

# Argonne National Laboratory

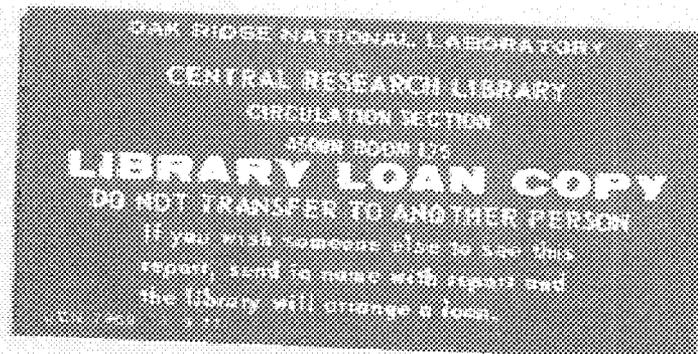
## Sampling and Analysis Data Document

This document contains uninterpreted sampling and analytical data. The data will be interpreted by the DOE Environmental Survey Team and used to modify, as appropriate, the tentative Survey findings contained in the Environmental Survey Preliminary Report. Final Survey findings will be presented in the Environmental Survey Summary Report.

### DRAFT

Volume II A

June 1989



DEPARTMENT OF ENERGY  
ENVIRONMENTAL SURVEY





# ARGONNE NATIONAL LABORATORY SAMPLING AND ANALYSIS DATA DOCUMENT

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June 1989

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U.S. Department of Energy  
Office of Environmental Audit

Prepared by:  
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Oak Ridge National Laboratory

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**Appendix A**

**UPDATED LIST OF SAMPLING AND ANALYTICAL REQUESTS**



TABLE A.1  
 ARGONNE SITE ENVIRONMENTAL SAMPLES  
 WITH FIELD QC SAMPLES  
 SORTED BY ENVIRONMENTAL PROBLEM AND REQUEST NUMBER

REQ NUMB	PROB NUMB	ST	DATE COLL. DD/MM/YY	LOCATION	TYPE	MEDIA	NUMB SAMP		TYPE	ANIONS		METALS		O&G		PET HYDRO		SOIL GAS		PES/H/PCB		SEMI VOLS		VOLS		RADS							
							ACTU	PLAN		ACTU	PLAN	ACTU	PLAN	ACTU	PLAN	ACTU	PLAN	ACTU	PLAN	ACTU	PLAN	ACTU	PLAN	ACTU	PLAN	ACTU	PLAN	ACTU	PLAN	ACTU	PLAN	ACTU	PLAN
AR300	1		07/11/87	NPDESOUTF1	EFFLUENT	SUR WATER	1	1	GRAB	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1	1	0	0						
AR300	1		07/11/87	NPDESOUTF1	EFFLUENT	SUR WATER	1	1	T COM	0	0	1	1	0	0	0	0	0	0	1	1	1	1	0	0	0	0						
AR300	1		10/11/87	NPDESOUTF1	EFFLUENT	SUR WATER	1	1	GRAB	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1	1	0	0						
AR300	1		10/11/87	NPDESOUTF1	EFFLUENT	SUR WATER	1	1	T COM	0	0	1	1	0	0	0	0	0	0	1	1	1	1	0	0	0	0						
AR300	1		12/11/87	NPDESOUTF1	EFFLUENT	SUR WATER	2	2	QC FL	0	0	1	1	0	0	0	0	0	0	0	0	0	0	1	1	0	0						
AR300	1		12/11/87	NPDESOUTF1	EFFLUENT	SUR WATER	1	1	GRAB	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1	1	0	0						
AR300	1		12/11/87	NPDESOUTF1	EFFLUENT	SUR WATER	1	1	T COM	0	0	1	1	0	0	0	0	0	0	1	1	1	1	0	0	0	0						
AR302	2		04/11/87	SAYMILL CR	BACKGROUND	SEDIMENT	3	3	GRAB	3	3	3	3	3	3	0	0	0	0	3	3	3	3	3	3	3	0	0					
AR307	3		05/11/87	NPDESOUTF2	SEEP	SUR WATER	1	1	GRAB	1	1	1	1	1	1	0	0	0	0	1	1	1	1	1	1	0	0						
AR307	3		06/11/87	NPDESOUTF2	SEEP	SUR WATER	1	1	GRAB	1	1	1	1	1	1	0	0	0	0	1	1	1	1	1	1	0	0						
AR307	3		06/11/87	NPDESOUTF2	SEEP	SUR WATER	1	1	QC RN	1	1	1	1	0	1	0	0	0	0	1	1	1	1	1	1	0	0						
AR307	3		10/11/87	NPDESOUTF2	SEEP	SUR WATER	1	1	GRAB	1	1	1	1	1	1	0	0	0	0	1	1	1	1	1	1	0	0						
AR308	4		04/11/87	COAL PILE	RUNOFF	SEDIMENT	3	3	GRAB	3	3	3	3	3	3	0	0	0	0	3	3	3	3	3	3	0	0						
AR308	4		04/11/87	COAL PILE	RUNOFF	SUR WATER	1	1	QC RN	1	1	1	1	1	1	0	0	0	0	1	1	1	1	1	1	0	0						
AR309	5		04/11/87	B815 SEWER	DRAINAGE	SUR WATER	1	1	GRAB	0	0	1	1	1	1	0	0	0	0	0	1	1	1	1	1	0	0						
AR309	5		05/11/87	B815 SEWER	DRAINAGE	SUR WATER	1	1	QC FL	0	0	1	1	1	1	0	0	0	0	0	0	0	0	0	0	0	0						
AR309	5		05/11/87	B815 SEWER	DRAINAGE	SUR WATER	1	1	GRAB	0	0	1	1	1	1	0	0	0	0	1	1	1	1	1	1	0	0						
AR309	5		06/11/87	B815 SEWER	DRAINAGE	SUR WATER	1	1	GRAB	0	0	1	1	1	1	0	0	0	0	1	1	1	1	1	1	0	0						
AR310	5		04/11/87	B815 SEWER	DRAINAGE	SEDIMENT	3	3	GRAB	0	0	3	3	3	3	0	0	0	0	3	3	3	3	3	3	0	0						
AR311	6		07/11/87	NPDES10WTP	DISCHARGES	SUR WATER	1	1	GRAB	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1	1	0	0						
AR311	6		07/11/87	NPDES10WTP	DISCHARGES	SUR WATER	1	1	QC RN	0	0	1	1	0	0	0	0	0	0	1	1	1	1	0	0	0	0						
AR311	6		10/11/87	NPDES10WTP	DISCHARGES	SUR WATER	1	1	T COM	0	0	1	1	0	0	0	0	0	0	1	1	1	1	0	0	0	0						
AR311	6		10/11/87	NPDES10WTP	DISCHARGES	SUR WATER	1	1	GRAB	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1	1	0	0						
AR311	6		10/11/87	NPDES10WTP	DISCHARGES	SUR WATER	1	1	T COM	0	0	1	1	0	0	0	0	0	0	1	1	1	1	0	0	0	0						
AR311	6		12/11/87	NPDES10WTP	DISCHARGES	SUR WATER	1	1	GRAB	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1	1	0	0						
AR311	6		12/11/87	NPDES10WTP	DISCHARGES	SUR WATER	1	1	T COM	0	0	1	1	0	0	0	0	0	0	1	1	1	1	0	0	0	0						
AR400	7		10/11/87	B31 TAP WA	WELLS	GRN WATER	1	1	GRAB	1	1	1	1	0	0	0	0	0	0	1	1	1	1	1	1	1	1						
AR401	7		10/11/87	B32 TAP WA	WELLS	GRN WATER	1	1	QC FL	1	1	1	1	0	0	0	0	0	0	0	0	0	0	0	0	1	1						
AR401	7		10/11/87	B32 TAP WA	WELLS	GRN WATER	1	1	GRAB	1	1	1	1	0	0	0	0	0	0	1	1	1	1	1	1	1	1						
AR402	7		10/11/87	B163 TAP W	WELLS	GRN WATER	1	1	GRAB	1	1	1	1	0	0	0	0	0	0	1	1	1	1	1	1	1	1						
AR403	7		10/11/87	B264 TAP W	WELLS	GRN WATER	1	1	GRAB	1	1	1	1	0	0	0	0	0	0	1	1	1	1	1	1	1	1						
AR404	8	DELETED		B6 UGRD TA	TANKS	SOIL	0	0	GRAB	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0						
AR405	8		17/11/87	B212 U. TA	TANKS	SOIL	6	6	GRAB	0	0	0	0	0	0	6	6	0	0	0	0	0	0	0	0	0	0						
AR406	9	DELETED		WELL #6	WELL	GRN WATER	0	0	BAILR	0	2	0	2	0	0	0	0	0	0	0	2	0	2	0	2	0	2						
AR406	9		07/12/87	WELL #9	WELL	GRN WATER	2	2	PUMP	2	2	2	2	0	0	0	0	0	0	2	2	2	2	2	2	2	2						
AR407	9	DELETED		WELL #6	WELL	SOIL	0	0	GRAB	0	6	0	6	0	0	0	0	0	0	0	6	0	6	0	6	0	6						
AR407	9	DELETED		WELL #9	WELL	SOIL	0	0	GRAB	0	6	0	6	0	0	0	0	0	0	0	6	0	6	0	6	0	6						
AR407	9		01/12/87	WELL #9	WELL	SOIL	1	1	GRAB	1	1	1	1	0	0	0	0	0	0	1	1	1	1	1	1	1	1						
AR407	9		02/12/87	WELL #9	WELL	SUR WATER	1	1	QC RN	1	1	1	1	0	0	0	0	0	0	1	1	1	1	1	1	1	1						
AR407	9		03/12/87	WELL #6	WELL	SOIL	1	1	GRAB	1	1	1	1	0	0	0	0	0	0	1	1	1	1	1	1	1	1						
AR408	9	DELETED		800 LF NEM	WELL	GRN WATER	0	0	BAILR	0	1	0	1	0	0	0	0	0	0	0	1	0	1	0	1	0	1						
AR408	9		07/12/88	800 LF NEM	WELL	GRN WATER	1	1	BAILR	0	1	0	1	0	0	0	0	0	0	0	1	0	1	1	1	0	1						
AR411	9		17/11/87	800 LANDFI	WELL	GRN WATER	2	2	BKGRN	2	2	2	2	0	0	0	0	0	0	2	2	2	2	2	2	2	2						

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TABLE A.1  
 ARGONNE SITE ENVIRONMENTAL SAMPLES  
 WITH FIELD QC SAMPLES  
 SORTED BY ENVIRONMENTAL PROBLEM AND REQUEST NUMBER

REQ NUMB	PROB NUMB	ST COLL.	DATE DD/MM/YY	LOCATION	TYPE	MEDIA	NUMB SAMP		ANIONS		METALS		ORG		PET HYDRO		SOIL GAS		PES/H/PCB		SEMIVOLS		VOLS		RADS					
							ACTU	PLAN	ACTU	PLAN	ACTU	PLAN	ACTU	PLAN	ACTU	PLAN	ACTU	PLAN	ACTU	PLAN	ACTU	PLAN	ACTU	PLAN	ACTU	PLAN	ACTU	PLAN	ACTU	PLAN
							AL	NED	AL	NED	AL	NED	AL	NED	AL	NED	AL	NED	AL	NED	AL	NED	AL	NED	AL	NED	AL	NED	AL	NED
AR412	9		04/11/87	PLOT M	WELL	GRN WATER	2	4	BAILR	0	4	0	0	0	0	0	0	0	0	0	2	4	2	4	0	4				
AR413	9		09/12/87	PLOT M	WELL	GRN WATER	2	2	PUMP	2	2	2	2	0	0	0	0	0	2	2	2	2	2	2	2					
AR413	9		09/12/87	PLOT M NEW	WELL	GRN WATER	1	1	QC FL	1	1	1	1	0	0	0	0	0	0	0	0	0	0	1	1					
AR413	9		09/12/87	PLOT M NEW	WELL	GRN WATER	1	1	QC RN	1	1	1	1	0	0	0	0	0	1	1	1	1	1	1	1					
AR414	9	DELETED		DH3 & BH.4	WELL	GRN WATER	0	6	BAILR	0	6	0	6	0	0	0	0	0	0	0	0	6	0	6	0	6				
AR415	9		05/11/87	PLOT M	WELL	GRN WATER	1	1	BAILR	1	1	1	1	0	0	0	0	0	0	1	1	1	1	1	1					
AR415	9		05/11/87	PLOT M	WELL	GRN WATER	1	1	PUMP	1	1	1	1	0	0	0	0	0	0	1	1	1	1	1	1					
AR416	9	DELETED		319 AREA	WELL	GRN WATER	0	2	BAILR	0	2	0	2	0	0	0	0	0	0	2	0	2	0	2	0	2				
AR417	9	DELETED		319 LANDF.	WELL	SOIL	0	4	GRAB	0	4	0	4	0	0	0	0	0	4	0	4	0	4	0	4					
AR417	9		07/12/87	319 LANDF.	WELL	SOIL	1	1	GRAB	1	1	1	1	0	0	0	0	0	1	1	1	1	1	1	1					
AR418	9	DELETED		PLOT M	WELL	GRN WATER	0	2	BAILR	0	2	0	2	0	0	0	0	0	0	0	0	2	0	2	0	2				
AR418	9	DELETED		PLOT M	WELL	GRN WATER	0	1	QC FL	0	1	0	1	0	0	0	0	0	0	0	0	0	0	0	0	1				
AR419	9		05/11/87	PLOT M	WELL	GRN WATER	1	1	BAILR	1	1	1	1	0	0	0	0	0	0	0	1	1	1	1	1	1				
AR419	9		06/11/87	PLOT M	WELL	GRN WATER	1	1	BAILR	1	1	1	1	0	0	0	0	0	0	0	1	1	1	1	1	1				
AR420	9		06/11/87	317-319 LF	WELL	GRN WATER	2	2	BKGRN	2	2	2	2	0	0	0	0	0	2	2	2	2	2	2	2					
AR420	9		11/11/87	317-319 LF	WELL	GRN WATER	1	1	QC RN	1	1	1	1	0	0	0	0	0	1	1	1	1	1	1	1					
AR500	10		05/11/87	RET. TANKS	WASTEWATER	SUR WATER	1	1	QC RN	1	1	1	1	0	0	0	0	0	1	1	1	1	1	0	0					
AR500	10		05/11/87	RET. TANKS	WASTEWATER	SEALED CO	4	8	GRAB	2	4	2	4	0	0	0	0	0	2	4	2	4	4	8	0	0				
AR500	10		09/11/87	RET. TANKS	WASTEWATER	SEALED CO	11	17	GRAB	6	9	6	9	0	0	0	0	0	6	9	6	9	11	17	0	0				
AR500	10		10/11/87	RET. TANKS	WASTEWATER	SEALED CO	4	4	GRAB	2	2	2	2	0	0	0	0	0	1	2	2	2	4	4	0	0				
AR500	10		11/11/87	RET. TANKS	WASTEWATER	SUR WATER	1	1	QC FL	1	1	1	1	0	0	0	0	0	0	0	0	0	0	0	0	0				
AR500	10		11/11/87	RET. TANKS	WASTEWATER	SEALED CO	11	17	GRAB	6	9	6	9	0	0	0	0	0	4	9	6	9	11	17	0	0				
AR500	10		12/11/87	RET. TANKS	WASTEWATER	SEALED CO	4	4	GRAB	2	2	2	2	0	0	0	0	0	2	2	2	2	3	4	0	0				
AR500	10		13/11/87	RET. TANKS	WASTEWATER	SEALED CO	7	9	GRAB	4	5	4	5	0	0	0	0	0	4	5	4	5	7	9	0	0				
AR500	10		17/11/87	RET. TANKS	WASTEWATER	SEALED CO	4	4	GRAB	2	2	2	2	0	0	0	0	0	2	2	2	2	4	4	0	0				
AR500	10		18/11/87	RET. TANKS	WASTEWATER	SEALED CO	6	6	GRAB	3	3	3	3	0	0	0	0	0	3	3	3	3	6	6	0	0				
AR501	11		11/11/87	DRYING BED	SLUDGE	SEDIMENT	6	6	GRAB	6	6	6	6	0	0	0	0	0	6	6	6	6	5	6	6	6				
AR501	11		11/11/87	DRYING BED	SLUDGE	SUR WATER	1	1	QC RN	1	1	1	1	0	0	0	0	0	0	1	1	1	1	1	1	1				
AR502	12		06/11/87	B145 FLUE	DISCHARGES	SEDIMENT	3	3	GRAB	3	3	3	3	0	0	0	0	0	0	0	0	0	0	0	0	0				
AR503	12		06/11/87	B145 FLUE	DISCHARGES	SUR WATER	3	3	GRAB	3	3	3	3	0	0	0	0	0	3	3	0	0	0	0	0	0				
AR503	12		06/11/87	B145 FLUE	DISCHARGES	SUR WATER	1	1	QC RN	1	1	1	1	0	0	0	0	0	1	1	0	0	0	0	0	0				
AR504	13		11/11/87	B148 FLUE	SEEPAGE	SOIL	6	6	GRAB	6	6	6	6	0	0	0	0	0	0	0	0	0	0	0	0	0				
AR504	13		11/11/87	B148 FLUE	SEEPAGE	SUR WATER	1	1	QC RN	1	1	1	1	0	0	0	0	0	0	0	0	0	0	0	0	0				
AR505	14		06/11/87	317 AREA	BURN PILE	SOIL	2	2	GRAB	2	2	2	2	0	0	0	0	0	2	2	2	2	2	2	0	0				
AR506	15		06/11/87	B. 378/382	LEAD	SOIL	9	9	GRAB	0	0	9	9	0	0	0	0	0	0	0	0	0	0	0	0	0				
AR507	16		06/11/87	BLDG 108	SILT	SEDIMENT	3	3	GRAB	0	0	3	3	0	0	0	0	0	3	3	3	3	3	3	0	0				
AR508	16		06/11/87	BLDG 108	SILT	SUR WATER	1	1	QC RN	0	0	0	0	0	0	0	0	0	1	1	1	1	1	1	0	0				
AR508	16		06/11/87	BLDG 108	SILT	UNSEAL CO	3	3	GRAB	0	0	0	0	0	0	0	0	0	3	3	3	3	3	3	0	0				
AR800	17		06/11/87	319 LANDF	STREAM	SEDIMENT	6	6	GRAB	0	0	6	6	0	0	0	0	0	6	6	6	6	6	6	0	0				
AR801	17		16/11/87	319 LNDF-S	LANDFILL	SOIL	3	6	GRAB	0	0	3	6	0	0	0	0	0	3	6	3	6	3	6	0	0				
AR802	17		09/11/87	319 LDF-NM	BACKGROUND	SOIL	9	9	BKGRN	9	9	9	9	0	0	0	0	0	9	9	9	9	9	9	8	9				
AR802	17		09/11/87	319 LDF-NM	BACKGROUND	SUR WATER	1	1	BKGRN	1	1	1	1	0	0	0	0	0	1	1	1	1	1	1	1	1				

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TABLE A.1  
 ARGONNE SITE ENVIRONMENTAL SAMPLES  
 WITH FIELD QC SAMPLES  
 SORTED BY ENVIRONMENTAL PROBLEM AND REQUEST NUMBER

REQ NUMB	PROB NUMB	ST COLL.	DATE DD/MM/YY	LOCATION	TYPE LOCATION	MEDIA	NUMB SAMP		ANIONS		METALS		O&G		PET HYDRO		SOIL GAS		PES/H/PCB		SEMIVOLS		VOLS		RADS		
							ACTU	PLAN	AL	INED	AL	INED	AL	INED	AL	INED	AL	INED	AL	INED	AL	INED	AL	INED	AL	INED	AL
AR803	18	DELETED		317 AREA	DRAINAGE	SOIL	0	1	GRAB	0	1	0	1	0	0	0	0	0	0	0	1	0	1	0	1	0	0
AR803	18		10/11/87	317 AREA	DRAINAGE	SOIL	1	1	GRAB	1	1	1	1	0	0	0	0	0	0	1	1	1	1	1	1	0	0
AR803	18		12/11/87	317 AREA	DRAINAGE	SOIL	4	4	GRAB	4	4	4	4	0	0	0	0	0	0	4	4	4	4	3	4	0	0
AR804	18		05/11/87	SE 317 ARE	DRAINAGE	SEDIMENT	3	3	GRAB	3	3	3	3	0	0	0	0	0	0	3	3	3	3	3	3	0	0
AR804	18		05/11/87	SE 317 ARE	DRAINAGE	SUR WATER	1	1	QC RN	1	1	1	1	0	0	0	0	0	0	1	1	1	1	1	1	0	0
AR805	19		05/11/87	319 LANDFI	LANDFILL	SEDIMENT	3	3	GRAB	3	3	3	3	0	0	0	0	0	0	3	3	3	3	3	3	0	0
AR806	20		13/11/87	570 MTP	LAGOON	SOIL	7	8	GRAB	7	8	7	8	0	0	0	0	0	0	7	8	7	8	6	8	7	8
AR806	20		13/11/87	570 MTP	LAGOON	SUR WATER	1	1	QC RN	1	1	1	1	0	0	0	0	0	0	1	1	1	1	1	1	1	1
AR807	21		16/11/87	NIKE SITE	DRAINS	SOIL	6	6	GRAB	0	0	6	6	0	0	0	0	0	0	0	0	6	6	5	6	0	0
AR807	21		17/11/87	NIKE SITE	DRAINS	SOIL	2	2	GRAB	0	0	2	2	0	0	0	0	0	0	0	0	2	2	2	2	0	0
AR808	21	DELETED		NIKE SITE	DRAINS	SOIL	0	1	GRAB	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
AR808	21		17/11/87	NIKE SITE	DRAINS	SOIL	3	3	GRAB	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
AR808	21		17/11/87	NIKE SITE	DRAINS	SUR WATER	1	1	QC RN	0	0	0	0	0	0	0	0	0	0	1	0	1	0	0	0	0	0
AR809	22		10/11/87	UNDRWTRS P	POND	SEDIMENT	3	3	GRAB	0	0	3	3	0	0	0	0	0	0	0	0	0	0	0	0	0	0
AR809	22		10/11/87	UNDRWTRS P	POND	SUR WATER	1	1	QC RN	0	0	1	1	0	0	0	0	0	0	0	0	0	0	0	0	0	0
AR810	23		14/11/87	BLDG 19/34	D&D	SOIL	9	9	GRAB	9	9	9	9	0	0	0	0	0	0	0	0	9	9	9	9	0	0
AR811	24		16/11/87	317 VAULT	LOM L. MAST	SOIL	6	6	GRAB	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	6	6	
AR812	25		13/11/87	CP-3 AREA	SITE A	SOIL	6	6	GRAB	0	0	6	6	0	0	0	0	0	0	0	0	6	6	6	6	0	0
AR813	25		13/11/87	SITE A	SITE A	SOIL	7	7	GRAB	0	0	7	7	0	0	0	0	0	0	0	0	0	0	0	0	0	0
AR814	25	DELETED		SITE A	SITE A	AIR	0	3	GRAB	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
AR814	25		15/11/87	SITE A	SITE A	AIR	1	1	QC FL	0	0	0	0	0	0	0	0	0	0	1	0	0	0	0	0	0	0
AR814	25		15/11/87	SITE A	SITE A	AIR	3	3	GRAB	0	0	0	0	0	0	0	0	0	0	3	3	0	0	0	0	0	0
AR815	25	DELETED		SITE A	SITE A	SOIL	0	12	GRAB	0	0	0	12	0	0	0	0	0	0	0	0	0	0	0	0	0	0
AR815	25		13/11/87	SITE A	SITE A	SUR WATER	1	1	QC RN	0	0	1	1	0	0	0	0	0	0	1	1	1	1	1	1	0	0
AR816	26		12/11/87	SUNOCO STA	GAS SPILL	SOIL	1	2	GRAB	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
AR816	26		12/11/87	SUNOCO STA	GAS SPILL	AIR	3	3	QC FL	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
AR816	26		12/11/87	SUNOCO STA	GAS SPILL	AIR	4	4	GRAB	0	0	0	0	0	0	0	0	0	0	3	4	0	0	0	0	0	0
AR817	27		09/11/87	B145 DRUM	DRUM	SOIL	2	2	GRAB	0	0	0	0	0	0	0	0	0	0	0	0	2	2	2	2	0	0
AR818	28	DELETED		PLOT M	SEEP	SUR WATER	0	1	QC RN	0	0	0	1	0	0	0	0	0	0	0	0	0	1	0	1	0	0
AR818	28	DELETED		PLOT M	SEEP	GRN WATER	0	3	GRAB	0	3	0	3	0	0	0	0	0	0	0	0	0	3	0	3	0	0
AR819	28		04/11/87	PLOT M	SEEP	SEDIMENT	3	3	GRAB	3	3	3	3	0	0	0	0	0	0	0	0	3	3	2	3	0	0
ARN01	99		05/11/87		TRIP BLANK	WATER	1	1	QC BL	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1	1	0	0
ARN05	99		06/11/87		TRIP BLANK	WATER	1	1	QC BL	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1	1	0	0
ARN07	99		06/11/87		TRIP BLANK	WATER	1	1	QC BL	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1	1	0	0
ARN08	99		09/11/87		TRIP BLANK	WATER	1	1	QC BL	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1	1	0	0
ARN11	99		09/11/87		TRIP BLANK	WATER	1	1	QC BL	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1	1	0	0
ARN12	99		10/11/87		TRIP BLANK	WATER	1	1	QC BL	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1	1	0	0
ARN15	99		10/11/87		TRIP BLANK	WATER	1	1	QC BL	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1	1	0	0
ARN16	99		11/11/87		TRIP BLANK	WATER	1	1	QC BL	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1	1	0	0
ARN19	99		11/11/87		TRIP BLANK	WATER	1	1	QC BL	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1	1	0	0
ARN21	99		12/11/87		TRIP BLANK	WATER	1	1	QC BL	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1	1	0	0
ARN24	99		12/11/87		TRIP BLANK	WATER	1	1	QC BL	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1	1	0	0

TABLE A.1  
 ARGONNE SITE ENVIRONMENTAL SAMPLES  
 WITH FIELD QC SAMPLES  
 SORTED BY ENVIRONMENTAL PROBLEM AND REQUEST NUMBER

REQ NUMB	PROB NUMB	ST COLL.	DATE DD/MM/YY	LOCATION	TYPE	MEDIA	NUMB SAMP		ANIONS		METALS		O&G		PET HYDRO		SOIL GAS		PES/H/PCB		SEMIVOLS		VOLS		RADS					
							ACTU	PLAN	AL	PLAN	ACTU	PLAN	ACTU	PLAN	ACTU	PLAN	ACTU	PLAN	ACTU	PLAN	ACTU	PLAN	ACTU	PLAN	ACTU	PLAN	ACTU	PLAN	ACTU	PLAN
							AL	NED	AL	NED	AL	NED	AL	NED	AL	NED	AL	NED	AL	NED	AL	NED	AL	NED	AL	NED	AL	NED	AL	NED
ARN26	99		16/11/87		TRIP BLANK	WATER	1	1	QC	BL	0	0	0	0	0	0	0	0	0	0	0	0	1	1	0	0				
ARN28	99		16/11/87		TRIP BLANK	WATER	1	1	QC	BL	0	0	0	0	0	0	0	0	0	0	0	0	1	1	0	0				
ARN29	99		16/11/87		TRIP BLANK	WATER	1	1	QC	BL	0	0	0	0	0	0	0	0	0	0	0	0	1	1	0	0				
ARN34	99		19/11/87		TRIP BLANK	WATER	1	1	QC	BL	0	0	0	0	0	0	0	0	0	0	0	0	1	1	0	0				
ARN36	99		19/11/87		TRIP BLANK	WATER	1	1	QC	BL	0	0	0	0	0	0	0	0	0	0	0	0	1	1	0	0				
AR406	99		07/12/87	TRIP BLANK		WATER	2	2	QC	BL	0	0	0	0	0	0	0	0	0	0	0	0	1	2	0	0				
AR408	99		07/12/87	TRIP BLANK		WATER	1	1	QC	BL	0	0	0	0	0	0	0	0	0	0	0	0	0	1	0	0				
AR413	99		09/12/87	TRIP BLANK		WATER	2	2	QC	BL	0	0	0	0	0	0	0	0	0	0	0	0	0	2	0	0				
TOTAL							280	362			129	178	191	256	17	18	18	29	10	16	123	178	160	222	195	276	54	91		

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**Appendix B**

**BACKGROUND CONCENTRATION LEVELS OF ANALYTES**



Appendix B

BACKGROUND CONCENTRATION LEVELS OF ANALYTES OF ANALYTES

B.1 Radionuclides

B.1.1 Surface Water

The attached tables provide data on background concentration levels of contaminants in surface water near Argonne National Laboratory. Sawmill Creek runs through the ANL grounds. The 16K sampling site (Table B.1) is upstream from ANL. Sawmill Creek then drains into the Des Plaines River. Samples in rows labeled A (Table B.2) are upstream from the mouth of Sawmill Creek, and can therefore be considered as background. There are also samples from the Illinois River (Table B.3). These samples are from below the point where the Des Plaines River empties into the Illinois River. Apparently, however, the dilution by that point is sufficient that these samples do not contain any noticeable contamination from ANL.

B.1.2 Groundwater

From Table 4.10 of ANL-88-13, the levels of radionuclides in tap water were as follows:

Alpha (nonvolatile)	0.5	pCi/L
Beta (nonvolatile)	3.5	pCi/L
Tritium	< 100	pCi/L
Strontium-90	< 0.25	pCi/L
Radium-226	0.15	pCi/L
Uranium (natural)	0.31	pCi/L

B.1.3 Soil

The top 5 cm of soil at remote (offsite) locations were sampled to determine concentrations of radionuclides. The average of off-site results are as follows:

Potassium-40	19.23	± 2.05	pCi/g
Cesium-137	0.888	± 0.23	pCi/g
Radium-226	1.42	± 0.17	pCi/g
Thorium-228	1.01	± 0.20	pCi/g
Thorium-232	0.91	± 0.17	pCi/g
Plutonium-238	0.8	± 0.1	fCi/g
Plutonium-239	17.8	± 4.6	fCi/g
Americium-241	6.4	± 4.0	fCi/g

### B.1.4 Sediment

The average concentrations of radionuclides in (river) bottom sediment at remote (offsite) locations are as follows:

Potassium-40	17.41	± 3.78	pCi/g
Cesium-137	0.13	± 0.09	pCi/g
Radium-226	1.11	± 0.20	pCi/g
Thorium-228	0.75	± 0.17	pCi/g
Thorium-232	0.68	± 0.18	pCi/g
Plutonium-238	0.2	± 0.1	fCi/g
Plutonium-239	2.9	± 2.0	fCi/g
Americium-241	0.8	± 0.6	fCi/g

## B.2 Chemical Constituents

### B.2.1 Surface Water

The concentrations of chloride, sulfate, and total dissolved solids (TDS) are as follows:

Chloride	147	± 43	mg/L
Sulfate	90	± 30	mg/L
TDS	586	± 97	mg/L

Some additional background chemical data from Sawmill Creek, just upstream from the waste water outfall are given in Table B.4. Some background field data (pH and temperature) for surface water are also given.

