory. Internal audits shall be conducted by DCL QA personnel at a minimum rate of twice per month.

10.0 CORRECTIVE ACTION

When, as a result of audit procedures or the analysis of quality control samples, the analytical or other laboratory systems are found to be unsatisfactory, a corrective action is initiated. The unsatisfactory situation may be either immediate or long term in nature. Immediate short term problems may include unsatisfactory performance on quality control samples (which may be more involved than simply out-of-control data), errors or omissions in the compilation of the data package, or other problems peculiar to a single lot of samples. Long-term problems include trends or cycles in quality control sample analysis data, standard and solution preparation control, staff training in analytical and quality control procedures, or other problems which affect several analytical methods or multiple lots of samples.

To enhance the timeliness of corrective action and thereby reduce the generation of unacceptable data, problems identified by assessment procedures are resolved at the lowest possible management level. Problems that cannot be resolved at this level are reported to the Quality Assurance Coordinator (QAC) for resolution. The QAC determines the management level at which the problem can best be resolved, and notifies the appropriate manager. Weekly progress reports detail all problems and subsequent resolutions.

Steps included in the corrective action system include:

1. Defining the problem;
2. Assigning responsibility for problem investigation;
3. Investigating and determining the cause of the problem;
4. Assigning responsibility for problem resolution; and
5. Verifying that the resolution has corrected the problem.

Problems requiring corrective action may not be easy to identify or define. The situation may not be producing out-of-control data, but simply producing data not of the quality desired. The project manager, section managers, analysts, and the quality assurance staff combine efforts in solving long-term unsatisfactory situations.

All corrective actions are documented by Quality Assurance. Final corrective action reports, which relate to a particular lot analysis, are included in the data package for that lot.

11.0 QUALITY CONTROL REPORTS

DCL provides weekly quality assurance evaluation reports to USAEC, in conjunction with weekly interim technical reports from project management. The QA reports include charts and tables of quality control data, a control chart checklist delineating contracts and lots, and copies of Corrective Action Reports (CARs). These CARs include explanations of analytical or quality control problems and discussions of the corrective actions taken to alleviate those problems. Observations of data trends or situations which could develop into problems are also discussed in this report, as well as preliminary acceptance or rejection of analytical data.

APPENDIX E

**ENVIRONMENTAL SCIENCE & ENGINEERING, INC.
MASTER QUALITY ASSURANCE PLAN
FOR
U.S. ARMY ENVIRONMENTAL CENTER**

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Contract No. DAAA15-87-D-0015
Delivery Orders 0066 and 0067

MASTER QUALITY ASSURANCE PLAN

February, 1992

Distribution limited to U.S. Government Agencies only for protection of privileged information evaluating another command: April, 1990. Requests for this document must be referred to: Commander, U.S. Army Toxic and Hazardous Materials Agency, Aberdeen Proving Ground, MD. 21010-5401

Prepared for:

U.S. ARMY TOXIC AND HAZARDOUS MATERIALS AGENCY
Aberdeen Proving Ground, MD. 21010-5401

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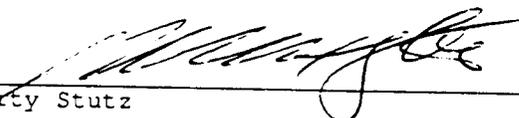
MASTER QUALITY ASSURANCE PROJECT PLAN

FOR ANALYTICAL SERVICES
PROVIDED TO

UNITED STATES ARMY TOXIC AND HAZARDOUS MATERIALS AGENCY (USATHAMA)

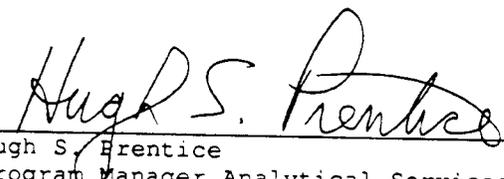
THROUGH VARIOUS CONTRACTS AND SUBCONTRACTS

APPROVALS:



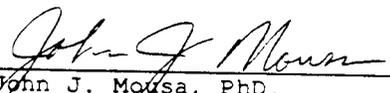
Marty Stutz
Contractor Officer Representative, USATHAMA

4 MAR 92
Date



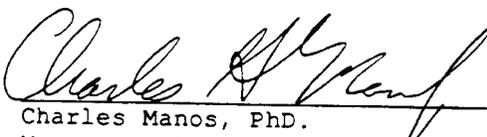
Hugh S. Prentice
Program Manager Analytical Services, ESE, Inc.

2/28/92
Date



John J. Mousa, PhD.
Manager Analytical Services, ESE, Inc.

2/28/92
Date



Charles Manos, PhD.
Manager, QA Division, ESE, Inc.

2/28/92
Date



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2/28/92
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CYANIDE IN WATER

<u>SHORT NAME</u>	<u>METHOD</u>	<u>STORET</u>	<u>LONG NAME</u>	USATHAMA			CLP
				<u>CRL</u>	<u>UCR</u>	<u>SLOPE</u>	<u>CRL</u>
CYN	TF18 (335.2)	99315	CYANIDE	2.50	50.0	1.00	10

Number in () is the EPA Method Number.

TF22 (353.2)
NITRATE/NITRITE IN WATER BY TECHNICON

<u>SHORT NAME</u>	<u>STORET</u>	<u>LONG NAME</u>	<u>CRL</u>	<u>UCR</u>	<u>SLOPE</u>
NIT	630	NITRATE PLUS NITRITE	10	200	0.999

Number in () is the EPA Method Number.

TT10 (300.0)
ANIONS IN WATER BY IC

<u>SHORT NAME</u>	<u>STORET</u>	<u>LONG NAME</u>	<u>CRL</u>	<u>UCR</u>	<u>SLOPE</u>
BR	71870	BROMIDE	1000	25000	1.03
CL	98555	CHLORIDE	2120	30000	0.911
F	98556	FLUORIDE	1230	10000	1.03
SO4	98581	SULFATE	10000	600000	1.00

Number in () is the EPA Method Number.

UF03
NITROCELLULOSE IN WATER BY TECHNICON

<u>SHORT NAME</u>	<u>STORET</u>	<u>LONG NAME</u>	<u>CRL</u>	<u>UCR</u>	<u>SLOPE</u>
NC	99574	NITROCELLULOSE	553	6000	0.826

There is not an EPA Method Number Available for this method.

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HSL METALS IN WATER

SHORT NAME	METHOD	STORET	LONG NAME	USATHAMA			CLP
				CRL	UCR	SLOPE	CRL
AL	SS10	1105	ALUMINUM	141	45000	0.891	200
SB	(200.7)	1097	ANTIMONY	38	6000	0.844	60
BA		1007	BARIUM	5	10000	1.08	200
BE		1012	BERYLLIUM	5	1000	0.893	5
CA		82032	CALCIUM	500.0	20000	0.974	5000
CD		1027	CADMIUM	4	5000	1.000	5
CR		1034	CHROMIUM	6	5000	1.010	10
CO		1037	COBALT	25	50000	0.879	50
CU		1042	COPPER	8.1	10000	0.985	25
FE		1045	IRON	42.7	500000	0.907	100
MG		82033	MAGNESIUM	500	20000	0.988	5000
MN		1055	MANGANESE	2.75	2000	0.934	15
NI		1067	NICKEL	34.3	15000	0.860	40
K		82034	POTASSIUM	375	12500	0.881	5000
NA		82035	SODIUM	500	50000	0.954	5000
ZN		1092	ZINC	21.1	20000	0.949	10
PB	SD20 (239.2)	1051	LEAD	1.26	100	0.922	5
AG	SD23 (272.2)	1077	SILVER	0.25	10	1.06	10
V	SD19 (200.7)	1087	VANADIUM	3.82	200	0.909	40
AS	SD22 (206.2)	1002	ARSENIC	2.54	100	0.938	10
SE	SD21 (270.2)	1147	SELENIUM	3.02	100	0.939	5
TL	SD09 (279.2)	1059	THALLIUM	6.99	25	0.950	10
HG	SB01 (245.1)	71900	MERCURY	0.243	10	1.03	.2

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UH14 (615)
HERBICIDES IN WATER BY HPLC

<u>SHORT NAME</u>	<u>STORET</u>	<u>LONG NAME</u>	<u>CRL</u>	<u>UCR</u>	<u>SLOPE</u>
245TP	39760	SILVEX	0.181	1.36	0.931
24D	39730	2,4-D	0.802	2.52	0.646

UL04
ORGANOSULFUR PESTICIDES IN WATER BY GCFF

<u>SHORT NAME</u>	<u>STORET</u>	<u>LONG NAME</u>	<u>CRL</u>	<u>UCR</u>	<u>SLOPE</u>
BTZ	81512	BENZOTHAZOLE	2.11	42.2	0.927
CPMS	98562	P-CHLOROPHENYLMETHYL SULFIDE	1.26	25.3	0.824
CPMSO	98561	P-CHLOROPHENYLMETHYL SULFOXIDE	4.23	106	0.743
CPMSO2	98560	P-CHLOROPHENYLMETHYL SULFONE	4.72	106	0.866
DITH	98563	1,4-DITHIANE	1.11	22.2	0.831
DMDS	81580	DMDS	1.14	22.8	0.801
OXAT	98564	1,4-OXATHIANE	1.98	39.5	0.829

There is not a EPA Method Number available for this method.

UN07 (622)
ORGANONITROGEN/ORGANOPHOSPHORUS PESTICIDES IN WATER BY GC-NPD

<u>SHORT NAME</u>	<u>STORET</u>	<u>LONG NAME</u>	<u>CRL</u>	<u>UCR</u>	<u>SLOPE</u>
DDVP	99897	VAPONA	0.25	5.0	0.884
ATZ	39033	ATRAZINE	0.512	5.0	1.04
MLTHN	39530	MALATHION	0.25	5.0	0.999
PRTHN	39540	PARATHION	0.25	5.0	0.983
SUPONA	98632	SUPONA	0.25	4.7	1.00

UN08 (607)
NITROSAMINES IN WATER BY GC-NPD

<u>SHORT NAME</u>	<u>STORET</u>	<u>LONG NAME</u>	<u>CRL</u>	<u>UCR</u>	<u>SLOPE</u>
24DNT	34611	2,4-DINITROTOLUENE	0.341	5.0	0.979
26DNT	77541	2,6-DINITROTOLUENE	0.250	5.0	0.964
NB	34447	NITROBENZENE	0.285	5.0	1.000
NNDNPA	34428	N-NITROSO, DI-N-PROPYLAMINE	0.294	5.0	1.030
NNDPA	34433	N-NITROSODIPHENYLAMINE	0.250	5.0	0.968

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PESTICIDES/PCBs IN WATER

SHORT NAME	METHOD	STORET	LONG NAME	USATHAMA			CLP
				CRL	UCR	SLOPE	CRDL
ABHC	UH13	39337	BHC, A	0.038	0.638	0.941	0.05
AENSLF	(608)	34361	ENDOSULFAN, A	0.022	0.575	1.020	0.05
ALDRN		39330	ALDRIN	0.092	0.606	0.756	0.05
BBHC		39338	BHC, B	0.018	0.600	0.891	0.05
BENSLF		34356	ENDOSULFAN, B	0.013	0.575	1.060	0.10
DBHC		34259	BHC, D	0.029	0.594	1.150	0.05
DLDRN		39380	DIELDRIN	0.018	0.600	1.040	0.10
ENDRN		39390	ENDRIN	0.018	0.594	1.320	0.10
ENDRNA		34366	ENDRIN	0.026	0.713	1.000	-
			ALDEHYDE				
ESFSO4		34351	ENDOSULFAN	0.079	0.675	0.961	0.10
			SULFATE				
HPCL		39410	HEPTACHLOR	0.042	0.619	0.849	0.05
HPCLE		39420	HEPTACHLOR	0.024	0.613	1.010	0.05
			EPOXIDE				
LIN		39782	LINDANE	0.051	0.619	0.964	0.05
MEXCLR		39480	METHOXYCHLOR	0.057	1.160	1.260	0.5
PPDDD		39310	DDD-PP	0.019	0.581	1.170	0.10
PPDDE		39320	DDE-PP	0.025	0.675	0.999	0.10
PPDDT		39300	DDT-PP	0.034	0.663	0.949	0.10
TXPHEN		39400	TOXAPHENE	1.350	11.60	1.00	1.0
CLDAN		39350	CHLORDANE+	0.246	5.300	0.962	-
PCB016	UH02	98140	PCB 1016	0.160	6.4	0.826	0.05
PCB221*	(608)	98351	PCB 1221	0.160	6.4	0.826	0.05
PCB232*		98352	PCB 1232	0.160	6.4	0.826	0.05
PCB242*		98353	PCB 1242	0.190	6.3	0.925	1.0
PCB248*		98802	PCB 1248	0.190	6.3	0.925	1.0
PCB254*		98354	PCB 1254	0.190	6.3	0.925	1.0
PCB260		98139	PCB 1260	0.190	6.3	0.925	1.0
KEND@			ENDRIN KETONE				0.10

* The detection limits for these analytes are uncertified.

+ The CLP Target Compound List has both alpha, and gamma-Chlordane listed separately, the method above is only certified for total Chlordane.

@ This analyte is done with method UM18 (extractable organics by GC/MS)
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UT02
IMPA, MPA IN WATER BY IC

<u>SHORT NAME</u>	<u>STORET</u>	<u>LONG NAME</u>	<u>CRL</u>	<u>UCR</u>	<u>SLOPE</u>
FC2A		FLUOROACETIC ACID	100	9000	1.006
IMPA		ISOPROPYLMETHYL PHOSPHONIC ACID	100	9000	0.991
MPA		METHYLPHOSPHONIC ACID	128	9000	1.023

No EPA Method Number is available at this time.

UW17
NITROGUANIDINE IN WATER BY HPLC

<u>SHORT NAME</u>	<u>STORET</u>	<u>LONG NAME</u>	<u>CRL</u>	<u>UCR</u>	<u>SLOPE</u>
NGD	97796	NITROGUANIDINE	30.9	620	0.956

UW18
PHENOLS IN WATER BY HPLC

<u>SHORT NAME</u>	<u>STORET</u>	<u>LONG NAME</u>	<u>CRL</u>	<u>UCR</u>	<u>SLOPE</u>
246TCP	36421	2,4,6-TRICHLOROPHENOL	1.9	111	0.743
24DCLP	34601	2,4-DICHLOROPHENOL	0.617	115	0.739
2CLP	34586	2-CHLOROPHENOL	1.69	88	0.623
2NP	34591	2-NITROPHENOL	0.363	27.2	0.684
46DN2C	34657	2-METHYL-4,6-DINITROPHENOL	0.295	25.8	0.700
4CL3C	34452	4-CHLORO-3-METHYL PHENOL	5.56	69.7	0.693
4NP	34646	4-NITROPHENOL	0.27	31.9	0.762
PCP	39032	PENTACHLOROPHENOL	1.49	16.4	0.891
PHENOL	34694	PHENOL	7.99	103.0	0.681

UW19
PETN/NITROGLYSERIN IN WATER BY HPLC

<u>SHORT NAME</u>	<u>STORET</u>	<u>LONG NAME</u>	<u>CRL</u>	<u>UCR</u>	<u>SLOPE</u>
NG	99808	NITROGLYCERIN	10.0	200	1.04
PETN	99620	PENTAERYTHRITOL TETRANITRATE	20.0	400	1.05

There is no EPA Method Number available for this method.

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U002 (601/602)
VOLATILE ORGANICS IN WATER BY GC

<u>SHORT NAME</u>	<u>STORET</u>	<u>LONG NAME</u>	<u>CRL</u>	<u>UCR</u>	<u>SLOPE</u>
111TCE +	34506	1,1,1-TRICHLOROETHANE	2.9	50	1.08
112TCE	34511	1,1,2-TRICHLOROETHANE	0.332	49	1.10
11DCE +	34501	1,1-DICHLOROETHENE	0.393	51	1.06
11DCLE	34496	1,1-DICHLOROETHANE	0.334	49.5	1.03
12DCLE	34531	1,2-DICHLOROETHANE	2.95	49	1.08
12DCLP	34541	1,2-DICHLOROPROPANE	3.16	49	1.09
2CLEVE	34576	2-CHLOROETHYL VINYL ETHER	22.1	49.5	1.11
BRDCLM	32101	BROMODICHLOROMETHANE	3.06	50.5	1.11
C13DCP	34704	CIS-1,3-DICHLOROPROPENE	3.23	48.5	1.08
C2H3CL	39175	VINYL CHLORIDE	2.07	50	1.16
C2H5CL	34311	CHLOROETHANE	1.6	50	1.24
C6H6 +	34030	BENZENE	0.651	49	1.07
CCL3F	34488	TRICHLOROFLUOROMETHANE	0.503	51.5	1.06
CCL4	32102	CARBON TETRACHLORIDE	2.81	49	1.03
CH2CL2	34423	METHYLENE CHLORIDE	3.1	49	1.05
CH3BR	34413	BROMOMETHANE	2.68	50	1.20
CH3CL	34418	CHLOROMETHANE	1.98	50	1.04
CHBR3	32104	BROMOFORM	4.03	52	1.06
CHCL3 +	32106	CHLOROFORM	1.26	50	1.05
CLC6H5 +	34301	CHLOROBENZENE	0.582	50.5	0.988
DBRCLM	34306	DIBROMOCHLOROMETHANE	0.352	51.5	1.10
ETC6H5 +	34371	ETHYLBENZENE	0.857	49.5	0.999
MEC6H5 +	34010	TOLUENE	0.716	49.5	0.990
T13DCP	34699	TRANS-1,3-DICHLOROPROPENE	0.326	49.5	1.10
TCLEA	34516	1,1,2,2-TETRACHLOROETHANE	1.09	52	0.935
TCLEE +	34475	TETRACHLOROETHENE	0.677	51	0.996
TRCLE +	39180	TRICHLOROETHENE	3.59	50	1.05
XYLEN	81551	XYLENE	1.73	102	0.995
13DCLB	34566	1,3-DICHLOROBENZENE	1.34	50	0.921
13DMB	77348	1,3-DIMETHYLBENZENE	1.56	49.5	1.00
CCL2F2	34668	DICHLORODIFLUOROMETHANE	2.04	50	1.25
CL2BZ	81524	DICHLOROBENZENE	6.22	111	0.942
T12DCE	34546	TRANS-1,2-DICHLOROETHENE	0.427	49	1.04

+ These compounds are used as control spikes for this method.
CRL CERTIFIED REPORTING LIMIT IN (micrograms per liter)
UCR UPPER CERTIFIED RANGE IN (micrograms per liter)
SLOPE REPRESENTS AVERAGE ACCURACY OVER THE CERTIFIED RANGE

The numbers in () are the EPA Method Numbers for this method.

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METHOD LM18 (8270); EXTRACTABLE ORGANICS IN SOIL BY GC/MS FOR BOTH
 PRIORITY POLLUTANTS AND HAZARDOUS SUBSTANCE LIST COMPOUNDS

SHORT NAME	STORET	LONG NAME	POLL.	HAZARDOUS SUBST. LIST	USATHAMA CRL	USATHAMA UCR	USATHAMA SLOPE	CLP CRDL
124TCB	99492	1,2,4-TRICHLOROBENZENE	Y	Y	0.04	13	0.801	0.3
12DCLB	99470	1,2-DICHLOROBENZENE	Y	Y	0.11	13	0.734	0.3
13DCLB	99472	1,3-DICHLOROBENZENE	Y	Y	0.13	13	0.724	0.3
14DCLB	99469	1,4-DICHLOROBENZENE	Y	Y	0.098	13	0.715	0.3
245TCP	97732	2,4,5-TRICHLOROPHENOL	N	Y	0.10	13	0.897	2
24DCLP	99498	2,4-DICHLOROPHENOL	Y	Y	0.18	13	0.909	0.3
24DMPN	99499	2,4-DIMETHYLPHENOL	Y	Y	0.69	1.3	0.917	0.3
24DNP	99495	2,4-DINITROPHENOL	Y	Y	1.2	6.7	0.816	2
24DNT	99474	2,4-DINITROTOLUENE	Y	Y	0.14	13	0.936	0.3
2CLP	99497	2-CHLOROPHENOL	Y	Y	0.06	13	0.745	0.3
2CNAP	99464	2-CHLORONAPHTHALENE	Y	Y	0.036	13	0.847	0.3
2MNAP	97733	2-METHYLNAPHTHALENE	N	Y	0.049	6.7	0.828	0.3
2MP	97461	2-METHYLPHENOL	N	Y	0.029	1.3	0.490	0.3
2NANIL	97728	2-NITROANILINE	N	Y	0.062	13	0.865	2
2NP	99495	2-NITROPHENOL	Y	Y	0.14	13	0.915	0.3
33DCBD	99471	3,3-DICHLOROBENZIDINE	Y	Y	6.3	13	0.633	0.7
3NANIL	9772	3-NITROANILINE	N	Y	0.45	13	0.909	2
46DN2C	99686	2-METHYL-4,6-DINITROPHENOL	Y	Y	0.55	13	1.060	2
4BRPPE	99462	4-BROMOPHENYLPHENYL ETHER	Y	Y	0.033	6.7	0.921	0.3
4CL3C	99683	3-METHYL-4-CHLOROPHENOL	Y	Y	0.095	13	0.894	0.3
4CLPPE	99465	4-CHLOROPHENYLPHENYL ETHER	Y	Y	0.033	13	0.826	0.3
4MP	97460	4-METHYLPHENOL	N	Y	0.24	1.3	0.439	0.3
4NANIL	97730	4-NITROANILINE	N	Y	0.41	13	0.739	2
4NP	99496	4-NITROPHENOL	Y	Y	1.4	33	0.921	2
ANAPNE	99450	ACENAPHTHENE	Y	Y	0.036	13	0.826	0.3
ANAPYL	99451	ACENAPHTHYLENE	Y	Y	0.033	6.7	0.881	0.3
ANTRC	99452	ANTHRACENE	Y	Y	0.033	13	0.870	0.3
B2CEXM	99459	BIS(2-CHLOROETHOXY) METHANE	Y	Y	0.059	13	0.863	0.3
B2CIPE	99461	BIS(2-CHLOROISOPROPYL) ETHER	Y	Y	0.2	13	0.819	0.3
B2CLEE	99458	BIS(2-CHLOROETHYL) ETHER	Y	Y	0.033	6.7	0.802	0.3

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UW22
THIODIGLYCOL IN WATER BY GCFP

<u>SHORT NAME</u>	<u>STORET</u>	<u>LONG NAME</u>	<u>CRL</u>	<u>UCR</u>	<u>SLOPE</u>
TDGCL	99797	THIODIGLYCOL	48.8	4880	0.687
TDGCLA	97399	THIODIGLYCOLIC ACID	52.7	1780	0.930

No EPA Method Number is available at this time.

UW32 (609)
NITROAROMATICS IN WATER BY HPLC

<u>SHORT NAME</u>	<u>LONG NAME</u>	<u>CRL</u>	<u>UCR</u>	<u>SLOPE</u>
HMX	CYCLOTETRAMETHYLENE TETRANITRAMINE	1.21	120.8	1.00
RDX +	CYCLONITE	1.17	116.8	0.952
135TNB+	1,3,5-TRINITROBENZENE	0.449	59.2	0.993
13DNB	1,3-DINITROBENZENE	0.611	55.0	0.95
NB +	NITROBENZENE	0.645	29.0	0.919
TETRYL	NITRAMINE	1.56	107.5	1.00
246TNT+	2,4,6-TRINITROTOLUENE	0.635	112.0	0.911
26DNT	2,6-DINITROTOLUENE	0.0738	24.4	0.985
24DNT +	2,4-DINITROTOLUENE	0.0637	21.2	0.929
4A26DT	4-AMINO-2,6-DINITROTOLUENE	1.57	20.8	1.12
2A46DT	2-AMINO-4,6-DINITROTOLUENE	0.158	22.0	0.973
2NT	2-NITROTOLUENE	0.406	122.6	0.936
3NT	3-NITROTOLUENE	1.40	116.8	0.934
4NT	4-NITROTOLUENE	1.11	120.4	0.913

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LN05 (8140)
ORGANONITROGEN/ORGANOPHOSPHORUS PESTICIDES IN SOIL BY GCNP

<u>SHORT NAME</u>	<u>STORET</u>	<u>LONG NAME</u>	<u>CRL</u>	<u>UCR</u>	<u>SLOPE</u>
ATZ	98655	ATRAZINE	0.25	2.00	1.12
DDVP	98646	VAPONA	0.452	5.00	1.02
MLTHN	98648	MALATHION	0.580	5.00	1.17
PRTHN	98658	PARATHION	0.733	5.00	1.23
SUPONA	98656	SUPONA	0.25	5.00	1.28

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LH11 (8150)
HERBICIDES IN SOIL BY GC-EC

<u>SHORT NAME</u>	<u>STORET</u>	<u>LONG NAME</u>	<u>CRL</u>	<u>UCR</u>	<u>SLOPE</u>
245TP	97483	SILVEX	8.5	109	0.907
24D	99239	2,4-D	17.7	202	1.080

Number in () is the EPA Method Number.

LL03
ORGANOSULFUR PESTICIDES IN SOIL BY GCFP

<u>SHORT NAME</u>	<u>STORET</u>	<u>LONG NAME</u>	<u>CRL</u>	<u>UCR</u>	<u>SLOPE</u>
BTZ	97302	BENZOTHAZOLE	1.08	13.2	0.788
CPMS	98653	4-CHLOROPHENYLMETHYL SULFIDE	1.08	21.6	0.999
CPMSO	98654	4-CHLOROPHENYLMETHYL SULFOXIDE	2.25	45.0	1.02
CPMSO2	98703	4-CHLOROPHENYLMETHYL SULFONE	2.37	47.4	0.790
DITH	98650	1,4-DITHIANE	1.47	11.4	0.916
DMDS	98697	DIMETHYLSULFIDE	0.69	13.8	0.946
OXAT	98644	1,4-OXATHIANE	0.85	17.1	0.930

There is no EPA Number available for this method.