The data in Table B.5 are from wells at the ANL site and from treated water. With regard to these data, the last paragraph on page 89 of ANL-88-13 states: "Samples from the wells and treated water were analyzed for the inorganic constituents listed in Table B.5. The results are similar to those obtained in the past and are levels normally found, except for the copper concentration of 83 ug/L in Well #1." Given that statement, I might consider using these data (except for the copper noted above) as background, or at least as an upper limit on background.

### REFERENCE:

Golchert, N.W., and T.L. Duffy. 1988. 1987 Annual Site Environmental Report for Argonne National Laboratory. ANL-88-13, Argonne National Laboratory, Argonne, Illinois 60439.

Table B.1. Radionuclides in Sawmill Creek Water, 1987

Type of Activity	No. of Samples	Avg.	Concentrations (pCi/L)	
			Min.	Max.
Alpha (Nonvolatile)	12 2.4	1.9 ± 0.1	0.8	
Beta (Nonvolatile)	12	7 ± 1	4	9
Hydrogen-3	12	< 138	< 100	321
Strontium-90	12	0.31 ± 0.01	< 0.25	0.48
Cesium-137	10	-	-	< 1.0
Uranium** (Natural)	12	2.0 ± 0.1	0.8	3.2
Neptunium-237	11	-	-	< 0.001
Plutonium-238	12	-	-	< 0.001
Plutonium-239	12	-	-	< 0.001
Americium-241	12	< 0.001	< 0.001	0.001
Curium-246 and/or Californium-252	12	-	-	< 0.001
Curium-244 and/or Californium-249	12	-	-	< 0.001

\*\* Uranium concentrations in units of mg/L can be obtained by multiplying the concentration given by 1.48.

Table B.2. Radionuclides in Des Plaines River Water, 1987

Type Of Activity	No. of Samples	Avg.	Concentration (pCi/L)	
			Min.	Max.
Alpha (Nonvolatile)	11	1.7 ± 0.2	1.3	2.4
Beta (Nonvolatile)	11	12 ± 3	8	19
Hydrogen-3	11	< 132	< 100	247
Strontium-90	11	0.29 ± 0.07	< 0.25	0.43
Uranium** (Natural)	11	1.3 ± 0.4	0.3	2.5
Neptunium-237	10	-	-	< 0.001
Plutonium-238	11	-	-	< 0.001
Plutonium-239	11	-	-	< 0.001
Americium-241	11	-	-	< 0.001
Curium-242 and/or Californium-252	11	-	-	< 0.001
Curium-244 and/or Californium-249	11	-	-	< 0.001

\*\* Uranium concentrations in units of ug/L can be obtained by multiplying the concentration given by 1.48.

**Table B.3. Radionuclides in Illinois River Water, 1987  
(Concentrations in pCi/L)**

Date Collected	Location	Alpha*	Beta*	Hydrogen-3	Uranium** (natural)	Plutonium-239
May 19	McKinley Woods State Park	0.9 ± 0.3	9.5 ± 0.4	138 ± 96	0.6 ± 0.1	< 0.001
May 19	Below Dresden Power Station	1.4 ± 0.2	3.4 ± 0.2	< 100	1.4 ± 0.1	< 0.001
May 19	Morris	2.1 ± 0.3	7.5 ± 0.3	156 ± 97	0.9 ± 0.1	-
May 19	Starved Rock State Park	1.4 ± 0.3	6.7 ± 0.3	112 ± 96	0.9 ± 0.1	-
October 1	McKinley Woods State Park	0.5 ± 0.2	7.7 ± 0.3	229 ± 74	0.5 ± 0.1	< 0.001
October 1	Below Dresden Power Station	0.7 ± 0.2	6.4 ± 0.3	231 ± 74	0.8 ± 0.1	< 0.001
October 1	Morris	0.6 ± 0.1	6.0 ± 0.3	181 ± 73	0.5 ± 0.1	-
October 1	Starved Rock State Park	1.0 ± 0.2	7.4 ± 0.3	182 ± 73	0.7 ± 0.1	-

\*Nonvolatile activity.

\*\*Uranium concentrations in units of µg/L can be obtained by multiplying the concentration by 1.48.

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Table B.4. Data for Sawmill Creek 15m (50ft) Upstream From  
 Waste Water Outfall, 1987

Constituent	Location	No. of Samples	Concentration (mg/l)		
			Avg.	Min.	Max.
Ammonia Nitrogen	7M (Up)	24	0.1 ± 0.0	0.1	0.1
Chloride	7M (Up)	24	148 ± 41	44	443
Dissolved Oxygen	7M (Up)	24	10.8 ± 1.0	6.7	15.5
Dissolved Solids	7M (Up)	24	592 ± 93	336	1110
pH*	7M (Up)	24	--	7.8	8.9
Sulfate	7M (Up)	24	92. ± 13	48	140
Temperature**	7M (Up)	24	14.1 ± 3.4	0.3	28.1

\*Unit  
 \*\*Degrees centigrade

Table B.5. Inorganic Constituents in Domestic Water, 1987  
 (Concentrations in mg/L)

Inorganic Constituent	Well Number				Treated Water
	1	2	3	4	
Aluminum	< 0.06	< 0.06	< 0.06	< 0.06	< 0.06
Antimony	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5
Arsenic	<0.004	< 0.004	< 0.004	< 0.004	< 0.004
Barium	0.094	0.081	0.052	0.050	0.050
Beryllium	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
Cadmium	< 0.004	< 0.004	< 0.004	< 0.004	< 0.004
Chromium	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02
Copper	0.083	< 0.02	< 0.022	< 0.02	< 0.02
Lead	< 0.01	< 0.01	< 0.01	0.12	< 0.004
Manganese	0.035	0.019	0.016	0.014	< 0.01
Mercury	< 0.0002	< 0.0002	< 0.0002	< 0.0002	< 0.0002
Molybdenum	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5
Nickel	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02
Selenium	< 0.002	< 0.002	< 0.002	< 0.002	< 0.002
Silver	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03
Sodium	36.9	24.4	22.4	21.1	21.7
Thallium	< 0.3	< 0.3	< 0.3	< 0.3	< 0.3
Vanadium	< 0.01	< 0.01	< 0.01	0.01	< 0.01
Zinc	< 0.02	0.027	0.016	0.011	0.011
Chlorides	79	55	49	42	58
Fluorides	0.24	0.29	0.30	0.33	0.3
Sulfates	140	130	100	140	150
Turbidity (NTU)	11.4	6.4	7.2	7.1	1.9



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U.S. Department of Energy  
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1000 Independence Avenue  
Washington, DC 20585

Dear Ms. Knight:

Enclosed is the final report by Jesse Gerard of LEMSCO for an on-site radiation measurement evaluation and the final report by Cynthia Miller, Jeffrey Worthington, and Betty Malone of Techlaw for an on-site evidentiary audit carried out at the Oak Ridge National Laboratory on August 25, 1987.

J. Gerard's report includes a completed copy of the new checklist for radiation measurement quality assurance support patterned after those established for the inorganic and organic technical areas under the Contract Laboratory Program (CLP) of the EPA. He outlined during the visit and the debriefing the data items required for a full data package for the sample designated group(s) that will get the full audit. ORNL will cooperate in furnishing this material.

The evidentiary audit covered all areas of the laboratory involved with the DOE environmental survey even though no technical evaluation was made during this visit for the organic and inorganic laboratory areas.

Of the four items noted in the Techlaw report as being repeated from the previous audit of June 10, 1987, the one of rewriting SOPs to may be the most extensive in effort but once done, will be the easiest to maintain or adapt in the future. The most difficult item of the four to keep from reappearing is the one involved with accounting for errors and error correction in the data documents. Training is important and supervisors have to vigilantly watch that proper correction is applied when bad data is to be identified as such. The other recommendations, both previous and from this audit can easily be addressed by following the procedures in the SOPs when they have been revised.

Sincerely,



Harold A. Vincent  
Chemist  
Quality Assurance Research Branch  
Quality Assurance and Methods Development Division

Enclosures

cc:

William Laing, ORNL  
Pamela Howell, ORNL  
Jeff Wade, ORNL

LABORATORY EVIDENCE AUDIT REPORT

FEB 22 1988

OAK RIDGE NATIONAL LABORATORY  
ANALYTICAL CHEMISTRY DIVISION  
MARTIN MARIETTA ENERGY SYSTEMS, INC.

OAK RIDGE, TENNESSEE

AUGUST 25, 1987

Martin Marietta Energy Systems, Inc. (MMES)  
Analytical Chemistry Division  
Oak Ridge National Laboratory  
Oak Ridge, TN 37830  
(615) 574-4898

William Laing	- Inorganic Chemistry Section Manager <sup>1,2,3</sup>
Bruce Clark	- Program Manager, Environmental Restoration and Facilities Upgrade Program <sup>1,2,3</sup>
Pam Howell	- Quality Control Officer <sup>1,2,3</sup>
Julian Hackney	- Analyst <sup>2</sup>
Julia Thompson	- Analyst <sup>2</sup>
Sophie Bobrowski	- Analyst <sup>2</sup>
Wayne Greist	- Group Leader, Separations and Synthesis <sup>2</sup>
Amelia Herndon	- Analyst <sup>2</sup>
W. Rogers	- Analyst <sup>2</sup>
Jeff Wade	- Group Leader, Low-Level Radio- chemical Analysis <sup>1,2,3</sup>
Robert Holmes	- Analyst <sup>2</sup>
K. Webb	- Analyst <sup>2</sup>
Sandra Glover	- Analyst <sup>2</sup>
B. Tomkins	- Analyst <sup>2</sup>
N. Ferguson	- Analyst <sup>2</sup>
J. Hayden	- Analyst <sup>2</sup>

---

<sup>1</sup>Present during pre-audit debriefing.

<sup>2</sup>Contacted during audit.

<sup>3</sup>Present during post-audit briefing.

USEPA/EMSL - Las Vegas, NV  
(702) 798-2129

Harold Vincent - Chemist

EMSL/LEMSCO - Las Vegas, NV  
(702) 798-3146

Earl Whittaker - Staff Scientist  
Jesse Gerard - Staff Scientist

NEIC/CEAT (TechLaw) - Denver, CO  
(303) 233-1248

Cynthia Miller - Staff Associate  
Jeffrey Worthington - Associate Consultant  
Elizabeth Malone - Associate Consultant

This work was conducted on behalf of the Environmental Protection Agency's (EPA) National Enforcement Investigations Center (NEIC) under EPA Contract #68-01-7369.

## INTRODUCTION

The National Enforcement Investigations Center (NEIC) assigned the Contract Evidence Audit Team (CEAT) to perform an evidence audit on Martin Marietta Energy Systems (MMES) Analytical Chemistry Division Laboratory located at Oak Ridge National Laboratory in Oak Ridge, Tennessee. The laboratory is receiving, preparing, and analyzing samples using USEPA Contract Laboratory Program (CLP) protocols for the Department of Energy's (DOE) Environmental Survey.

The purpose of this audit was to determine if laboratory policies and procedures are in place to satisfy evidence handling requirements. The report specifies the corrective action needed to meet EPA Evidence Audit Requirements.

The audit was conducted on August 25, 1987 in conjunction with a technical audit performed by representatives from the USEPA Environmental Monitoring Systems Laboratory (EMSL) at Las Vegas, Nevada.

The following operations, accompanying documentation, and written standard operating procedures (SOPs) were reviewed: sample receiving, sample storage, sample tracking (from receipt to completion of analysis), and analytical project file organization and assembly.

## EXECUTIVE SUMMARY

This was the third audit of MMES conducted by USEPA representatives in support of the DOE Environmental Survey Program. The previous audit was conducted on June 8-9, 1987 and resulted in nine recommendations. Four of the nine recommendations have not been addressed or corrected. The recommendations from the previous audit still requiring corrective action are:

1. The laboratory's written SOPs should be revised to include accurate descriptions of the actual laboratory procedures in the following areas:
  - a. Sample Receiving
  - b. Sample Storage
  - c. Sample Identification
  - d. Sample Security
  - e. Sample Tracking
  - f. Analytical Project File Organization and Assembly
2. Corrections to documents should be made by drawing a single line through the error and initialing and dating the correction. Correction fluid should not be used on Environmental Survey project-related documents.

3. Laboratory personnel should record the appropriate information on the Organic Sample Control and Chain-of-Custody Sheet or indicate that the activity was not performed.
4. Airbills should be routinely placed in the receiving document files.

The following six findings (non-conformances to Evidence Audit Requirements) were identified during the present audit and are discussed in this report:

#### Findings

1. Written SOPs did not contain accurate descriptions of the actual laboratory procedures used for the following:
  - a. Sample Receiving
  - b. Sample Storage
  - c. Sample Identification
  - d. Sample Security
  - e. Sample Tracking
2. Information was obliterated or rendered unreadable.
3. Error corrections were not consistently signed and dated by the analysts.
4. Entries in the explosives laboratory logbook are not consistently signed and dated.
5. Sample receiving information on the Organic Sample Control and Chain-of-Custody Sheet is not recorded in the space provided.
6. Airbills are not always placed in the receiving document file.

As a result of these findings, the following recommendations were made:

#### Recommendations

1. The laboratory's written SOPs should be revised to include accurate descriptions of the actual laboratory procedures in the following areas:

- a. Sample Receiving
  - b. Sample Storage
  - c. Sample Identification
  - d. Sample Security
  - e. Sample Tracking
2. Corrections to supporting documents and raw data should be made by drawing a single line through the error and entering the correct information.
  3. Corrections and additions to supporting documents and raw data should be dated and initialed.
  4. Logbook entries should be dated and signed by the analyst or individual performing the activity at the time the activity was performed.
  5. Laboratory personnel should record the appropriate information on the Organic Sample Control and Chain-of-Custody Sheet or indicate that the activity was not performed.
  6. Airbills should be routinely placed in the receiving document files.

The audit was concluded August 25, 1987. Audit participants are listed on the cover page of this report.

## PROCEDURAL AUDIT

The procedural audit consisted of review and examination of actual and written SOPs and accompanying documentation for the following laboratory operations: sample receiving, sample storage, sample identification, sample security, sample tracking (from receipt to completion of analysis), and analytical project file organization and assembly.

### Sample Receiving

Samples are received at the shipping/receiving area of the laboratory which is located approximately one mile from the laboratory building. A receiving clerk signs the airbills, and the sample containers are delivered to Building 4500S by the facility's delivery service. The Federal Express couriers may deliver the sample containers directly to Building 4500S on Saturdays.

Barry Grant, the designated sample custodian, takes possession of the containers. B. Grant inspects the custody seals and open the containers in the sample receiving area of Building 4500S. The custodian signs and dates the chain-of-custody records, checks for the presence/absence of receiving documents, and verifies the agreement/non-agreement among information recorded on the sample shipping documents. The sample custodian records the receiving information on the Shipping Container Sample Log-In Form.

According to Bruce Clark, problems associated with sample condition or documentation and their resolution are noted in the "Comments" column of the Shipping Container Sample Log-In Form and the "Remarks" column of the Field Chain-of-Custody Record. Also, according to Bruce Clark, tag numbers not referenced on shipping documents are recorded on the Field Chain-of-Custody Record.

A Request for Analytical Services Form is also received with the samples. This form contains information regarding sample identification and requested analyses.

An internal chain-of-custody receipt record is completed for each batch of samples received at the facility. This document is sent with the sample when delivered to the analyst. A unique laboratory identification number is assigned to each sample when the sample arrives at the laboratory where the analysis is to be performed. Each laboratory (inorganic, organic, radiochemistry) has the same method for assigning identification numbers. The

year is the first two (2) digits, the month is the second two (2) digits, the day is the third two (2) digits, and the sequence order representing the order in which the sample was checked in for that day is the last two (2) digits.

#### Inorganic Sample Receiving

The sample custodian makes a copy of the Request for Analytical Services Form and writes a request number on the original form. A sample identification number is then assigned to each inorganic sample, and the numbers are recorded on the original request and on the Sample Log-In Sheet.

Copies of the Request for Analytical Services Form are sent to each inorganic laboratory to serve as notification of sample arrival. The samples are placed in a storage area located adjacent to the sample receiving area.

#### Organic Sample Receiving

The sample custodian sends a Request for Analytical Services form to the organic analysis department to inform the department of the arrival of samples. The organic laboratory assigns identification numbers to each sample and places them in storage.

#### Radiochemistry Sample Receiving

A copy of the Request for Analytical Services Form is also sent to the radiochemistry laboratory. The radiochemistry laboratory assigns identification numbers to each sample and places them in storage.

Written SOPs for sample receiving have been developed and implemented. The auditor read these SOPs, and they did not accurately describe the procedures in use for sample receiving. These SOPs are documented in Quality Assurance/Quality Control Standard Operating Procedure and Sample Receipt and Handling.

#### Sample Storage, Identification, and Security

Storage, identification, and security procedures are described in the four sections below.

##### Inorganic Sample Storage and Identification

Inorganic samples are stored in the Building 4500S storage room located immediately adjacent to the sample receiving room. Samples designated for Inductively Coupled Plasma (ICP) analysis may also be stored in the same storage room. If samples are delivered on Saturday, all samples could be stored here.

Inorganic samples are identified with the field identification number and the assigned laboratory number. Sample preparation containers are identified with the laboratory number, percent acid, and sample weight or volume.

In Building 1505, samples are stored in a locked three-door refrigerator located in the hallway near the entrance to the atomic absorption (AA) laboratory. Prepared AA metals samples are stored in locked cabinets in the AA laboratory. Samples and digestates for AA and mercury analysis are also stored in locked cabinets in Building 2026 Annex.

Samples prepared for AA and mercury analysis (digestates) are identified with the field identification number and the laboratory number. Sample preparation containers are identified with the laboratory number.

#### Organic Sample Storage and Identification

Organic samples are stored in the sample preparation laboratory located in Building 4500S. Extracts are stored in a refrigerator located adjacent to the analysis area.

Organic samples are identified with the field number and the assigned laboratory number. Sample extract vials are marked with a marking pen or sticker indicating the assigned laboratory number.

#### Radiochemistry Sample Storage and Identification

Samples requiring radiochemistry analysis are stored in the locked custody room located in the radiochemistry department in Building 4500S. These samples are identified with the field identification number and the assigned laboratory number.

#### Security

The refrigerators and sample storage areas are locked at night. The facility is surrounded by a fence. Visitors must enter through a visitor screening center, obtain an identification tag, and sign in before they are allowed to enter the facility. The visitors are not escorted when entering the facility. This was discussed during the post-audit debriefing. The AA preparation and analysis laboratories in Building 1505 are locked at night.

Written SOPs for sample storage, identification, and sample security have been developed and implemented. The auditors read these SOPs, and they described the procedures in the laboratory; however, they did not accurately describe the storage areas in

the laboratory that will be used for Environmental Survey samples. The SOPs are documented in the laboratory SOPs Quality Assurance/Quality Control Standard Operating Procedures and Sample Receipt and Handling.

### Sample Tracking

All samples are currently received at the "inorganic receiving area" of Building 4500S. Metals samples requiring ICP analysis are also prepared and analyzed in Building 4500S.

Cyanide, oil and grease, ion chromatography, and radio-chemistry tests are performed in Building 4500S. Asbestos analyses are performed in Building 4500N.

Metal samples for AA analysis are delivered to Building 1505. These samples are then taken to the Building 2026 Annex where they are prepared (digested). The mercury fraction is analyzed by cold vapor AA in Building 2026 Annex. The AA metals digestates are returned to Building 1505 where they are analyzed by Furnace AA.

The preparation and analysis of "explosives" samples are performed in Building 2026 Annex.

Samples may be tracked through the laboratory from receipt to completion of analysis by using the following documents:

1. Shipping Container Sample Log-In Forms
2. Request for Analytical Services (Several Copies)
3. Receipt Record/Chain-of-Custody Forms
4. ICP Preparation Logs
5. ICP Preparation Control Worksheets
6. ICP Analysis Logbooks (ICP EPA/CLP Program Log)
7. Log-In Books (AA and Hg Samples)
8. Contract Laboratory Samples - Flame AA and Furnace AA Analyses Building 1505 Logbook
9. Contract Laboratory Samples Preparation and Mercury Analysis Building 2026 Annex Log (AA and Hg Preparation, Hg Analysis)
10. AA Analysis Control Worksheets
11. CLP Logbooks (Cyanide Preparation and Analyses)
12. Phenol Analysis Logbooks
13. Ion Chromatography Analysis Control Worksheets
14. Asbestos Samples Pantex (Asbestos Determinations)
15. CLP Logbooks (Oil and Grease Determinations)
16. Oil and Grease Analysis Control Worksheets
17. Uranium Analysis Control Worksheets

18. CLP Logbooks (Explosives Weight and Identification Number)
19. HPLC Sample Logbooks (Explosive Analyses)
20. Sample Preparation Logsheets (Organic Preparation)
21. GC/MS Instrument Operations Logsheets
22. GC Instrument Operations Logsheets
23. Chain-of-Custody Record Low-Level Radiochemical Analysis Group
24. Alpha/Beta Worksheets
25. Gamma Scan Worksheets

The procedures and documentation used to track inorganic and organic samples and radiochemistry samples are described in the following three sections.

### Inorganic Sample Tracking

Copies of the Request for Analytical Services Forms (with the assigned inorganic batch number) are sent to the appropriate inorganic laboratories by B. Grant to serve as notification of the arrival of samples. Preparation of samples for ICP analysis are documented in the ICP preparation logbook entitled Logbook for P.E. and EPA Sample J. H. Hackney, 4500 SR-147. ICP preparation information is also recorded on an ICP Preparation Control Worksheet. The ICP analyses are recorded in the logbook entitled ICP EPA/CLP Program Log.

Metals samples for AA analysis are brought to Building 1505 after the laboratory personnel signs the Receipt Record/Chain-of-Custody Record.

The samples are then delivered to Building 2026 where mercury and inorganic sample digestions are recorded in a logbook entitled Contract Laboratory Samples Preparation and Mercury Analysis Building 2026 Annex Log. The mercury analyses are performed in Building 2026 and recorded in the same logbook as well as a Mercury Control Worksheet. The transfer of samples to Building 2026 and back to Building 1505 is recorded in the Log-In Book.

The prepared metal digestates are returned to Building 1505 for analysis and are accompanied by the logbook (Contract Laboratory Samples - Flame AA and Furnace AA Analyses Building 1505 Logbook). The AA analyses are recorded in the previously described logbook and on AA Control Worksheets.

Cyanide analyses are performed in Building 4500S and are recorded in a logbook entitled CLP. Ion chromatography analysis is performed in Building 4500S. The analyses are recorded on Ion Chromatography Control Worksheets. The instrument produces a strip chart.

Asbestos determinations are performed in Building 4500N. This analysis is recorded in a logbook entitled Asbestos Samples Pantex. The laboratory has not analyzed any samples for phenols. According to J. Stewart, a logbook for phenols analysis will be initiated when samples arrive with a request for phenols analysis. Oil and grease determinations are recorded in a logbook entitled CLP and the Oil and Grease Analysis Control Worksheets.

#### Organic Sample Tracking

Organic samples are brought to the organic sample preparation area with a Request for Analytical Services Form and an Analytical Chain-of-Custody Form/Receipt Record that had been initiated by the sample receiving department. This record was previously described in the inorganic sample tracking section.

The preparation chemist signs the custody form and initiates the Record Receipt/Chain-of-Custody. The auditors observed that the receiving information was not consistently recorded on this form.

The preparation chemist assigns a batch number to the Request for Analytical Services Form, copies the request form, and then tapes the copy into a logbook entitled No. 4 Sample Log.

Extraction data is recorded on the Sample Preparation Logsheet. Copies of this logsheet are also taped into the No. 4 Sample Log.

The analysis of the volatile fraction is recorded on the GC/MS Instrument Operations Logsheet (GC/MS Logbook). The analysis of the base/neutral/acid fraction is recorded in a separate GC/MS logbook.

The pesticides analysis is recorded on the GC Instrument Operations Logsheet (Logbook).

The explosive analysis is recorded in the HPLC Sample Log. The weight of each sample is recorded in a CLP logbook. The auditors observed that the information in both logbooks were not consistently dated and signed.

#### Radiochemistry Sample Tracking

The transfer of samples to the radiochemistry laboratory is recorded on the Chain-of-Custody Record Low-Level Radiochemical Analysis Group (LLRAG) Form in addition to the previously mentioned Receipt Record/Chain-of-Custody. This form is also used to track the sample through the radiochemistry laboratory.

Summaries of preparation and analyses radiochemistry are recorded in the untitled radiochemistry logbook. Alpha and beta counts are recorded on the Alpha/Beta Worksheet. Gamma scans are recorded on the Gamma Scan Worksheet.

The uranium analysis is recorded on the Uranium Analysis Control Worksheet.

Written SOPs for sample tracking have been developed and implemented. The auditor read these SOPs, and they did not accurately describe the documents used to track samples and the analytical paths of the various sample fractions. The written SOPs are documented in Quality Assurance/Quality Control Standard Operating Procedures and Sample Receipt and Handling.

#### Analytical Project File Organization and Assembly

Receiving documents are currently filed in the laboratory receiving room. Preparation logbooks remain in the possession of the analysts. Analysis logbooks are kept in the analytical area of the laboratory. The Organic Chain-of-Custody Forms are kept in files in the organics laboratory office. Airbills are retained by the receiving clerk.

The laboratory has not developed actual or written procedures for the organization and assembly of laboratory documents related to the receipt, storage transfer, preparation, and analysis of Environmental Survey samples. (Technical direction has not been received from DOE in this area.)

#### EVIDENCE AUDIT

The evidence audit consisted of review and examination of analytical project file documentation. Completed analytical project files have not been assembled, numbered, or inventoried. Thus, the auditors could make no observations concerning the completeness and consistency of analytical project files.

#### AUDIT FINDINGS

The following six findings (non-conformances to Evidence Audit Requirements) are based on the results of the procedural and evidence audits.

#### Findings

1. Written SOPs did not contain accurate descriptions of the actual laboratory procedures used for the following:

- a. Sample Receiving
  - b. Sample Storage
  - c. Sample Identification
  - d. Sample Security
  - e. Sample Tracking
2. Information was obliterated or rendered unreadable.
  3. Error corrections were not consistently signed and dated by the analysts.
  4. Entries in the explosives laboratory logbook are not consistently signed and dated.
  5. Sample receiving information on the Organic Sample Control and Chain-of-Custody Sheet is not recorded in the space provided.
  6. Airbills are not always placed in the receiving document file.

#### SUMMARY

A debriefing session was held on August 25, 1987 with MMES personnel. During this debriefing, the evidence auditors made the following recommendations based on the findings discussed in this report:

1. The laboratory's written SOPs should be revised to include accurate descriptions of the actual laboratory procedures in the following areas:
  - a. Sample Receiving
  - b. Sample Storage
  - c. Sample Identification
  - d. Sample Security
  - e. Sample Tracking
2. Corrections to supporting documents and raw data should be made by drawing a single line through the error and entering the correct information.
3. Corrections and additions to supporting documents and raw data should be dated and initialed.
4. Logbook entries should be dated and signed by the analyst or individual performing the activity at the time the activity was performed.

5. Laboratory personnel should record the appropriate information on the Organic Sample Control and Chain-of-Custody Sheet or indicate that the activity was not performed.
6. Airbills should be routinely placed in the receiving document files.



Environmental Programs Office  
1050 E. Flamingo Road, Suite 120, Las Vegas, Nevada 89119

FEB 22 1988

January 28, 1988

United States Environmental  
Protection Agency  
P.O. Box 93478  
Las Vegas, Nevada 89193-3478

ATTENTION: DR. HAROLD VINCENT, QAD

VIA: <sup>7/24/88</sup> ~~2/2/88~~ M. T. HOMSHER

SUBJECT: ON-SITE RAD PREASSESSMENT EVALUATION OF OAK RIDGE  
NATIONAL LABORATORY (ORNL/X-10).

Dear Dr. Vincent:

This is the detailed RAD Preassessment Evaluation Report for  
ORNL/X-10. A preliminary report was sent to you on September 2,  
1987. Due to a lack of funds, this report is about four months  
beyond its due date.

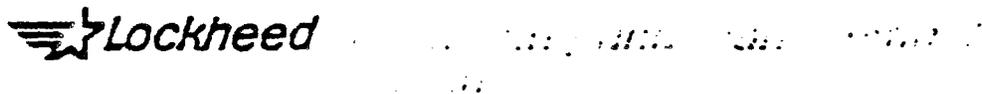
Very truly yours,

Jesse T. Gerard  
Staff Scientist  
QA Department

JTG/ahh

cc: M. T. Homsher      D. W. Bottrell  
R. D. Flotard      K. J. Cabbie  
J. D. Petty      J. Huber  
C. S. Soong      E. Whittaker  
J. O. 70.23      WP-1916C  
DES 9-122

ATTACHMENT



Environmental Programs Office  
1050 E. Flamingo Road, Suite 120, Las Vegas, Nevada 89119

January 19, 1988

United States Environmental  
Protection Agency  
P.O. Box 93478  
Las Vegas, Nevada 89193-3478

ATTENTION: DR. HAROLD C. VINCENT

SUBJECT: RAD PREASSESSMENT ON-SITE EVALUATION OF OAK RIDGE  
NATIONAL LABORATORY (ORNL/X-10) ON AUGUST 25, 1987

Dear Dr. Vincent:

The subject RAD preassessment on-site evaluation has been completed and the following items must be given attention in order to improve data integrity.

1. Logbooks and laboratory notebooks were not signed and dated by personnel or verified by signing and dating by the supervisor. This was the case across the board for all techniques. Additionally, notebook/logbook changes were not crossed out and initialed by personnel making the changes.
2. It is recommended that an instrument logbook be maintained for the  $\gamma$ -ray spectroscopy area with instrument settings etc., entered.
3. It is recommended that manual validation checks of computer generated data/results be performed randomly at a fixed frequency. For example, rather than blindly accepting computer data reduction results of  $\gamma$ -ray spectra it is recommended that manual checks be made (printing out digital channel data and hand calculation/calculator computation of peak areas) to ensure that something has not gone wrong and that the method of computer integration is appropriate for the situation. Results of the computer versus hand calculated final results should be documented in a logbook/notebook in a continuing fashion easy to follow with time. Retain calculations and data for archival purposes.

4. At present, ORNL is not storing raw data for archival purposes. Raw data being data directly output from the equipment (instrument settings, etc., for runs would be available in logbooks), onto disks or tapes, etc. Raw data is data on which a decision has not been irreversibly made so that at a future date, one can return to the original data/instrument output (in the case of  $\gamma$ -spectroscopy all 2000/4000 channels) as versus data reduced in a fashion so that original instrument output data cannot be regenerated. It is recommended that all data output directly from equipment be stored on disk, or tape, etc., for future retrieval. The capability already exists to do this at ORNL but it is not being done.
5. Written SOPs were not available for the overall program sample receipt and storage area - nor were appropriate portions available to the sample custodian.
6. As a general recommendation, it is suggested that survey program wide Gross  $\alpha$  and Gross  $\beta$  procedures for soils, sludges etc., be used that can provide comparable data such as consistent comparably low detection limits as well as good precision and accuracy. The variation of capabilities of procedures among different laboratories is wide and since the site survey plans are beginning to depend more heavily on survey/screening techniques such as Gross  $\alpha$ , Gross  $\beta$  and  $\gamma$ -scan it is very important that comparable data be generated across all sites especially since these results will be used to prioritize sites for further work. These procedures for water and air filters seem to be quite acceptable and comparable and seem to be well documented.
7. While analyses are being performed (or planned) for Gross  $\alpha$ , Gross  $\beta$ ,  $\gamma$ -Scan,  $^3\text{H}$ , Tot.U etc., in soils and sludges, validated "Survey Analysis and Sampling Manual Appendix 4: Radiochemical Analyses" procedures for ORNL (X-10) could not be found.
8. Based on conversations on July 27, 1987 at a meeting in Las Vegas, K. Knight expressed support for all DOE Laboratories participating in the Environmental Program to also participate in the EML PE program and EPA drinking water PE/IC samples. It is recommended that ORNL participate on a full regular basis in those programs for those radionuclides/parameters associated with the DOE Environmental Survey Program for matrices involved in site analyses requested of them. Past participation generally is good and quite comprehensive but ORNL participation does not cover all parameters required for the DOE Environmental Survey Program even though available in the PE samples.

DR. HAROLD C. VINCENT  
RAD PREASSESSMENT ON-SITE EVALUATION....  
PAGE III

9. Data audit sample reporting requirements for reporting of data/results on samples to be audited were discussed and it was generally felt and agreed that lab personnel understood what was required.

Details of some of the above items may be found in the text of this report. An evidentiary audit was conducted simultaneously. Their findings will be provided in a separate report.

Laboratory: Oak Ridge National Laboratory (X-10)  
Date: August 25, 1987  
Type of Evaluation: RAD Preassessment On-Site Evaluation

Personnel Contacted:

NAME	TITLE
Bruce R. Clark	Coordinator, DOE Environmental Survey Program
Pamala Howell	QA Specialist
Jeff W. Wade	Supervisor of RAD Analytical Area
Bill Laing	Section Head QA Office
Joe Stewart	Fluorimetry Expert

Laboratory Evaluation Team:

Jesse T. Gerard	RAD QA Evaluator
Earl Whittaker	RAD QA Evaluator
Harold Vincent	Task Monitor DOE Site Survey Program
Cynthia L. Miller	Techlaw (CEAT) Auditor
Betty C. Malone	Techlaw (CEAT) Auditor
Jeff Worthington	Techlaw (CEAT) Auditor

A. Procedural Changes the Laboratory Agreed to Implement

The following comments refer to deficiencies noted in the Laboratory Evaluation Checklist (Attachment 1).

For comments see page 1, 2, and 3 above and also page 6, item D.

B. Review of Environmental Measurements Laboratory and EPA Drinking Water Performance Evaluation Samples

The results of both were discussed with the laboratory personnel:

For comments see page 2, item 8 above.

C. Review of Data Audit

The following comments refer to the Summary/Conclusions of the data audit for Problem No \_\_\_\_\_, Request No. \_\_\_\_\_ (Attachment 2.)

<u>Report</u>		
<u>Item #</u>	<u>Comments</u>	<u>Action*</u>

Information on samples for data audits has not been received yet-as this stage is just beginning to evolve. See page 3, item 9 above for comment.

D. Issues to be Resolved by DOE Headquarters

As is required for items page 1, 2 and 3 since this is a preassessment evaluation.

Attachment 1

Laboratory Evaluation Checklist

I. Organization and Personnel (Page 1 of 2)

ITEM	YES	NO	COMMENT
Laboratory or Project Manager (individual responsible for overall technical effort)  Name: <u>Bruce R. Clark</u>	x		615-574-6896
Name: <u>Jeff W. Wade</u> Job Title: <u>Supervisor RAD Anal. Chem.</u>	x		615-574-4528
Name: <u>Bill Laing</u> Job Title: <u>Section Head, QA Office</u>	x		
Name: <u>Joe Stewart</u> Job Title: <u>Fluorimetry Expert</u>	x		615-574-4895
Name: _____ Job Title: _____			
Name: _____ Job Title: _____			
Name: _____ Job Title: _____			
Do personnel assigned to this project have the appropriate background to successfully accomplish the objectives of the program?	x		



II. Sample Receipt and Storage Area (Page 1 of 1)

ITEM	YES	NO	COMMENT
Are written Standard Operating Procedures (SOPs) developed for receipt and storage of samples?		x	For RAD area, yes.
Is the appropriate portion of the SOP available to the sample custodian at the sample receipt/storage area?		x	For RAD area, yes.
Are adequate facilities provided for storage of samples.	x		
Are the sample receipt/storage and records maintained in a manner consistent with program needs?	x		
Are standards stored separately from sample digestates?	x		
Has the supervisor of the individual maintaining the notebook/bench sheet/logbook personally examined and reviewed the notebook/bench sheet/logbook periodically, and signed his/her name therein, together with the date and appropriate comments as to whether or not the document is being maintained in an appropriate manner?		x	

Additional Comments

Main DOE Environmental Survey Receipt and Storage SOPs were not completed at this point in time.

III. Sample Preparation Area (Page 1 of 2)

When touring the facilities, give special attention to: (a) the overall appearance of organization and neatness, (b) the proper maintenance of facilities and instrumentation, (c) the general adequacy of the facilities to accomplish the required work.

ITEM	YES	NO	COMMENT
Is the laboratory maintained in a clean and organized manner?	x		
Does the laboratory appear to have adequate workspace (120 sq. feet, 6 linear feet of unencumbered bench space per analyst)?	x		
Are contamination-free areas provided for trace level analytical work? (Low level and high activity areas separated.)	x		
Are the hoods in good condition and functional?	x		
Are chemical waste disposal policies/procedures well defined and followed by the laboratory?	x		
Does the laboratory have a source of distilled/demineralized water?	x		
Is the conductivity of distilled/demineralized water routinely checked and recorded?		x	Not needed?
Is the analytical balance located away from draft and areas subject to rapid temperature changes?	x		
Has the balance been calibrated within one year by a certified technician?	x		Quarterly.
Is the balance routinely checked with the appropriate range of class S weights daily before use and are the results recorded in a logbook?	x		Contracted.
Is the sample preparation portion of the SOP available to the analyst at the sample preparation area?	x		
Are unexpired standards used to prepare instrument calibration standards?	x		
Are fresh analytical standards prepared at a frequency consistent with good QA?	x		
Are chemicals and standards dated upon receipt?	x		
Are reference materials properly labeled with concentrations, date of preparation, and the identity of the person preparing the sample?	x		
Is a spiking/calibration standards preparation and tracking logbook(s) maintained?	x		
Are the primary standards traceable to NBS standards where possible?	x		
Do the analysts record bench data in a neat and accurate manner?	x		



IV. Sample Analysis Instrumentation (Page 1 of 11)

A. Gamma-Ray Spectrometer

	Manufacturer	Model	Automated Sample Exchanger Used	Installation Date
1. Spectrometer		GeLi-		
ID# 1		(1)LGC2250LATT		
2	Two PyT's	(2)LGC2250LATT	Manual	5 years old
Data System	ND-9900			
2. Spectrometer		Ge-		
ID# 3		(3)2020		
4	Two Canberra's	(4)2001	Manual	6 years old
Data System	ND-9900			
3. Spectrometer		Ge-		
ID# 5		(5)OTZDS30-25185		
6	Two Tennelec's	(6)CPZDS30-25185	Manual	< 1 year old
Data System	ND-9900			
4. Spectrometer				
ID#				
Data System				
5. Spectrometer				
ID#				
Data System				
6. Spectrometer				
ID#				
Data System				

Spectrometers 1, 2, 3, 4 are approx. 20% effic., 5 is 25% and 6 is 30% - 3 inch lead chambers used. ND-9900 controls all 6 detectors.

IV. Sample Analysis Instrumentation (Page 2 of 11)

A. Gamma-Ray Spectrometer

ITEM	YES	NO	COMMENT
Are operating manuals readily available to the operator?	x		
Are calibration protocols available to the operator?	x		
Are energy, efficiency, FWHM values, gains and check standard results kept in a permanent record so that instrument performance can be measured over time?		x	Yes, except for Inst., logbook settings i.e., gains etc.
Is there a methods manual (SOP) available to the operator?	x		
Are NBS traceable standards used for calibration?	x		
Duplicate samples analyzed? (Frequency)	x		1/10, 1 per batch
Spike/standard samples and blanks? (Frequency)	x		1/20, 1/10, 1 per batch.
Is a permanent service record maintained in a logbook?	x		
How is the data reduced-off line computer, dedicated system or other?	x		Dedicated.
Are radioisotopic or interelement correction factors updated every six months or more frequently?		x	Avoided.
Is service maintenance by contract?	x		
Is preventative maintenance applied?	x		

Additional Comments

Blindly takes computer output without performing manual validation checks (see item 3, page 1).

Does not store raw data for archival purposes even though capability exists to do so (see item 4, page 2).

Calibrates efficiency, resolution etc., each day and maintains results in logbook with printout.

IV. Sample Analysis Instrumentation (Page 3 of 11)

B. Alpha Spectrometer

	Manufacturer	Model	Automated Sample Exchanger Used	Installation Date
1. Spectrometer ID#				
1, 2, 3, 4	Tennelec	Si(Li) TC-256	Manual	2 years old
Data System	ND-9900			
2. Spectrometer ID#				
5, 6, 7, 8	Tennelec	Si(Li) TC-256	Manual	2 years old
Data System	ND-9900			
3. Spectrometer ID#				
9, 10, 11, 12	Tennelec	Si(Li) TC-256	Manual	2 years old
Data System	ND-9900			
4. Spectrometer ID#				
Data System				
5. Spectrometer ID#				
Data System				
6. Spectrometer ID#				
Data System				

3-Four simultaneously operated  $\alpha$ -spectrometers for a total of 12 available. 1024 channels used for spectra. ND-9900 controls all detectors. All are part of the same system so there is only one model number TC-256.

B. Alpha Spectrometer

ITEM	YES	NO	COMMENT
Are operating manuals readily available to the operator?	x		
Are calibration protocols available to the operator?	x		
Are energy, efficiency, FWHM values, gains and check standard results kept in a permanent record so that instrument performance can be measured over time?	x		
Is there a methods manual (SOP) available to the operator?	x		
Are NBS traceable standards used for calibration?	x		
Duplicate samples analyzed? (Frequency)	x		1/10, 1 per batch
Spike/standard samples and blanks? (Frequency)	x		1/10, 1/20, 1 per batch.
Is a permanent service record maintained in a logbook?	x		
How is the data reduced--off line computer, dedicated system or other?	x		Dedicatd.
Are radioisotopic or interelement correction factors updated every six months or more frequently?		x	Avoided-not applicable.
Is service maintenance by contract?	x		
Is preventative maintenance applied?	x		

Additional Comments

Calibrates efficiency and resolution etc., each day and maintains results in logbook with printouts.

IV. Sample Analysis Instrumentation (Page 5 of 11)

C. Low Background Gas Flow Proportional Counting System (Gross Alpha and Gross Beta)

	Manufacturer	Model	Sample Capacity	Installation Date
1. Instrument				
ID#				
Gross $\alpha$ /B Ctr	Tennelec	LB5100	Multiple	3 years old.
Window		Voltage		Operating $\alpha$ =750
Density or Thickness	260 $\mu\text{g}/\text{cm}^2$	Plateau	Not available	Voltage $\beta$ =1470
		Span and Slope	Not available	Gas p-10(Ar, Me)
(Rack of 4) x 3 = 12 at a time				
2. Instrument				
ID#				
$^{90}\text{Sr}$ Ctr	Tennelec	LB4000	Manual	Not Available
Window		Voltage		Operating $\alpha$ =1200
Density or Thickness	260 $\mu\text{g}/\text{cm}^2$	Plateau	Not available	Voltage $\beta$ =1913
		Span and Slope	Not available	Gas p-10. (Ar, Me)
3. Instrument				
ID#				
Window		Voltage		Operating
Density or Thickness		Plateau		Voltage
		Span and Slope		Gas
4. Instrument				
ID#				
Window		Voltage		Operating
Density or Thickness		Plateau		Voltage
		Span and Slope		Gas
5. Instrument				
ID#				
Window		Voltage		Operating
Density or Thickness		Plateau		Voltage
		Span and Slope		Gas

1 system of each type. The second one is the older of the two.

IV. Sample Analysis Instrumentation (Page 6 of 11)

C. Low Background Gas Flow Proportional Counting System (Gross Alpha and Gross Beta)

ITEM	YES	NO	COMMENT
Are operating manuals readily available to the operator?	x		
Are calibration protocols available to the operator?	x		
Are calibration results kept in a permanent record so that instrument performance can be measured over time?	x		
Is there a methods manual (SOP) available to the operator?	x		
Are NBS traceable standards used for calibration?	x		
Is a permanent service record maintained in a logbook?	x		
How is the data reduced--off line computer, dedicated system or other?	x		Each has its own microprocessor-HP
Is calibration done at least daily or batch frequency?	x		
Duplicate samples analyzed? (Frequency)	x		1/10, 1 per batch
Spike/standard samples and blanks? (Frequency)	x		1/10 stds, 1/20 spikes, 1/batch.
Are self-absorption curves readily available to analyst (curves reestablished last 3 months)?	x		Daily checked.
Is service maintenance by contract?	x		
Is preventative maintenance applied?	x		

Additional Comments

Calibrates efficiency, etc., each day and maintains results in logbook with printouts.

IV. Sample Analysis Instrumentation (Page 7 of 11)

D. Liquid Scintillation (LS) Spectrometer

	Manufacturer	Model	Sample Capacity	Installation Date
1. LS Spectrometer ID# 1	Packard	460C	Multiple	5-6 years old

Data System Data output by system is manually feed into area computer

2. LS Spectrometer  
ID#

Data System

3. LS Spectrometer  
ID#

Data System

4. LS Spectrometer  
ID#

Data System

5. LS Spectrometer  
ID#

Data System

6. LS Spectrometer  
ID#

Data System

1 liquid scintillation system only.

IV. Sample Analysis Instrumentation (Page 8 of 11)

D. Liquid Scintillation (LS) Spectrometer

ITEM	YES	NO	COMMENT
Are operating manuals readily available to the operator?	x		
Are calibration protocols available to the operator?	x		
Are calibration results (i.e., sensitivity) kept in a permanent record so that instrument performance can be measured over time?	x		
Is there a methods manual (SOP) available to the operator?	x		
Are NBS traceable standards used for calibration?	x		
Is a permanent service record maintained in a logbook?	x		
How is the data reduced-off line computer, dedicated system or other?	x		Raw data input into area computer manually.
Duplicate samples analyzed? (Frequency)	x		1/10, 1 per batch
Spike/standard samples and blanks? (Frequency)	x		Stds 1/10, spikes 1/20, 1 per batch
Is calibration done at least daily or batch frequency?	x		Per setup or each day.
Are multiple discriminator channels available? (List how many.)	x		3.
Refrigeration?		x	
External Standard?	x		
Is service maintenance by contract?	x		
Is preventative maintenance applied?	x		

Additional Comments

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IV. Sample Analysis Instrumentation (Page 9 of 11)

E. Fluorometer/Spectrophotometer

	Manufacturer	Model	Type: Fluorometer or Spectrophotometer	Installation Date
1. Instrument ID# 1	ORNL In-House	Q1165	Fluorophotometer Serial #12	Fluorometer Not Available
2. Instrument ID#				
3. Instrument ID#				
4. Instrument ID#				
5. Instrument ID#				
6. Instrument ID#				
7. Instrument ID#				
8. Instrument ID#				
9. Instrument ID#				
10. Instrument ID#				
11. Instrument ID#				
Tot.U-Induction Furnace Method. One system only.				

IV. Sample Analysis Instrumentation (Page 10 of 11)

E. Fluorometer/Spectrophotometer

ITEM	YES	NO	COMMENT
Are operating manuals readily available to the operator?	x		
Are calibration protocols available the the operator?	x		
Are calibration results (i.e., sensitivity) kept in a permanent record so that instrument performance can be measured over time?	x		
Is there a methods manual (SOP) available to the operator?	x		
Are NBS traceable standards used for calibration?	x		
Is a permanent service record maintained in a logbook?	x		
How is the data reduced--off line computer, dedicated system or other?	x		Output from INST. Manual Calc.-- Calib Curves.
Is calibration redone at least every 3 months?	x		Daily Check.
Duplicate samples analyzed? (Frequency)	x		1/10, 1 per batch
Spike/standard samples and blanks? (Frequency)	x		Stds 1/10, Spikes 1/20, 1 per batch
Is service maintenance by contract?	x		
Is preventative maintenance applied?	x		

Additional Comments

Fluorometer (Tot.U) is not located in the RAD area. Uranium in RAD area is usually by  $\alpha$ -spectrometry. There is only one unit. It is part of Inorg. Section Eval. also.

IV. Sample Analysis Instrumentation (Page 11 of 11)

F. Thermal Ionization Mass Spectrometer (TIMS)

	Manufacturer	Model	Installation Date
1. Instrument ID#			
2. Instrument ID#			
3. Instrument ID#			

ITEM	YES	NO	COMMENT
Are operating manuals readily available to the operator?			
Are calibration protocols available to the operator?			
Are calibration results kept in a permanent record so that instrument performance can be measured over time?			
Is there a methods manual (SOP) available to the operator?			
Are NBS traceable standards used for calibration?			
Is a permanent service record maintained in a logbook?			
How is the data reduced-off line computer, dedicated system or other?			
Is calibration/recalibration done at least with batch frequency?			
Duplicate samples analyzed? (Frequency)			
Spikes/standard samples and blanks? (Frequency)			
Is service maintenance by contract?			
Is preventative maintenance applied?			

Additional Comments

ORNL (X-10) - does not have a TIMS Unit.



VI. Quality Control Manual and SOP's (Page 1 of 1)

ITEM	YES	NO	COMMENT
Does the laboratory maintain a Quality Control Manual?		x	See below.
Does the manual address the important elements of a QC program, including the following:		x	See below.
a. Personnel?		x	See below.
b. Facilities and equipment?		x	See below.
c. Operation of instruments?		x	See below.
d. Documentation of procedures?		x	See below.
e. Preventative maintenance?		x	See below.
f. Reliability of data?		x	See below.
g. Data validation?		x	See below.
h. Feedback and corrective action?		x	See below.
Are files of outdated SOP's stored for reference		x	See below.

Additional Comments

QA/QC Division (Pam. Howell) - contents of manual in preparation at this point in time - so these questions can't be answered yet.



## Internal Correspondence

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MARTIN MARIETTA ENERGY SYSTEMS, INC.

November 3, 1987

Robert B. Fitts

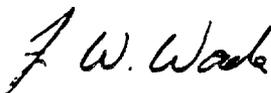
Response to the On-Site Evaluation and Evidentiary Audit Carried out at  
the Oak Ridge National Laboratory on 8/25/87

- Item #1 - Notebooks are now reviewed once a week by the laboratory supervisor, notebook/logbook changes are made by drawing a line through the entry and then initialed by the technician making the change.
- Item #2 - We have been keeping a logbook (containing QA/QC data) for each instrument, we are now keeping a logbook that contains instrument settings, etc.
- Item #3 - We process a standard or standard spike and a duplicate with every tenth sample. The computer generated data/results are checked by such QA/QC measures. All instruments are monitored on a daily basis by counting known standards before the day's counting begins. The recommendation that we perform manual data reduction on gamma spectra is unfounded.
- Item #4 - We are now storing all gamma spectra for the survey indefinitely on floppy disks. Previously, the data was held for thirty days.
- Item #5 - We have a written SOP for sample receiving, login, and chain-of-custody. The SOP is and has been available to everyone.
- Item #6 - This recommendation should be addressed by the RAD Committee, not our laboratory.
- Item #7 - All of our procedures should be in the survey manual, they were submitted months ago.
- Item #8 - We are heavily involved in the EPA-Las Vegas PE/IC samples. The data from past work is available from me or from EPA-LV.

November 3, 1987

We measure radionuclides in water and air filters and these analyses cover all parameters required in a water matrix for the survey. As of 11/1/87, soil samples were not available from EPA-LV.

Sincerely,



J. W. Wade  
Analytical Chemistry Division

JWW:sdc

cc: B. R. Clark  
D. L. Dihel  
P. L. Howell  
W. R. Laing  
J. R. Stokely



JUL 05 1988 *AS*

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
OFFICE OF RESEARCH AND DEVELOPMENT  
ENVIRONMENTAL MONITORING SYSTEMS LABORATORY-LAS VEGAS  
P.O. BOX 93478  
LAS VEGAS, NEVADA 89193-3478  
(702/798-2100 - FTS 545-2100)

JUN 17 1988

Mr. William B. Laing  
Program Manager  
Oak Ridge National Laboratory, X-10  
P.O. Box 2008, 4500s, MS-127  
Oak Ridge, TN 37831

Dear Mr. Laing:

Enclosed is the final report by Jesse Gerard of LEMSCO for an on-site RAD audit carried out by Gerard, Jane Huber, and Earl Whittaker of Lockheed EMSCO and the final report by Mary Franquemont of Techlaw for an on-site evidentiary audit carried out at the OAK RIDGE NATIONAL LABORATORY on May 5th, 1988.

The report by Gerard's group includes a completed copy of the new checklist for radiation measurement quality assurance support, a summary of ORNL, X-10 activity in comparative performance evaluation sample programs, a discussion of their SDG data package activity, and general comments regarding RAD laboratory. the EPA. He outlined during the visit and the debriefing the data items required for a full data package for the sample designated group(s) that will get the full audit. ORNL will cooperate in furnishing this material.

The evidentiary audit centered on those areas of the laboratory involved with the RAD measurements of the DOE environmental survey. We particularly avoided probing into areas primarily in the organic area so as not to interrupt ongoing data handling priorities. Mary Franquemont reviewed custody and documentation in the high explosives laboratory area. The sample receiving and distribution was reviewed by both groups.

Robert Heinrich of ANL was the representative of the RAD committee at this audit.

Please respond to the issues, comments and recommendations presented in these reports and describe any corrective actions or

changes related to the report items. In order to maintain our document scheduling, we should expect to receive your reply by June 10th, 1988. Thank you for your cooperation in this matter.

Sincerely,



Harold A. Vincent  
Chemist, Quality Assurance Research Branch  
Quality Assurance and Methods Development Division

Enclosures

cc:

D. Karen Knight, DOE HQ (w/enclosures)  
James Stokely, ORNL  
Robert Heinrich, ANL  
Pamela Howell, ORNL  
Jeff Wade, ORNL  
Jessie Gerard, LEMSCO  
Jane Huber, LEMSCO  
Earl Whittaker, LEMSCO



Environmental Programs Office  
1050 E. Flamingo Road, Suite 120, Las Vegas, Nevada 89119  
(702) 734-3200

May 25, 1988

United States Environmental  
Protection Agency  
P.O. Box 93478  
Las Vegas, Nevada 89193-3478

ATTENTION: DR. HAROLD VINCENT

VIA: R. D. FLOTARD *R. D. Flotard*

SUBJECT: ROUTINE ON-SITE LABORATORY EVALUATION OF OAK RIDGE  
NATIONAL LABORATORY (ORNL/X-10) FOR RAD ANALYSIS ON MAY  
5, 1988.

Dear Dr. Vincent:

The Routine On-Site Laboratory Evaluation of Oak Ridge National Laboratory on May 5, 1988, has been completed. The following items must be given attention in order to improve data integrity:

1. It is recommended that radionuclide standards (or any other radioactive materials such as QAQC liquids, standards, PE samples etc.) not be stored in the same room with Environmental Survey Program samples to be analyzed (or already analyzed) to prevent possible cross contamination - especially since standard radionuclides can be orders of magnitude higher than environmental samples.
2. It is recommended that personnel working with samples wear rubber gloves due to biological hazards etc. from samples such as sewer water, sludges etc.
3. It is suggested (optional since organic and inorganic auditing sections of survey program need to have input also) for the main sample receipt and storage area - where there is quite a bit of crowding due to many, already analyzed, liquid samples - that a secondary storage area also be used. It probably is best to keep only the present/unanalyzed samples in the main

area. This probably would also keep any possible cross contamination from one site's samples to the next at a minimum. Hence, if the main area is kept for incoming as yet unanalyzed samples (and is kept clean) with analyzed unused samples being returned to a secondary storage, possible cross contamination would be minimized in addition to decreasing crowding.

4. Data audit package submittal for LLNL and SNLL was discussed. Future data audit packages should be submitted to Dr. H. Vincent at EPA who will then forward them to Jane Huber at Lockheed-EMSCO for review. Resubmissions are to be sent directly to Jane Huber. It was agreed that only one gamma spectrum plot will be submitted per site per matrix for each data package plus any unusual ones from the site since spectrum plots are so difficult to make at ORNL. The spectra plots for all audited samples if they can be done easier in the future should be submitted though. In regard to the data package submitted by ORNL - total uranium even though done in the inorganic section is to be included with the radioanalytical data package. The necessary radioanalytical forms for uranium samples will be submitted by ORNL. Jeff Wade provided (during meeting) necessary copies of analytical procedures and SOPs necessary to perform the audits. The ORNL personnel seem to have a good understanding of data package submittal requirements at this point in time.
5. The RAD area sample log-in was discussed during the meeting (post lab team meeting) and it was decided that this was in the category of convenience (all requirements for survey were already being satisfied) but that any information collected on survey samples should be a part of case file. Since any information collected on the samples are to be part of the case file it was decided information should be collected in a manner that is consistent with the file and that the log-in information kept in logbooks on a continuing basis should be reviewed, signed and dated by personnel logging in samples and checked by supervision of area. In addition, information collected probably should be such that other customer's (not part of survey program) information should be separate and not appear with survey sample information.
6. Analyses are being performed (or planned) for tritium in soil and total uranium in soil or sediments but validated Survey Analysis and Sampling Manual Appendix 4: Radiochemical Analysis procedures for ORNL could not be found. Also, the

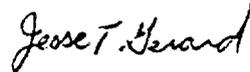
reference to total strontium seems to be confusing. Total strontium usually refers to an inorganic analysis such as by atomic absorption, gravimetric procedure etc. and in the case of strontium would include non-radioactive natural strontium isotopes.

7. There was a lack of visibility (all areas) of SOPs posted. It is recommended that SOPs - on matters such as instrument operating procedures, calibration procedures, administrative procedures, etc., in addition to methods SOPs or Appendix 4 type of operating procedures-be posted/readily available.
8. ORNL has participated extensively in both the EPA and EML QA programs. Generally results are quite good-improvements necessary are discussed below. The overall EML average score for  $^{239}\text{Pu}$  in water, air, soil and vegetation was  $66.3 \pm 21.4$  compared to EML known values and  $65.6 \pm 19.6$  compared to grand average values. There was a consistently low bias in the plutonium values for all matrices except air. For the EPA QA program (water) ORNL had 14 outliers or extreme outliers out of 41 parameters during the baseline period. This was mainly due to a dilution error for PE samples on 10/87 of a factor of 2 (1/8 instead of 1/16). Using correct calculations ORNL overall scores changed from 67.8 to 81.0, compared to known values. ORNL will be more careful about their dilution instructions in the future. Other parameters needing special attention are alpha, beta and  $^{226}\text{Ra}$  in water matrix.
9. Previous visit recommendation and checklist items were reviewed - logbook/notebook/data sheet signatures, instrument logbooks, validation checks (Qual. and Quant.), raw data storage, SOPs not available for overall program sample receipt and storage etc., Appendix 4 procedures, EPA/EML participation, etc. and appropriate changes have been made or appropriate courses of action are being followed/or are in process.
10. For the fluorometry area (Total U) it is recommended that there should be some kind of direct printout of calibration data and sample results or storage of direct instrument reading/results etc. (computer, disk, tape ----) for documentation purposes. SOPs were not readily available/posted. The permanent service record logbook should be made more readily available. Also see item 6 above.

DR. HAROLD VINCENT  
ROUTINE ON-SITE EVALUATION OF OAK RIDGE NATIONAL LABORATORY  
PAGE 4

Details of some of the above items may be found in the text of this report. An evidentiary audit was conducted simultaneously by the Contract Evidence Audit Team (CEAT) Techlaw. Their findings will be provided in a separate report.

Very truly yours,



Jesse T. Gerard  
Staff Scientist  
Quality Assurance Department

JTG/ahh

cc: M. T. Homsher  
J. D. Petty  
E. L. Whittaker  
J. Huber  
D. W. Bottrell  
K. J. Cabbie  
J.O. 70.23  
QA - 5-174  
WP-2306C

ATTACHMENT: (On-Site Laboratory RAD Evaluation)



Summary of Laboratory Evaluation

A. Procedural Changes the Laboratory Agreed to Implement

The following comments refer to deficiencies noted in the Laboratory Evaluation Checklist (Attachment 1).

For comments, see pages 1-4 above and also page 8, Item D.

B. Review of Environmental Measurements Laboratory and EPA Drinking Water Performance Evaluation Samples

The results of both were discussed with the laboratory personnel:

For comments, see page 3, Item 8 above. Information on the Claude Sill Samples have not been received yet. ORNL has received and is analyzing the Claude Sill samples. They will be scored on their performance with those samples in a manner similar to the above mentioned two programs.

C. Review of Data Audit

The following comments refer to the Summary/Conclusions of the data audit for Problem No \_\_\_\_\_, Request No. \_\_\_\_\_ (Attachment 2.)

Report  
Item #

Comments

Action\*

Information on samples for data audits for LLNL and SNLL is being received and is just beginning to evolve. See page 2, Item 4 above for comments.

D. Issues to be Resolved by DOE Headquarters

As is required for items pages 1-4 above since this is an on-site evaluation.

## Attachment 1

## Laboratory Evaluation Checklist

I. Organization and Personnel (Page 1 of 2)

ITEM	Present		COMMENT
	YES	NO	
Laboratory or Project Manager (individual responsible for overall technical effort)  Name: <u>R. B. Fitts</u>	x		
Name: <u>J. R. Stokely</u> Job Title: <u>RAD Coordinator, DOE Envir. Surv. Program.</u>	x		(615) 574-4907
Name: <u>J. W. Wade</u> Job Title: <u>Supervisor, RAD Anal. Chem.</u>	x		(615) 574-4528
Name: <u>D. L. Dihel</u> Job Title: <u>Radiochemist</u>	x		(615) 574-4910
Name: <u>N. E. Owen</u> Job Title: <u>Sample Custodian</u>	x		
Name: <u>W. R. Laing</u> Job Title: <u>Section Head, Inorg. Chem., ACD</u>	x		
Name: <u>W. H. Griest</u> Job Title: <u>Supervisor, Separations and Synth.</u>	x		
Do personnel assigned to this project have the appropriate background to successfully accomplish the objectives of the program?	x		

I. Organization and Personnel (Page 2 of 2)

ITEM	Present		COMMENT
	YES	NO	
Quality Assurance Supervisor Name: <u>P. L. Howell</u>	x		
Support-Electronic Technician Name: _____			
Is the organization adequately staffed to meet project commitments in a timely manner?	x		
Were all personnel involved with the analysis available during the evaluation? (List those not present.)	x		

Additional Comments

Additional personnel present/contacted: J. C. Price - Chemist, Chemical and Physical Anal., Supervisor.