LN01
NITROSAMINES IN SOIL BY GCNP

<u>SHORT NAME</u>	<u>STORET</u>	<u>LONG NAME</u>	<u>CRL</u>	<u>UCR</u>	<u>SLOPE</u>
24DNT	98575	2,4-DINITROTOLUENE	0.092	1.00	0.737
26DNT	98573	2,6-DINITROTOLUENE	0.055	1.00	0.736
NB	99485	NITROBENZENE	0.0962	5.00	0.751
NNDMA	99486	N-NITROSODIMETHYLAMINE	0.108	0.50	0.330
NNDNPA	99487	N-NITROSODIPROPYLAMINE	0.231	1.00	0.561
NNDPA	99488	N-NITROSODIPHENYLAMINE	0.163	5.00	0.938

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LW12 (8090)
NITROAROMATICS IN SOIL BY HPLC

<u>SHORT NAME</u>	<u>LONG NAME</u>	<u>ACCU</u>		
		<u>CRL</u>	<u>UCR</u>	<u>SLOPE</u>
135TNB	1,3,5-TRINITROBENZENE	0.488	24.4	0.991
13DNB	1,3-DINITROBENZENE	0.496	24.8	0.952
246TNT	2,4,6-TRINITROTOLUENE	0.456	22.8	1.01
24DNT	2,4-DINITROTOLUENE	0.424	21.2	0.938
26DNT	2,6-DINITROTOLUENE	0.524	26.2	0.977
HMX	CYCLOTETRAMETHYLENE TETRANITRAMINE	0.666	33.3	1.000
NB	NITROBENZENE	2.41	27.4	0.793
NG	NITROGLYCERIN	4.00	200.0	0.931
PETN	PETN	4.00	80.0	0.969
RDX	CYCLONITE	0.587	21.9	0.929
TETRYL	NITRAMINE	0.731	20.2	1.130

No EPA Method Number is available at this time.

LW15
NITROGUANIDINE IN SOIL BY HPLC

<u>SHORT NAME</u>	<u>STORET</u>	<u>LONG NAME</u>	<u>CRL</u>	<u>UCR</u>	<u>SLOPE</u>
NG	9779	NITROGUANIDINE	0.475	9.5	0.901

There is no EPA Number available for this method.

LW18
THIODIGLYCOL IN SOIL BY HPLC

<u>SHORT NAME</u>	<u>STORET</u>	<u>LONG NAME</u>	<u>CRL</u>	<u>UCR</u>	<u>SLOPE</u>
CLC2A	97285	CHLOROACETIC ACID	18.0	302	0.837
TDGCL	99798	THIODIGLYCOL	3.94	102	1.07

No EPA Method Number is available at this time.

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LO02 (8010/8020)
VOLATILE ORGANICS IN SOIL BY GC

<u>SHORT NAME</u>	<u>STORET</u>	<u>LONG NAME</u>	<u>CRL</u>	<u>UCR</u>	<u>SLOPE</u>
111TCE +	98692	1,1,1-TRICHLOROETHANE	0.04	5.0	0.988
112TCE	98693	1,1,2-TRICHLOROETHANE	0.081	5.0	0.957
11DCE +	98789	1,1-DICHLOROETHENE	0.051	5.0	0.941
11DCLE	98683	1,1-DICHLOROETHANE	0.055	5.0	0.948
12DCLE	98684	1,2-DICHLOROETHANE	0.071	5.0	0.902
12DCLP	98790	1,2-DICHLOROPROPANE	0.043	5.0	1.00
2CLEVE	98796	2-CHLOROETHYL VINYL ETHER	0.075	5.0	0.799
BRDCLM	98783	BROMODICHLOROMETHANE	0.047	5.0	0.921
C13DCP	98791	CIS-1,3-DICHLOROPROPENE	0.062	5.0	0.860
C2H3CL	98795	VINYL CHLORIDE	0.031	5.0	0.921
C2H5CL	98786	CHLOROETHANE	0.029	5.0	0.961
C6H6 +	98699	BENZENE	0.085	5.0	0.952
CCL3F	98794	TRICHLOROFLUOROMETHANE	0.037	5.0	0.929
CCL4	98680	CARBON TETRACHLORIDE	0.044	5.0	0.965
CH2CL2	98689	METHYLENE CHLORIDE	0.083	5.0	0.956
CH3BR	98785	BROMOMETHANE	0.031	5.0	0.899
CH3CL	98787	CHLOROMETHANE	0.18	5.0	0.933
CHBR3	98784	BROMOFORM	0.031	5.0	0.856
CHCL3 +	98682	CHLOROFORM	0.038	5.0	0.969
CLC6H5 +	98681	CHLOROBENZENE	0.026	5.0	0.925
DBRCLM	98788	DIBROMOCHLOROMETHANE	0.081	5.0	0.957
ETC6H5 +	98688	ETHYLBENZENE	0.062	5.0	1.03
MEC6H5 +	98691	TOLUENE	0.028	5.0	0.970
T13DCP	98792	TRANS-1,3-DICHLOROPROPENE	0.081	5.0	0.957
TCLEA	98793	1,1,2,2-TETRACHLOROETHANE	0.045	5.0	0.906
TCLEE +	98690	TETRACHLOROETHENE	0.045	5.0	0.906
TRCLE +	98694	TRICHLOROETHENE	0.049	5.0	0.972
XYLEN	97353	XYLENE	0.086	10	1.01
13DCLB	99468	1,3-DICHLOROBENZENE	0.032	5.0	1.01
13DMB	98799	1,3-DIMETHYLBENZENE/ M-XYLENE	0.056	5.0	1.01
CCL2F2	97015	DICHLORODIFLUOROMETHANE	0.032	5.0	0.921
CL2BZ	98803	DICHLOROBENZENE	0.06	10	0.990
T12DCE	98687	TRANS-1,2-DICHLOROETHYLENE	0.063	5.0	0.948

+ THESE ARE THE CONTROL ANALYTES FOR THIS METHOD
CRL CERTIFIED REPORTING LIMIT IN (micrograms per gram)
UCR UPPER CERTIFIED RANGE IN (micrograms per gram)
SLOPE REPRESENTS AVERAGE ACCURACY OVER THE CERTIFIED RANGE

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1.0 INTRODUCTION

A Quality Assurance Project Plan (QAPP) is required by the U.S. Army Toxic and Hazardous Materials Agency (USATHAMA) and the U.S. Environmental Protection Agency (EPA) for all environmental monitoring and measurement efforts mandated or supported by EPA. The QAPP documents the policies, organization, objectives, functional activities, and procedures for the identification and documentation of the precision, accuracy, completeness, and representativeness of the data produced.

USATHAMA has documented QA requirements for laboratories performing analyses in support of environmental programs (USATHAMA 1985, 2nd ed. and January 1990). The ESE Analytical Services Division performs analytical services for USATHAMA by several contractual avenues: ESE has a prime contract with USATHAMA (Contractor Laboratory Analytical Support Services - CLASS) managed by the Analytical Services Division; ESE has subcontracts with other consulting firms for their prime contracts with USATHAMA (RIFS and ATEPS Contracts); ESE has prime contracts with USATHAMA (RIFS and ATEPS Contracts) and the Analytical Services Division serves as Task Managers.

Analytical methods and QA/QC requirements are consistent for all USATHAMA work no matter which contractual avenue is used. Therefore the ESE Analytical Services Division and Quality Assurance Division have prepared this Master QA plan to be used as an appendix in support of installation specific USATHAMA project plans. This Master QA plan has been prepared following the organizational guidelines contained in the U.S. Environmental Protection Agency guidelines QAMS - 005/80. Project or installation specific requirements different from the requirements outlined in this Master QA plan will be coordinated with the ESE Analytical Services and QA Divisions. The different requirements will be documented in the appropriate USATHAMA project or installation plans and a copy provided to the ESE Analytical Services and QA Divisions for implementation.

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10. Acceptance of charts by USATHAMA or response to comments,
11. Review and validation of chemical analysis
12. Data Management procedures for submitting chemical data to USATHAMA data base,
13. Submission of final files defining work performed for the delivery order or contract effort (Class Order Lot Assignment program - COLA),
14. Preparation and submission of final delivery order reports or project files which include all project related communications, chemical analysis lot folders, data reports, and verification of final USATHAMA data base with what ESE submitted.

1.2 SITE DESCRIPTION

Each CLASS delivery order and RATS or ATEPS work order will be site specific. Actual descriptions of the site are not required in this Master QAPP.

1.3 PROGRAM ORGANIZATION AND RESPONSIBILITIES

This QAPP functions according to the USATHAMA QA Program. The ESE Analytical Services Division is an approved USATHAMA laboratory monitored by USATHAMA project chemists in the Technical Support Division. The ESE Analytical Services and QA Divisions have organized a program team to support the analytical needs of various USATHAMA contracts. Only key personnel have been identified since some shifting of personnel can occur. The program organization chart is shown in Fig. 1.3-1. The Quality Assurance Division which is independent of the Analytical Services Division has an assigned program team to perform specified duties. The Analytical Services Division has in addition its own QA supervisor. The QA/QC responsibilities of each of the participants are outlined in the following subsections.

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Some appendices of this Master QA plan contain specific protocols unique to the USATHAMA CLASS contract. These specific ESE/USATHAMA protocols are unable to be used for the other contracts because of the contract structure. The objective of USATHAMA CLASS contract and all USATHAMA work is to provide defined analytical services by certified laboratories to meet data quality and schedule requirement for remedial investigation projects, emergency response situations, or other required projects. Since the CLASS contract supplies work through separate delivery orders and the work could go to different laboratories, this Master QA plan has been prepared document procedures so analytical work can be assigned on short notices to approved laboratories and avoid costly delays.

1.1 SCOPE OF WORK AND SCHEDULE

It is not always known when CLASS delivery orders or other contract work will be awarded therefore a specific scope of work and schedule cannot be defined in this Master QA Plan. Work scopes are defined in each CLASS delivery order with a tentative schedule, however, schedules are finalized through communications with the USATHAMA Contracting Officer's Representative (COR), ESE Program Manager or Task Manager, and the sampling team. Specific scopes of work and schedules for work performed through the other contracts are defined in the appropriate work plans.

Each CLASS delivery order and other contract effort has similar work components that can be defined and scheduled. This Master QA plan defines the required standard procedures. The work components are:

1. Management and administration,
2. Pre-sampling organization,
3. Defining field groups, preparing sampling kits,
4. Shipment of kits and instructions to field personnel,
5. Communications with field team,
6. Field team communications and delivery of samples to the laboratory,
7. Laboratory communications to USATHAMA COR defining sample receipt,
8. Chemical analysis,
9. Submittal of QC charts to USATHAMA for approval,

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1.3.1 ANALYTICAL SERVICES PROGRAM MANAGER

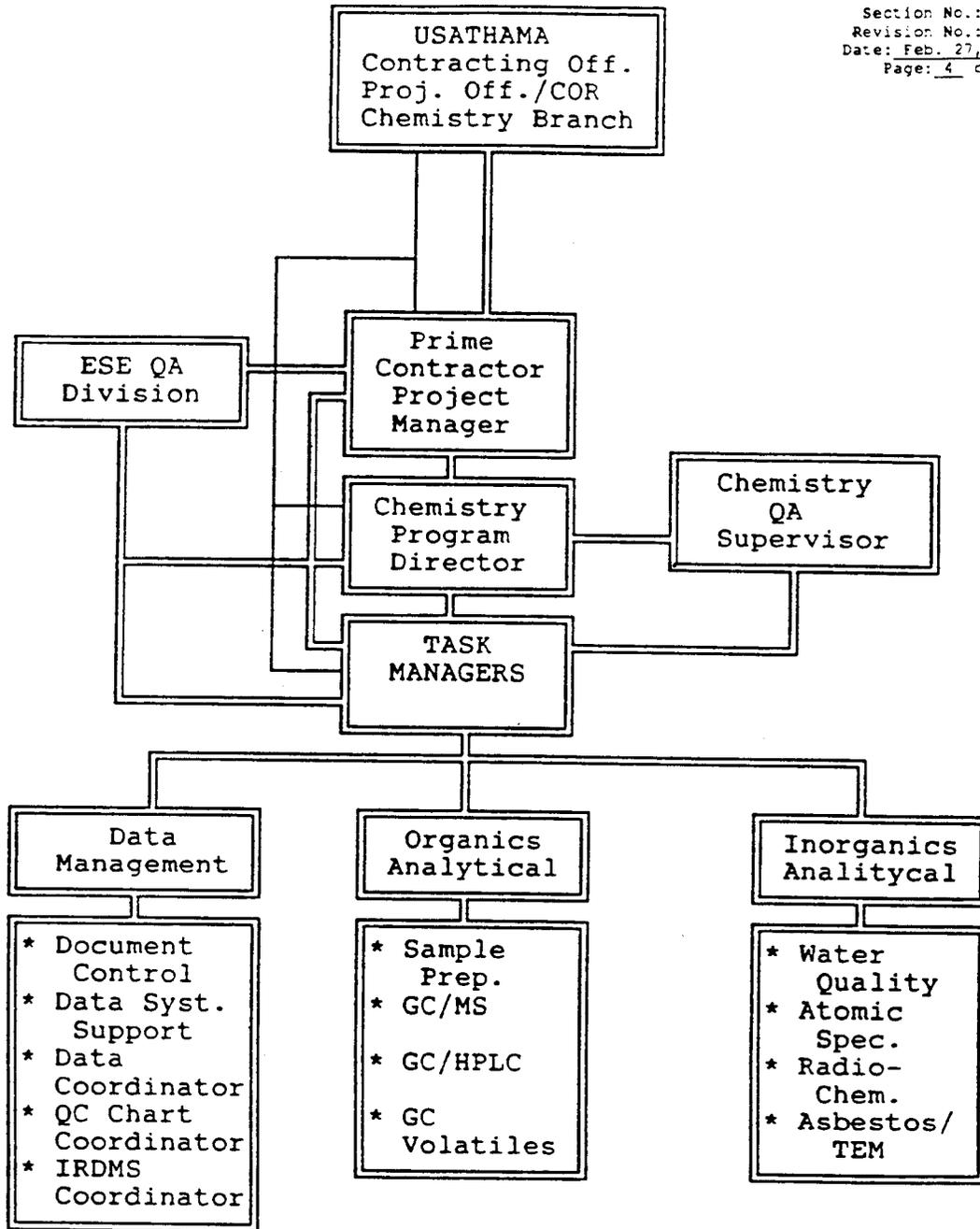
The Analytical Services Program Manager, Mr. Hugh Prentice serves in the QA function as a primary technical reviewer of project deliverables. Responsibilities also include assignment of Task Managers or serving as a Task Manager, review of progress of data deliverables such as QC chart submittals and validation of lot folders, project costs and profitability, review and monitoring of corrective actions, liaison with the USATHAMA CLASS COR and Technology Division Chemists. He works closely with the Data Management Task Manager and support personnel to monitor program progress. The Program Manager also provides authority in support of the Project QA Staff in the performance of his/her duties.