II. Sample Receipt and Storage Area (Page 1 of 2)

ITEM	YES	NO	COMMENT
Are written Standard Operating Procedures (SOPs) developed for receipt and storage of samples?		x	For RAD area, yes see comment 1.
Is the appropriate portion of the SOP available to the sample custodian at the sample receipt/storage area?		x	For RAD area, yes see comment 1.
Are adequate facilities provided for storage of samples.		x	Crowded, see comment 2.
Are the sample receipt/storage and records maintained in a manner consistent with program needs?	x		
Are standards stored separately from sample digestates?	x		Also-see comment 3.
Has the supervisor of the individual maintaining the notebook/bench sheet/logbook personally examined and reviewed the notebook/bench sheet/logbook periodically, and signed his/her name therein, together with the date and appropriate comments as to whether or not the document is being maintained in an appropriate manner?	x		Also-see comment 4.

Additional Comments

1. Main DOE Environ. Survey Receipt and Storage SOPs were being revised at this point in time.
2. See comment Page 1, Item 3.
3. See comments Page 1, Item 1.
4. See comments Page 2, Item 5.

II. Sample Receipt and Storage Area (Page 2 of 2)

ITEM	SITE	YES	NO	COMMENT
<p>Have all samples been received to date from each of the sites listed? Give Date <u>May 5, 1988.</u></p>	Rocky Flats	x		
	Pantex	x		
	LLNL & SNLL	x		
	ANL	x		
	BNL	x		All in, yes? none anal.yet
<p>Have all samples to be analyzed from each of the sites listed been completed to date? (Results finalized by all laboratory personnel and turned in for reporting.)</p>	Rocky Flats	x		
	Pantex	x		
	LLNL & SNLL	x		
	ANL	x		
	BNL		x	Just starting.

Additional Comments:

Caroline Granger - RAD sample custodian.

III. Sample Preparation Area (Page 1 of 3)

When touring the facilities, give special attention to: (a) the overall appearance of organization and neatness, (b) the proper maintenance of facilities and instrumentation, (c) the general adequacy of the facilities to accomplish the required work.

ITEM	YES	NO	COMMENT
Is the laboratory maintained in a clean and organized manner?	x		
Does the laboratory appear to have adequate workspace (120 sq. feet, 6 linear feet of unencumbered bench space per analyst)?	x		
Are contamination-free areas provided for trace level analytical work? (Low level and high activity areas separated.)	x		
Are the hoods in good condition and functional?	x		
Are chemical waste disposal policies/procedures well defined and followed by the laboratory?	x		
Does the laboratory have a source of distilled/demineralized water?	x		
Is the conductivity of distilled/demineralized water routinely checked and recorded?		x	Not needed?
Is the analytical balance located away from draft and areas subject to rapid temperature changes?	x		
Has the balance been calibrated within one year by a certified technician?	x		Quarterly.
Is the balance routinely checked with the appropriate range of class S weights daily before use and are the results recorded in a logbook?	x		Contracted.
Is the sample preparation portion of the SOP available to the analyst at the sample preparation area?	x		
Are unexpired standards used to prepare instrument calibration standards?	x		
Are fresh analytical standards prepared at a frequency consistent with good QA?	x		
Are chemicals and standards dated upon receipt?	x		
Are reference materials properly labeled with concentrations, date of preparation, and the identity of the person preparing the sample?	x		
Is a spiking/calibration standards preparation and tracking logbook(s) maintained?	x		
Are the primary standards traceable to NBS standards where possible?	x		
Do the analysts record bench data in a neat and accurate manner?	x		

III. Sample Preparation Area (Page 2 of 3)

ITEM	YES	NO	COMMENT
Are digestion logbooks/bench sheets maintained in a neat and organized manner?	x		
Is an adequate drying oven available with a temperature measurement device?	x		
Has the supervisor of the individual maintaining the notebook/bench sheet personally examined and reviewed the notebook/bench sheet periodically, and signed his/her name therein, together with the date and appropriate comments as to whether or not the notebook/bench sheet is being maintained in an appropriate manner?	x		

Additional Comments

1. Also see page 1, item 2.



IV. Sample Analysis Instrumentation (Page 1 of 23)

A. Gamma-Ray Spectrometer

	Manufacturer	Model	Automated Sample Exchanger Used	Installation Date
1. Spectrometer		Geli-		
ID# 1		(1)LGC2250LATT		
2	Two PyT's	(2)LGC2250LATT	Manual	5 years old
Data System	ND-9900			
2. Spectrometer		Ge-		
ID# 3		(3)2020		
4	Two Canberra's	(4)2001	Manual	6 years old
Data System	ND-9900			
3. Spectrometer		Ge-		
ID# 5		(5)OTZDS30-25185		
6	Two Tennelec's	(6)CPZDS30-25185	Manual	< 1 year old
Data System	ND-9900			
4. Spectrometer				
ID#				
Data System				
5. Spectrometer				
ID#				
Data System				
6. Spectrometer				
ID#				
Data System				

Spectrometers 1, 2, 3, 4 are approx. 20% effic., 5 is 25% and 6 is 30% - 4 inch lead chambers used. ND-9900 controls all 6 detectors. All detectors are now 4 inch lead shielded which is a partial change from last time.

IV. Sample Analysis Instrumentation (Page 2 of 23)

A. Gamma-Ray Spectrometer

ITEM	YES	NO	COMMENT
Are operating manuals readily available to the operator?	x		
Are calibration protocols available to the operator?		x	Being written now for 9900 system.
Are energy, efficiency, FWHM values, gains and check standard results kept in a permanent record so that instrument performance can be measured over time?	x		Computer file control charts being kept now.
Is there a methods manual (SOP) available to the operator?		x	Being written now.
Are NBS traceable standards used for calibration?	x		
Duplicate samples analyzed? (Frequency)	x		1/20, 1 per batch
Spike/standard samples and blanks? (Frequency)	x		1/20, 1 per batch
Is a permanent service record maintained in a logbook?	x		
How is the data reduced-off line computer, dedicated system or other?	x		Dedicated.
Are radioisotopic or interelement correction factors updated every six months or more frequently?		x	Avoided.
Is service maintenance by contract?	x		ORNL Division.
Is preventative maintenance applied?	x		

Additional Comments

1. Also see page 3, Item 9.
2. Calibrates efficiency, resolution etc., each day and maintains results in logbook with printout. Detailed extensive rework every 6 months or if check standards indicate something is wrong. Computer file control charts - peak centroids, FWHM, Eff. -  $^{60}\text{Co}$  (1173, 1332 Kev) and  $^{137}\text{Cs}$  (661 Kev) etc.
3. Qualitative and quantitative validation - at least one per parameter, per site and any samples varying substantially from rest of site samples - to begin with BNL.

IV. Sample Analysis Instrumentation (Page 3 of 23)

A. Gamma-Ray Spectrometer

ITEM	YES	NO	COMMENT
Are more than one site's samples being analyzed at this time? (List sites).		x	All done to BNL. Not yet start BNL
Are all samples for gamma-spectroscopy analysis completed for this site? How many samples have been analyzed by gamma-ray spectroscopy for this site?		x	96 samples received for gamma spec.-BNL.
For this site, what QA/QC has lab collected? Starting with <u>energy calibration</u> , give total number or frequency (per set, daily, etc.), from logbook and notebook entries or computer listings.	x		43 gamma-ANL-samples done. Not start BNL.
Detector <u>efficiency calibration</u> done for this site's analyses? Give total number or frequency (monthly, etc.) done per geometry from logbooks and notebooks or computer listings.	x		Not start BNL.
<u>Duplicates?</u> Give total number or frequency (1/20, per batch, per day, etc.), done for site from logbook and notebook entries or computer listings.	x		ANL-5 Duplicates. Not start BNL.
<u>Blanks and/or backgrounds?</u> Give total number or frequency (1/20, per batch, per day, etc.), done for site from logbook and notebook entries or computer entries.	x		1/20, per batch. Not start BNL.
Is more than one <u>counting geometry</u> used? List number for each matrix used for this site.	x		See comment 1.
Are <u>PE samples</u> from internal sources being analyzed? Give total number done during this site's analyses and list radionuclide(s).	x		Each week-ANL 7 std.samples analyzed. Not start BNL
<u>Chemical yields</u> done (if chemistry)? List radionuclides involved.		x	No chem.
<u>Spike recoveries</u> (liquid, solids, etc.), for samples if chemistry or for efficiency or geometry checks etc. List radionuclides used.	x		For ANL-2 for water samples.

Additional Comments

1. There are 2 or 3 counting geometries used - soil petrie dish, and 900 cc marinelli beakers.

IV. Sample Analysis Instrumentation (Page 4 of 23)

A. Gamma-Ray Spectrometer

For the BNL site, list the DOE sample numbers for which gamma spectroscopy analysis has been performed. If other than for a gamma scan-list specific radionuclides analyzed.

BNL - No samples analyzed yet (96 samples received).

ANL - 43 samples analyzed.

Auditor has computer listing of all RAD samples for 2 sites listed above giving parameters, matrices etc. Auditor also has RAD FORM copies of results for ANL gamma scans.

For this site, give the information requested below for samples that analyst has checked in detail to qualitatively and quantitatively validate analysis results for site. (Should have at least one validation check per parameter, per site, and on any samples varying substantially from rest of site samples.)

<u>Sample No.</u>	<u>Radionuclide/ Parameter</u>	<u>Detector ID/ Number Used</u>	<u>Comments</u>
<u>Discussed Qual. and Quant. Validation concepts - will do starting with BNL.</u>			
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

IV. Sample Analysis Instrumentation (Page 5 of 23)

B. Alpha Spectrometer

	Manufacturer	Model	Automated Sample Exchanger Used	Installation Date
1. Spectrometer				
ID#				
1, 2, 3, 4	Tennelec	Si(Li) TC-256	Manual	2 years old
Data System				
	ND-9900			
2. Spectrometer				
ID#				
5, 6, 7, 8	Tennelec	Si(Li) TC-256	Manual	2 years old
Data System				
	ND-9900			
3. Spectrometer				
ID#				
9, 10, 11, 12	Tennelec	Si(Li) TC-256	Manual	2 years old
Data System				
	ND-9900			
4. Spectrometer				
ID#				
13,14,15,16	Tennelec	Si(Li), Tc-256 One Tc-257	Manual	2 years old
Data System				
	ND-9900			
5. Spectrometer				
ID#				
Data System				
6. Spectrometer				
ID#				
Data System				

4-Four simultaneously operated  $\alpha$ -spectrometers for a total of 16 available. 1024 channels used for spectra. ND-9900 controls all detectors. The last 4 detectors were added since the last on-site.

IV. Sample Analysis Instrumentation (Page 6 of 23)

B. Alpha Spectrometer

ITEM	YES	NO	COMMENT
Are operating manuals readily available to the operator?	x		
Are calibration protocols available to the operator?	x		Updating now.
Are energy, efficiency, FWHM values, gains and check standard results kept in a permanent record so that instrument performance can be measured over time?	x		Logbook.
Is there a methods manual (SOP) available to the operator?	x		Updating now.
Are NBS traceable standards used for calibration?	x		
Duplicate samples analyzed? (Frequency)	x		1/20, per batch.
Spike/standard samples and blanks? (Frequency)	x		1/20, per batch.
Is a permanent service record maintained in a logbook?	x		
How is the data reduced-off line computer, dedicated system or other?	x		See comment 1.
Are radioisotopic or interelement correction factors updated every six months or more frequently?		x	Avoided.
Is service maintenance by contract?	x		Another ORNL-Div.
Is preventative maintenance applied?	x		

Additional Comments

1. Prints out about 512 channels-manually integrates peaks, enters data, etc. into nearby computer for calculations etc.
2. Calibrates efficiency and resolution etc. each day and maintains results in logbook with printouts.

IV. Sample Analysis Instrumentation (Page 7 of 23)  
 B. Alpha Spectrometer

ITEM	YES	NO	COMMENT
Are more than one site's samples being analyzed at this time? List site(s).		x	Not yet start BNL
Are all samples for alpha spectrometry completed for this site? How many samples have been analyzed by alpha spectrometry for this site?		x	ANL all done. Not yet start BNL.
For this site, what QA/QC has the laboratory collected? Starting with <u>energy calibration</u> , give total number of calibrations or frequency (per set, daily, etc.), from logbooks and notebook entries or computer listings.	x		35 samples were done for ANL - 32Pu, 3U. Not start BNL.
Detector <u>efficiency calibration</u> done for this site's analysis? Give total number or frequency (daily, monthly, etc.), done per geometry from logbook and notebook entries or computer listings.	x		Not start BNL.
<u>Duplicates?</u> Give total number or frequency (1/20, per batch, per day, etc.), done for site from logbook and notebook entries or computer listings.	x		6 duplicates were done for ANL-Pu and U. Not start BNL.
<u>Blanks and backgrounds?</u> Give total number or frequency (1/20, per batch, per day, etc.), done for site from logbook and notebook entries or from computer listings.	x		3 blanks were done for ANL-PU and U. Not start BNL.
Is more than one <u>counting geometry</u> used? List number used for this site.		x	1 count geometry only.
Are <u>PE samples</u> from internal sources being analyzed? Give total number done during this site's analyses and list radionuclides.	x		Weekly--for ANL 6 for PU, 1 for U.
<u>Chemical yields?</u> List both radionuclide and non radionuclide(s) tracers involved.	x		Every Sample - <sup>242</sup> Pu, <sup>232</sup> U.
<u>Spike recoveries?</u> List radionuclides involved and frequency (1/20, per batch, per day, etc.), or total number done from logbook and notebook entries or computer listings.	x		For ANL - 2 for Pu, 1 for U.
<u>Self-absorption correction curves?</u> List radionuclides involved. List number of times curves were updated during analyses for this site.		x	Avoided.

Additional Comments:

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IV. Samples Analysis Instrumentation (Page 8 of 23)

B. Alpha Spectrometer

For the BNL site, list the DOE sample numbers for which alpha spectroscopy analysis has been performed and also list radionuclides determined.

BNL - No samples analyzed yet.

ANL - 35 samples were analyzed (32Pu, 3U).

Auditor has computer listing of all RAD samples for both sites giving parameters, metrics etc., and also has RAD FORM copies of results for ANL Pu and U.

For this site, give the information requested below for samples that analyst has checked in detail, to qualitatively and quantitatively validate analysis results for this site for both chemical separation and instrumentation parts. (Should have at least one validation check per parameter per site and any samples varying substantially from rest of site's samples.)

<u>Sample No.</u>	<u>Radionuclide/ Parameter</u>	<u>Detector ID/ Number Used</u>	<u>Comments</u>
<u>AR811031B</u>	<u>238+239Pu</u>	<u>#1</u>	<u>Has printout of spectrum, calculations, computer printout of results, chem sepn spike, results data, verifies program calculates (qual. and quant. valid.) correctly, tracer etc.</u>
<u> </u>	<u> </u>	<u> </u>	<u> </u>
<u> </u>	<u> </u>	<u> </u>	<u> </u>
<u> </u>	<u> </u>	<u> </u>	<u> </u>
<u> </u>	<u> </u>	<u> </u>	<u> </u>
<u> </u>	<u> </u>	<u> </u>	<u> </u>
<u> </u>	<u> </u>	<u> </u>	<u> </u>

IV. Sample Analysis Instrumentation (Page 9 of 23)

C. Low Background Gas Flow Proportional Counting System (Gross Alpha and Gross Beta)

	Manufacturer	Model	Sample Capacity	Installation Date
1. Instrument				
ID#				
Gross $\alpha$ / $\beta$ Ctr	Tennelec	LB5100	Multiple	3 years old.
Window		Voltage		Operating $\alpha$ =750
Density or Thickness	260 ug/cm <sup>2</sup>	Plateau	Not available	Voltage $\beta$ =1470
		Span and Slope	Not available	Gas p-10(Ar,Me)
(Rack of 4) x 3 = 12 at a time				
2. Instrument				
ID#				
<sup>90</sup> Sr Ctr	Tennelec	LB4000	Manual	Not Available
Window		Voltage		Operating $\alpha$ =1200
Density or Thickness	260 ug/cm <sup>2</sup>	Plateau	Not available	Voltage $\beta$ =1913
		Span and Slope	Not available	Gas p-10, (Ar Me)
3. Instrument				
ID#				
Window		Voltage		Operating
Density or Thickness		Plateau		Voltage
		Span and Slope		Gas
4. Instrument				
ID#				
Window		Voltage		Operating
Density or Thickness		Plateau		Voltage
		Span and Slope		Gas
5. Instrument				
ID#				
Window		Voltage		Operating
Density or Thickness		Plateau		Voltage
		Span and Slope		Gas

1 system of each type. The second one is the older of the two.

IV. Sample Analysis Instrumentation (Page 10 of 23)

C. Low Background Gas Flow Proportional Counting System (Gross Alpha and Gross Beta)

ITEM	YES	NO	COMMENT
Are operating manuals readily available to the operator?	x		
Are calibration protocols available to the operator?	x		Updating.
Are calibration results kept in a permanent record so that instrument performance can be measured over time?	x		
Is there a methods manual (SOP) available to the operator?	x		Updating.
Are NBS traceable standards used for calibration?	x		
Is a permanent service record maintained in a logbook?	x		
How is the data reduced-off line computer, dedicated system or other?	x		Each has its own microprocessor-HP
Is calibration done at least daily or batch frequency?	x		
Duplicate samples analyzed? (Frequency)	x		1/10, 1 per batch
Spike/standard samples and blanks? (Frequency)	x		1/10 stds, 1/20 spikes, 1/batch.
Are self-absorption curves readily available to analyst (curves reestablished last 3 months)?	x		Daily checked - 6 mo. reestabl.
Is service maintenance by contract?	x		
Is preventative maintenance applied?	x		

Additional Comments

Calibrates efficiency, etc., each day and maintains results in logbook with printouts.

IV. Sample Analysis Instrumentation (Page 11 of 23)

C. Low Background Gas Flow Proportional Counting System (Gross Alpha and Gross Beta)

ITEM	YES	NO	COMMENT
Are more than one site's samples being analyzed at this time? List site(s).		x	Just starting BNL.
Are all samples for alpha and beta counting completed for this site? How many samples have been analysed for this site?		x	ANL-17 gross $\alpha/\beta$ -34 Sr. Just starting BNL
For this site, what QA/QC has the laboratory collected? Starting with detector energy calibration/discriminator checks, give total number or frequency (daily, per set, etc.), from logbook and notebook entries or computer listings.	x		Each day, per batch.
Detector efficiency or performance checks done for this site's analyses? Give total number or frequency (daily, per set, etc.), done per geometry from logbook and notebook entries or computer listings.	x		Each day, per batch.
Duplicates? Give total number or frequency (1/20, per batch, per day, etc.), done for site from logbook and notebook entries or computer listings.	x		ANL-3 for Gross $\alpha$ / $\beta$ -4 for Sr. Just starting BNL
Blanks and backgrounds? Give total number or frequency (1/10, 1/20, per batch, per day, etc.) done for site from logbook and notebook entries or from computer listings.	x		Each day for 4100 Each wk. for 5100 For ANL-2 for $\alpha/\beta$ -2 for Sr.
Is more than one counting geometry used? List number used for this site.		x	1 only.
Are PE samples from internal sources being analyzed? Give total number done during this site's analyses and list radionuclides.	x		For ANL-6 for $\alpha/\beta$ -4 for Sr.
Chemical yields? List both radionuclide and non radionuclide(s) involved.	x		For $^{90}\text{Sr}$ .
Spike recoveries? List radionuclides involved and frequency (1/20, per batch, per day, etc.), or total number done from logbook and notebook entries or computer listings.	x		For ANL-2 for Gross $\alpha/\beta$ -2 for Sr. Just starting BNL
Self-absorption correction curves? List radionuclides involved. List number of times curves were updated during analyses for this site.	x		Gross $\alpha/\beta$ , $^{90}\text{Sr}$ . For ANL-1 time. Just starting BNL

Additional Comments:

IV. Samples Analysis Instrumentation (Page 12 of 23)

C. Low Background Gas Flow Proportional Counting System (Gross Alpha and Gross Beta)

For the BNL site, list the DOE sample numbers for which alpha and beta analysis has been performed and also list radionuclides determined.

BNL - No samples analyzed yet.

ANL - 17 Gross alpha, 17 Gross beta, 34 strontium samples were analyzed.

Auditor has computer listing of all RAD samples for both sites giving parameters, matrices etc. and also has RAD FORM copies of results for ANL gross alpha, beta and strontiums.

For this site, give the information requested below for samples that analyst has checked in detail, to qualitatively and quantitatively validate analysis results for this site for both chemical separation and instrumentation parts. (Should have at least one validation check per parameter per site and any samples varying substantially from rest of site's samples.)

<u>Sample No.</u>	<u>Radionuclide/ Parameter</u>	<u>Detector ID/ Number Used</u>	<u>Comments</u>
<u>Discussed Qual. and Quant. validation concepts - will do starting with BNL---</u>			
<u>They seem to be doing most of what is required now.</u>			
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

IV. Sample Analysis Instrumentation (Page 13 of 23)

D. Liquid Scintillation (LS) Spectrometer

	Manufacturer	Model	Sample Capacity	Installation Date
1. LS Spectrometer ID# 1	Packard	460C	Multiple	5-6 years old
Data System Data output by system is manually feed into area computer				
2. LS Spectrometer ID#				
Data System				
3. LS Spectrometer ID#				
Data System				
4. LS Spectrometer ID#				
Data System				
5. LS Spectrometer ID#				
Data System				
6. LS Spectrometer ID#				
Data System				

1 liquid scintillation system only. Used mainly for <sup>3</sup>H.

IV. Sample Analysis Instrumentation (Page 14 of 23)

D. Liquid Scintillation (LS) Spectrometer

ITEM	YES	NO	COMMENT
Are operating manuals readily available to the operator?	x		
Are calibration protocols available to the operator?	x		Updating.
Are calibration results (i.e., sensitivity) kept in a permanent record so that instrument performance can be measured over time?	x		
Is there a methods manual (SOP) available to the operator?	x		Updating.
Are NBS traceable standards used for calibration?	x		
Is a permanent service record maintained in a logbook?	x		
How is the data reduced-off line computer, dedicated system or other?	x		Raw data input into area computer manually.
Duplicate samples analyzed? (Frequency)	x		1/10, 1 per batch
Spike/standard samples and blanks? (Frequency)	x		1/20, 1 per batch
Is calibration done at least daily or batch frequency?	x		Per setup or each day.
Are multiple discriminator channels available? (List how many.)	x		3.
Refrigeration?		x	
External Standard?	x		
Is service maintenance by contract?	x		
Is preventative maintenance applied?	x		

Additional Comments

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IV. Sample Analysis Instrumentation (Page 15 of 23)

D. Liquid Scintillation (LS) Spectrometer

ITEM	YES	NO	COMMENT
Are more than one site's samples being analyzed at this time? List site(s).		x	Just starting BNL
Are all samples for liquid scintillation completed for this site? How many samples have been analysed for this site?		x	ANL-36 tritiums Just starting BNL
For this site, what QA/QC has the laboratory collected? Starting with <u>energy calibration/discriminator checks</u> give total number of calibrations or frequency (per set, daily, etc.), from logbooks and notebook entries or computer listings.	x		Tritium std. each day. Eff. each week with set of samples. Each day/per batch.
<u>Detector efficiency or performance checks</u> done for this site's analysis? Give total number or frequency (daily, per set, etc.), done per geometry from logbook and notebook entries or or computer listings.	x		Each sample (Quench Corr.).
<u>Duplicates?</u> Give total number or frequency (1/10, 1/20, per batch, per day, etc.), done for site from logbook and notebook entries or computer listings.	x		For ANL-4 Tritium duplicates Just starting BNL
<u>Blanks and backgrounds?</u> Give total number or frequency (1/10, 1/20, per batch, per day, etc.) done for site from logbook and notebook entries or from computer listings.	x		For ANL-4 tritium blanks Just starting BNL
Are <u>PE samples</u> from internal sources being analyzed? Give total number done during this site's analyses and list radionuclides.	x		For ANL-4 (wkly). Just starting BNL
<u>Chemical yields?</u> List both radionuclide being determined and being added.			Not applic?
<u>Spike recoveries?</u> List radionuclides involved and frequency (1/10, 1/20, per batch, per day, etc.), or total number done from logbook and notebook entries or computer listings.	x		For ANL-2 Just starting BNL
<u>Quench corrections?</u> Method used to correct quenching-external standard, repurification of sample, etc., - list.	x		External Standard

Additional Comments:

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IV. Samples Analysis Instrumentation (Page 16 of 23)

D. Liquid Scintillation (LS) Spectrometer

For the BNL site, list the DOE sample numbers for which liquid scintillation has been performed also list radionuclides determined.

BNL - No samples analyzed yet.

ANL - 36 tritium samples were analyzed.

Auditor has computer listing of all RAD samples for both sites giving parameters, matrices etc. and also has RAD FORM copies of results for ANL tritiums.

For this site, give the information requested below for samples that analyst has checked in detail, to qualitatively and quantitatively validate analysis results for this site for both chemical separation and instrumentation parts. (Should have at least one validation check per parameter per site and any samples varying substantially from rest of site's samples.)

<u>Sample No.</u>	<u>Radionuclide/ Parameter</u>	<u>Detector ID/ Number Used</u>	<u>Comments</u>
<u>BNL - No samples analyzed yet.</u>			
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

IV. Sample Analysis Instrumentation (Page 17 of 23)

E. Fluorometer/Spectrophotometer

	Manufacturer	Model	Type: Fluorometer or Spectrophotometer	Installation Date
1. Instrument ID# 1	ORNL In-House	Q1165 Fluorophotometer Serial #12	Fluorometer	Not Available
2. Instrument ID#				
3. Instrument ID#				
4. Instrument ID#				
5. Instrument ID#				
6. Instrument ID#				
7. Instrument ID#				
8. Instrument ID#				
9. Instrument ID#				
10. Instrument ID#				
11. Instrument ID#				

Tot.U-Induction Furnace Method. One system only.

IV. Sample Analysis Instrumentation (Page 18 of 23)

E. Fluorometer/Spectrophotometer

ITEM	YES	NO	COMMENT
Are operating manuals readily available to the operator?	x		
Are calibration protocols available the the operator?	x		
Are calibration results (i.e., sensitivity) kept in a permanent record so that instrument performance can be measured over time?	x		
Is there a methods manual (SOP) available to the operator?	x		
Are NBS traceable standards used for calibration?	x		
Is a permanent service record maintained in a logbook?	x		See comment 1.
How is the data reduced-off line computer, dedicated system or other?	x		Now, direct reading from instrument.
Is calibration redone at least every 3 months?	x		Daily Check.
Duplicate samples analyzed? (Frequency)	x		See comment 2.
Spike/standard samples and blanks? (Frequency)	x		Stds 1/10, Spikes 1/20, 1 per batch
Is service maintenance by contract?	x		
Is preventative maintenance applied?	x		

Additional Comments

1. Instrument personnel fixing the instrument — Keep the logbook — Not readily available.
2. Triplicates are ran for each sample.
3. Fluorometer (Tot.U) is not located in the RAD area. Uranium in RAD area is usually by  $\alpha$ -spectrometry. There is only one unit. It is part of Inorg. Section Eval. also.
4. It is recommended that there should be some kind of instrument printout of calibration data and sample results or storage of direct instrument results for documentation purposes.

IV. Sample Analysis Instrumentation (Page 19 of 23)

E. Fluorometer/Spectrophotometer

ITEM	YES	NO	COMMENT
Are more than one site's samples being analyzed at this time? List site(s).		x	See comment 1.
Are all samples for fluorometry (spectrophotometry) completed for this site? How many samples have been analysed for this site?	x		For ANL, yes.
For this site, what QA/QC has the laboratory collected? Starting with <u>Calibration checks</u> , checks give total number of calibrations or frequency (per set, daily, etc.), from logbooks and notebook entries or computer listings.	x		
<u>Duplicates</u> ? Give total number or frequency (1/10, 1/20, per batch, per day, etc.), done for site from logbook and notebook entries or computer listings.	x		
<u>Blanks and backgrounds</u> ? Give total number or frequency (1/10, 1/20, per batch, per day, etc.) done for site from logbook and notebook entries or from computer listings.	x		
Are <u>PK samples</u> from internal sources being analyzed? Give total number done during this site's analyses.	x		Weekly.
<u>Spike recoveries</u> ? List radionuclides involved and frequency (1/10, 1/20, per batch, per day, etc.), or total number done from logbook and notebook entries or computer listings.	x		1/20, per set.
<u>Quench corrections</u> ? Method used to correct quenching, standards closely bracket sample value, dilution method, etc., list.	x		See comment 2.

Additional Comments:

1. ORNL is not doing BNL total uranium.
2. Pellet-300 mg samples extracted 3M HNO<sub>3</sub> and diluted out to reduce quenching.

IV. Sample Analysis Instrumentation (Page 20 of 23)

E. Fluorometer/Spectrophotometer

For the BNL site, list the DOE sample numbers for which fluorometry/spectrophotometry has been performed also list parameters determined.

All BNL total U samples are being done at K-25 (Total U).

For ANL - 18 samples.

There was no listing of Tot.U analyses or RAD FORM copies of results for ANL readily available for the auditors.

For this site, give the information requested below for samples that analyst has checked in detail, to qualitatively and quantitatively validate analysis results for this site for both chemical separation and instrumentation parts. (Should have at least one validation check per parameter per site and any samples varying substantially from rest of site's samples.)

<u>Sample No.</u>	<u>Radionuclide/ Parameter</u>	<u>Detector ID/ Number Used</u>	<u>Comments</u>
<u>AR420016F</u>	<u>Tot.U</u>	<u>#1</u>	<u>Direct reading of Mg/L from Inst.</u>
<u>AR420016G</u>	<u></u>	<u></u>	<u>Calibrated during each set samples.</u>
<u>AR420016H</u>	<u></u>	<u></u>	<u>As SOPs were not posted there was</u>
<u>AR420016I</u>	<u></u>	<u></u>	<u>confusion as to what was being done.</u>
<u>AR420016G</u>	<u></u>	<u></u>	<u></u>
<u></u>	<u></u>	<u></u>	<u></u>
<u></u>	<u></u>	<u></u>	<u></u>

IV. Sample Analysis Instrumentation (Page 21 of 23)

F. Thermal Ionization Mass Spectrometer (TIMS)

	Manufacturer	Model	Installation Date	
1. Instrument ID#	Not applicable.			
2. Instrument ID#				
3. Instrument ID#				
ITEM	YES	NO	COMMENT	
Are operating manuals readily available to the operator?				
Are calibration protocols available to the operator?				
Are calibration results kept in a permanent record so that instrument performance can be measured over time?				
Is there a methods manual (SOP) available to the operator?				
Are NBS traceable standards used for calibration?				
Is a permanent service record maintained in a logbook?				
How is the data reduced-off line computer, dedicated system or other?				
Is calibration/recalibration done at least with batch frequency?				
Duplicate samples analyzed? (Frequency)				
Spikes/standard samples and blanks? (Frequency)				
Is service maintenance by contract?				
Is preventative maintenance applied?				

Additional Comments

ORNL (X-10) - does not have a TIMS unit.

IV. Sample Analysis Instrumentation (Page 22 of 23)

F. Thermal Ionization Mass Spectrometer (TIMS)

ITEM	YES	NO	COMMENT
Are more than one site's samples being analyzed at this time? List site(s).			
Are all samples for TIMS completed for this site? How many samples have been analyzed for this site?			
For this site, what QA/QC has the laboratory collected? Starting with <u>calibration verification</u> checks give total number of calibrations or frequency (before and after each set of samples, daily, 1/10, 1/20, etc.), from logbooks and <u>notebook entries or computer listings</u> .			
<u>Duplicates?</u> Give total number or frequency (1/10, 1/20, per batch, per day, etc.), done for site from logbook and notebook entries or <u>computer listings</u> .			
<u>Blanks and backgrounds?</u> Give total number or frequency (1/10, 1/20, per batch, per day, etc.) done for site from logbook and notebook entries or <u>from computer listings</u> .			
Are <u>PE samples</u> (isotopic ratio types) from internal sources being analyzed? Give total number done during this site's analyses.			

Additional Comments:

Not applicable.

IV. Samples Analysis Instrumentation (Page 23 of 23)

F. Thermal Ionization Mass Spectrometer (TIMS)

For the \_\_\_\_\_ site, list the DOE sample numbers for which mass spectrometry has been performed and also list parameters determined.

Not applicable.

For this site, give the information requested below for samples that analyst has checked in detail, to qualitatively and quantitatively validate analysis results for this site for both chemical separation and instrumentation parts. (i.e., sample purity to ensure other heavy metals or oxides are not present to give false measurements - more of a problem with samples than high purity standards).

<u>Sample No.</u>	<u>Radionuclide/ Parameter</u>	<u>Detector ID/ Number Used</u>	<u>Comments</u>
<u>Not applicable.</u>			



VI. Quality Control Manual and SOP's (Page 1 of 1)

ITEM	YES	NO	COMMENT
Does the laboratory maintain a Quality Control Manual?"		x	See below.
Does the manual address the important elements of a QC program, including the following:		x	See below.
a. Personnel?		x	See below.
b. Facilities and equipment?		x	See below.
c. Operation of instruments?		x	See below.
d. Documentation of procedures?		x	See below.
e. Preventative maintenance?		x	See below.
f. Reliability of data?		x	See below.
g. Data validation?		x	See below.
h. Feedback and corrective action?		x	See below.
Are files of outdated SOP's stored for reference		x	See below.

Additional Comments

QA/QC Division (Pam. Howell) - contents of manual in preparation at this point in time - so these questions can't be answered yet.

VII. Summary Checksheet (Page 1 of 1)

ITEM	YES	NO	COMMENT
Do responses to the evaluation indicate that project and supervisory personnel are aware of QA/QC and its application to the project?	x		
Have responses with respect to QA/QC aspects of the project been open and direct?	x		
Has a cooperative attitude been displayed by all project and supervisory personnel?	x		
Have any QA/QC deficiencies been discussed before leaving?	x		
Is the overall quality assurance adequate to accomplish the objectives of the project?	x		
Have corrective actions recommended during previous evaluations been implemented? If not, provide details in Section VII.B.	x		See comment 1.

Additional Comments

1. See Page 3, Item 9.

LABORATORY EVIDENCE AUDIT REPORT

MARTIN MARIETTA ENERGY SYSTEMS, INC.  
ANALYTICAL CHEMISTRY DIVISION

OAK RIDGE, TENNESSEE

May 5, 1988

Martin Marietta Energy Systems, Inc. (MMES)  
Analytical Chemistry Department  
Oak Ridge, TN 37830  
(615) 574-9768

Jim Stokley - Section Head<sup>1,2,3</sup>  
Jeff Wade - Group Leader - Low Level Radio-  
chemical Analysis Group<sup>1,2,3</sup>  
Bob Fitts - OR Survey Program Manager<sup>1</sup>  
W. R. Laing - Section Head<sup>1,3</sup>  
Wayne Greist - Group Leader - High Explosives  
Analysis Group<sup>1,2,3</sup>  
John Canton - Group Leader - Organic Analysis<sup>2</sup>  
Donald Dihel - Chemist<sup>1,2,3</sup>  
J. C. Price - Chemist<sup>1,2,3</sup>  
Bruce Tomkins - Chemist<sup>2</sup>  
Nancy Owen - Laboratory Aide/Sample Custodian<sup>1,2</sup>  
Carolyn Granger - Sample Custodian<sup>2</sup>  
Pamela Howell - Quality Assurance Supervisor<sup>1,3</sup>

University of Chicago - Argonne National Laboratory - Argonne, IL  
(312) 972-4340

Robert Heinrich - DOE Environmental Survey Radio-  
chemistry Analytical Manager

USEPA/EMSL - Las Vegas, NV  
(702) 798-2129

Harold Vincent - Chemist

- <sup>1</sup>present during pre-audit briefing  
<sup>2</sup>contacted during audit  
<sup>3</sup>present during post-audit debriefing

EMSL/LEMSCO - Las Vegas, NV  
(702) 798-31456

Jesse Gerard - Staff Scientist  
Earl Whittaker - Staff Scientist  
Jane Huber - Senior Associate Scientist

NEIC/CEAT (TechLaw) - Denver, CO  
(303) 233-1248

Mary Franquemont - Associate Consultant

This work was conducted on behalf of the Environmental Protection Agency's (EPA) National Enforcement Investigations Center (NEIC) under EPA Contract #68-01-7369.

## INTRODUCTION

The National Enforcement Investigations Center (NEIC) assigned the Contract Evidence Audit Team (CEAT-TechLaw) to perform an evidence audit of Martin Marietta Energy Systems (MMES) Analytical Chemistry Department Laboratory. The laboratory is receiving, preparing, and analyzing samples using USEPA Contract Laboratory Program (CLP) protocols for the Department of Energy's (DOE) Environmental Survey.

The purpose of this audit was to determine if laboratory policies and procedures are in place to satisfy evidence handling requirements. The report specifies the corrective action needed to meet EPA Evidence Audit Requirements.

The audit was conducted on May 5, 1988 in conjunction with a technical audit performed by representatives from the USEPA Environmental Monitoring Systems Laboratory at Las Vegas, Nevada.

The following operations, accompanying documentation, and written standard operating procedures (SOPs) were reviewed: sample receiving, sample storage, sample tracking (from receipt to completion of analysis), and analytical project file organization and assembly.

## EXECUTIVE SUMMARY

This was the third audit of MMES conducted by USEPA representatives in support of the DOE Environmental Survey Program. The previous audit was conducted on August 26, 1987 and resulted in ten recommendations. One of the ten recommendations has not been addressed or corrected. The recommendation from the previous audit still requiring corrective action is:

1. Written SOPs do not adequately describe sample tracking procedures.

The following eight findings (non-conformances to Evidence Audit Requirements) were identified during the present audit and are discussed in this report:

### Findings

1. If sample tag numbers are not listed on the chain-of-custody record, the sample custodian does not record the numbers.
2. Copies of notebook pages containing sample number cross-reference information are not included in the case files.

3. The Analytical Chemistry Data Sheets do not contain the name of the laboratory.
4. The preparation of water samples in the high explosive laboratory is not documented.
5. Written SOPs do not specify which laboratory within the Analytical Chemistry Department to which they apply.
6. Written SOPs for sample tracking do not describe or include examples of the documents used within the laboratories.
7. The High Explosives laboratory does not have written SOPs.
8. Written SOPs do not exist for case file organization and assembly.

As a result of these findings, the following recommendations were made:

#### Recommendations

1. If sample tag numbers are not listed on the chain-of-custody record, the sample custodian should record the numbers.
2. Copies of notebook pages containing sample number cross-reference information should be included in the case files.
3. The Analytical Chemistry Data Sheets should contain the name of the laboratory.
4. The preparation of water samples in the High Explosives laboratory should be documented.
5. Written SOPs should specify which laboratory within the Analytical Chemistry Department to which they apply.
6. Written SOPs for sample tracking should describe and include examples of the documents used within the laboratories.
7. Written SOPs should be developed for the High Explosives laboratory.
8. Written SOPs should be developed for case file organization and assembly.

The audit was concluded May 5, 1988. Audit participants are listed on the cover page of this report.

## PROCEDURAL AUDIT

The procedural audit consisted of review and examination of actual and written SOPs and accompanying documentation for the following laboratory operations: sample receiving, sample storage, sample identification, sample security, sample tracking (from receipt to completion of analysis), and analytical project file organization and assembly.

### Sample Receiving

Samples are received at the receiving department of the Gaseous Diffusion Plant. The airbill is signed by the receiving clerk, and a copy of the airbill along with the unopened cooler are transferred to the sample receiving area (Room S-159A) in Building 4500S.

The condition of the coolers and custody seals is inspected by the sample custodian, N. Owens. The coolers are unpacked and the custodian inspects the condition of the samples, checks for the presence or absence of shipping documents, signs the chain-of-custody records, and verifies the agreement/non-agreement of information recorded on the shipping and sample documents. Sample receiving information is recorded on the Shipping Container Sample Log-In Form which contains the following information:

1. Samples Received - Date/Time
2. Container Identification
3. Custody Seals - Present/Absent, Intact/Broken/
4. Chain-of-Custody Record - Present/Absent
5. Airbill Copy - Present/Absent
6. Sample Tags - Present/Absent
7. Do All Documents Agree - Yes/No
8. Condition of Samples - All Intact/Some Broken/Some Leaking
9. Sample Identification Numbers
10. Comments
11. Sample Custodian Signature

Next, the sample custodian initiates the Request for Analytical Services form which contains the following information:

1. Customer
2. Building
3. Date
4. Request Number
5. Charge Number
6. Matrix
7. Sample Description

8. Department Code
9. Series
10. Frequency
11. Deadline Date
12. Compliance Sample
13. Sample Number
14. Sample Identification Number
15. Collection Date
16. Analysis
17. Method/Detection Limits
18. Comments
19. Requestor

The sample custodian does not enter the sample identification numbers. Instead, the forms are distributed to the appropriate laboratory where the sample identification numbers are assigned.

To transfer samples to the appropriate laboratory, the sample custodian initiates the Receipt Record/Chain-of-Custody which contains the following information:

1. Case Number
2. Assigned Request Number
3. Sample Numbers
4. Comments
5. Released By: Date/Time
6. Received By: Date/Time/Laboratory

Written SOPs for sample receiving have been developed and implemented. The auditor read these SOPs and found that they accurately described the procedures used for sample receiving. The written SOPs are documented in Duties and Responsibilities of Sample Custodian for the DOE Environmental Survey (SOP: 001, April 11, 1988) and Sample Receiving and Inspection for the DOE Environmental Survey Program (SOP: 002, April 11, 1988).

#### Sample Storage, Identification, and Security

##### Low Level Radiochemistry (LLR) Sample Storage and Security

Samples transferred to the LLR laboratory are stored in boxes on shelves located in Room F-50 of Building 4500S. The door to the storage room is locked when no one is in the LLR receiving area. In addition, the door to the receiving area contains a combination lock and is locked during non-business hours.

## High Explosives (HEX) Sample Storage and Security

Samples transferred to the HEX laboratory are stored in a locked refrigerator located on the second floor of Building 2026. The gas chromatograph (GC) laboratory in which the samples are stored is locked during non-business hours.

### Additional Security

The laboratory is located within a designated secure federal facility. All persons entering the fenced perimeter must pass through a guard station and present valid identification that they are an employee at the facility. The employees also pass their identification through a card reader which records their entry or exit from the facility. Visitors to the facility must sign-in at the guard gate and be escorted during their visit to the facility. Visitors that are not with an escort or any person not displaying an identification badge will be detained by site security personnel.

### Sample Identification

Each laboratory assigns its samples numbers from the Analytical Chemistry Department's computer network system. Laboratory personnel request the quantity of sample numbers that are needed. The computer assigns the numbers by year, month, and date of request and the sequence of required numbers. For example, if five numbers are needed on May 5, 1988, the computer could assign numbers 880505-10 through 880505-14. The assigned numbers are written or typed on labels which are attached to the sample containers. Prepared samples have the sample identification numbers written on the containers.

Written SOPs for sample storage and security have been developed and implemented for the LLR laboratory. The auditor read these SOPs and they accurately described procedures used for storage and security. The written SOPs are documented in Sample Storage for the Low Level Radiochemical Analysis Laboratory (SOP: LLL007, April 28, 1988) and Sample Security for the Low Level Radiochemical Analysis Laboratory (SOP: LLL008, April 28, 1988).

Written SOPs for sample storage, identification, and security of all DOE Environmental Survey Program samples have been developed and implemented. The auditor read these SOPs and they accurately described the procedures in use. The written SOPs are documented in Sample Storage for the DOE Environmental Survey Program (SOP: 007, April 11, 1988), Sample Login and Identification for the DOE Environmental Survey Program (SOP: 004, April 11, 1988), and Sample Security for the DOE Environmental Survey Program (SOP: 008, April 11, 1988).

The auditor observed that the HEx laboratory did not have written SOPs for sample storage and security.

### Sample Tracking

Environmental survey samples may be tracked through the LLR and HEx laboratories from receipt to completion of analysis by using the following documents:

1. Shipping Container Sample Login Form
2. Request for Analytical Services
3. Receipt Record/Chain-of-Custody
4. Chain-of-Custody Records for Low Level Radiochemistry Analysis Group
5. Notebook for cross-reference sample identification numbers
6. Site-specific analysis logbook
7. Analytical Chemistry Data Sheet
8. Soil Sample Preparation Logbook
9. Gas Chromatogram (GC) Injection Logbook

Documents 1 through 3, listed above, are used to document the condition of samples upon receipt and the transfer of samples to the proper laboratory for analysis.

### LLR Sample Tracking

When samples are transferred to the LLR laboratory, C. Granger, the LLR sample custodian, signs the Receipt Record/Chain-of-Custody. She completes the Request for Analytical Services by entering the sample identification numbers on the form. These numbers are also listed in a loose-leaf notebook which contains a record of all samples received by the LLR laboratory. The auditor noted that copies of the notebook pages are not filed with other Environmental Survey sample data.

Next, C. Granger stores the samples and initiates the Chain-of-Custody Record for Low Level Radiochemical Analysis Group (LLRAG) which contains the following information:

1. Material(s) Released to LLRAG By
2. Releaser's Identification Code (Request Number)
3. Date/Time
4. Received in LLRAG By
5. LLRAG Request Number
6. Log Number(s) (Sample Identification Numbers)
7. Storage Location
8. Log Number(s) (of Samples Removed from Storage)
9. Assigned To

10. Date/Time
11. Location
12. Returned To
13. Date/Time

The preparation and analysis of LLR samples is documented in site-specific logbooks. Information for each sample includes date, activity, sample numbers, analyst, comments, and transfer to the count room.

Analytical Chemistry Data Sheets are used to record count room activities. The sheets include sample numbers, dates, analysts, and results. The auditor noted that the pre-printed sheets did not contain the name of the laboratory.

#### High Explosives Sample Tracking

High explosive samples are transferred from the sample receiving area to J. Caton in the organics laboratory. J. Caton assigns the sample identification numbers, completes the Request for Analytical Services, and initiates a Receipt Record/Chain-of-Custody before the samples are transferred to Building 2026.

W. Greist or B. Tomkins receives the samples in Building 2026 and places them in the storage refrigerators.

Preparation of soil samples is documented in Soil Sample Preparation Logbooks. Information such as weight of sample, sample number, date, method, and analyst is recorded. The preparation of water samples is not documented.

The analysis of high explosive samples is recorded in a GC Injection Logbook which contains the sample numbers, date, and analyst.

Written SOPs for sample tracking for all DOE Environmental Survey Program samples have been developed and implemented. The auditor read these SOPs and they generally described the procedures in use for sample tracking. The SOPs did not include descriptions or examples of documents used to track samples within the laboratory. These SOPs are documented in Sample Tracking for the DOE Environmental Survey Program (SOP: 010, April 11, 1988) and Sample Chain-of-Custody for the DOE Environmental Survey Program (SOP: 011, April 11, 1988).

Written SOPs for sample tracking of LLR samples have been developed and implemented. The auditor read these SOPs and they accurately describe procedures in use for sample tracking in the

LLR laboratory. These SOPs are documented in Sample Tracking for the Low Level Radiochemical Analysis Laboratory (SOP: LLL010, April 29, 1988) and Sample Chain-of-Custody for the Low Level Radiochemical Analysis Laboratory (SOP: LLL011, April 29, 1988).

Written SOPs for sample tracking within the high explosive laboratory have not been developed.

#### Analytical Project File Organization and Assembly

Logbooks currently remain in the possession of the analysts. Sample receiving records are filed by request number and kept in the sample receiving area. Laboratory chain-of-custody records and other documents are filed by request number and kept in the appropriate laboratory.

Written SOPs for analytical project file organization and assembly have not been developed.

#### EVIDENCE AUDIT

The evidence audit consisted of review and examination of analytical project file documentation. Completed analytical project files have not been assembled, numbered, or inventoried. Thus the auditor could make no observations concerning the completeness and consistency of analytical project files.

#### AUDIT FINDINGS

The following eight findings (non-conformances to Evidence Audit Requirements) are based on the results of the procedural and evidence audits.

#### Findings

1. If sample tag numbers are not listed on the chain-of-custody record, the sample custodian does not record the numbers.
2. Copies of notebook pages containing sample number cross-reference information are not included in the case files.
3. The Analytical Chemistry Data Sheets do not contain the name of the laboratory.
4. The preparation of water samples in the high explosive laboratory is not documented.
5. Written SOPs do not specify which laboratory within the Analytical Chemistry Department to which they apply.

6. Written SOPs for sample tracking do not describe or include examples of the documents used within the laboratories.
7. The High Explosives laboratory does not have written SOPs.
8. Written SOPs do not exist for case file organization and assembly.

#### SUMMARY

A debriefing session was held on May 5, 1988 with MMES personnel. During this debriefing, the evidence auditor made the following recommendations based on the findings discussed in this report:

1. If sample tag numbers are not listed on the chain-of-custody record, the sample custodian should record the numbers.
2. Copies of notebook pages containing sample number cross-reference information should be included in the case files.
3. The Analytical Chemistry Data Sheets should contain the name of the laboratory.
4. The preparation of water samples in the High Explosives laboratory should be documented.
5. Written SOPs should specify which laboratory within the Analytical Chemistry Department to which they apply.
6. Written SOPs for sample tracking should describe and include examples of the documents used within the laboratories.
7. Written SOPs should be developed for the High Explosives laboratory.
8. Written SOPs should be developed for case file organization and assembly.

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*Fam*  
AUG 1988 34

OAK RIDGE NATIONAL LABORATORY  
OPERATED BY MARTIN MARIETTA ENERGY SYSTEMS, INC.

POST OFFICE BOX 2008  
OAK RIDGE, TENNESSEE 37831

August 18, 1988

Harold Vincent  
EPA-LV  
P. O. Box 93478  
Las Vegas, NV 89193-3478

Dear Harold:

This letter is to inform you of actions taken as a result of the RAD audit at ORNL on May 5, 1988.

Item 1. Standards and samples stored together:  
Standards, controls, etc. have been moved out of the sample storage/chain of custody room and into a laboratory area where no other samples are stored (4500S, F-50).

Item 2. Wear rubber gloves when handling certain samples:  
Laboratory personnel are now wearing gloves in the lab to prevent exposure to biological hazards that could be present in sludge, sewer water, etc.

Item 3. Crowded conditions in the central sample receiving area:  
We are still crowded here but since this visit we have purchased 2 large glass door refrigerators like those you see at the local 7-11 store. These allow more efficient use of refrigerator space and ease of locating samples. We are slowly disposing of old survey samples as the reports are prepared.

Item 4. Data audit package submittal requirements:  
All of these requirements have been met.

Item 5. Information collected on survey samples should be part of the case file:  
We will place in the case file a copy of our manual login records. We will continue to log survey and other non-survey samples into the same logbook. The sample login form has been improved and should meet all audit requirements.

Item 6. Missing procedures and changes in procedure titles:  
The procedure for uranium in soil (OR-030) is in the survey manual. A procedure for tritium in water is in the manual (OR-101), tritium in soil is determined by a modification of this method and this modification will be described in detail in the case narratives. The strontium procedure is titled "Total Radioactive Strontium" not "Total Strontium." A fluorometric procedure from the Environmental Survey Manual, Appx. D, is now available in the laboratory.

Item 7. SOP's not readily available:  
SOP's are now in clearly labeled notebooks and are kept at each working area. Instrument SOP's are now located near each instrument.

Item 8. Bias in EML and EPA QA programs:  
For the Feb. 1988 EML QA samples, ORNL's average score for Plutonium in all matrices was 80 (known) and 95 (mean) and no bias was indicated. Our scores are 87 for gross alpha and 72 for gross beta in the EMSLV QA samples so far this year. We have always used a Sr(Y)-90 equivalent when reporting gross beta because of the concern about strontium here at ORNL. EMSLV uses Cs-137 to prepare the gross beta and if a Sr(Y)-90 equivalent is used the result will be low. In the future we will report our results for gross beta to EMSLV as a Cs-equivalent; this should correct the negative bias.

Item 9. Recommended changes in logbook, data storage and SOP's:  
These changes have been made or are in process.

Item 10. No direct printout of fluorometric uranium results, no record logbook:

Direct printout of calibration data and sample results is not possible with the present instrument. We have purchased a laser fluorimeter with an RS232 port so interfacing with a printer or a computer is possible. A record logbook has been started which records all calibration standards and quality control results and lists the customer samples analyzed with these standards and controls.

Please call (615/574-4852) if you have any questions on this audit response.

Sincerely,

  
W. R. Laing  
ACD Team Leader

WRL:lp

cc: P. L. Howell  
J. R. Stokely  
J. W. Wade  
R. B. Fitts

**Draft - Do Not Cite**  
**ANL Data Document**  
**Issue Date: June 1989**  
**Revision: 01**

### **Internal Quality Assurance Reviews**

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OAK RIDGE NATIONAL LABORATORY

OPERATED BY MARTIN MARIETTA ENERGY SYSTEMS, INC

POST OFFICE BOX 1  
OAK RIDGE, TENNESSEE 37831

April 21, 1988

Distribution

Martin Marietta Energy Systems QA Audit of  
The Oak Ridge Environmental Survey Program

Attached is the final report from the internal QA Audit of the Oak Ridge National Laboratory activities for the DOE Environmental Survey Program. The audit was commissioned by me and, for the ORNL Analytical Chemistry Division (ACD) by D. Shults, Director of the ORNL ACD at the request of D. K. Knight, the DOE Environmental Survey Program Manager.

I would welcome any comments you might wish to make regarding this report.

Sincerely,



Robert B. Fitts, Program Manager  
DOE Environmental Survey  
Environmental Sciences Division

REF:tmp

DISTRIBUTION

B. R. Appleton  
J. T. Bradbury  
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R. F. Swiger  
R. S. Wiltshire

Internal Correspondence

MARTIN MARIETTA ENERGY SYSTEMS, INC.

APR 22 1988

April 18, 1988

R. B. Fitts

DOE Environmental Survey Program - Final Quality Assurance (QA) Review of the ORNL Analytical Chemistry Division's Organic, Inorganic, Radiochemical, and High Explosives Analysis Laboratories

Please find attached the report from the above activities. In order to understand the final report, please reference the cover letter for the Review of the Pantex Site Organic Data Generated by the ORNL Analytical Chemistry Division (ACD), dated March 23, 1988. This cover letter is included with this report as Attachment 6.

Due to the urgency of this situation we have distributed draft reports to the labs. Further distribution should be made by your office. Please request corrective actions and allow P. L. Howell to track, review and verify adequacy of the completed action items as per the Charter, dated February 25, 1988.

All of the requested QA reviews of the ORNL ACD's Organic, Inorganic, Radiochemical and High Explosives analysis labs are now complete. Any additional information concerning the reviews (review notes, evidentiary information) is available to you upon request.

Should you have further concerns or questions about anything in the reports or QA concerns in your program, please call me or P. E. Melroy, ORNL's Quality Manager.

*D. W. Frazier*

D. W. Frazier, 1000, MS-335, ORNL (6-0347)

DWF:cet (QA-88-30)

**Attachments:**

1. Copy of Sample control and Chain-of-Custody Sheet with suggested additions
2. Letter - Oak Ridge Environmental Survey Program Review - Final Review and Recommendations - To Frazier, From McMahon
3. Lists of the revised Organic and Inorganic Standard Operating Procedures reviewed
4. Total list of organic SOP's to be revised
5. Total list of inorganic SOP's to be revised
6. Cover letter and Review Report (from L. W. McMahon) from the Pantex site data review

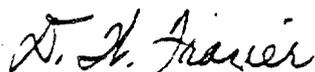
Final Report of the Second Quality Assurance (QA) Review of the ORNL Analytical Chemistry Division's Organic, Inorganic, Radiochemistry, and High Explosives Analysis Laboratory Participating in the DOE Environmental Survey Program

Issued to:

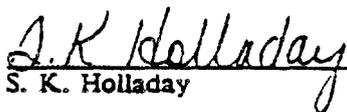
R. B. Fitts

April 18, 1988

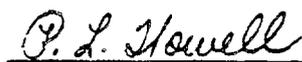
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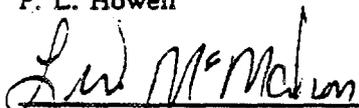
D. W. Frazier, Review Team Leader



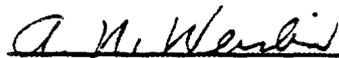
S. K. Holladay



P. L. Howell



L. W. McMahon



A. N. Weisbin



A. A. Halouma

## INTRODUCTION

On April 11-12, 1988, the QA review team consisting of A. A. Halouma, S. K. Holladay, P. L. Howell, L. W. McMahon, A. N. Weisbin and D. W. Frazier met with ORNL personnel W. R. Laing, J. E. Caton Jr., W. H. Griest, J. C. Price, J. W. Wade, C. A. Treese, J. A. Hayden, and S. J. Bobrowski, prior to beginning the review of the subject laboratories. A checklist including the areas of concern for the review had been provided prior to the activity. The status of the corrective action items from the EPA audits of the program conducted in June 1987 and January 1988 and from the first QA review were also addressed. This report will reflect, as best could be determined, the status of subject labs readiness to be audited by the EPA in connection with the requirements of the statement of work. Since this is the final report, items from the first report are included to provide a comprehensive overall summary of this status.

## SCOPE

This QA review was requested by R. B. Fitts, Program Manager of the Oak Ridge Environmental Survey Program (ORESP) and ORNL Analytical Chemistry Division (ACD) Director W. D. Shults, to obtain an independent evaluation of participant's compliance to established guidelines to the Contract Laboratory Program protocol. The Draft of ORNL USEPA Contract Laboratory Program (CLP) Statement of Work for Organic and Inorganic Analysis Multi-Media, Multi-Concentration SOW No. 787, and good lab practices were used as the basis for the review. The team began in the Sample Receiving Laboratory and proceeded to review the Organic, Inorganic, Radiochemistry, and High Explosives analysis laboratories.

## COMMENDABLE EFFORTS NOTED

### ORGANICS LABORATORY

1. Volatile organic matrix spikes, tune criteria, and surrogate recoveries are being reviewed on a batch-to-batch basis - relates a good effort to comply with protocol in spite of man-power needs.
2. Instrument run log notebooks were well thought-out and designed.
3. There was an excellent effort to develop software to produce the required PCB/Pesticide CLP forms. Further efforts to include additional useful information to the Form 1D was made prior to the second QA review.
4. Review of the linearity of standards, surrogate recoveries, matrix spikes and matrix spike duplicates is now evident in the Organic labs prior to sample reporting.
5. There has been a commendable effort put forth to address the corrective action items from the EPA audits and the first QA review.

6. The semivolatile data evaluation, although not complete at the time of this second review, is moving toward completion since additional instrumentation has been ordered and further training in the use of the software is scheduled with Hewlett Packard Company representatives.
7. The final report of Pantex VOA data has been generated to correctly state quantitative values, positive contaminate identifications, documentation of deviations from the protocol, and documentation of corrective actions taken for out-of-control conditions.

#### INORGANICS LABORATORY

8. Applicable inorganic technical and CLP procedures were made accessible in notebooks for use by each analyst - very good practice.
9. Exemplary documentation of notebooks in compliance to the CLP protocol in the ICP and Atomic Absorption labs.
10. Revised standard operating procedures, and implementation thereof has begun.
11. All biographical data on personnel was well documented.
12. Certification records were available on all personnel including the EPA procedures that they were certified to perform - excellent.
13. CAPA Sample Prep lab notebooks and records were exemplary.
14. A holding time traceability system has been established in this section, and is being tested in the organic section. By request number the sample is compared to the holding time date and to the program due date, whichever date is earlier is printed as the deadline.

Status: Incomplete

#### ATOMIC ABSORPTION LAB

14. Training records to CLP procedure are complete.
15. Procedures in use were on hand for analysts use.

#### MERCURY LAB

16. The sample prep and mercury labs were very well organized.

#### RADIOCHEMISTRY

17. Chain-of-custody system for paperflow and sample management appeared to be an effective system for the present set-up.
18. Documentation of instrument maintenance, specific weekly counting activities, instrument setting log, and QC were found to be exemplary.

## HIGH EXPLOSIVES LABORATORY

19. Even though this lab is not under the CLP protocol, several SOP's were written to cover the involvement in the program.
20. Data transfer and CLP form generation are being patterned after the PCB/PEST Form I and are quite comprehensive - excellent effort.

## ASBESTOS LAB

21. Involvement for the Environmental Survey in the Asbestos lab was found to be very well organized, instrument and standard operating procedures were in place, training - past and future plans were excellent, master log book is noteworthy, lab security is well thought out and implemented, and waste management was handled by sending all of the sample (including the portion analyzed) back to the customer, just an exemplary effort.

## DEFICIENCIES/RECOMMENDATIONS

### GENERAL:

This review included a more thorough study of the standard operating procedures (SOP) throughout the labs. A. N. Weisbin, spent a considerable amount of time reviewing newly written SOP's against the CLP requirements. The list of Organic and Inorganic SOP's reviewed and conclusions drawn can be found in Attachment\_3 to this report. Consider that the recommendations and comments in the attachment are the team's recommendations to be incorporated into the SOP.

1. There were too many different forms requiring varying information, and inconsistently used for the same purpose in use throughout the laboratories, which made sample tracking very difficult. Although the number of forms has not decreased, the Organic lab has re-designed their chain-of-custody form to reflect only the needed information.

**Recommendation** This applies specifically to work under the CLP protocol; Use a centralized receiving record, or a log to record the incoming samples.

### Comment

- A. The Organic Lab Chain-of-Custody form has been revised to reflect their informational needs. Three suggested additions are included for your consideration as a result of previous audits (1) the number of containers received, (2) the site name, and (3) state whether the container holds a sample or an extract. (See Attachment 1, copy of the form.)
- B. In order for sample tracking to be more efficient, consider numbering the forms to cross-reference Request for Analytical Services form with the Chain-of-Custody form.
- C. There is now a central sample tracking system in place.

Status: Complete

2. There is no consistent documentation to the customer concerning as-received sample nonconformances.

Recommendation: Written documentation of sample nonconformances should accompany phone calls to notify the customer. An entry can be made directly on the Request for Analytical Services form. This could be called out in the Sample Receiving and Inspection for the DOE Environmental Survey Program Standard Operating Procedure.

Comment: This item is covered in Draft SOP-002, Sample Receiving and Inspection for the DOE Environmental Survey Program.