1.3.2 PROGRAM QA SUPERVISOR AND QA STAFF

The Program QA Supervisor, Joe Owusu-Yaw, monitors the conduct of all USATHAMA analytical efforts. The Program QA Supervisor is not directly subordinate to anyone responsible for analysis and reports only to the ESE USATHAMA Program Director or the ESE Analytical Services Program Director. The Program QA Supervisor oversees the performance of the QA Staff in the QC chart submittal and lot folder validation process of the ESE laboratory data. The QA Staff (Program QA Supervisor and any QA/QC Coordinator) monitors the chemical analysis effort in the laboratory to ensure compliance with USATHAMA QA requirements and those of this Master QA Plan. The QA Staff does not necessarily audit and monitor field sampling activities for CLASS contract delivery orders, but does for other USATHAMA contracts.

The Program QA Supervisor directly supervises the performance of the QA/QC Coordinator and may audit the performance of any required subcontractor to ensure that the requirements of the QAPP are followed in sampling and analysis activities. The Program QA Supervisor directs the development of the QAPP and approves any deviations or changes to QA/QC requirements. USATHAMA Chemistry Branch and the Contracting Officer's Representative (COR) must approve any changes to the QA/QC program. The project QA Supervisor maintains liaison with the Program Team and the USATHAMA Chemistry Branch.

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== Formal Lines of Communications
— Informal Lines of Communications

Source: ESE, 1990.

Figure 1.3-1 PROGRAM ORGANIZATIONAL CHART

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1.3.3 TASK MANAGER

The Analytical Services Division Task Manager is responsible for effective day-to-day coordination of all analytical activity. He/she is responsible for review and approval of all chemical analysis data generated for the task. The Task Manager's QA/QC responsibilities are to ensure that QC requirements of the QAPP are implemented; provide guidance and technical support in resolution of QC problems (review QC chart corrective actions); support QA/QC preparation of control samples; and provide guidance in preparation of analytical lots to ensure efficient, comprehensive analysis of all required parameters. The Task Manager also provides additional authority, when needed, to support the Project QA Staff in analytical matters and must approve all revisions of the QAPP regarding analytical activities. Several Task Managers are available they are Joe J. Vondrick, Jackie Hargrove, David Greer, Hugh Prentice, and Paul Geiszler.

1.3.4 ANALYST SUPERVISORS

Analyst Supervisors are responsible for provision of accurate laboratory data produced by analysts under their supervision. They are responsible for ensuring that all QC procedures are followed and documented. All raw data must be entered into the ESE CLASS system and lot folders must be completed with all the required documentation. The Supervisor or designate must review and ensure that the documentation is complete. The QA/QC role of the Supervisors is to enforce the required QA/QC procedures.

1.3.5 ANALYSTS

It is the responsibility of the analysts to perform the required QA/QC procedures and to document all observations and calculations in the proper notebooks or standard forms. At the time analyses are initiated, the analyst defines what samples will be analyzed from the appropriate available numbers system and obtains a lot folder assignment, lot folder forms and recent QC Charts from the Data Management Data Coordinator. ESE field group and sequence numbers are provided so the Data Coordinator can properly update the Chemtrack system to track deadlines.

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The Project QA Staff's specific responsibilities are to:

1. Provide an independent overview of the QC practices within each respective organization to ensure that all QC requirements of the QAPP are completed;
2. Maintain and review all QC records, including control charts, and to provide copies of QC records to USATHAMA on a weekly basis;
3. Prepare or review those sections of all interim and final project reports dealing with QC data;
4. Monitor the establishment of testing lots (batches) in coordination with the Analytical Services Program Team and the introduction of appropriate control samples in each lot;
5. Monitor the logging-in of samples, as well as sample preservation, handling, subsampling, and transport throughout the project;
6. Review all data batches for proper QC procedures, audit data files for correct entry of all data, and approve data for transmittal to IRDMS;
7. Monitor the maintenance records on Standard Analytical Reference Materials (SARMs) or interim reference materials;
8. Maintain a vigil of the entire laboratory (and field operations, when applicable) to detect conditions that might jeopardize control of the various analytical and sampling systems;
9. Ensure by field visits, when applicable, that appropriate sampling, field testing, and field analysis procedures are followed and that correct QC checks are being made and
10. Inform the project management concerning non-conformance with the QAPP and provide documentation of said non-conformance, recommend the corrective actions, and document their completion.

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2. Data Systems Support Coordinator - Enters prefield setup form information into the CLASS system to produce sample labels and chain-of-custody forms. He is responsible for entering completed chain of custody forms to log in samples into the data management system. He also is responsible for filing and storage of validated USATHAMA lot folders until transmittal is required to USATHAMA.
3. Data Coordinator - Assigns lot names and distributes lot folder forms to the analysts requesting a lot assignment. He finalizes data batches and distributes completed lot folders to the appropriate people in the review and validation chain (Analyst, Department Manager, Task Manager, and QA Staff). He also updates the Chemtrak data base which documents the status of QC chart submittals, lot folder review and IRDMS delivery deadlines. He produces weekly reports for the Program Management team to review in weekly management meetings.
4. QC Chart Coordinator - Receives QC spike recovery data from the analysts and produces QC charts. He distributes the charts and corrective action explanations throughout the QC chart review chain (Analyst, Department and Task Manager and QA Staff), and is responsible for documenting and entering status updates for QC charts in the Chemtrak Data base.
5. IRDMS Coordinator - Receives validated lot folders which include files formatted to be read into the IRDMS. He processes the transfer files through the USATHAMA group and record check programs at ESE and then electronically transfers the files to the USATHAMA IRDMS. He is responsible for maintaining documentation on transfers to IRDMS and weekly feedback from the USATHAMA Contractor responsible for the IRDMS system.

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During analyses the analyst performs preliminary QC checks to determine system performance and control. Recovery data are then submitted to the QC Chart Coordinator for official plotting and submittal to USATHAMA. Analysts must ensure that each batch of data being generated meets all analytical criteria specified by the method. Following completion of the instrumental analyses, the analyst must ensure that the data are entered correctly into the ESE CLASS system and complete all lot folder documentation. The analyst must also bring any unusual observation or analytical problem to the immediate attention of his/her Supervisor, the Program or Task Manager, or the QA Staff. The analyst or field team member must ensure that all instruments are calibrated and the calibration recorded in permanent records. Each analyst is also responsible for ensuring that sufficient quantities of reagents of adequate quality are available for the performance of the required analyses.

1.3.6 DATA MANAGEMENT TASK MANAGER

The Analytical Services Division Data Management Task Manager is also in charge of the laboratory information data management system. This computerized data management system, Chemical Laboratory Analysis Scheduling System (CLASS), is described in Appendix E.1. The ESE CLASS system is designed to interact with all USATHAMA program protocols up to submission of data to the IRDMS. Mrs. Virginia O'Brien is the Data Management Task Manager. Her specific duties for the USATHAMA program include supervision of personnel that provide support services for various QA/QC and project management needs. The following support services are performed:

1. Document Control Coordinator - Stores and distributes certified methods and Master QAPP to the appropriate people. He maintains the computerized methods distribution program, and is responsible for reviewing and updating the USATHAMA methods file stored in the ESE CLASS system. The methods file allows automatic production of the correct method information into the IRDMS transfer file.

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by the lab requires verification of proper preservation, documentation, and chain-of-custody. Achievement of analytical extraction and analysis holding times are also required (see Appendix A for summary tables).

Completeness is a measure of the amount of data obtained from a measurement system compared to the amount that was expected under normal conditions. Data completeness required for a certain delivery order is not necessarily known. However, programs in place at USATHAMA which require input from the contractor laboratories (called the COLA program) enables USATHAMA to confirm that all data required by the delivery order have been delivered and elevated to USATHAMA Level, III (for definition see USATHAMA QA Program Plan, 2nd Edition, March 1987). USATHAMA approves final delivery order billings by running this final check and thereby ensures completeness. Early identification of incomplete data losses occurs during the weekly control chart review process.

Comparability is the confidence with which one data set can be compared with another. All data will be calculated and reported in units consistent with standard procedures so that the results of the analyses can be compared with those of other laboratories. The objectives of the ESE Analytical Services Division for comparability are:

- to demonstrate traceability of standards to NIST, EPA or USATHAMA sources;
- to report results from similar matrices in standard units;
- to apply appropriate levels of quality control within the context of the laboratory QA program;
- to participate in interlaboratory studies to document laboratory performance.

By using traceable standards and standard methodology, the analytical results can be compared to other laboratories operating similarly.

USATHAMA certification is required to provide initial performance data based on standard matrix control spikes. Daily control spikes are subsequently used to document conformance with certification and to update method precision and accuracy estimates (this is done through a control chart process). Performance data obtained during certification

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**2.0 QA OBJECTIVES FOR MEASUREMENT OF DATA
IN TERMS OF PRECISION, ACCURACY, COMPLETENESS,
REPRESENTATIVENESS, AND COMPARABILITY**

2.1 ANALYTICAL MEASUREMENT DATA

In general all analyses to be performed for CLASS contract Delivery Orders must yield data of a quality sufficient to support human risk assessment. According to EPA Data Quality Objectives Guidance (EPA, 1987), analytical Level 3 or higher will be required. Data quality of a lower analytical level (Level 2) may be requested, an example would be soil gas analyses.

Analyses performed for Delivery Orders will use certified USATHAMA analytical procedures for analysis of water and soil/sediment. These procedures, in many cases, are equivalent to EPA analytical methods. However, some EPA methods do not currently exist for the exotic compound analyses required at various Army Installations (e.g. explosives, agent breakdown products, etc). Comparability of analyses is based on their similarity to EPA methods and review and acceptance of certified methods by USATHAMA (part of USATHAMA certification review process compares data obtained by other labs certifying the same method). Quarterly contractor meetings also review analytical problems, corrective actions and lessons learned from the contractor laboratories.

Data representativeness is defined as the degree to which the sample and results obtained accurately represents the area sampled. Controlling elements are: sampling requirements and protocols; maintenance of sample integrity; and comparability and performance of analytical methods used. Sampling requirements and protocols are not be controlled by the ESE Analytical Services Division, therefore QA objectives can not be defined in this Master QA plan. Maintenance of sample integrity, however, can be documented from field sampling to the lab and is controlled within the lab. Prior to lab receipt, representativeness is controlled by providing sampling kits, preservatives and preservation instructions, chain-of-custody forms, and coolers for sample shipment. Sample receipt

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Moving Average Precision: Evaluated based on the difference between the highest and lowest recovery of the three recent daily standard matrix control spikes (defined in Sec. 11.0).

Moving Average Accuracy: Evaluated based on the average of individual recoveries of the three recent daily standard matrix control spikes (defined in Sec. 11.0).

Units: Volume in liters (L) [e.g., micrograms per liter (ug/L)] indicates a water matrix. Control spikes are added to organic-free laboratory water. Weight in grams (g) [e.g., micrograms per gram (ug/g)] indicates a soil/sediment matrix. Control spikes are added to a standard USATHAMA soil that has been chemically characterized.

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include: certified reporting limits (CRL's); upper certified limits (UCL's) above which samples require dilution; method precision and accuracy data; and initial control chart limits for the required daily control spike levels. Acceptance criteria for analytical data generated is based on control chart limits which are a measure of laboratory control for that method. Method performance criteria can be used to help judge acceptance of analytical results. Weekly control chart explanations and corrective actions are supplied to USATHAMA for approval.