Status: Complete

3. The lack of man-power which was evident in the sample receiving area during the first review is being handled.

Recommendation: During the interim, it will be necessary to properly train temporary personnel. The use of a simple stepwise checklist made up from the SOP to assure that everything gets done can be used, or simply train some relief personnel to the SOP for back-up (especially in the sample receiving areas.)

Comment: This item is also covered by Draft SOP-002, as in item #4.

Status: Complete

4. Different AnaLis sample identification numbers were assigned to the same sample for multi-analysis (VOA, SVO, ICP, Hg, etc.) was found to be inefficient and time consuming when compiling data reports for a sample.

Recommendation: Consider centralization of the sample log-in function. Man-power and terminals for this function could yield a more efficient sample tracking system with several avenues to data retrieval at one source. Consideration of this for the CLP program is strongly advised by the QA review team.

Comment: Lab personnel have developed a sample tracking system which allows samples to be located via request numbers or assigned lab numbers. Therefore a central login would not be necessary.

Status: Complete

5. A lack of awareness of the Analytical Chemistry Division's general policy for sample disposal was Train employees in the use of applicable SOPs.

Comment: Draft SOP-013 will be issued by June 1, 1988. Training of the sample receiving personnel to the SOP has already taken place.

Status: Incomplete

6. Printed forms were completely filled in. This was much improved over the situation observed during the last review.

Comment: This area should be monitored on an unscheduled basis to assure that it is continuously being done.

Status: Complete

7. Personnel should be made aware of the data validation process. A documented data validation process is scheduled to be written to cover this issue.

Comment: Standard operating procedures to be revised or written should have targeted completion dates.

Status: Incomplete

8. Date of receipt on chemicals were inconsistently applied.

Recommendation: Management must assure that policy regarding age of chemicals used for any aspect of analysis is set up and implemented. This allows chemicals to be used on a first-in first-out basis.

Status: Incomplete

9. Non-target parameter laboratories have very little familiarity with QA/QC and evidentiary requirements.

Recommendation: Strongly consider conducting documented QA/QC discussions at regular intervals during general meetings or separately, whichever meets the need. Regular meetings should document attendance if safety or QA/QC is discussed and kept in training file.

Status: Incomplete

10. Non-target parameter labs were found to be weak in the implementation of standard operating procedures (SOPs).

Recommendation: Train employees in the use of applicable SOPs.

Status: Incomplete

11. Glassware Cleaning procedures, posted above sinks for easy reference by user, were not signed and dated by management.

**Recommendation:** All Technical and Standard Operating Procedures should be signed and dated by applicable management to show that the procedure is an official document.

**Status:** Incomplete

12. Notebook reviews were being performed, but repeated obliterations without initials or dates of the action were found.

**Recommendations:** Instructions for how to fill out a notebook are available in the Martin Marietta Energy System's laboratory notebooks and handling of errors is a part of the instructions. Training to these instructions should be a part of the regular group meetings for old and new hires. An error should have a single line drawn through it, initialed, and dated.

**Comment:** Draft SOP-003, Requirements for Recording and Correcting Lab Entries for the Environmental Survey Program has been written to address this deficiency. Training of all ACD employees to the SOP has been planned and will be complete by June 1, 1988.

**Status:** Incomplete

13. The mechanism for handling future CLP work has changed. Future work will incorporate analyst review and interpretation of all data prior to reporting quantitative values, and to assure that the required QC criteria are met before proceeding with the analysis.

**Status:** To be monitored during analysis of next CLP samples.

#### ORGANIC LABORATORIES

14. Although writing and revision of SOP's are in progress, it is doubtful that all of the SOP's called out on the list supplied to the team will be completed prior to another EPA audit.

**Recommendation:** Prepare an action plan for completing the writing and revision of SOP's, with specifics, such as SOP name, completion date, review and comment due date, issue date, training to SOP completion date, and show evidence that the plan is being followed. Be reasonable in this activity, set dates that can be achieved, but dates that reflect urgency to have this activity completed.

**Status:** Incomplete

15. While tracking an Argonne CLP sample, it was noted that there was no Chain-of-Custody form, nor original request for services resulting in an incomplete paperflow.

**Recommendation:** Prepare a receiving and completed data package checklist to be reviewed for essential paperwork in a CLP package for each file.

**Comment:** This type problem will be handled with the implementation of the appropriate SOP's. However, this is still a concern until the SOP's are implemented. A copy of this checklist was supplied to the lab by L. W. McMahon.

**Status:** Incomplete

16. Training to the CLP protocol is being planned for the Organic labs staff. Arrangements are being made to obtain the services of EPA personnel to conduct the training in mid-May.

**Status:** Incomplete

17. There was insufficient data handling software/hardware during the first review. Presently, arrangements have been made with Hewlett-Packard Company representative to further train staff to use the new RTA System, and two additional Scan Boxes have been ordered to make the system efficient which will increase data evaluation productivity.

**Status:** Incomplete

18. There is now documentation of corrective actions in the GC-MS and PCB/PEST labs.

**Status:** Complete

19. The daily check on the refrigerator temperature is now being performed and recorded. Temperature excursions are handled by adjusting the controls until the event is under control. The Temperature Controlled Sample Storage Areas: Records and Maintenance SOP, is to be written and implemented. The Organic Analysis lab supervisor has committed to supply the team with a schedule for the completion of the organic SOP's.

**Status:** Incomplete

20. Sample concentration data is now being flagged to show the appropriate blanks concentrations.

**Status:** Incomplete

21. Data validation will be performed by two people in the GC-MS lab, as well as by the Group Supervisor, when possible, in a manner that will expedite sample analysis and data handling.

**Comment:** Unscheduled monitoring should confirm continued practice.

**Status:** Complete

22. There was evidence that only <sup>two</sup> ~~three~~ performance evaluation samples out of five quarters were completed and reported.

**Recommendation:** In order to access the labs ability and capability to operate under the CLP protocol, the performance evaluation samples must be completed and reported to show good faith that the samples can be analyzed as necessary.

**Status:** Incomplete

#### PESTICIDE/PCB LABORATORY

During this QA review, L. W. McMahon reviewed in detail the PCB/PEST data as it is now being evaluated and the semivolatile data as it is presently generated using the Aquarius software. Please find a draft version of his report to me in Attachment 2, dated April 15, 1988 entitled Oak Ridge Environmental Survey Program Review - Final Review and Recommendations. The recommendations stated in his report are official recommendations of the QA review team and will be considered as such.

22. Lack of sufficient number of Gas Chromatographs (GC) and personnel for project workload was noted during the first review. At present, another GC has been borrowed for CLP work until a recently ordered system is in-house and set up. Management is actively interviewing to add personnel to the workforce. There can be no date set for personnel addition, this activity will have to be monitored closely to expedite the process.

**Status:** Incomplete

23. A better understanding of the CLP protocol is now evident, such as personnel now are aware that the Form VIII Evaluation Standards must be within specification prior to sample analyses; that the raw data reported on Form I is the laboratory validated results, and that tentatively identified compounds must be referenced on Form X. However, the following recommendations must be made in an effort to strengthen this area.

**Recommendation:**

- Give SAIC hardcopy of data to use to verify the final electronic CLP form generation.
- Continue to put the PCB/PEST data together in the CLP package.
- Report all quantitation data as estimated flagged with a "J".
- If matrix spike recovery = 0, the data associated with it should be flagged as not useful.
- Alter computer program on sample calculation for the following; discontinue averaging the response factors, and quantitate on the nearest appropriate Individual A or B standard.
- All organic staff need additional training to the CLP protocols.

24. SAIC should take out the packed and capillary column data that they now have and replace it with the data on the present Form I.

Status: Incomplete

25. Case narrative should explain the rationale for altering Forms II and VIII and should also address Form III.

Status: Incomplete

26. Confirm via comparison the information on the forms vs the information in the AnaLis database.

Status: Incomplete

#### VOLATILE ORGANICS

The status of the VOA data was reported in a letter to D. W. Frazier, from L. W. McMahon entitled Review of Pantex Data at ORNL 2/23/88 - 2/26/88, dated March 2, 1988. (See Attachment 7.)

#### SEMI-VOLATILE ORGANICS

29. The evaluation of the raw data generated on the GC-MS Chem stations is now taking place through the use of the RTA to produce the CLP forms. The information is being assembled into CLP data packages.

Status: Incomplete

30. The review team has similar concerns with the semi-volatile organic data as with the volatile organic data, such as matrix spike results being outside the QC window, detection limits and results needing to be corrected for moisture content, and positive hits reported as estimated values. The number of CLP non-conformances is probably not so extensive that the data should all be declared as Level III quality. This conclusion was based on the evaluation of limited data available at the time of the review. The semi-volatile organic data evaluation by the labs' staff was not complete. It has been predicted that this data evaluation will not be complete for several weeks.

Status: Incomplete

#### HIGH EXPLOSIVES LAB

31. Sample receipt is inadequate. Chain-of-custody is not carried through to receiving personnel at Bldg. 2026 from ORNL Receiving personnel.

Recommendation: Some type of arrangements will be made and documented with ORNL Receiving such that someone in the Lab must sign for the incoming samples. They are presently left at the front door of the High Explosives lab Bldg. 2026 until the cooler is found.

Status: Incomplete

## INORGANIC LABS

## GENERAL:

32. Control work sheets containing the results of analysis are now being put into laboratory notebooks in the % solid and fluorometric Uranium analysis lab.

Status: Complete

33. Notebook entries are being made in black ink.

Status: Complete

34. Violations of error correction protocol (single line through error, initials, and date) were observed in notebooks throughout the lab.

Recommendation: See recommendation under Deficiency #12.

Status: Incomplete

35. The review of the notebooks by supervision or designee obliterated actual data in several notebooks.

Recommendation: An area on the data page should be allotted for witnesses signatures and/or stamps.

Status: Unscheduled monitoring to confirm continued action.

## ICP LAB

36. Lack of back-up instrumentation presently on line in the ICP laboratory.

Recommendation: Provide documented policy or agreements for back-up in case the present ICP instrument fails.

Comment: To date the team has not received any assurances that this concern has been handled.

Status: Incomplete

## CYANIDE LAB

37. There is a need for awareness of the methods used in the lab (SW-846, EPA-600, and CLP method EPA-335.2) for different types of samples.

Recommendation: Train employees so that they will be aware of such information.

Comment: This can be handled in regular group discussion meetings.

Status: Incomplete

38. There was no awareness that there are specified concentrations with which the instrument should be calibrated.

a. This was reflected in the lack of frequent instrument standardizations.

b. General lab QA/QC not strictly followed;

- Conductivity of water is not recorded.

- Balance is not regularly calibrated.

**Recommendation:** Implement SOP's to alleviate this situation.

**Comment:** Assure that employees in this lab are following the QA/QC procedures for the ACD as well as for the Environmental Survey Program.

**Status:** Incomplete

39. There was no SOP for washing glassware at the sink.

**Recommendation:** Post SOP at sink in the Cyanide analysis lab.

**Status:** Incomplete

40. Reagents should be dated upon receipt before storage in the refrigerator.

**Recommendation:** Initial and date all incoming reagents, standards, etc. for use in sample analysis to allow first-in first-out usage of supplies.

**Status:** Requires unscheduled monitoring for continuous action.

#### RADIOCHEMISTRY LAB

41. Procedures are still in the old format, but updating to conform to the NQA-1 format is in progress.

**Recommendation:** Document expected completion of this activity.

**Status:** Incomplete

42. The Environmental Survey Manual is in the process of assigning ESM numbers for the Radiochemical procedures.

**Status:** Complete

43. The Sample Receiving, Logging and Distribution procedure was found to be inadequate. There is no QA input and it is not written in procedural format.

**Recommendation:** This procedure is a strawman and is in need of being completed, "adding the meat of how to do the receiving, logging and distribution." The SOP is a part of the QA process and was written so that when it is implemented will assure that these processes don't fall through a crack.

Status: Incomplete

**ASBESTOS LAB**

44. Standard operating procedures for this lab are not written, but a system is definitely in place.

Recommendation: Inorganic lab SOP's should include the Asbestos lab in all areas.

Status: Incomplete



# DRAFT

Internal Correspondence

MARTIN MARIETTA ENERGY SYSTEMS, INC.

ATTACHMENT 2  
Detailed Review of PCB/PEST Data Evaluations

April 15, 1988

D. W. Frazier

Oak Ridge Environmental Survey Program Review - Final Review and Recommendations

During the second review on April 11, Mike Guerin's and John Hayden's comments and questions expressed previously (Pantex PCB/Pesticide Data Review, Guerin to Frazier, March 25, 1988) regarding the pesticide/PCB data were addressed. I will note how these issues were resolved and then offer some conclusions from the review.

Issues Noted in Guerin's Memo

1. The data packages reviewed on February 23-26 did not reflect extensive data evaluation and checks. Contradictory results were reported within the data set (duplicate Form I's with different results), within AnaLIS, and within the SAIC database. Two causes for this were identified; misunderstanding by the laboratory about how to present CLP data and transfer of raw data to SAIC. As of the second review on April 11 the lab is reprocessing the CLP packages to reflect the necessary data checks and evaluation.
2. The calibrations did not meet the CLP linearity requirement. Specific instruction is found on pages D-32 through D-35 and E-52 of the 10/86 SOW. The additional 5 point standards used by the lab to demonstrate linearity were at a higher concentration range than required. In addition the response factors used for calculations were averaged. This process was reviewed with John Hayden on 4/11 and his questions regarding the linearity and continuing calibration requirements were resolved.
3. To insure SAIC database is correct, hard copies of the lab evaluated data will be given to SAIC.
4. Abnormalities previously noted in computer generated forms have been corrected.
5. After re-evaluating the blank data and correcting the Form I data, the concern about blank contamination has been resolved. The single positive hit must be addressed in the case narrative.
6. Over the past year to 18 months, EPA-EMSL has been quite nebulous regarding the use of an appropriate surrogate as well as the value of Dibuty/Chlorandate (DBC) recovery data. The lab was operating under the assumption that mirex was an acceptable alternative to DBC. In terms of the SOW used for the DOE Survey work it was not. However, while no criteria is available to evaluate mirex recovery, it can be used to make some technical judgement as to how well the overall extraction and analysis process is working. This issue must also be addressed in a case narrative. (Analysis data to evaluate mirex is provided as Attachment 6.)

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7. The questions posed by the Guerin memo were addressed on 4/11 with John Hayden as follows:
  - (a) A single Form I is used to report quantitative, confirmed data. Raw data from both columns is to be included in the package. The data reported on Form I is the laboratory validated results.
  - (b) If the linearity check from EVAL A, B, and C exceeds 10% for aldrin, endrin, or DBC discontinue the analysis, troubleshoot the equipment/technique, and meet this requirement before continuing analysis. If DDT exceeds the 10% requirement see paragraph 4.5.4.4, page E-59 of the 10/86 SOW. The footnote on Form VIII PEST-1 refers to DDT only.
  - (c) There is no reference to tentatively identified compounds on Form X.

#### OVERALL ASSESSMENT OF PANTEX PESTICIDE/PCB DATA

While appropriate to make professional judgments and express concerns on the validity of data, the additive nature of QC factors out of specification is difficult to express. The reviewer as well as the laboratory has a responsibility to inform users of the data of all concerns in order to assist that user in avoiding inappropriate use of the data while at the same time not precluding data necessary to facilitate the progress of projects requiring the availability of the data. While data which does not meet specified requirements is never fully acceptable, this line-of-thought is consistent with EPA guidance on laboratory data evaluation (Technical Directive Document No. HQ-8410-01, Laboratory Data Validation, Functional Guidelines For Evaluating Pesticide/PCB's Analysis, May 28, 1985). Using guidance from this document, I suggest reporting the data annotated as outlined below while fully explaining any non-conformance in the case narratives. I suggest this for the following reasons:

1. Factors beyond the control of the laboratory were a cause of many QC non-conformances.
  - (a) There was miscommunication between management and the lab concerning project requirements, capabilities available at the time of Pantex sampling, and capacity to handle the workload within the time frame allotted.
  - (b) There were continuing changes in program requirements, by DOE-HQ, concerning the CLP reporting requirements and documentation, and
  - (c) Continuing changes to the Sampling and Analysis Plan even during sampling.
2. Making data available in this manner will facilitate the progress of the Pantex project.

I. Suggested procedure to annotate Pantex Pesticide/PCB data

Sample Holding Times - If 40 CFR 136 holding times are exceeded, flag all positive results as estimated (J) and sample quantitation limits as estimated (UJ) and annotate data to the effect that holding times were exceeded.

II. Pesticides Instrument Performance -

1. DDT Retention Time - If the retention time of DDT is less than 12 minutes, a close examination of the chromatography is necessary to assure that adequate separation of individual components is achieved. If adequate separation is not achieved, all affected compound data are unusable and must be flagged with (R).

2. Retention Time Windows - Retention time windows are used in qualitative identification. When these retention time windows have not been met, positive results should be considered tentative (N).

3. DDT/Endrin Degradation Check

a. DDT breakdown is greater than 20%;

(1) All quantitative results for DDT should be considered estimated and flagged with (J).

(2) Qualitative and quantitative results for DDD and DDE should be considered estimated and tentatively identified and flagged with (JN).

(3) All other pesticide PCB results should be inspected very closely to determine their validity.

b. If Endrin breakdown is greater than 20%;

(1) All quantitative results for endrin should be considered estimated and flagged with (J).

(2) Qualitative and quantitative results for Endrin ketone should be considered as tentative and flagged with (NJ).

(3) All other results should be inspected very closely to determine their validity.

4. Retention Time Check

a. If the retention time shift for DBC is greater than 2.0% for packed column or greater than 0.3% for capillary column, the analysis should be

considered unusable for that sample(s) with discernable chromatographic peaks and results flagged with an (R).

- b. The absence of a DBC peak does not constitute a violation of the above condition since DBC may be absent due to low recovery of dilution.

### III. Calibration

1. Initial Calibration - If criteria for linearity are not met, all associated quantitative results should be considered estimated and flagged with (J).
2. Continuing Calibration
  - a. If the % Difference between calibration factors during the 12 hour period is greater than 15% for the compound(s) being quantitated, flag all associated positive quantitative results as estimated and flagged with (J).
  - b. If the % difference is > 20% than the CRL0D is estimated and flagged with (UJ).

### IV. Matrix Spike/Matrix Spike Duplicate

1. No action is taken on Matrix Spike/Matrix Spike Duplicate (MS/MSD) Data alone to qualify an entire Case.
2. The results of the matrix spike and matrix spike duplicate can be used in conjunction with other QC criteria to aid the user in applying more informed professional judgement when necessary.
3. On a sample-by-sample basis, the following suggestion on using MS/MSD results is provided for the specific sample spiked. If the results are positive (above detection limit) and the percent recovery is zero, the results of the unspiked sample for which (MS/MSD) were performed are flagged with a (J) as estimated. If the results are less than the detection limit and spike recovery is zero, the results for the spiked compound(s) with zero recovery for the unspiked MS/MSD sample should be flagged as unusable with an (R). Multiple zero recoveries for compounds may suggest more general application of qualifiers.

- VII. Compound Identification - Compound results reported without meeting qualitative criteria for two column confirmation should be flagged as not detected with a (U), using professional judgement to assign appropriate Sample Detection Limit.

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April 15, 1988

#### Status of Laboratory Operations for Future Work

The laboratory personnel have a better understanding of CLP QA/QC requirements and are working within their means to insure capabilities are in place to handle future work. The Hewlett Packard (HP) RTA system is operational. On-site training by HP personnel, well versed in the use of Aquarius software is scheduled for mid-May. Two scan boxes previously recommended to increase productivity for semivolatile data processing has been ordered.

Communication between the sampling team and analytical team has improved and the sampling schedule at INEL has been lengthened in an attempt to resolve capacity issues in light of holding time concerns. Since 300 volatile organics will exceed the labs capacity, the aide of one or more other laboratories should be arranged as soon as possible.

#### Review of Sampling and Data Management Activities in Support of DOE Survey

On the morning of April 13, a short time was spent with Donna Pickel, John Murphy, and Karen Daniels reviewing the ORNL field participation in the Pantex project. Murphy reiterated the evolution of program requirements regarding field QC activities and their subsequent implementation by the ORNL team. At Murphy's initiative he has updated his on-site NPDES sampling program to include many of the DOE Environmental Survey program field QC protocols and intends further QC improvements to the RCRA sampling as well. From this discussion it appears the participation of the ORNL sampling team in the DOE Environmental Survey has resulted in improvements to the on-site monitoring programs at ORNL. Murphy provided the review team a written response to the review team checklist which addressed the documentation techniques, disposal procedures, sampling plan deviations, and training and personnel qualifications.

I would offer a single suggestion as to how this work effort has been documented in that the field log sheets should be bound by 19-hole punch spiral binder prior to archival in the case file. This should serve as better binding for storage than the staples and loose-leaf binders used during assimilation.

Karen Daniels is responsible for the data management activities. Much of this work has been contracted to SAIC. A review of SAIC work was reported earlier (McMahon to Frazier, March 18, 1988). Again, I would reiterate the recommendation that the data management teams review hard copy, lab generated CLP forms against the electronic database to insure that lab evaluated data is the data represented in the

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database. Furthermore, a meeting between lab personnel and the data management team will likely be needed to insure the annotated lab data is properly interpreted. Dealing with CLP QA/QC requirements is equally new to the data management team. I believe a training program, by lab personnel experienced in the generation of CLP data, would be beneficial for the data management team and strengthen the communication skills needed to deal with the CLP lab.

Please call me if I can provide further information.

L. W. McMahon, 9704-1, MS-001, Y-12 (4-7535)

cc: T. R. Butz/C.C. Hill  
L. L. McCauley/C.W. Kimbrough

ATTACHMENT 3

Organic Lab - List of New Standard Operating Procedures (SOP) Reviewed

A. N. Weisbin  
4-11-88

Recommendations and Comments:

- SOP #1                    Sample Login and Identification for the DOE Environmental Survey Program (Draft dated 3-12-88 - not approved)
- 7.2.10 - "Arrange for the proper and secure storage of all samples" - too general.
  - Delete "...QA/QC section, if not applicable", statement.
- SOP #6                    Personnel Signature and Initial Record
- SOP #4                    Sample Storage for the DOE Environmental Survey Program (Refrigerators)
- SOP #2                    Duties and Responsibilities of Sample Custodian
- SOP #3                    Sample Chain of Custody
- Procedure should address answers to questions of "Who signs what?" (signature and date) "Who has ultimate responsibility?"
- SOP #5                    Sample Storage Area Security
- SOP #8                    Sample Tracking
- How are corrections made? Signed for? Attachments?
- SOP #9                    Sample Preparation Bench Sheet
- Sect. 6.2. - How will the sample be identified?
  - Sect. 6.3. - Incomplete
- SOP #17                    Document Flow
- Incomplete
  - Need responsible person also for each document.

Inorganic - List of Standard Operating Procedures (SOP) Reviewed

A. N. Weisbin

4-12-88

Recommendations and Comments:(Applies to all SOP's)

1. Recommend that the Scope and Purpose be separated.
2. Recommend that the QA/QC applicability statement be deleted.
3. Suggest that the summary should be "requirements".
4. Suggest that the list of forms be an attachment in the procedure.

SOP # 001                    Duties and Responsibilities of Sample Custodian for the DOE Environmental Survey Program

SOP # 002                    Sample Receiving and Inspection

Suggestion:                - 7.4.11 Reference secure storage and login procedures...  
Be specific, reference which secure storage and which login procedure will be used.

SOP # 003                    Requirements for Recording and Correcting Laboratory Entries for the Environmental Survey Program

SOP # 004                    Sample Login and Identification

SOP # 005                    Personnel Signature and Initial Record

SOP # 006                    Monitoring Analytical Balance Performance

SOP # 007                    Sample Storage

SOP # 008                    Sample Security

SOP # 009                    Monitoring Cold Storage Temperatures

SOP # 011                    Sample Chain-of-Custody

SOP # 013                    Sample disposal

See comprehensive listing of all SOP's in Attachment 5 to this report.

ATTACHMENT 4

STANDARD OPERATING PROCEDURES

ORGANIZATIONAL

1. SAMPLE LOGIN AND IDENTIFICATION ✓
2. DUTIES AND RESPONSIBILITIES OF SAMPLE CUSTODIAN ✓
3. SAMPLE CHAIN-OF-CUSTODY ✓
4. SAMPLE STORAGE ✓
5. SAMPLE STORAGE AREA SECURITY ✓
6. PERSONNEL SIGNATURE AND INITIAL RECORD ✓
7. SAMPLE IDENTIFICATION
8. TRACKING SAMPLE ANALYSES ✓
9. SAMPLE REQUEST LOG NOTEBOOK
10. SAMPLE PREPARATION LOG
11. SAMPLE PREPARATION BENCH SHEET ✓
12. VOLATILES ANALYSIS INJECTION LOG
13. SEMIVOLATILES ANALYSIS INJECTION LOG
14. GCMS BACKLOG SHEET
15. PESTICIDES/PCBS ANALYSIS INJECTION LOG
16. PROGRESS REPORT
17. DOCUMENT FLOW ✓
18. DOCUMENT CONTROL
19. ORGANIC GCMS DATA REVIEW
20. REVIEW OF SAI-TREATED VOLATILES DATA
21. ORGANIC PESTICIDES DATA REVIEW
22. ORGANIZATION AND ASSEMBLY OF CASE FILE
23. ORGANIZATION AND ASSEMBLY OF EPA ORGANIC DATA PACKAGE
24. DOCUMENT/DATA PACKAGE SHIPPING
25. TRACEABILITY OF STANDARDS
26. ORGANIC STANDARDS STORAGE AND CUSTODY
27. ORGANIC REAGENT TRACEABILITY
28. TRACEABILITY OF MATRIX AND SURROGATE SPIKING SOLUTIONS
29. STORAGE OF MATRIX AND SURROGATE SPIKING SOLUTIONS
30. REQUIREMENTS FOR RECORDING, VALIDATING, AND CORRECTING ENTRIES
31. TEMPERATURE CONTROLLED SAMPLE STORAGE AREAS: RECORDS AND MAINTENANCE
32. CLEANING OF GLASSWARE
33. BALANCE OPERATION CHECK
34. DISPOSAL OF ENVIRONMENTAL SAMPLES
35. LABORATORY SAFETY

**DRAFT**

C-132

*Susan Holliday (4-11-'88)*

ATTACHMENT 5

STANDARD OPERATING PROCEDURES  
FOR THE DOE ENVIRONMENTAL SURVEY PROGRAM

- X • DUTIES AND RESPONSIBILITIES OF SAMPLE CUSTODIAN
- X • SAMPLE RECEIVING AND INSPECTION
- X • REQUIREMENTS FOR REPORTING AND CORRECTING LABORATORY ENTRIES
- X • SAMPLE LOGIN AND IDENTIFICATION
- X • SAMPLE STORAGE
- X • SAMPLE SECURITY
- X • SAMPLE CHAIN-OF-CUSTODY
- X • SAMPLE TRACKING
- X • PERSONNEL SIGNATURE AND INITIAL RECORD
- X • MONITORING COLD STORAGE TEMPERATURES
- X • SAMPLE DISPOSAL
- X • MONITORING ANALYTICAL BALANCE PERFORMANCE
- DOCUMENT CONTROL
- ANALYTICAL PROJECT FILE ORGANIZATION
- CASE FILE ASSEMBLY
- DATA MANAGEMENT AND SECURITY
- MONITORING WATER QUALITY
- CLEANING GLASSWARE

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X = DRAFT COMPLETED

Sophie Bobrowski  
Analytical Chemistry Division  
April 11, 1988

Attachment 6

Oak Ridge Environmental Survey Program - Review of the Pantex Site Organic Data  
Generated by the ORNL Analytical Chemistry Division (ACD)

Issued to:

R. B. Fitts

March 23, 1988

Issued by:

*D. W. Frazier*

D. W. Frazier, Review Team Leader

*A. H. Halouma*

A. H. Halouma

*P. L. Howell*

P. L. Howell

*L. W. McMahon*

L. W. McMahon

## Internal Correspondence

MARTIN MARIETTA ENERGY SYSTEMS, INC.

March 23, 1988

R. B. Fitts

DOE Environmental Survey Program - Review of the Pantex Site Organic Data  
Generated by the ORNL Analytical Chemistry Division (ACD)

In January 1988, EPA representatives reviewed the Pantex Site data generated by the ORNL ACD Organic labs. As a result of that audit, the data was declared suspect. A quality assurance review team was chosen at MM-ES to conduct an independent review of the data. On February 23, 24, & 25, 1988, this activity took place to assess the status or usefulness of the data in light of the comments made, and to document an independent evaluation of the participant's compliance to established guidelines as stated in the CLP statement of work.

Selected organic data, generated by ORNL, on environmental samples collected at Pantex as part of the DOE Environmental Survey were reviewed by the team. The following summary will discuss our conclusions based on compliance to requirements of the CLP protocol or from a view of the data being legally defensible versus actual usefulness from a technical point of view. However, prior to stating the conclusions drawn from the review, the team requests that the following issues/comments be recognized and considered.

## 1. Recognize:

- a. That the Organic Lab employees were directed to analyze the sample set from Pantex within the holding times and produce data. The lab received 195 volatile organic analyses (VOA), 203 semivolatile organic (SVO), and 154 PCB/Pesticides to be analyzed by two employees for 75% of the project, (25% of the samples were analyzed by one person) on 4 GC/MS instruments equipped with auto-samplers, two gas chromatographs with auto-samplers (which were not operational 100% of the project) operated by one or two employees;
- b. That these samples came in one delivery;
- c. That laboratory capacity was estimated to be 40 samples per week for the three parameters including sample preparation.

## 2. Recognize:

- a. That long hours and diligent efforts were expended by all concerned to produce the data within the specified holding times.

R. B. Fitts  
Page 2  
March 23, 1988

- b. It was readily apparent that sufficient staff and instrumentation were not available to handle the workload from the Pantex Site.
  - c. Furthermore, it is suspected that sufficient laboratory capacity does not exist in any single DOE laboratory to handle this project given the short holding times associated with the organic samples.
  - d. At the time of the Pantex sample analyses, only 10% of the data was to be reported as full CLP data packages.
3. **Recognize:**
- a. That the ORNL Organic lab, like the other DOE laboratories, was unaccustomed to providing the level of documentation required by CLP.
  - b. There is a definite learning curve which all laboratories, including ORNL, must undergo before producing CLP level data efficiently and in quantity.
4. **Consider:**
- a. The results in light of the CLP statement of work which when adhered to, should produce data that is legally defensible in a court of law.
  - b. That technically, in a broad sense, most of the data is useful for the volatile organics (both soil and water samples).

It is with these issues in mind that the review is summarized below. Specific comments and notes from the review can be supplied upon request.

The VOA data, although not documented to the degree that a third party could recreate the analysis, were retrievable. The level of CLP non-compliances was not unreasonable for the two soil data sets reviewed considering the time frame available for the analyses to be completed. On the other hand, the VOA data reviewed for two water data sets had numerous errors which caused serious concerns. The chief cause of non-compliances appeared to have been a lack of communication or interpretation of CLP requirements, insufficient software to allow timely data interpretation by the analysts, and insufficient time and resources to properly document required information to the level required by the CLP.

1. **Recommendation:** The final report of Pantex VOA data should be regenerated to correctly state quantitative values, positive contaminant identifications, documentation of deviations from the protocol, and documentation of corrective actions taken for out-of-control conditions.

The most serious concerns were with the Pesticide/PCB data. There was an excellent effort to produce the forms electronically, however, the evaluation of the required QC samples was less than adequate. According to the data reviewed, quantitative values

R. B. Fitts  
Page 3  
March 23, 1988

appeared to be reported based on raw electronic data, rather than analyst review and interpretation which is essential.

The linearity evaluation check did not meet CLP requirements on any of the analysis batches. In conclusion, there were enough errors found in the documentation to cause the team to doubt the validity of the results to be reported. Considering that the Gas Chromatograph Electron Capture detector data is more difficult to reconstruct, and that all linearity checks were outside the QC window, it is doubtful that useful data can be regenerated from the raw electronic data, as with the VOA's.

2. **Recommendation:** Future CLP work should incorporate analyst review and interpretation of all data prior to reporting quantitative values, assure that the required QC criteria are met before proceeding with the analysis.

The laboratory evaluation and interpretation of the Semivolatile data had not been completed at the time of the review. There was insufficient data to evaluate the usefulness of the Pantex semivolatiles analysis.

3. **Recommendation:** Due to the length of time since the analysis were performed and the target completion date, the evaluation of this data should be given top priority in order to ultimately generate the necessary CLP forms to complete the data package.

A major concern of the team was the data that SAIC and DEM have in the Pantex data base. None of the data in the SAIC database should be considered as laboratory evaluated and approved. SAIC has provided a useful service which aided the laboratory process raw data, and generate CLP forms. However, it appeared that SAIC and DEM had misinterpreted raw data as final analysis results. The data required processing and laboratory evaluation prior to being put onto the final CLP forms. To reiterate, a considerable amount of data review and evaluation is required on the part of the laboratory before any of the Organic analytical results from the Pantex site can be considered final.

4. **Recommendation:** All of the data in the SAIC data bases should be discarded, and only the final results, validated by laboratory staff should be included in the data. The team understands that the release of the data prior to validation was to aide in the development of the required software. However, there was insufficient resources for the amount of review that this entailed.

R. B. Fitts  
Page 4  
March 23, 1988

Should you have any questions concerning this report please call me.

D. W. Frazier, 1000, MS-335, ORNL (6-0347)

DWF:cet (QA-88-26)

MAR 8 1988

**MARTIN MARIETTA**

Internal Correspondence

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MARTIN MARIETTA ENERGY SYSTEMS, INC.

March 2, 1988

D.W. Frazier

Review of Pantex Data at ORNL 2/23/88 - 2/26/88

Selected Organic data, generated by ORNL, on environmental samples collected at Pantex as part of the DOE Environmental Survey were reviewed by myself as a member of the review team on February 23-24 and 26. The purpose of the review was to assess the usefulness of the data in light of comments made by DOE and EPA during a program review in January.

Before stating conclusions drawn from the review, please allow me to make a few pertinent comments. The long hours and diligent efforts by the analysts and chemists who have worked on the Pantex analyses should be recognized. It is readily apparent that sufficient staff and instrumentation were not available to handle the workload from Pantex. Furthermore, I suspect sufficient laboratory capacity did not exist in any single DOE laboratory to handle this project given the short holding times associated with the organic samples. Compounding this issue is the fact that ORNL, like the other DOE laboratories, was unaccustomed to providing the level of documentation required by CLP. There is a definite learning curve which all laboratories, including ORNL, must undergo before producing CLP level data efficiently and in quantity. It is with these issues in mind that my review is summarized below. Specific comments and notes from the review are included in the attachment.

The VOA data, although not documented to the degree that a third party could recreate the analysis, were retrievable. The level of CLP non-compliances was not unreasonable for the two data sets I reviewed. The chief cause of non-compliances appear to have been lack of communication as to CLP requirements and insufficient software to allow timely data interpretation by the analysts. The final report of this data should be regenerated to correctly state quantitative values and positive contaminate identifications. Considering the samples were relatively "clean", useful information can still be gathered provided the issues noted in the attachment are addressed.

The most serious concerns are with the Pesticide/PCB data. Based on the data presented it appears quantitative values were reported based on raw electronic data rather than analyst review and interpretation. The linearity evaluation check did not meet CLP requirements on any of the analysis batches. Enough errors were found in the documentation to create doubt in the validity of the results reported. Considering that the GC ECD

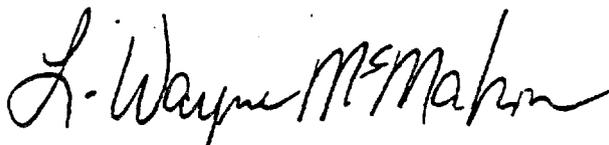
D. W. Frazier  
Page 2  
March 2, 1988

data is more difficult to reconstruct, and that all linearity checks were outside the QC window, it is doubtful that useful data can be regenerated as with the VOA's.

The laboratory evaluation and interpretation of the Semivolatile data had not been completed at the time of the review. Insufficient data exists to evaluate the usefulness of the Pantex semivolatiles.

A major concern is the data SAIC and DEM have in the Pantex data base. No data in the SAIC database should be considered as laboratory evaluated and approved data. SAIC has provided a useful service in aiding the laboratory process raw data. However, it appears SAIC and DEM have misinterpreted raw data, requiring processing, and laboratory evaluation as final analysis results. This is not the case!! A considerable amount of data review and evaluation is required on the part of the laboratory before any of the Organic analytical results from the Pantex site can be considered final.

Please call me if I can provide any other information.



L.W. McMahon, 9704-1, MS-001, Y-12 (4-7535) - NoRC

LWM:da

Attachment: As stated

cc/attach: T. R. Butz/C. C. Hill  
L. L. McCauley/C. W. Kimbrough

VOA Data Reviewed at ORNL 2/23-2/26

VOA Data - Two sets of VOA soil data were reviewed. The sample sets were selected at random from the GC/MS Instrument Operations Logbook. The laboratory personnel stated that the VOA data had been compiled as CLP packages for delivery to EMSL-LV but the laboratory records had been dismantled and the VOA data filed by run day with all like forms combined as a case file of Pantex data. This has resulted in renumbering of the pages as well as duplication of many forms and raw data thus making the data review more difficult.

The lab has prepared Instrument Operation Logbooks which detail the analysis sequence. The logbooks were very useful in defining an analysis batch. The lab staff detailed how the data was compiled for the Pantex data. SAIC has written software to aid in calculations and preparation of the VOA CLP forms. The software provided by SAIC has been most useful in "crunching numbers" but has generated a large amount of "Form I data" which needs to be carefully scrutinized by the laboratory.

The area report tables and quant reports output by the Laboratory Chem Station Data Systems were often included with the raw data along with a second report table "from a Lotus File". The documentation as it exists is often conflicting and leads to many questions. Laboratory staff were needed to explain how certain response factors and quantitative numbers were obtained. The explanation was always provided. The documentation, as it exists, can not be used to reconstruct the analysis without the aid of the individual performing the analysis. Also, there is no indication that the detection limits for soils or quantitative results for soils have been corrected to allow for percent moisture.

I. VOA analyses of 6/7/87, Instrument 0

- Logbook shows sequence of analysis as follows for VOA's requested on Pantex requisition number 91283.

<u>LAB Ident.</u>	<u>Description</u>
BFB Tune	
06707201	50 ppb CCC run
067VW801	Blank 6/7
870607-016	PX012031
-017	PX012019
-018	PX053052
-019	PX053052
-020	PX053041
-021	PX045018
-022	PX045029
-023	PX045030

The last sample of this set (PX045030) was ran outside the twelve window of tune, CCC, and blank requirements. However, the BFB tune file was not altered during the entire Pantex project according to the chemist. The tune and CCC run of the following days run were within spec.

- Form V, BFB tune. The computer generated form V misstates the ion abundance criteria for mass 174 as "> 2% of mass 174". The correct statement should be > 50% of mass 95. The bar graph and mass listing are within requirements and the tune as reported for mass 174 is correct.
- Form VII, Continuing Calibration Check (CCC) - The 50ppb CCC and SPCC requirements were met.
- Lab Blank. Methylene chloride (11.5 ppb) and acetone (10.4 ppb) are reported. This trace level of background is typical for organic laboratories. Only mass spectra of Methylene chloride is given and no standard spectra are included.
- Form II, Surrogates - 25 of 27 surrogates reported with this set are within the QC window.
- Form III, Matrix Spike (MS) and Matrix Spike Duplicate (MSD) - No Matrix spikes were analyzed with this set. The analyst misinterpreted the CLP procedure to require only one set of matrix spikes per twenty samples without regard to matrix type. A water MS and MSD were analyzed with a set of water samples (on instrument G) on this same day. However, this does not meet the requirement of MS and MSD for the soil sample set under review.
- Form IV, Blank data. A water blank, rather than a blank of similar matrix was analyzed. The form correctly reflects the samples associated with this set and that the last analysis was outside the twelve hour window.
- Form VIII, internal standard areas - All internal standard areas were within the QC windows established.
- Form I, results. The laboratory personnel stated that the completed Form I's were still being reviewed to insure flags were properly assigned to the data. It was also reported that the data had already been delivered as a complete CLP package.

A large number of compounds, from several samples, are reported to be present at a level less than the required reporting detection limit (an estimated value) and thus are flagged with a J. Many compounds are reported as "0 J ug/kg". No spectra were included for the majority of compounds

reported as estimated values. It appears that the data on the Form I's represent positive hits of the quantitation ion rather than reported final results based on review of mass spectral data. These positive hits may in fact be due to background or "electronic noise".

The J flag should be used to note the concentration of a tentatively identified compound as estimated or to flag a Target Compound as being present but at a level less than the quantitation limit. In either case, a conclusion that a compound is present in the sample is to be based on mass spectral data that matches standard spectra or that meets the identification criteria based on spectral interpretation. The Form I data reviewed in this set appears to report a positive identification for many compounds, however a review of the raw data indicates few positive identifications based on mass spectral data. Only one sample appears to have a target compound significantly above the quantitation limit. Toluene is reported at 58 ppb in sample PX045029. Raw and background subtracted spectra are included which identify toluene as being present but the CLP required standard reference spectra is missing.

Mass Spectral data for this set should be reviewed to determine presence of Target Compounds. The Form I results should be regenerated to reflect actual reportable results. On regenerating the results the % moisture determination is to be used to calculate actual detection limits and quantifiable results. Lab personnel stated that no results had been corrected for moisture at the time of this review.

- Form VI, Calibration data. The last calibration date was 6/2. The response factors were reviewed and the calculations spot checked. The calibration data were acceptable.

## II. VOA Analyses of 6/12/87, Instrument 0

- Sample ID's and order of analysis taken from GC/MS Instrument Operations Logbook

<u>Lab Ident.</u>	<u>Description</u>
BFB Tune	
50 ppb	50 ppb CCC run
870611-226	PX020019
870611-227	PX020020
870611-228	PX020031
870611-229	PX020042
870611-230	PX020053
870611-231	PX020064
870611-231	PX020064 MS
870611-231	PX020064 MSD

The last run was again outside the twelve hour window of BFB tune, CCC, and instrument blank requirements.

- Form V, BFB Tune. Ion Abundance Criteria statement for mass 174 is incorrect as noted previously. All mass % relative abundances on the computer generated form are " 0 ". The zeros have been stricken and hand entry of data recorded without any notations. Bar graph and mass listing met requirements.
- Form VII, Continuing Calibration Check. SPCC and CCC requirements were met. Two area report tables, with different areas are included with the documentation. Input from lab personnel was needed to determine which areas were used to determine the response factors.
- Lab Blank. The Form I report for the lab blank reports Methylene Chloride, Acetone, and 2-butanone at 5 ppb or above. Many compounds are reported to be present at less than 1ppb (0J). The only spectra documenting the presence of any compounds was for methylene chloride and the standard reference spectra was missing for it.
- Form II, Surrogate recovery. 26 of 27 surrogate recoveries were within QC window.
- Form III, Matrix Spike results. 9 of 10 Matrix spike recoveries were within the QC window while the relative percent difference between duplicates was in the QC window for all 5 matrix spike compounds. However the Form III was not properly completed to report these results.

A report of MS and MSD data, generated by SAIC, was reviewed ( Summary of Pantex Volatiles, Run = 0612). This output has MS and MSD % recoveries which differ from the Quant reports in the lab.

- Form IV, Blank Data. Time of analysis reported for last sample run shows the run to be outside the twelve hour window. A water blank was utilized.
- Form VIII, Internal Standard Area - The sample identifications on the form do not differentiate the MS and MSD runs from the sample run. 24 of 27 internal areas met the QC window. The three outside the window represent all three standards from the final run of the day (PX020064 MSD). The peak areas from this run differ by a factor of approximately 50 from the other runs in this set.
- Form I, Results. In general many positive results are reported as estimated values (flagged with J) but the raw data does not substantiate these results. As with the set of data previously discussed, the Form I's need considerable rework to reflect the chemist interpretation on the data.

In addition, all detection limits and results should be corrected for moisture content.

Examples of problems are noted:

- \* PX020019 - acetone and MEK results should be flagged with a B. Only spectra included is that of methylene chloride. Three copies of Form I are included: Two appear to be duplicates, a third reports different results.
- \* PX020020 - Many positive hits reported as estimated values, no spectra to support identifications.
- \* PX020042 - Methylene chloride and acetone are correctly flagged with B's, MEK is not. Duplicate pages in the package complicate the review process.
- \* PX020064 MSD - No Form I included, only TIC and quant report. The total-ion-chromatogram for this sample indicates very low response of internal standards and surrogates. The pattern of the TIC indicates that perhaps the purge and trap device malfunctioned on this run. This is also likely to be the cause for the three internal standards from this run to be outside the QC window.

For these reasons and for those cited on the first set, the mass spectral data should be reviewed to determine presence and absence of target compounds and Form I data regenerated to reflect data review by the laboratory.

- Form VI, Calibration data - The same calibration file (6/2/87) was used for this set.

#### Summary of Pantex PCB/Pesticide Data Reviewd at ORNL 2/23 -2/26

It was readily apparent that considerable time and effort had gone into the development of software to "crunch the numbers" and generate the CLP Pesticide/PCB forms. However, a review of the data also reveals that the software is still in a development stage. While the GC/MS data readily lends itself to computerization, the day-to-day GC data evaluation is based more on operator experience and day-to-day interpretation of chromatographic patterns. Decisions must be made daily, often hourly on various operating conditions that may influence the results (background, sample matrix, and late eluting peaks that interfere with the next run for example). Programming these decisions into computer software is complex at best and lab personnel should be commended for progress to date.

*date.*

to the Pantex data a number of concerns must be expressed. The most pressing concern is that "electronic data" (i.e. raw, unevaluated data) has been accepted by SAIC prior to laboratory evaluation. In addition, the bulk of the documentation appears to report analysis results based solely on electronic processing rather than operator evaluation.

More difficulty was experienced in determining a sample "batch" for the review. The chemist was uncertain if the samples had been analyzed in such a manner as to relate a blank, Matrix spike (MS) and Matrix Spike Duplicate (MSD) with a given set of samples. A review of the Analytical Services Form, Sample Preparation Logsheet, and GC Instrument Operations Logsheet revealed the following samples from Pantex Request # 91339 as a "batch".

<u>Laboratory Indent.</u>	<u>Description</u>
870615-213	PX052017
870615-214	PX052028
870615-215	PX052039
870615-216	PX052040 *
870615-217	PX052051
870615-218	PX052062
870615-219	PX052073
870615-220	PX052084
870615-221	PX052095
870615-222	PX052108
870615-223	PX052119
870615-224	PX052120
870615-225	PX052131
PX91339SB	Blank

\* Prepared as unspiked, matrix spike and matrix spike duplicate

The three forms were needed to relate this as a batch since;

- Only Pantex sample identifications were used on the GC log
- Only Lab sample identifications were used on the Sample Prep Log
- Only the Service request form relates both lab and Pantex Identifications
- The GC log omits the first numerical digit of the Pantex sample identification due to field size allowed by the computer program.

This set of samples were received on 6/15, extracted on 6/26 and analyzed on 9/15 thru 9/17 ( 1 day beyond extraction holding time, and 52 days beyond analytical holding time).

- Form II, Surrogate Recovery - Mirex was used as the surrogate rather than Dibutylchlorodate (DBC). Assuming the QC advisory guidelines for DBC can be extended to mirex, 9 of 16 surrogates are outside the advisory

window. Since Mirex was used as the surrogate rather than DBC, the number of non-compliances can not be evaluated.

- Form III, Matrix Spike (MS) and Matrix Spike Duplicate (MSD) - The form reviewed had the proper header (Soil Pesticide Matrix Spike) but the QC limits stated on the form were those for water. The comments on the form state that the samples were prepared incorrectly with no further explanation of what was done incorrectly. 12 of 12 MS recoveries were outside QC limit while the form data reports 1 of 6 % RPD out. In fact 5 of 6 RPD were out with only dieldrin reproducing with 0% recovery. The chemist stated that the computer was not programmed (at the time of the Pantex project) to report negative % RPD as out-of-control since the CLP procedure did not specify negative values as out-of-control. In fact the absolute value should be considered and it was implied that the computer program had been so modified.

Sample PX052040 and been analyzed unspiked and as MS and MSD. The matrix spike compounds were gamma-BHC, heptachlor, aldrin, dieldrin, endrin, and 4,4'-DDT. The analysis results of this sample (Form I data, unspiked), matrix spike, and matrix spike duplicate are noted below. Also included are the sample results as reported in ANALIS.

<u>Compound Reported</u>	<u>Packed Column Form I</u>	<u>ANALIS</u>	<u>Form I MS</u>	<u>Form I MSD</u>
alpha-BHC			83.59	19.07
beta-BHC	8.07			
Endosulfan I	16.03	8.00	29.47	13.34
4,4'-DDT		16.00		19.32
aldrin			20.16	

Besides the fact that poor recoveries were obtained on the spiked samples, the presence or absence of other contaminants in the sample are questionable based on the various results reported above.

- Form IV, Blank data. Samples associated with this set are noted. The Form I report for the blank (PX91339SB) shows 16 ug/kg heptachlor. The quant report for this blank (part of the raw data) reports 19.14 ug/Kg beta-BHC and 30.00 ug/Kg Heptachlor. Data from other blanks (PX91306SB, PX91306SB, PX91275WB) analyzed as part of the Pantex project were reviewed. It was noted that aldrin, heptachlor epoxide, endosulfan II and heptachlor were reported at levels of 12.44 to 53.67 ug/kg.

- Form VIII, Evaluation Standards Summary. The percent relative standard deviation (%RSD) of calibration factors for aldrin, endrin, DBC, and DDT is not to exceed 10% on the quantitation (packed) column.

The procedure makes an exception to this rule for DDT. This linearity check for each 72 hour run sequence for the Pantex project was reviewed and is summarized below.

Date of analyses	Number compounds exceeding 10% RSD	Smallest % RSD Reported for outliers
7/30 - 8/2	3 of 4	18
8/6 - 8/12	3 of 4	15
9/10 - 9/13	3 of 4	13
9/14 - 9/15	2 of 4	10
9/28 - 10/1	4 of 4	15
10/1 - 10/2	4 of 4	38
10/14 - 10/21	4 of 4	38
10/15 - 10/17	4 of 4	30

Based on EPA data evaluation criteria, all quantitative results would be questioned.

Summary of Pantex Semivolatile Data Reviewed at ORNL 2/23-2/26

SAIC has worked with lab personnel to develop software to generate the CLP documentation for the Semivolatiles as they did for the Volatiles. Although considerable work has been completed, data processing for the semivolatiles has not been completed to the extent of the Volatiles. It was explained that as semivolatiles are analyzed by GC/MS, data files containing peak number for identification purposes, retention time, quantitation mass, and peak area of the quantitative ion are uploaded to SAIC for processing. The laboratory received back from SAIC not analyses batches but the entire set of Pantex data. Corrections were made to the output from SAIC and returned. The next submission contained data which had been corrected for dilution factors. A third submission was in the laboratory for evaluation at the time of the review.

While the SAIC work has been helpful to the laboratory, it has not provided the timely processing of data needed by lab personnel to effectively evaluate the data. The Semivolatile data for Pantex is at best in the very early stage of evaluation by the laboratory.

A review of data to date included Pantex samples from requestion 91332. The samples were extracted on 6/24/87 and analyzed on 11/2/87, beyond the analytical holding time. Data for a second set of samples, analyzed on 8/10 were also reviewed. The amount of data available at the time of the review is insufficient to make an evaluation of its acceptability for the DOE Survey Program. A few comments are noted on the available data below.

- The two instrument tunes for DFTPP reviewed met the tune criteria.
- The instrument calibration of 11/1 had only the response factors for the SPCC and CCC compounds calculated. This is the minimum information needed to determine if samples can be run. The lab is dependent on the SAIC software to calculate all response factors.
- On the CCC run of 8/10 the percent difference in RF from the calibration run for hexachlorobutadiene exceeded the 25% requirement (31.39%). All other CCC and SPCC compounds (16 of 17) were within established QC window.
- No blank, MS, or MSD data were located for the set analyzed on 11/2/87.
- Surrogate recoveries had not been determined for the majority of analyses. An SAIC report of analysis results on sample 870615-132 (PX015023) dated 2/23/88 was reviewed. The report included results with and without correction for the dilution factor. The dilution factor was recorded as 35. Assuming the surrogate spike levels were as designated in the CLP, the recoveries were calculated as shown below.

<u>Surrogate Compound</u>	<u>Assumed Spike Level</u>	<u>% Recovery at at DF of 1</u>	<u>% Recovery at at DF of 35</u>
Nitrobenzene-d5	50 ug/L	12	218
2-Fluorobiphenyl	50	15.2	272
p-terphenyl-d14	50	17.6	311
Phenol-d6	100	16.7	589
2-Fluorophenol	100	11.6	408
2,4,6-TBP	100	33.6	1180

Phenol-d6 and 2,4,6-TBP are within the QC window assuming the dilution factor was 1 and not 35. However, an assessment of surrogate recoveries would premature at this stage since the laboratory is still processing the data.

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**Draft-Do Not Cite  
ANL Data Document  
Issue Date: June 1989  
Revision: 01**

**ORNL Results of Inorganic and Organic Performance Evaluation Studies**

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PERFORMANCE EVALUATION SCORES FOR ORNL

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Date Received	Code	Score
07/19/88	QB4FY88 Inorganic	89.5 (CAR)
04/20/88	QB3FY88 Inorganic	96.3
01/22/88	QB2FY88 Inorganic	94.1
10/22/87	QB1FY88 Inorganic	86.5 (CAR)
08/11/87	QB4FY87 Inorganic	96.0
04/28/88	QB3FY88 Organic	78.7 (CAR)
01/25/88	QB2FY88 Organic	62.3 (CAR)
08/17/87	WP-019 Nontarget inorganic	Acceptable
02/24/88	WP-020 Nontarget inorganic	Acceptable
08/31/88	WP-021 Nontarget inorganic	Acceptable
10/23/87	QB1FY88 Organic	*
08/13/87	QB4FY87 Organic	*

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\* Did not report samples for scoring (see attached letter).

CAR = Corrective Action Required

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Internal Correspondence

MARTIN MARIETTA ENERGY SYSTEMS, INC.

September 15, 1988

R. B. Fitts

Quarterly Blind (QB) Samples for Organic Analysis

This memo is my response to your request for information about the QB samples that EPA sent to the Organic Services Group in support of the Environmental Survey Program. We received eight samples:

<u>No.</u>	<u>QB Number</u>	<u>Period</u>	<u>Received</u>	<u>Reported</u>
1	QB6437	Qtr 1, FY87	10/16/86	12/31/86
2	QB6666	Qtr 2, FY87	1/23/87	4/10/87
3	QB7144	Qtr 3, FY87	4/28/87	No
4	QB7760	Qtr 4, FY87	8/13/87	No
5	QB8124	Qtr 1, FY88	10/23/88	No
6	QB8783	Qtr 2, FY88	1/25/88	3/31/88
7	QB9300	Qtr 3, FY88	4/28/88	6/1/88
8	QB10015	Qtr 4, FY88	7/28/88	In Process

As you can see, results from three consecutive samples were not reported to EPA. We did not complete the data packages for these three samples because of excessive workloads of higher priority at the time. I need to elaborate on this on a sample-by-sample basis in order to clearly show the conditions that existed at the time.

The analytical work was done on QB7144, but the data package was not completed. Samples from the Pantex site took precedence over QB7144. The Pantex sample consignment arrived over a 10-day period starting June 6, 1987. (For two days during this 10-day period we were audited by DOE/EPA including the DOE Manager.) At that time the available staff consisted of one secretary and myself to log in, distribute and report; three sample preparation technicians, (including two technicians borrowed when the samples arrived); two staff members in the GC/MC Laboratory; two persons in the gas chromatography laboratory; and one staff member along with his Group Leader to determine high explosives, soil gas, etc. Because this sample load far exceeded our capacity, we did not have time to finish assembling the data package for QB 7144. We simply could not get all pending work completed even when maximum amount of overtime was worked by all available staff. Preparation of water samples was performed by the two staff members assigned to the gas chromatography laboratory while the personnel assigned to the preparation laboratory devoted all efforts exclusively to soil preparation. The two-person GC/MS staff worked very long hours to complete volatiles analysis within holding times. Our main objective was to maximize the number of holding

times satisfied for (1) volatiles analysis and (2) pesticide and semivolatiles preparation. The great majority of these holding times were satisfied. In short, because our priorities were to analyze local samples and Survey samples before directing attention to the QB Sample, we did not have time to devote to the complete of the data package for QB7144. (Incidentally, at that time all CLP data packages coming from our laboratory had to be developed manually. All results were manually input and forms were handwritten. Thus QB7144 was never submitted to the EPA.)

Sample consignments from Lawrence Livermore and Sandia arrived in our organic analysis laboratory during the period of August 8 to August 17, 1987. QB7760 arrived on August 13, 1987. Work still remained to be done on the analysis of semivolatiles and pesticides from Pantex. At this time our preparation capacity was slightly greater, (three technicians in the preparation laboratory prepared water samples and satellite laboratory from another Section in the Division prepared all soil samples). However, the two-person gas chromatography staff was still working on the analysis of Pantex pesticide samples as well as samples received locally. The real limitation at this time was the GC/MS staff where the most knowledgeable person was not available because of a traffic accident. An able technician was borrowed to bring the staff level to two. However, the borrowed technician was completely unfamiliar with this laboratory operation and arrived during a period of intense activity. Thus the contribution of the second GC/MS staff member was not optimal. Problems for the GC/MS effort were compounded by the fact that the Lawrence Livermore/Sandia sample load contained nearly three hundred volatile organic samples. (Our capacity for volatiles at that time was 30-60 volatile organic samples per month.) Our priority was to analyze local and Survey samples before expending the significant amount of time required to manually complete and assemble a CLP data package for QB7760. Thus, we never began to assemble QB7760.

QB8124 arrived on October 23, 1987. At that time we had analyzed only 15 of the semivolatiles prepared from the Lawrence Livermore/Sandia samples. The decision was made to send approximately three-fourths of the Lawrence Livermore/Sandia semivolatile sample preparation to other laboratories so that we could get ready for Survey samples from Argonne and adhere to all holding times. Thus QB8124 was prepared and analyzed, but the manual data treatment and package preparation could not be completed before the Survey samples from Argonne arrived (November 11, 1987 to November 23, 1987). Even with a larger staff, [2 persons in receiving/distribution/reporting; 3 persons in GC/MS; 3 persons in sample preparation plus the satellite laboratory for soils sample preparation; and 3 persons in the gas chromatography (pesticide/PCB) laboratory], much overtime was required to service local samples and complete the analysis of the Argonne Survey samples in the permitted time frame. The data gathering phase of the analysis of the Argonne samples was completed in late December 1987. At that time, much of our efforts had to be directed toward an audit by DOE/EPA which was scheduled for January 14-15, 1988. Since the next quarter QB was scheduled to arrive shortly (in January),

Sept. 15, 1988

we decided not to complete the data package for QB8124, but rather to put our effort on the next (second quarter) QB sample.

QB8783 was received on January 25, 1988. It was submitted to the EPA on March 31, 1988.

QB9300 was received on April 28, 1988, and the results were submitted to the EPA on June 1, 1988. This was the first QB sample for which a great majority of the forms were processed electronically. During April and May 1988 the GC/MS staff (now consisting of four persons) received training in data processing from data system experts provided by the vendor that had supplied the data system. Since that time, data processing for complete packages (CLP) has progressed rapidly.

QB10015 was received on July 28, 1988, (during another audit/data review). Data packages for volatiles and semivolatiles have been assembled and are in the review process.

This memo is only an abbreviated history of our handling of the several QB samples that we have received. If I can provide further information, please let me know.

*John Caton*

J. E. Caton, 4500S, MS-6120 (4-4861)

JEC:db

cc: M. R. Guerin  
P. L. Howell  
W. R. Laing  
W. D. Shultz

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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
OFFICE OF RESEARCH AND DEVELOPMENT  
ENVIRONMENTAL MONITORING SYSTEMS LABORATORY-LAS VEGAS  
P.O. BOX 93478  
LAS VEGAS, NEVADA 89193-3478  
(702/798-2100 - FTS 848-2100)

OCT 24 1988

Mr. William R. Laing  
Oak Ridge National Laboratory  
P. O. Box 2008, 45005 MS-127  
Oak Ridge, TN 37831

Dear Mr. Laing:

The results of the participation of your laboratory in the EMSL-LV fourth quarter inorganic performance evaluation study (QB4, FY88, INORGANIC) are enclosed. This includes copies of the analysis reports for inorganics in soil and water samples. The reports also present statistical information on the numbers of laboratories that had difficulties with specific analytes.

The score for your laboratory was 89.5. The DOE environmental survey requires a formal response from each laboratory, describing any changes or actions taken to identify and correct any deficiencies and to improve laboratory performance. That response will become part of the quality assurance record for analytical work done by your laboratory for sites in the DOE environmental survey. In order to meet schedule times for data document publication, corrective action responses should be sent within 15 days of receipt of this letter.

This office will be glad to furnish any counsel and further information regarding this work.

Sincerely,

A handwritten signature in cursive script that reads "Harold A. Vincent".