USATHAMA requires contract laboratories to control the data quality they produce through pre-analysis certification and subsequent daily control spikes which produce precision and accuracy data. Precision and accuracy estimates of the generated data can also be produced by replicate or collocated sampling and matrix spikes, however, these are required on a case-by-case basis and would be either inherent in the sampling design or called for especially in the delivery order.

Precision and accuracy criteria to be used are continually updated by the control chart process. Current control spike limits for the certified methods are presented in Appendix A. Daily control spikes are not performed for every analyte in every method. Selected analytes are spiked for multi-analyte methods for method control purposes. The terms used in Appendix A Tables are briefly explained in the following paragraphs. Items in the table that are not applicable are denoted by NA.

Precision: Evaluated based on the percent difference of duplicate daily standard matrix control spikes (defined in Sec. 11.0).

Accuracy: Evaluated based on the average percent recovery of daily standard matrix control spikes (defined in Sec. 11.0).

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glass bottles or bottles wrapped to prevent light exposure will be used for all samples to be analyzed for organic species. Plastic containers will be constructed from linear polyethylene. The holding times listed in Appendix B apply to both water and soil/sediment samples.

Appendix B identifies sampling containers and the proper preparation of sampling containers to ensure that all samples properly represent constituents within the environmental matrix sampled. Responsibility for providing the sampling team with properly prepared sampling containers and preservation reagents rests with the ESE Analytical Services Division Task Manager, based on the notification of the sampling schedule by the Field Team Leader and/or ESE Task Manager.

Also presented in Appendix B is an example SOP of communications provided to the sampling team.

The following sections document QC practices related to sampling procedures followed by ESE personnel. This information, which is consistent with the USATHAMA Jan. 1990 QA Program Plan, will be provided to other sampling teams if requested.

3.1 VOLATILE COMPOUNDS

Loss of volatile compounds from water samples can occur through headspace and/or evaporation. Care should be taken to preclude aeration of the sample, to completely fill bottles with the samples without any air space, and to analyze within the specified holding times.

Volatile compounds may be analyzed in soil samples, using a solvent extraction step such as methanol extraction or by adding the soil to organic-free water in a sealed purging device. Care must be taken to place samples in an air-tight container immediately upon collection.

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3.0 SAMPLING PROCEDURES

This section describes the QC procedures to be followed during environmental matrix sampling for samples received for analysis by USATHAMA CLASS contract delivery orders. To ensure samples representative of the system under study, samples must be collected in properly cleaned containers, promptly and properly preserved, and transported to the laboratory in a manner that minimizes the chance for significant change in constituents. The type of sample (grab, composite, etc.) and the location rationale of the sample point cannot be controlled by this document and should be described in the specific contractor's Sample Design Plan. Proven sampling, preservation, and shipping methods that comply with USATHAMA and EPA specifications will be used. USATHAMA specifications will take precedence over any other specifications unless otherwise required in the delivery order.

The contractor Field Team Leader is responsible for proper sample collection, documentation, preservation, and shipment. The contract laboratory is required to identify documentation, preservation and/or shipment problems to the USATHAMA COR along with recommended actions for guidance concerning stop analysis. The Project QA Staff monitors the receipt of samples and monitors compliance with preservation and holding time specifications.

Typically, a copy of ESE's computerized sample logsheet will accompany the samples as part of the chain-of-custody record (Appendix B). Other chain-of-custody forms may be submitted by the firm doing the field sampling.

The Field Team Leader is responsible for proper sampling, labeling of samples, preservation, and shipment of samples to the laboratory in a proper manner to meet required holding times. Tables in Appendix B identify the preservation methods, holding times, and ESE sampling container and fraction codes that will be used for the analytes. Amber

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well and then appropriately discarded to avoid cross-contamination.

During the sampling of each monitor well, the following data will be collected:

- (1) Well number;
 - (2) Date and Time;
 - (3) Static water level;
 - (4) Depth of well;
 - (5) Pumping rate and duration of pumping, if applicable;
 - (6) Volume of water removed;
 - (7) Drawn-down water level;
 - (8) In situ water quality measurements such as pH, specific conductance, and temperature;
 - (9) Fractions sampled and preservation;
 - (10) Miscellaneous observations; and
 - (11) Signature of sampler and date.
4. The sample will be collected in a manner that will minimize aeration and prevent oxidation of reduced compounds in the sample: well water will not be agitated by the bailer when the bailer is lowered into and out of the monitor well. The sample container will be gently filled to overflowing without air bubbles and tightly capped. For volatiles, the bottles will be checked to verify that no air has been entrained. If a volatile bottle is contaminated by dropping the septum or touching the septum or lips of the bottle, it will be discarded and a clear bottle issued and labeled. Under no circumstances will volatile fractions be transferred from other sampling containers. Volatile fractions will not be filtered.
5. Samples for metals analyses may or may not be filtered depending on whether dissolved or total metals are required. Samples for total metals analysis will not be filtered. Samples for metal dissolved analyses will be vacuum filtered in the field through a 0.45-micrometer (μm) filter, chilled to

3.2 GROUNDWATER

Groundwater sampling should not be performed until after newly installed monitor wells have been allowed to reach equilibrium (no less than 14 days after well development). All observations and pertinent data developed during groundwater sampling will be recorded in a field notebook similar to that used for surface water sampling. The following procedures will be followed during sampling:

1. The depth to water will be measured and recorded in the field notebook.
2. Samples will be taken after the fluid in the screen, well casing, and annulus has been exchanged five times. The amount of fluid exchanged will be measured and recorded in the field notebook. All water purged from monitor wells prior to sampling will be collected and transported to the South Balloon Treatment System for treatment. All sampling will be accomplished by a dedicated bailer constructed of polyvinyl chloride (PVC). No glue will be used in the construction of these bailers.
3. To protect the wells from contamination during sampling, the following guidelines will be followed.
 - a. A separate bailer (and rope) will be supplied for each well. After use, the bailer will be rinsed with water from the approved source, tagged, wrapped in aluminum foil, and stored in a secure area on-site. Each well will be sampled with a dedicated bailer and, therefore, collection of rinsate for rinsate blank analyses will be unnecessary.
 - b. When a pump is used to purge the standing water from the well, the pump, rope, and associated hoses will be thoroughly cleaned between the samples by steam cleaning and allowed to air dry.
 - c. All sampling equipment will be placed on disposable polyethylene plastic sheeting spread on the ground at the well to prevent soil contamination of the groundwater samples. Each polyethylene sheet is to be used at only one

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A single mid-current sampling point will be used. Sampling will take place at approximately 1/2 to 2/3 of the water depth at its deepest point.

Sampling the edge of the canal from the bank will be avoided, if possible. If unavoidable, sampling will be on the outside of a bend where the current flows along the bank. This will avoid collection of stagnant water of a quality that does not represent that of the main flow. Care will be taken to sample at a point on the canal with complete vertical and lateral mixing. Samples will not be taken immediately below a waste source or tributary, unless there is a specific reason to do so.

In the canal, fractions will be taken as a grab sample. The sample container will be held just beneath the surface of the water and allowed to fill.

Prior to sample collection, each sample bottle will be rinsed with the stream water immediately downstream from the sampling point. Surface water samples generally will not be filtered prior to analysis. The need to filter surface water is a project-specific decision that depends on whether dissolved or total contaminants are of interest. Sample fractions for analysis of volatiles and grease/oil will not be filtered.

3.4 SOILS

Appropriate point sampling or compositing techniques will be used to ensure that the sample is representative of the area sampled and the type of information (e.g., depth of contamination) desired. Soil samples will be placed in an amber or foil-wrapped, wide-mouthed glass jar with Teflon[®]-lined lid. Sampling equipment will be decontaminated by the following method:

1. Brush the equipment to remove gross contamination,
2. Steam clean, and
3. Allow to air dry prior to collecting the next sample.

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- 4 degrees Celsius (C), appropriately preserved, and immediately transported to the laboratory.
6. Each sample bottle and cap will be triple rinsed with water from the well at the time of sampling.
 7. On-site measurements of water quality will include conductivity, pH, and temperature. Calibration standards will be run prior to each set of measurements. Calibration standards for conductivity will consist of solutions of potassium chloride having conductivities of approximately 1,400, 700, and 150 micromhos per centimeter (umhos/cm). pH buffer solutions at pH 7.0, 10.0, and 4.0 will be used to calibrate pH meters.

3.3 SURFACE WATER (IF REQUIRED)

Prior to surface water sampling, the following data will be noted and recorded in the field notebook:

1. Site number or location;
2. Date;
3. Time (24-hour system);
4. Antecedent weather conditions, if known;
5. In situ parameter measurements;
6. Fractions and preservatives;
7. Any other pertinent observations (odor, etc.); and
8. Signature of sampler and date.

At the conclusion of each day in the field, the Field Team Leader will review each page of the notebook for errors and omissions and then date and sign each reviewed page.

All field instrument calibrations will be recorded in a designated portion of the notebook at the time of the calibration. Adverse trends in instrument calibration behavior will be corrected.

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will be performed on the exposed ends of the sample at this time and also included into the physical log.

5. After the sample liners are sealed, they will be placed on ice and delivered to the laboratory.
6. Sample control and tracking information will be recorded in bound field logbooks with prenumbered pages and will include the following information: boring number and location, date, drilling equipment, driller's name, sampler's name, method of sampling, and soil sample physical description. If more than one notebook is required, each notebook will reference all other notebooks. Sample containers will be labeled to include boring number, depth interval, date, project name, project number, and sampler's initials.
7. At the completion of sampling a given borehole, remaining drill cuttings and the borehole will be handled/grouted and sealed in accordance with USATHAMA geotechnical requirements.

Observations recorded in the field notebook at time of soil sampling will consist of:

1. Site identification;
2. Description of location, including distance from reference point to sample point;
3. Date;
4. Time (24-hour system);
5. Description of vegetation;
6. Characteristics of soil;
7. Sample number;
8. Fractions and preservations;
9. Other observations; and
10. Signature of sampler and date.

Prior to drilling, and between each boring, the drill rig and downhole flight augers will be steam cleaned at the designated on-site rig decontamination station.

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Sample containers will be labeled with a preprinted label, chilled to 4°C, and shipped under ice in a cooler to the laboratory for analysis. No plastic should be allowed to contact soil samples requiring organic analysis.

Surface soil (upper 2 ft) samples will be collected with a hand piston sampler or other appropriate device.

A drill rig utilizing hollow-stem augers is often used to collect subsurface (0 to 15 ft) soil boring samples at selected on-site locations. The following procedures outline a typical soil boring sampling:

1. Soil sampling intervals will be determined by the data requirements at each site.
2. Auger flights will be advanced only to the top of the soil sampling interval. Steam-cleaned flights will be added as needed during the drilling operation. Sampling will be accomplished using a 140-lb hammer to advance a 2-inch modified California split-spoon sampler through and ahead of an 8-inch hollow-stem flight auger. The number of blows required to advance the sampler through 6-inch depth intervals will be recorded.
3. Split-spoon samplers will be initially rinsed in deionized water, and final rinsed with pressurized steam. The same split-spoon sampler will be used only throughout a composite interval (i.e., to a 4.5-ft depth), after which a clean split-spoon sampler will be used.
4. Liners may be used inside of the sampler to collect the sample. When removed from the split spoon, the sample's physical characteristics will be described (e.g., color, lithology, general appearance, etc.). Visible indication of contamination will be noted at this time. A field organic vapor analysis

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4.0 SAMPLE CUSTODY

ESE maintains and documents chain-of-custody as described by the National Enforcement Investigation Center (NEIC) of EPA, which defines sample chain-of-custody as follows:

1. The sample is in your actual possession;
2. The sample is in your view after being in your physical possession;
3. The sample was in your possession, and then you locked or sealed it to prevent tampering; or
4. The sample is in a secure area.

A critical step in the processing of samples involves initial check-in and preparation for analysis. Proper chain-of-custody, efficient processing to meet holding times, and avoidance of cross contamination are vital to the integrity of the final data.

Samples are received by the Chemical Analysis Supervisor or his/her designate. The samples are unpacked, and the logsheets are compared with the contents. Samples are scheduled for processing, and the logsheets are given to the Data Management Coordinator, who activates the sample numbers for analysis. If any sample processing is required, it will take place immediately.

Samples are received and checked into the coldroom as described by ESE Analytical Services Standard Operating Procedure (SOP) 4122-04 and shown in Fig. 4.0-1 (selected relevant SOP's from this manual are included in Appendix A). Only the sample custodian replaces samples to the shelves; all other employees replace samples to the return shelf. The coldroom door is always locked when no one is inside.

Samples are not stored in laboratories, but when samples are in a laboratory awaiting analysis, they will be secured by one of the four ways listed previously.

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3.5 SEDIMENTS (IF REQUIRED)

All sediment samples will be collected with a hand piston sampler or other appropriate device. After sampling, depth of water at each sampling point will be measured and recorded. Sampling equipment will be decontaminated using the same procedure described in Sec. 3.4 (soils).

Sediment samples will be placed in amber glass or foil-wrapped containers with Teflon[®]-lined lids, shipped under ice, and stored at 4°C.

Observations recorded in the field notebook at time of soil sampling will consist of:

1. Site identification;
2. Description of location, including distance from reference point to sample point;
3. Date;
4. Time (24-hour system);
5. Description of vegetation;
6. Characteristics of soil;
7. Sample number;
8. Fractions and preservations;
9. Other observations; and
10. Signature of sampler and date.

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that can be manually processed through the limiting step of the method during a single time period (not to exceed 1 day, 24 hours, as defined by the process). The samples will be placed into analytical lots based on analysis and sample matrix type. The number of samples per lot will depend on the number of samples that can be conveniently and efficiently analyzed in one 24-hour day. The sample digestion/extraction or instrumental step may be the rate-limiting step. Other factors that should be taken into consideration in establishing lot size include: (1) type and complexity of analysis; (2) sample holding time and (3) time constraints imposed by well development, sampling, and shipping. The batch lot will be optimized to provide efficient analysis while meeting the holding time criteria for the samples.

Every attempt will be made to maximize the number of samples per lot within the constraints of the daily rate-limiting step. Small lot sizes may be necessary due to the limited number of samples being collected at any particular installation, especially complex sample analysis or extraction procedures, or holding time constraints.

The following QA procedures will be implemented to monitor sample management and handling:

1. The Project QA Staff will ensure that samples are being labeled, preserved, stored, and transported according to the prescribed methods.
2. The Project QA Staff will monitor the introduction of control samples (spikes and blanks) into the sample flow.
3. The Project QA Staff will prepare, review and comment on control chart explanations, corrective actions, submittals, and evaluate responses and required action by USATHAMA responses.

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Each sample or sample fraction removed from the coldroom will be recorded on a check-in/check-out form posted outside the coldroom door. The sample number, date of removal, and the person's initials will be clearly recorded.

Each sample or sample fraction returned to the coldroom will be recorded on the same logsheet. The sample number, date of return, and the person's initials will be clearly recorded. Samples and sample fractions will be returned to the return shelf only.

After the sample extraction is completed, at all times under secure custody as defined previously, the extraction technician completes an Extract Custody Form and then transfers the extracts to the analyst. The analyst then stores the extracts in a secure area and places the Extract Custody Form in the lot folder.

The extracts will be stored at all times in a secure area and will not be discarded until permission is received from the USATHAMA Project Officer.

Sample log-in at the laboratory will be monitored by the Project QA Staff. The Project QA Staff will periodically check the computer logsheet for verification of complete conformance of the log to the sample set and verification of the information contained on the sample labels. Any inconsistencies or unusual circumstances, such as broken or leaking containers, improper preservation, or noncompliance with holding or shipping requirements, will be identified in writing to the Project Manager and the Project QA Staff. Corrective action will be recommended and approved by the Project Manager and the Project QA Staff.

Establishment of USATHAMA lots will be performed by the Information Services Group and monitored by the Project QA Staff. After the samples have been logged into the laboratory sample management system, the analyst will request a lot folder assignment when analysis or extraction begins. A lot is the maximum number of samples, including QC samples,

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5.0 CALIBRATION PROCEDURES

Every analytical method performed has been certified by USATHAMA procedures and/or is comparable to EPA approved or other validated, standard method. USATHAMA certified method writeups document the required initial and daily calibration requirements and QC checks for reference samples and continuing calibration checks. Standard reference material are obtained from USATHAMA (SARMS) and EPA. Additional reference materials are obtained commercially. USATHAMA methods require three independently prepared stock solutions from different sources to prepare: calibration standard stock solutions, control spike stock solutions, and reference sample stock solutions.

In general, acceptance criteria for calibration measurements are as follows:

1. Initial calibration curves encompass the upper and lower certified range of the analyte for that method. Curve diagnostics should be consistent with certification requirements (quadratic or linear) and calibration performance will be estimated using Lack-of-Fit.
2. Reference solution analyses must be performed at the time of initial calibration and results must be within criteria of originator or initial limits required by USATHAMA if independent reference solution is not available. Initial acceptance limits are adjusted (tightened) after a specified period of performance using the mean and standard deviation of performance data.
3. Continuing calibration checks are performed at least at the end of a run and recoveries must be within initial defined acceptance limits. Acceptance limits are adjusted (tightened) after a specified period of performance using mean and standard deviation of acceptable performance data.
4. Daily calibration checks can be performed for methods if desired. Daily calibration checks must verify applicability of the initial calibration curve which was confirmed with

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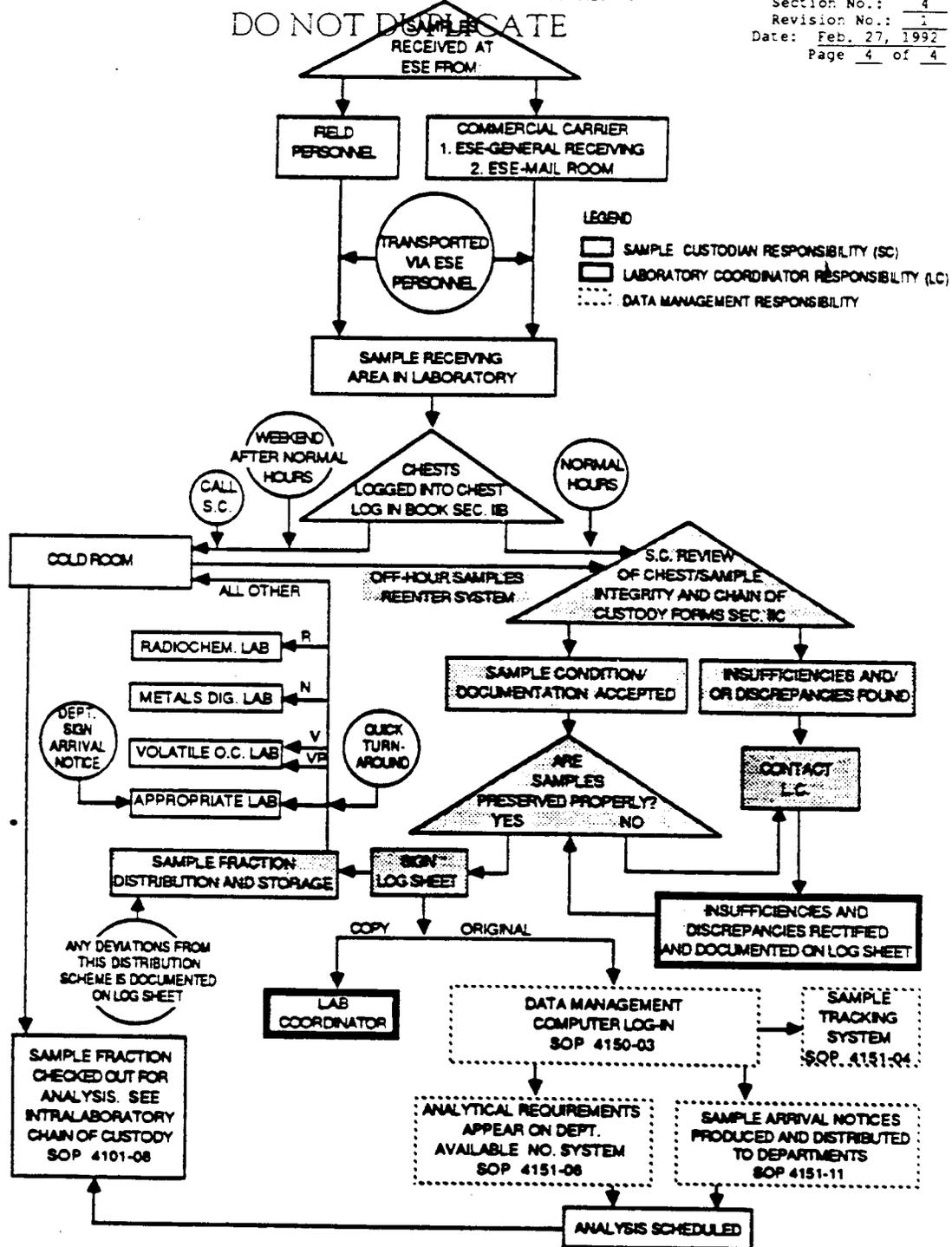


Fig. 4.0-1
SAMPLE RECEIPT, LOG-IN, AND
DISTRIBUTION FLOW CHART (SOP #4122-04)

SOURCE: ESE 1987.

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Failure of an instrument to maintain accurate calibration will be reported to the Field Team Leader, who must take immediate corrective action to ensure that accurate field data accompany any samples. The faulty instrument is tagged and cannot be used until repaired and recalibrated.

5.2 LABORATORY INSTRUMENTS

Daily QC of the analytical systems ensures that accurate and reproducible results are produced. The analyst must check instrumental calibration data for compliance with QC requirements. Unless specified differently in the approved USATHAMA methods, Table 5.2-1 describes the general instrumental QC checks to be implemented.

Initial calibration should be performed under the following conditions:

- (1) analysis is first setup or prior to the first set of samples,
- (2) the instrument has been idle for long periods of time,
- (3) the instrument detector has been subject to major maintenance,
- (4) the instrument fails the daily calibration QC checks, or
- (5) the instrument is used to analyze analytes different from those for which the instrument was calibrated previously.

When available, SARMS supplied by USATHAMA will be used to prepare calibration standards and spiking standards. SARMS or interim SARMS are materials that have undergone extensive purity and stability checks. If SARMS are not available or their quantities limited, "as is" chemicals may be used as interim reference materials. However, the "as is" material would be stored at 0°C and a portion retained for comparison with the approved SARMS when available.

Any "as is" chemical must be characterized for compound identity and purity and results provided to USATHAMA with the certification Performance Data Package. Organic standards will be characterized for purity using capillary gas chromatography/flame ionization detection (GC/FID) analysis and for identity using GC/MS analysis. Inorganic

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standards will be identified against known National Bureau of Standards (NBS) or EPA standards.

All reference compounds used in the USATHAMA projects will be stored at 0°C and protected from light. The Project QA Staff will request SARMS as required, monitor their use and maintain a record of receipt of SARMS.

5.2.1 DOCUMENTATION OF STANDARD PREPARATION

Standard preparation notebooks are kept to document preparation of independent stock and working solutions for: (1) calibration stock solutions, intermediates, and working solutions; (2) calibration reference working solutions (if reference is a concentrate or reference had to be prepared in the lab); and (3) control spike stock solutions, intermediates, and working solutions. Copies of these notebooks are provided in each analytical lot folder.

5.2.2 CALIBRATION CHECKS

Calibration standards are verified with independent reference solutions (when available, otherwise independent stocks solutions are prepared). The analysis of the reference standard is required with each initial calibration. If an initial calibration is run daily, then the reference sample is required on a weekly basis. Reference standards are not required when a daily calibration protocol is followed since the daily calibration standards must be verified to the initial calibration curve. Other calibration quality control involves analysis of continuing calibration check standard (or drift check). Acceptance criteria are documented in each method.

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Table 5.2-1. Summary of Instrumental Systems Control Requirements

Requirement	Analytical Control Limits
<u>Initial Calibration--Minimum Testing Range</u>	
Class 1	<ul style="list-style-type: none">o Calibration curve--concentration series 0X (blank), 0.5X, 1X, 2X, 5X, and *10X, where X is the target or certified reporting limit, as appropriateo *10X daily calibration standard at end of the dayo Check standard, *10X, at beginning and end of day
Class 1A	<ul style="list-style-type: none">o Calibration curve--concentration series 0X (blank), 0.5X, 2X, and *10Xo *10X daily calibration standard at end of the day
Class 1B	<ul style="list-style-type: none">o Calibration curve--concentration series 0X (blank), 0.5X, 2X, and *10Xo *10X daily calibration standard at end of the dayo Check standard, *10X, at beginning of the day
<u>Daily Calibration--Minimum Testing Range</u>	
Classes 1, 1A, 1B	<ul style="list-style-type: none">o *10X daily calibration standard analyzed at beginning and end of the day

*10X = 10-percent to 25-percent range extension, which allows for fluctuations from a theoretical 100-percent method recovery.

Source: ESE, 1988.

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6.0 ANALYTICAL PROCEDURES

6.1 RATIONALE

Four different levels of method certification (Class 1, 1A, 1B, and 2) are recognized by the December 1985 USATHAMA QA Program Plan (2nd Edition, March 1987). The difference between the classes is the procedure used to characterize laboratory performance of the method. Class 1 certifications are the most rigorous and Class 1 methods will typically be employed in this program. Class 1A certification is reserved exclusively for GC/MS methods, whereas Class 1B is reserved for low sample throughput methods (non-GC/MS). Class 2 certification is used for methods that screen for the presence or absence of contaminants. Each type of analysis requires a different level of documentation, including precision and accuracy data and a different set of daily or batch-related QC criteria. The following sections outline the testing procedures for Classes 1, 1A, and 1B that will be used to define the detection limit, precision, and accuracy of each analytical method. Class 2 will typically not be utilized for this task program.

Method certification in standard media will certify the laboratory to run analyses for a given analyte. Documentation of the analytical testing certification will be submitted to USATHAMA for approval before use of the analytical method for analysis according to the format described in Appendix A of the 1990 USATHAMA QA Program Plan.

ESE's current list of USATHAMA certified methods is provided in a summary table of Appendix A, additional tables in Appendix A list the certified analytes in each method. ESE will prepare from Appendix A, when required, an Appendix to this Master QA Manual that will provide summary descriptions of the USATHAMA methods to be employed for each specific project. In the event that additional methods are needed where no reliable methods exist, documentation for proposed methods development will be submitted to USATHAMA for approval prior to initiation of method development. The documentation package for the

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proposed method certification will include a description of the technical approach and an estimate of required resources.

6.2 METHOD CERTIFICATION

The following paragraphs describe the procedures to be used to certify analytical methods. All methods certification and documentation data will be developed in standard matrices.

The standard matrix for documentation of inorganic analyses (e.g., sulfate, nitrate, or metals) in water will be deionized water conforming to American Society for Testing and Materials (ASTM) Type I grade water. The standard matrix for documentation of organic analysis will be deionized, organic-free (ASTM Type II) water containing 100 milligrams per liter (mg/L) each of added sulfate and chloride.

The data for documentation of both inorganic and organic analyses in soils and aquatic sediments will be developed using an uncontaminated standard soil matrix obtained from USATHAMA. An aliquot of standard soil will be carried through each set of documentation samples to act as a blank. Added concentrations of the subject analyte(s) will be dissolved in a volume of solvent just sufficient to wet the soil. This solution is poured over the subsample of soil and allowed to stand for 1 hour; volatile organics will be allowed to stand for 15 minutes prior to beginning analysis; the solvent is allowed to evaporate.

If, and only if, a column is to be used for the extraction, the analyte may be dissolved in the minimum quantity of the solvent consistent with volumetric transfer. The solution is placed on the column and allowed to soak in before additional extracting solvent is introduced.

Certain compounds or elements (e.g., nitrate or iron) will be present as natural components of the soil. This background will be accounted for where it exists, and the certified reporting limit (CRL) for the particular method will be considered as the lowest level of analyte in the sample being analyzed which can be quantitatively differentiated

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from zero with 90-percent confidence using a complete, specific analytical method and for which precision and accuracy criteria are valid.

6.2.1 CLASS 1A GC/MS

The CRL of the total method will be estimated by spiking standard matrices of interest (water, soil, etc.) with the actual analytes and surrogate standards. The spikes must be within the working range of the instrument and in the following minimum sequence: 0 (blank), 0.5X, 2X, and 10X in duplicate, where X is the desired or required CRL. The analyte should be dissolved in a solvent to prepare the spiking solution. The spiked levels should be as close as possible to those listed, but a reasonable attempt at producing these levels will be considered acceptable. The spiked samples will be analyzed through the entire analytical method without dilution for analysis. After analysis, the CRL will be calculated using the USATHAMA reporting limit program. The CRL determined by this process will be reported as the CRL of the method.

In summary, certification of the GC/MS method requires the following:

1. A minimum of two spiked standard matrix samples at each of three concentration levels (0.5X, 2X, and 10X), plus a blank analyzed in a single day;
2. The CRL and accuracy calculated using the USATHAMA reporting limit program; and
3. Documentation of the procedures in USATHAMA format.

6.2.2 CLASSES 1 AND 1B NON-GC/MS ANALYSES

Requirements for certification of Classes 1 and 1B methods are as follows:

1. A minimum of one spiked sample at each of five concentration levels (0.5X, X, 2X, 5X, and 10X), plus a blank analyzed each day for 4 separate days. Extended range must include spike samples at 20X, 50X, 100X, 200X, 500X, and 1,000X.

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2. The CRL and accuracy will be calculated using the USATHAMA reporting limit program.
3. Documentation of the procedures in USATHAMA format.

6.3 PRECERTIFICATION AND CERTIFICATION CALIBRATION CURVES

Before initiating certification activities, a calibration curve will be constructed for the planned analytes at concentrations bracketing the anticipated testing range. The standards will be prepared and analyzed in duplicate. These precertification calibration curves will then be tested for lack of fit (LOF) and zero intercept (ZI) (App. E, 1985 USATHAMA QA Program Plan). The results will then be submitted to USATHAMA Analytical Branch for approval prior to certification initiation.

Decisions will be made as to whether or not the calibration is linear over the range. Those methods with nonlinear calibration curves will be handled on a case-by-case basis with specific controls on daily calibration written into the certified method.

All certification analyses must be preceded by instrument calibration. On the first day of certification analyses, initial calibration will be performed. Initial calibration will consist of a minimum of one blank and five calibration standards that bracket the tested concentration range. The slope of the initial calibration curve is compared to slopes obtained from the precertification calibration curve. All data must be collected during periods when instrument calibration is in control (within 10 percent of the mean response for inorganic analyses and within 25 percent of the mean response for all other analyses).

Separate master stock solutions for calibration and spiking will be utilized for daily control. A single master stock solution for the preparation of calibration standards and control spikes should be utilized only during certification.

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6.4 CLASS 1 AND CLASS 1B CERTIFICATION

During certification, a minimum of one standard sample at each concentration shall be analyzed each day for 4 separate days. Sample spikes at each concentration for each day shall be prepared from separate master stock solutions as the calibration standards.

The 4 days of analysis shall be consecutive or as close to consecutive as possible. Analysis refers to performance of the entire method, including spiking samples and sample preparation, not merely to instrumental measurement.

The CRL and the method accuracy for each analyte shall be calculated by the Contractor Laboratory using a software program based on the equations outlined in Sec. 11.0. Data generated over the 4 days of analysis shall be used in the calculations.

6.5 CLASS 1A CERTIFICATION

During certification, a minimum of two standard samples at each concentration shall be analyzed on a single day. Sample spikes at each concentration for each day shall be prepared from separate master stock solutions as the calibration standards.

Analysis refers to performance of the entire method, including spiking samples and sample preparation, not merely to instrumental measurement.

The CRL and the method accuracy for each analyte shall be calculated by the Contractor Laboratory using a software program based on the equations outlined in Sec. 11.0. Data generated over the single day of analysis shall be used in the calculations.

6.6 NON CERTIFIED METHODS

Certification is not required for all analytical methods, for example TOC, TOX, pH, and alkalinity are not considered certifiable by USATHAMA. Certain other methods have not been certified because USATHAMA has not yet officially required certification by ESE.

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7.0 DATA REDUCTION, VALIDATION, AND REPORTING

7.1 INTRODUCTION

There are many steps required to explain data reduction, validation, and reporting for USATHAMA projects. This section provides a summary of operations and procedures with references to the appropriate ESE SOP's for further detail. The ESE CLASS data management system (see Appendix E.1 for system description) is instrumental in ensuring that minimal manual entry errors and manual manipulations occur in providing a client with valid chemical data. USATHAMA requires the production of defined chemical data files and contractor transfer of those files to the USATHAMA IRDMS data base. ESE has a computerized data base that documents the control of data quality. Therefore, programs have been written to automatically produce USATHAMA chemical transfer files to prevent manual entry errors. Validation occurs both internally at ESE and with the USATHAMA IRDMS at each step of the process. As a final check, printouts from the IRDMS are obtained on computer files and verified with the existing ESE data base transferred.

The ESE data management system calculates concentrations and recoveries for all samples and QC from either raw data, manual entry, or computerized transmittal of raw data (i.e. instrument responses for calibration curves, samples and associated QC samples). The data management system allows for control of analytical data for samples by grouping environmental samples in "field groups". Each sample is assigned a defined analyses list or "STORET list" to ensure that all of the required analyses are performed. Each STORET number could have multiple method requirements, therefore "STORET*method code" combinations can be defined in the "STORET list" to control the type and criteria for various QC required. USATHAMA certified method numbers have been used as the "method code" for each STORET required for analysis by that method. The type of QC and required limits are updated and reviewed in each STORET*method code. When EPA STORET numbers are not available or applicable (different units required), ESE internally assigns a STORET number starting with 90000.

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Computerized output from the following instruments have been interfaced with the ESE CLASS system to minimize manual entry errors:

1. ICP and furnace instruments for metals analysis;
2. GC/MS instrumentation for VOA and semi-VOA analysis;
3. Ion chromatography instruments and TRACS autoanalyzer instruments; and,
4. Some GC instruments.

All other methods and instrument output require entry of raw data responses or final concentrations and the required QC data. Verified programs in the CLASS system then calculate results for samples and QC and compare them to the required acceptance limits. For the USATHAMA certified methods, specific data entry requirements are documented in individual method summaries which become part of the raw data lot folders.

USATHAMA requires lot name assignments for groups of samples requiring an analysis. The analyst obtains USATHAMA lot name assignments when a "batch" of samples are grouped for analysis or extraction/digestion for analysis. The CLASS system assigns "batch numbers" when analysts begin to enter raw data into the CLASS system. A separate data base has been built relating ESE batch numbers and USATHAMA lot names. This "Chemtrak" data base allows the Chemistry USATHAMA Program Manager and Project Team to monitor the status and priorities for QC chart and lot folder submission and validation.

USATHAMA lot folders constitute the formal documentation for all data reduction, validation, and final report files to the USATHAMA IRDMS. Historically, lot folder documentation requirements have typically called for 'stand alone' documentation and traceability. Currently, method specific requirements are in preparation and incorporation into each certified method. Lot folder document inventory formats including QA validation forms are provided in Appendix D.2. Road maps documenting

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comparability to EPA CLP files have been presented in Appendix D.3. Additional data reduction, validation, and reporting information is provided in Appendix E and in the following Sections.

7.2 DATA REDUCTION

7.2.1 CLASS 1A METHODS (GC/MS)

Results will be reported in terms of concentrations in the original matrix and will be reported unadjusted for accuracy for entry into the USATHAMA IRDMS. Results of samples that cannot be diluted within the certified range will be reported as greater than the upper limit of the certified range. Lack of indications of the presence of specific compounds to be reported will be reported as less than the certified reporting limit. Estimates of concentrations of species that have not been subjected to the method certification procedure and for which no standards are available, as in the GC/MS screening procedure, will be reported based on the response compared to the response of a reference compound or internal standard provided that: (1) the instrumental response of the species is at least 10 percent of the response of the internal standard, (2) the estimated concentration contains only one significant figure, (3) the estimated concentration is annotated as based on the reference compound, and (4) the estimated concentration is reported as the concentration in the original matrix assuming 100-percent recovery. Non-target compounds from the GC/MS screen will be reported as the compound in the USATHAMA database or as UNKXXX, where XXX is keyed to the relative retention times.

Results of the analyses will be entered into the USATHAMA IRDMS, as outlined in the Installation Restoration (IR) Data Management User's Guide (USATHAMA, 1988). The analyte concentration will be reported to two significant figures. Results obtained after dilution and results of screening for noncertified analytes will be reported to only one significant figure.

7.2.2 CLASSES 1 AND 1B NON-GC/MS

Estimates of concentration levels in QC and actual samples will be reported to USATHAMA according to the guidance as outlined in the

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program tasking and the IR Data Management User's Guide. Values less than the CRL will be reported as less than the certified limit.

If results for an analyte were obtained using the method without dilution, the analyte concentration in the sample may be reported up to three significant figures. If dilution is required, the result may be reported to only two significant figures.

The analyst performs the analysis of samples and control samples and plots QC sample results on control charts. The data are then processed through the Data Management System, where automated QC checks are performed, and the data are presented in standard laboratory and USATHAMA format. The Analyst Supervisor then reviews and approves the data. The Task Chemical Analysis Supervisor then reviews and approves the data and QC results and submits the data batch to the Project QA staff for review and approval.

7.2.3 DOCUMENTATION OF RAW DATA

The ultimate repository for information concerning analyses performed in the laboratory is the analyst's personal laboratory notebook and the instrument logbooks. Bound notebooks with prenumbered pages are maintained according to good laboratory practices. Entries will be completed in ink. Corrections will be made by drawing one line through the incorrect entry, entering the correct information, and initialling and dating the correction.

Each analyst is required to have a personal notebook designated by a unique number, and is responsible for maintaining complete laboratory notes. The QA/QC Coordinator may audit laboratory notebooks without notice. Method specific forms may be used to document laboratory data.

Laboratory notebooks or forms will not be taken from the laboratory without written permission of the Chemical Analysis Supervisor and the Task Manager. Every entry into the notebook or form should be dated and signed. Entries in the personal notebook or onto the form will vary

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depending on the role of the individual in the laboratory and the type of work being performed. At a minimum, the following information should be documented:

1. A reference to or a description of the procedures used for sample workup or analysis,
2. A summary of the samples extracted or analyzed,
3. Weighings and calculations of standard concentrations, and
4. Information on spiking procedures and observations and comments on the procedures or samples.

An instrument logbook will be maintained for required analyses. Each time an instrument is used for sample analysis, the following information is entered:

1. Date of analysis;
2. Project name and number;
3. Type and number of samples analyzed;
4. Time spent on analysis (start to finish);
5. Preventive maintenance performed, if any;
6. Time spent on preventive maintenance;
7. Instrument calibration performed, if any; and
8. Name of analyst.

Additional notes are made in the instrument logs when required. These notes are particularly important when abnormal instrument or analytical performance is observed. It is the analyst's responsibility to ensure that instrument logs are properly filled out and kept up to date. The QA Staff monitors and audits the status of instrument logbooks.

No samples are to be run on any instrument which fails calibration and not until it is clearly demonstrated that the instrument is back in control.

At the end of the project, copies of all logbooks containing information specific to the installation will be forwarded to USATHAMA, if requested. ESE corporate logbooks should be avoided; however, if such

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logbooks are used, certified copies of all relevant logbook pages will be submitted to USATHAMA upon request.

7.3 DATA VALIDATION PROCEDURES

The data processed through the ESE Data Management system, where automated QC checks are performed, are reviewed by the analyst supervisor, analytical Task Manager, and the QA supervisor. Each data package contains all items documented in Appendix D.2. This includes computerized batch reports, review checklists, and all raw data.

7.3.1 LABORATORY LOT FOLDER REVIEW

Once the analyst has completed the analyses for a 'Lot' of samples a USATHAMA Lot Folder is prepared (example in Appendix D.2) and submitted to the data management center. The Data Coordinator finalizes the results in the ESE data batch and incorporates the remaining information into the Lot folder. The laboratory review chain then continues with the Department manager or group leader review of the Lot folder. Finally the Task Manager reviews the Lot prior to QA validation. The Army Data review form (shown in Appendix D.2) is filled out upon completion of review.

7.3.2 INDEPENDENT QA AUDIT OF LOT FOLDERS

The Project QA Staff is responsible for audit reviewing for approval all data packets before transmittal of data to USATHAMA for entry into IRDMS. Further, all data packets transmitted to USATHAMA must be validated by the Project QA Supervisor or the QA/QC Coordinator. Validation involves a thorough review of the data documentation from reported results to raw data including recalculation of results of a selected subset of data.

For the efficient flow of laboratory data to USATHAMA, it is critical that the QA and supervisory reviews of data be organized in a planned methodology which includes successful interface with the data management program. Formal review sheets accompany chemical analysis results of each completed lot of samples. The data are routed to several key

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individuals for approval. Any changes to the data are documented on the formal review sheets so that the appropriate flags are incorporated in the USATHAMA lot file. Representative examples of these forms are presented in Appendix D.2.

Audits are performed on every data Lot to ensure that all QC checks required by the method were performed and acceptable. The use of method specific data review checklists ensure that a thorough Lot folder audit is done. This audit includes check of the control charts, method blanks, standard matrix recoveries, surrogate recoveries, calibration curves, certified reporting limits and units. Also included in the reviews are analysts's notebook pages, number of samples and identifications, dilutions, moisture content, sample weights, chain-of-custody forms, standard preparation notebooks, instrument logbooks, etc. After ensuring that all these items summaries on the method specific inventory are present and complete, selected data values are verified. Several lines of data in the IRDMS transfer file are selected by a random number generator according to MIL-STD-105D, April 29, 1963. One line of data represents one data point. The chosen data points are then traced back to the raw data to verify correctness.

Any discrepancies pertaining to any of the previously mentioned audits are directed to the analytical task manager for verification, clarification, and/or correction. Other queries regarding the data transmission file are addressed directly to Data management. After these processes are complete the Data Management group can transmit the data to USATHAMA for final group and record checks and entry into IRDMS.

Three data levels are used to indicate increasing QA and validation performed on the data. Data reviewed by ESE and transmitted to USATHAMA IRDMS are considered Level 1. Level 2 data has gone through final checks by PRI (the IRDMS contractor) and USATHAMA. Data are considered Level 3 when approved and transferred to the UNISYS system. Level 3 data are available to users to create reports and graphs, but data cannot be changed by contractors.

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7.4 DATA REPORTING - IRDMS RECORD AND GROUP CHECKS

After each data packet has been reviewed by key individuals and validated by QA staff, the electronic data file for the packet is loaded into the USATHAMA IRDMS system at ESE and is run through first record check and then group check. Every data point is checked using these two routines. IRDMS record check determines the following:

1. Data correctly formatted.
2. Lab certified for method on date of analysis.
3. Whether file name (such as CGW, CSW) and site type (BORE, WELL) combinations are valid.
4. Sample date, preparation/extraction date and analysis date are compared to determine any holding time violations or inconsistencies.
5. All test names are valid for the method.
6. Value compliance with Certified Reporting Limit and Upper Certified Limit or diluted within range.

IRDMS group check determines the following:

1. The existence of all station identifications for the lot data in the map file for the appropriate installation.
2. That all test names/analytes found in the QC are present in all the samples.
3. That all required QC spikes exist, and that all spiking levels are valid, as determined by the methods table, and that no aberrations exist in QC or sample data.

If any errors are found in group and record check which are not addressed on the lot cover sheet by the laboratory analysts, laboratory project coordinator, or the QA coordinator, the lot is returned to the laboratory project coordinator so that the problem can be rectified. If changes to the analytical data are required, the lot is then resubmitted to Quality Assurance, and after re-validation, it is again processed through IRDMS to assure that any errors have been corrected. Comments affecting the quality of data will be associated with each data point as necessary by the use of flagging codes. A description of these codes

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can be found in Appendix E. The flagging code will be placed in Column 29 on the same line as the appropriate test name and data point in the lot file that is submitted to USATHAMA. These codes will be part of the official database.

After the data in a lot have successfully passed QA validation and IRDMS record and group checks, a transfer file of the lot is created and sent to USATHAMA via telephone line. The data are again run through record and group checks by USATHAMA, and after passing the data checks, are elevated to Level 2.

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8.0 INTERNAL QUALITY CONTROL CHECKS

Several internal QC checks are required by the USATHAMA QA program that the laboratory must perform. Other possible internal QC checks are site specific and are not identified in this Master QA Plan. Site specific internal QC checks that might be defined and required in project specific QA plans are:

1. Collocated, split or replicate samples;
2. Matrix spikes or matrix spike duplicates; and,
3. Frequency of field, trip and equipment blanks.

If the above QC samples are required, decisions on how to implement them occur on a case-by-case basis. Generally, the only impacts are costs because protocols exist to introduce them in the analytical scheme.

Internal QC checks required for USATHAMA work are defined in each individual method write-up. The types of internal QC checks used are:

1. Use of Standard Analytical Reference Material (SARMS) for traceability of independent stock solutions prepared for calibration stocks, control spike stocks and reference solution stocks;
2. Verification of initial calibration curves with independent reference stock solutions;
3. Verification of initial calibration curves with daily calibration standards (if used some methods always use initial calibration);
4. Verification of continued calibration control by analysis of check standards to document calibration drift; and,
5. Analysis of control spikes to document method performance and control in respect to original certification and recent performance.

An attempt will be made to analyze all samples within the certified range of the analytical method. Dilution of a sample extract with extracting solvent or of the original sample matrix with

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distilled/deionized water should be performed if the concentration of analyte is greater than the certified range of the method.

8.1 SAMPLE PREPARATION

The following paragraphs describe the preparation of water, soil, sediment, and standard samples for analysis. The Project QA Staff will monitor the sample preparation procedure to assure compliance with USATHAMA requirements.

8.1.1 WATER SAMPLES

Water samples requiring filtration will be specified in the Project Work Plan. Generally, the filtrate will be analyzed for metals only. An attempt will be made to utilize filtration material that is compatible with the constituents of interest.

If samples containing high levels of contamination are expected, the suspected high-level samples will be filtered last and the suspected low-level samples filtered first to minimize the possibility of cross contamination. Samples for volatiles and oil/grease determination will never be filtered.

8.1.2 SOIL/SEDIMENT SAMPLES

Soil and sediment samples will be analyzed in the as-received condition. The soil/sediment samples will be made as homogeneous as possible by shaking and/or stirring with a spatula before a subsample is taken. The sub-sampling procedure does not apply to samples for volatiles analysis.

Percent moisture for soils and sediments will be determined prior to analysis by ASTM Method D2216-17 (ASTM, 1981).

8.1.3 STANDARD SAMPLES

Preparation of standard soil and water for methods development and analytical systems control is described in Sec. 6.2. Standard samples for soil analysis consist of samples of an approved uncontaminated soil obtained from USATHAMA.

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8.1.4 METHOD CONTROL QC SAMPLES

Control samples will be introduced into the train of actual samples as a monitor on the performance of the analytical system. Control samples will consist of spiked standard matrix samples and blanks. Results from spiked standard matrix samples will be used to construct control charts to monitor variations in the precision and accuracy of routine analyses. The specific type and number of control samples and the construction of control charts required for USATHAMA are summarized in Table 8.1-1.

8.1.4.1 Surrogate QC Spikes

Certain methods require the use of surrogates to help monitor method performance. When surrogates are required, they are spiked into all environmental samples, QC samples, and method blanks. The surrogates serve two main functions in the GC/MS methods: to control the method and to document the recoveries of compounds similar in chemical composition to the target compounds. The recoveries of the surrogates in the standard matrix spike analyzed with each analytical lot are plotted on \bar{X} and R control charts (control charts are discussed in Sec. 8.1.5.3). If any point on any of the surrogate control charts are outside criteria, either an acceptable explanation must be provided or the analytical lot will have to be reextracted and reanalyzed. Control charts are not prepared for the surrogates in the environmental samples.

The recoveries of the surrogates in the sample matrices are reported to the database and are used to help interpret the analytical results. Typically, if the recoveries of the standard matrix spikes are within precision and accuracy criteria, the method is considered "in control." Sample surrogate recoveries that are much lower or higher than the accuracy or precision criteria typically document that the analytical method is not totally applicable to that sample matrix. For example, if all the acid surrogate recoveries for a sample matrix were below criteria, then the analytical results for the acid extractable target compounds would be interpreted as estimated low due to matrix effects.

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Table 8.1-1. QC Requirements by Sample Lot

Requirement	Analytical Control Limits
Control Samples Non-GC/MS Methods	<p>At least one standard matrix method blank for each daily lot.</p> <p>Three standard matrix control spikes at approximately X, 10X, and 10X, where X is the CRL per daily lot.</p>
Control Samples GC/MS Method	<p>At least one standard matrix method blank for each daily lot spiked with deuterated surrogate standards at the 10X level.</p> <p>Each sample spiked with deuterated surrogate standards spiked at approximately 10X, where X is the concentration in the matrix corresponding to the CRL.</p>
Control Charts Non-GC/MS	<p>Plot average percent recovery value (\bar{X}) obtained from the duplicate 10X spikes within each lot for the accuracy control chart.</p> <p>Plot differences (R) between the percent recovery values of the duplicate 10X spikes within each lot for the precision control chart.</p> <p>Plot 3-point moving average percent recovery values (\bar{X}) obtained from the X single spikes within each lot for the moving average accuracy control chart.</p>

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Table 8.1-1. QC Requirements by Sample Lot (Continued, Page 2 of 2)

Requirement	Analytical Control Limits
Control Charts GC/MS Methods	Plot 3-point moving differences (R) of percent recovery values of the X single spike within each lot for the moving average precision control chart.
	Plot 3-point moving average percent recovery values (\bar{X}) obtained from the single 10X standard matrix spike within each lot for the moving average accuracy control chart.
	Plot 3-point moving differences (R) of the percent recovery values of the single X spike within each lot for the moving average precision control chart.

Source: ESE, 1990.

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8.1.5.2 Matrix Spike and Matrix Spike Duplicate QC Samples

A matrix spike (MS) and matrix spike duplicate (MSD) are not required by USATHAMA but can be requested. If requested, an MS and MSD would be analyzed at a minimum rate of 1 MS and 1 MSD per 20 environmental samples of the same matrix (aqueous versus solid). The MS/MSD would be spiked with the same target compounds that are used to spike the standard matrix. The recoveries of the MS/MSD in the sample matrix could then be reported to the database and are used to help interpret the analytical results. Typically, if the recoveries of the standard matrix spike are within precision and accuracy criteria, the method is considered "in control." Recoveries of target analytes in the MS/MSD that are much higher or lower than the accuracy or precision criteria typically document that the analytical method is not totally applicable to that sample matrix. For example, if the MS/MSD recoveries for a sample matrix were below criteria, then the analytical results for the samples in that batch would be interpreted as estimated low due to matrix effects.

8.1.5.3 Control Spikes and Charts for GC/MS Methods

The results of MS and MSD when required, will be reviewed in conjunction with the standard MS, surrogate, and other QC information to aid in determination of the usability of the data. A single control spike of surrogates per lot into standard matrix will be the basis for laboratory control of GC/MS methods. The exact level to be used for the surrogates in the volatiles method and the semi-volatiles method are included in the certified methods writeups and summarized in Appendix A.6. All actual samples will also be spiked with the same surrogate spiking solutions, but the recovery of surrogates from actual samples will not be used for control purposes. The recovery of surrogates from actual samples may be used by USATHAMA at a later time to assess matrix effects.

The percent recovery for each surrogate in the standard matrix spike will be used for control purposes rather than actual concentration. Since there is only one control sample per lot, normal \bar{X} and R (average and range) charts cannot be used. A 3-point moving accuracy and

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