Harold A. Vincent

Chemist, Quality Assurance Research Branch  
Quality Assurance and Methods Development Division

Enclosures

cc:  
Vincent Fayne, DOE HQ  
Alan Crockett, INEL

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**INORGANIC PERFORMANCE EVALUATION SAMPLE  
INDIVIDUAL LABORATORY SUMMARY REPORT  
FOR GS 4 FY 88**

LABORATORY NAME: Oak Ridge National (TN) (E2)  
 PERFORMANCE LEVEL: ACCEPTABLE - Corrective Action Necessary  
 LABORATORY RANK: Above = 29 Same = 0 Below = 17

I Score: 89.5  
 REPORT DATE: 9/26/1988  
 MATRIX: WATER

ELEMENT NAME	95 % CI		LAB RESULTS		GLAB NOT-ID	GLAB NIS-QMANT	PROGRAM DATA			TOTAL GLAB
	LOWER	UPPER	REPORTED VALUE	QUALIFIER CODE			GLAB FALSE POS	GLAB NEPK OUT	GLAB DUP OUT	
ALUMINUM	725	930	839		0	6	0	0	0	38
ANTIMONY	66.8	96	76		0	3	0	4	0	38
ARSENIC	26	39	31.1		0	1	0	2	1	38
BARIUM	2790	3260	3070		0	2	0	0	0	38
BERYLLIUM	30	40	36		0	0	0	0	0	38
CADMIUM	6.9	13	9.8		0	4	0	1	0	38
CALCIUM	5190	6370	5790		0	2	0	0	0	38
CHROMIUM	31	49	45		0	4	0	0	0	38
COBALT	72	96	82		0	1	0	0	0	38
COPPER	60	100	83		0	2	0	0	0	38
IRON	1600	1890	1690		0	2	0	2	0	38
LEAD	54	77	48.8	X	0	0	0	2	2	38
MAGNESIUM	7040	9040	8400		0	1	0	0	0	38
MANGANESE	46	57	54		0	2	0	0	0	38
MERCURY	6.3	10	8.7		0	3	0	0	0	38
NICKEL	113	163	137		0	3	0	0	1	38
POTASSIUM	8300	10700	9150		0	2	0	0	0	38
SELENIUM	11	19	15.3		0	0	0	3	1	38
SILVER	18.8	15	8.3	B	15	1	0	2	1	38
SODIUM	17100	22100	20300		0	3	0	0	0	38
THALLIUM	29	50	35.6		1	4	0	5	3	38
VANADIUM	34	60	72	X	0	6	0	0	0	38
ZINC	30	50	70	X	0	6	0	0	1	38

0 OF ELEMENTS NOT-IDENTIFIED: 0  
 0 OF ELEMENTS NIS-QMANTIFIED: 3  
 0 OF FALSE POSITIVES: 0

0 OF MATRIX SPIKES OUT: 0  
 WATER :

0 OF DUPLICATES OUT: 0  
 WATER :

**INORGANIC PERFORMANCE EVALUATION SAMPLE  
INDIVIDUAL LABORATORY SUMMARY REPORT  
FOR GS 4 FY 88**

LABORATORY NAME: Oak Ridge National (TD) (R2)  
 PERFORMANCE LEVEL: ACCEPTABLE - Corrective Actions Necessary  
 LABORATORY RANK: Above = 20 Same = 0 Below = 17

I Score: 89.5  
 REPORT DATE: 9/26/1988  
 MATRIX: SOIL

ELEMENT NAME	95 % CI		LAB RESULTS		#LABS NOT-ID	#LABS HIS-QUANT	PROGRAM DATA			TOTAL #LABS
	LOWER	UPPER	REPORTED VALUE	QUALIFIER CODE			#LABS FALSE POS	#LABS SPIKE OUT	#LABS DUP OUT	
ALUMINUM	4630	17500	12000		0	1	0	0	1	38
ANTIMONY	12.0	50	21		3	2	0	27	0	38
ARSENIC	242	378	318		0	6	0	2	2	38
BARIUM	94	146	119		0	3	0	1	0	38
BERYLLIUM	4.4	7.7	7.1	E	1	2	0	2	0	38
CADMIUM	13	20	15	E	0	7	0	2	0	38
CALCIUM	49000	61300	54000		0	4	0	0	0	38
CELESTIUM	42	62	49	E	0	2	0	1	0	38
COBALT	35	50	47		0	4	0	1	0	38
COPPER	1710	2100	1900		0	4	0	0	0	38
IRON	13500	26000	20500		0	4	0	0	0	38
LEAD	302	412	336		0	5	0	2	1	38
MAGNESIUM	29900	37900	35100		0	2	0	0	0	38
MANGANESE	4310	5660	5260		0	4	0	1	0	38
MERCURY	1.9	4.4	3.9		0	2	0	0	1	38
NICKEL	20	50	36		0	2	0	1	0	38
POTASSIUM	1000.8	1440	1026		0	5	0	0	0	38
SELENIUM	4.8	16	11.5		1	3	0	4	2	38
SILVER	3.8	10	8.2		0	4	0	5	2	38
SODIUM	6	6	290	E	0	0	1	0	0	38
THALLIUM	6.5	10	10		1	3	0	6	0	38
VANADIUM	24	50	41	E	0	2	0	1	0	38
ZINC	296	330	268		0	3	0	5	0	38

# OF ELEMENTS NOT-IDENTIFIED: 0  
 # OF ELEMENTS HIS-QUANTIFIED: 0  
 # OF FALSE POSITIVES: 0

# OF MATRIX SPIKES OUT: 2  
 SOIL : Ed, Ag

# OF DUPLICATES OUT: 0  
 SOIL :

OAK RIDGE NATIONAL LABORATORY

OPERATED BY MARTIN MARIETTA ENERGY SYSTEMS, INC.

POST OFFICE BOX 2008  
OAK RIDGE, TENNESSEE 37831

November 2, 1988

Vincent Fayne  
USDOE  
Forrestal Bldg, EH-24  
Independence Ave., SW  
Washington, DC 20585

Harold Vincent  
EMSL-LV  
P. O. Box 93478  
Las Vegas, NV 89193-3478

Gentlemen:

Oak Ridge National Laboratory participated in the EMSL-LV fourth quarter inorganic performance evaluation study (QB4, FY88, INORGANIC) receiving a score of 89.5. It is assumed, no detailed score sheet was received, that points were deducted for mis-quantification of lead (GFAAS), vanadium (ICP), and zinc (ICP) in the WATER sample. Additional points were deducted for matrix spike noncompliance results for antimony (ICP) and silver (ICP) in the SOIL sample.

Poor spike recovery for antimony in soil digestions continues to be a problem. As mentioned in previous response letters, the digestion technique is being evaluated. No progress has been made in correcting the problem as of this date. Recoveries for silver in soil digestions have never been a problem in the past, and no clear reason for the QB4 noncompliance has been found. Silver analyses will be monitored carefully during future DOE Site Survey work.

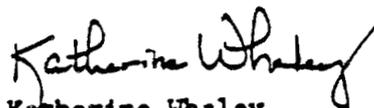
Vanadium on the JY48 suffers from adjacent channel interference from the strong emitter magnesium which cannot be accommodated using software driven interelement correction. Manual correction is required. A service call is expected shortly and this situation will be evaluated again.

It is believed that the poor zinc performance is a result of contamination during digestion, as the calibration verification and 2XCRDL standard results were in compliance. Greater effort will be made to ensure that digestion vessels and glass pipets are contamination free before use and that handling during digestion does not result in contamination.

All quality control parameters for lead analysis in the WATER sample were in compliance throughout the run. The sample was diluted to bring the observed result within the calibration range of the instrument and it is felt that the error stems from improper pipeting. Greater care will be taken in the future to ensure that pipets are calibrated and functioning properly.

Please call if you have any questions.

Sincerely,



Katherine Whaley  
ICP Spectroscopist



William Laing  
Program Manager

cc: R. B. Fitts

Bcc: Whaley  
Ferguson  
Halladay  
Hawell  
Boisrouski



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
OFFICE OF RESEARCH AND DEVELOPMENT  
ENVIRONMENTAL MONITORING SYSTEMS LABORATORY-LAS VEGAS  
P.O. BOX 93478  
LAS VEGAS, NEVADA 89193-3478  
(702/798-2100 - FTS 545-2100)

JUL 15 1988

Mr. William R. Laing  
Oak Ridge National Laboratory  
P. O. Box 2008, 45005 MS-127  
Oak Ridge, TN 37831

Dear Mr. Laing:

The results of the participation of your laboratory in the EMSL-LV third quarter inorganic performance evaluation study (QB3, FY88, Case Number 9302) are enclosed. This includes copies of the analysis reports for inorganics in soil and water samples. The reports also present statistical information on the numbers of laboratories having difficulties with specific analytes.

The score for your laboratory is higher than 90 so that no formal response is required describing any changes or corrective actions taken to improve the performance evaluation score. However, it is still prudent for your laboratory to examine all factors affecting the scoring and take any actions which would improve those scores.

This office will be glad to furnish any council and further information regarding this work.

Sincerely,

A handwritten signature in cursive script that reads "Harold A. Vincent".

Harold A. Vincent,  
Chemist, Quality Assurance Research Branch  
Quality Assurance and Methods Development Division

Enclosures

cc: (w/enclosure)  
D. K. Knight, DOE HQ

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INORGANIC PERFORMANCE EVALUATION SAMPLE  
 INDIVIDUAL LABORATORY SUMMARY REPORT  
 FOR Q3 3 FY 88

LABORATORY NAME: Oak Ridge National (TN) (C3)  
 PERFORMANCE LEVEL: ACCEPTABLE  
 LABORATORY RANK: Above = 6 Same = 1 Below = 30

% Score: 96.3  
 REPORT DATE: 6/15/1988  
 MATRIX: WATER

ELEMENT NAME	95 % CI		LAB RESULTS		PROGRAM DATA					TOTAL #LABS
	LOWER	UPPER	REPORTED VALUE	QUALIFIER CODE	#LABS NOT-ID	#LABS MIS-QUANT	#LABS FALSE POS	#LABS MSPK OUT	#LABS DUP OUT	
ALUMINUM	1790	2190	1960		0	3	0	0	0	38
ANTIMONY	86	156	115		2	3	0	3	0	38
ARSENIC	40	58	48.6		0	1	0	5	3	38
BARIUM	265	331	314		0	3	0	1	0	38
BERYLLIUM	5.0	6.7	5.9		2	1	0	0	0	38
CADMIUM	65	82	79		0	2	0	1	0	38
CALCIUM	8970	11000	10400		0	3	0	0	0	38
CHROMIUM	90	117	111		0	2	0	0	0	38
COBALT	61	87	78		0	1	0	0	0	38
COPPER	126	170	154		0	3	0	1	0	38
IRON	492	621	568		0	1	0	0	1	38
LEAD	5.0	7.5	5.2		3	0	0	4	2	38
MAGNESIUM	5740	6770	6940	X	0	4	0	0	0	38
MANGANESE	35	50	46		0	2	0	0	0	38
MERCURY	2.8	5.2	4.3		0	0	0	4	1	38
NICKEL	48	85	70		0	4	0	1	0	38
POTASSIUM	6700	8220	7800		0	4	0	0	0	38
SELENIUM	39	62	54.6		0	1	0	0	2	38
SILVER	10.0	15	11		13	2	0	4	3	38
SODIUM	8970	10900	10700		0	4	0	0	0	38
THALLIUM	17	31	21.4		1	4	0	7	0	38
VANADIUM	64	93	87		0	1	0	0	0	38
ZINC	124	178	166		0	2	0	0	0	38

# OF ELEMENTS NOT-IDENTIFIED: 0  
 # OF ELEMENTS MIS-QUANTIFIED: 1  
 # OF FALSE POSITIVES: 0

# OF MATRIX SPIKES OUT: 0  
 WATER :

# OF DUPLICATES OUT: 0  
 WATER :

INORGANIC PERFORMANCE EVALUATION SAMPLE  
 INDIVIDUAL LABORATORY SUMMARY REPORT  
 FOR QB 3 FY 88

LABORATORY NAME: Oak Ridge National (TN) (CJ)  
 PERFORMANCE LEVEL: ACCEPTABLE  
 LABORATORY RANK: Above = 6 Same = 1 Below = 30

% Score: 96.3  
 REPORT DATE: 6/15/1988  
 MATRIX: SOIL

ELEMENT NAME	95 % CI		LAB RESULTS		#LABS NOT-ID	#LABS NIS-QUANT	PROGRAM DATA			TOT #L
	LOWER	UPPER	REPORTED VALUE	QUALIFIER CODE			#LABS FALSE POS	#LABS MSPK OUT	#LABS DUP OUT	
ALUMINUM	8310	16200	13000		0	3	0	0	0	
ANTIMONY	c	c	10	U	0	0	0	27	1	
ARSENIC	2.0	2.3	1.4	B	7	7	0	4	2	
BARIUM	40.0	57	50		0	0	0	3	0	
BERYLLIUM	c	c	0.40	B	0	0	1	1	0	
CADMIUM	c	c	0.90		0	0	1	0	1	
CALCIUM	1000.0	4150	2570		0	0	0	0	0	
CHROMIUM	13	34	23		0	1	0	2	0	
COBALT	d	d	6.4		0	0	0	0	0	
COPPER	8.9	22	15		0	1	0	1	0	
IRON	8720	19000	14300		0	1	0	0	0	
LEAD	3.2	7.1	4.8		1	3	0	0	5	3
MAGNESIUM	3340	5550	4520		0	3	0	0	0	
MANGANESE	171	282	237		0	3	0	3	1	
MERCURY	c	c	0.04	B	0	0	2	2	2	
NICKEL	24	45	35		0	2	0	1	0	3
POTASSIUM	d	d	355	B	0	0	1	0	0	
SELENIUM	c	e	0.25	U	0	0	0	12	0	
SILVER	c	e	1	U	0	0	1	9	1	3
SODIUM	d	d	163	B	0	0	0	0	0	
THALLIUM	c	c	0.14	U	0	0	1	3	1	
VANADIUM	17	53	38	B	0	3	0	0	0	
ZINC	31	59	49		0	0	0	1	3	3

# OF ELEMENTS NOT-IDENTIFIED: 0  
 # OF ELEMENTS NIS-QUANTIFIED: 0  
 # OF FALSE POSITIVES: 0

# OF MATRIX SPIKES OUT: 1  
 SOIL : Sb

# OF DUPLICATES OUT: 0  
 SOIL :

September 21, 1988

Randal Scott  
Sampling & Analysis Program Manager  
Office of Environmental Audit and Compliance  
US Dept. of Energy  
Forrestal Bldg.  
1000 Independence Ave.  
Washington, DC 20585

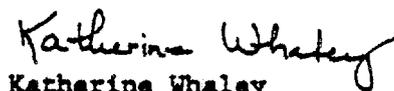
Dear Randal:

The score received by Oak Ridge National Laboratory, X-10, for the QB3-FY88 inorganic performance evaluation study was 96.3 percent. Points were deducted for mis-quantification of magnesium in the water sample and for nonconformance antimony spike results in the soil sample.

Associated calibration verification data for both elements were in control throughout analysis. Analysis results for re-digested QB2-FY88 water sample were within the control limits for magnesium. Assuming no instrument glitch at time of analysis, the problem would seem to be contamination at either/or both the preparation and/or analysis stages. We will more carefully clean our glassware and work spaces in the future.

In the case of antimony, the spike recovery for the water sample was in control. Historically we have had problems with loss of antimony during soil digestions involving the CLP procedure. Efforts are ongoing to ascertain at what point in the digestion the loss occurs.

Sincerely,

  
Katherine Whaley  
ICP Spectroscopist

  
W. R. Laing  
DOE Site Survey Program Manager  
Analytical Chemistry Division

KSW:WRL:lp

cc: Harold Vincent

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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
 OFFICE OF RESEARCH AND DEVELOPMENT  
 ENVIRONMENTAL MONITORING SYSTEMS LABORATORY-LAS VEGAS  
 P.O. BOX 93478  
 LAS VEGAS, NEVADA 89193-3478  
 (702/798-2100 - FTS 545-2100)

APR 12 1988

Mr. W. R. Laing  
 Oak Ridge National Laboratory  
 Building 4500 S. MS-131  
 Oak Ridge, TN 37831-6107

Dear Mr. Laing: *second*

The results of the participation of your laboratory in the EMSL-LV ~~first~~ quarter inorganic performance evaluation study (QB2, FY88, Case Number 8782) are enclosed. This includes copies of the analysis reports for inorganics in soil and water samples and a comparison table showing the distribution of scores of all laboratories participating. The number of misses for each element is also listed.

This office will be glad to furnish any council and further information regarding this work.

Sincerely,

*Harold A. Vincent*

Harold A. Vincent,  
 Chemist, Quality Assurance Research Branch  
 Quality Assurance and Methods Development Division

Enclosures

APR 20 1988

cc: Pamela Howell *Price*  
*Bobrowski*  
 cc: (w/o encl) *Whaley*  
 D. K. Knight, *Hordley*  
*Ferguson*  
*Hamden*  
*musick*  
*shultz*

*Another good PE score!*  
*Rerun this PE with the*  
*new QB3 which has*  
*just been received. You*  
*will be able to compare*  
*results with those reported*

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*Rev.*

*Whaley*

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INORGANIC PERFORMANCE EVALUATION SAMPLE  
 INDIVIDUAL LABORATORY SUMMARY REPORT  
 FOR Q3 2 FY 88

*Are for 30 labs  
 was 30. All  
 score was 94.1*

LABORATORY NAME: ORNL  
 PERFORMANCE LEVEL: ACCEPTABLE  
 LABORATORY RANK: Above = 11 Same = 1 Below = 18

Score: 94.1  
 REPORT DATE: 3/23/1988  
 MATRIX: WATER

ELEMENT NAME	95 % CI		LAB RESULTS		PROGRAM DATA					TOTAL #LABS
	LOWER	UPPER	REPORTED VALUE	QUALIFIER CODE	#LABS NLS ID	#LABS NLS-QUANT	#LABS FALSE POS	#LABS MSPK OUT	#LABS DUP OUT	
ALUMINUM	2540	3300	2990		0	1	0	0	0	31
ANTIMONY	0	111	82.9		3	0	0	1	3	31
ARSENIC	68	106	89.6		0	1	0	0	0	31
BARIUM	372	450	691	X	0	4	0	0	1	31
BERYLLIUM	38	51	44.7		0	1	0	0	0	31
CADMIUM	19	32	27.4	E	0	0	0	0	1	31
CALCIUM	12300	15500	14600		0	2	0	0	0	31
CHROMIUM	14	40	13		0	0	0	0	1	31
COBALT	66	113	91.7	E	0	0	0	0	0	31
COPPER	180	244	213		0	2	0	1	2	31
IRON	355	442	430	E	0	4	0	0	0	31
LEAD	12	25	17.7		0	0	0	3	2	31
MAGNESIUM	7830	9600	8970		0	2	0	0	0	31
MANGANESE	62	81	73.1	E	0	1	0	0	0	31
MERCURY	10	20	15.6		0	2	0	1	1	31
NICKEL	86	126	107		0	1	0	0	1	31
POTASSIUM	8810	12400	10600		0	2	0	0	0	31
SELENIUM	18	28	26		0	2	0	1	0	31
SILVER	e	e	9.5	B	0	0	0	5	0	31
SODIUM	6100	8320	7150		0	5	0	0	0	31
THALLIUM	51	88	58.8		0	1	0	7	1	31
VANADIUM	118	154	140		0	1	0	1	0	31
ZINC	47	66	57		0	5	0	1	2	31

# OF ELEMENTS NOT IDENTIFIED: 0  
 # OF ELEMENTS MISQUANTIFIED: 1  
 # OF FALSE POSITIVES: 0

# OF DUPLICATES OUT: 2  
 WATER : Sb, Ba  
 SOIL :

# OF MATRIX SPIKES OUT: 1  
 WATER :  
 SOIL : Sb

INORGANIC PERFORMANCE EVALUATION SAMPLE  
 INDIVIDUAL LABORATORY SUMMARY REPORT  
 FOR Q3 2 FY 88

*Our score*



LABORATORY NAME: ORNL  
 PERFORMANCE LEVEL: ACCEPTABLE  
 LABORATORY RANK: Above = 11 Same = 1 Below = 18

Score: 94.1  
 REPORT DATE: 3/23/1988  
 MATRIX: SOIL

ELEMENT NAME	95 % CI		LAB RESULTS		PROGRAM DATA					TOTAL #LABS
	LOWER	UPPER	REPORTED VALUE	QUALIFIER CODE	#LABS MIS ID	#LABS MIS-QUANT	#LABS FALSE POS	#LABS MSPK OUT	#LABS DUP OUT	
ALUMINUM	4790	11900	9690		0	2	0	0	0	31
ANTHONY	0	53	33		3	3	0	20	0	31
ARSENIC	17	28	21.8		0	4	0	7	1	31
BARIUM	156	189	169		0	3	0	1	0	31
BERYLLIUM	16	21	18		0	0	0	1	0	31
CADMIUM	9.7	17	13.1		0	0	0	1	0	31
CALCIUM	75301	184001	90700		0	2	0	0	0	31
CHROMIUM	16	51	30.8		0	2	0	0	0	31
COBALT	71	92	75.3	E	0	1	0	0	0	31
COPPER	88	112	94.5		0	3	0	1	0	31
IRON	12600	17400	15300	E	0	3	0	0	0	31
LEAD	164	226	180		0	4	0	2	0	31
MAGNESIUM	40801	57101	40400		0	2	0	0	0	31
MANGANESE	2810	3530	3220	E	0	7	0	1	0	31
MERCURY	12	24	17.6		0	3	0	2	1	31
NICKEL	26	54	37.9		0	2	0	3	0	31
POTASSIUM	0	1970	1690		0	4	0	0	0	31
SELENIUM	6.5	20	16		0	3	0	4	4	31
SILVER	33	52	45.6		0	3	0	5	1	31
SODIUM	4	4	361	B	0	0	0	0	0	31
THALLIUM	19	43	29.8		0	0	0	6	2	31
VANADIUM	41	70	50.3	E	0	1	0	0	0	31
ZINC	162	209	189		0	2	0	2	0	31

# OF ELEMENTS NOT IDENTIFIED: 0  
 # OF ELEMENTS MISQUANTIFIED: 0  
 # OF FALSE POSITIVES: 0

# OF DUPLICATES OUT: 2  
 WATER : Sb, Ba  
 SOIL :

# OF MATRIX SPIKES OUT: 1  
 WATER :  
 SOIL : Sb

- b CONFIDENCE INTERVALS (CI) WERE DERIVED FROM LABORATORY SUBMITTED VALUES. LESS THAN VALUES (<), U-VALUES, AND NON-SUBMITTED VALUES (-) WERE NOT USED IN THE CALCULATION OF THE CI.
- c CI WERE NOT SET SINCE 40 % OR MORE OF THE LABORATORIES SUBMITTED A NON-USABLE VALUE.
- d CI NOT USED. SEE SCORING NOTES, PROCEDURE FOR GRADING U-VALUES NO. 4.
- B INDICATES AN ESTIMATED VALUE LESS THAN THE CRDL. SAME AS (-)-FLAG.
- D INDICATES A DILUTION.
- E INDICATES A VALUE ESTIMATED OR NOT REPORTED DUE TO THE PRESENCE OF INTERFERENCES.
- G INDICATES VALUE DETERMINED BY THE METHOD OF STANDARD ADDITION.
- NR NOT REQUIRED.
- U ANALYZED FOR BUT NOT DETECTED.
- X VALUE WAS OUTSIDE BOTH THE WARNING AND THE ACTION LIMIT. POINTS DEDUCTED.
- 0 VALUE WAS OUTSIDE THE WARNING LIMIT ONLY. NO POINTS DEDUCTED.
- VALUE NOT SUBMITTED FOR THIS PARAMETER.
- INDICATES A FALSE POSITIVE BY BIRCH'S TEST. POINTS DEDUCTED.
- ? BEST ESTIMATE OF VALUE AND/OR QUALIFIER. POOR COPY AND/OR ILLEGIBLE VALUE SUBMITTED.
- < INDICATES A VALUE LESS THAN THE CRDL OR THE INSTRUMENT DETECTION LIMIT.
- ( ) INDICATES AN ESTIMATED VALUE LESS THAN THE CRDL. SAME AS B-FLAG.
- ( ) INDICATES AN ESTIMATED VALUE LESS THAN THE CRDL. SAME AS B-FLAG.
- D2 THE SAMPLE WAS DILUTED BY A FACTOR OF 2.
- D4 THE SAMPLE WAS DILUTED BY A FACTOR OF 4.
- D10 THE SAMPLE WAS DILUTED BY A FACTOR OF 10.
- D20 THE SAMPLE WAS DILUTED BY A FACTOR OF 20.
- D40 THE SAMPLE WAS DILUTED BY A FACTOR OF 40.
- D50 THE SAMPLE WAS DILUTED BY A FACTOR OF 50.
- W WARNING LIMIT (90 PERCENT CI).
- A ACTION LIMIT (95 PERCENT CI).

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SCORING NOTES: PROCEDURE FOR GRADING U-VALUES

1. ANY U-VALUE RESPONSE (INSTRUMENT DETECTION LIMIT) > CRDL FOR THE APPROPRIATE DILUTION, EVEN IF IT IS IN THE 95 % CI, CAUSES A POINT DEDUCTION. IF 25 % OR MORE OF THE LABORATORIES REPORT A U-VALUE OVER THE CRDL, NO POINTS ARE DEDUCTED FOR ANY LABORATORY, POSSIBLY INDICATING A MATRIX INTERFERENCE IN THE SAMPLE.
2. IF CRDL < LOWER CI, THEN USE CI AS SET.
3. IF LOWER CI < CRDL AND CRDL < UPPER CI, THEN SET LOWER CI TO ZERO (0). NO POINTS DEDUCTED FOR IDENTIFICATION OR QUANTITATION LESS THAN OR EQUAL TO THE CRDL.
4. IF CRDL > LOWER AND UPPER CI, THEN NO CI USED. PARAMETER DROPPED FROM THE SCORING. NO POINTS DEDUCTED FOR IDENTIFICATIONS OR QUANTITATIONS. FALSE POSITIVES POSSIBLE.

NOTE THAT ONLY CLP LABORATORIES WERE USED IN THE CALCULATION OF THE CI.

NOTE THAT A U-VALUE FOLLOWED BY X (U X) MEANS THAT POINTS WERE LOST FOR IDENTIFICATION AND QUANTITATION.

SUMMARY OF LABORATORY SCORES  
 QB 2 FY 88

CODE	SCORE	NOT ID	MISQUANT	FALSE POS	MSPK OUT	DUP OUT
A1	72.8	0	6	0	5	5
A2	91.8	0	2	0	3	0
A3	-	-	-	-	-	-
B1	99.5	0	0	0	1	0
B2	72.3	0	7	0	4	3
B3	79.1	0	6	0	1	1
C1	96.1	0	1	0	1	0
C2	-	-	-	-	-	-
C3	-	-	-	-	-	-
D1	-	-	-	-	-	-
D2	94.1	0	1	0	1	2
D3	83	0	5	0	1	0
E1	95.6	0	1	0	2	0
E2	91.8	0	2	0	1	1
E3	-	-	-	-	-	-
F1	-	-	-	-	-	-
F2	-	-	-	-	-	-
F3	-	-	-	-	-	-
G1	86.5	0	4	0	1	0
G2	83.5	0	5	0	0	0
G3	98.5	0	0	0	3	0
H1	-	-	-	-	-	-
H2	-	-	-	-	-	-
I1	-	-	-	-	-	-
I2	-	-	-	-	-	-
J1	75.5	0	6	0	9	1
J2	90	0	0	0	4	0
K1	95.1	0	1	0	3	0
K2	-	-	-	-	-	-
L1	96.6	0	1	0	0	0
L2	-	-	-	-	-	-
M1	93.1	0	1	0	3	2
M2	89.8	0	2	0	7	0
N1	76.8	0	6	0	5	1
N2	87.5	0	3	0	3	1
O1	-	-	-	-	-	-
O2	99	0	0	0	2	0
P1	94.1	0	1	0	3	1
P2	96.6	0	1	0	0	0
Q1	-	-	-	-	-	-
Q2	-	-	-	-	-	-
R1	-	-	-	-	-	-
R2	-	-	-	-	-	-
S1	69.3	0	10	0	0	0
S2	-	-	-	-	-	-
T1	78	0	5	0	7	2
T2	-	-	-	-	-	-
U1	71.9	0	8	0	5	1
U2	-	-	-	-	-	-
V1	97.5	0	0	0	3	1
V2	94.6	0	1	0	2	1
V1	-	-	-	-	-	-
V2	-	-	-	-	-	-
X1	-	-	-	-	-	-
X2	-	-	-	-	-	-
Y1	90.8	0	2	0	3	1
Y2	-	-	-	-	-	-
Z1	-	-	-	-	-	-
Z2	89	0	3	0	2	0

OAK RIDGE NATIONAL LABORATORY  
OPERATED BY MARTIN MARIETTA ENERGY SYSTEMS, INC.

POST OFFICE BOX X  
OAK RIDGE, TENNESSEE 37831

April 29, 1988

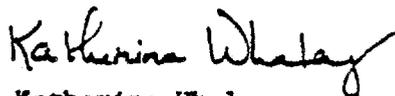
Harold Vincent  
US EPA, EMSL-LV, QAD  
P. O. Box 15027  
Las Vegas, NV 89114

Dear Mr. Vincent:

According to instructions received with the QB-2-88 performance evaluation score sheet package, any quantified value falling outside the acceptance window should be explained in writing. Our score for this set was 94.1. The result for Ba on the water sample fell outside the upper range unit. The high value is believed to be caused by contamination during preparation as the duplicate result was also out for Ba. The soil sample, prepared in Erlenmeyer flasks, was not contaminated. The beakers used in the preparation of water samples will be cleaned more carefully in the future.

If a letter is not required for scores greater than 90, please let me know.

Sincerely,



Katherine Whaley  
ICP Spectroscopist



W. R. Laing  
DOE Site Survey Program Manager

cc: Karen Knight

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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
OFFICE OF RESEARCH AND DEVELOPMENT  
ENVIRONMENTAL MONITORING SYSTEMS LABORATORY-LAS VEGAS  
P.O. BOX 93478  
LAS VEGAS, NEVADA 89193-3478  
(702/796-2100 - FTS 545-2100)

W. R. Laing  
Oak Ridge National Laboratory  
P.O. Box X MS 127  
Bethel Valley Rd.  
Oak Ridge, TN 37831-6127

Dear Mr. Laing:

For your information and review, enclosed are the results for your participation in the EMSL-LV First Quarter Inorganic Performance Evaluation Study (QB1 FY-88, Case No. 8123). Your laboratory code is on your scoresheet. The samples were prepared by the EMSL-LV and consisted of one soil sample and two water samples. The homogeneous soil sample and one of the water samples were spiked with inorganic parameters. The other water sample was a blank. The samples were to be prepared and analyzed by current IFB procedures as per contract. All laboratories received the samples single blind. Also enclosed is more general information about the Superfund Performance Evaluation Program.

Thank you for your participation in this study. I trust that this information will be beneficial in your pursuit of excellence as a member of the community of laboratories analyzing hazardous waste samples.

Sincerely,

A handwritten signature in black ink, appearing to read "Larry C. Butler".

Larry C. Butler, Ph.D.  
Supervisor, Performance Evaluation Program  
Quality Assurance Research Branch  
Quality Assurance Methods Development Division

**Enclosures**

cc: (w/out enclosures)  
Mike Hurd, OERR (WH-548A)  
Carla Dempsey, OERR (WH-548A)  
William Langley, OERR (WH-548A)

(Blank Page)



FILE: QB1I88R3.WK1

CODED SUMMARY OF SCORES  
FIRST QUARTER INORGANIC FY 88 CLP SINGLE BLIND  
(QB 1 FY 88, CASE NO. 8123)

-----

CODE	POINT SCORE	% SCORE	NO. OF DAYS LATE
----	-----	-----	-----
G1	100.0	100.0	0
F2	98.5	98.5	0
A1	95.6	95.6	0
Z1	94.6	94.6	0
J1	92.1	92.1	0
S1	91.8	91.8	0
K1	91.0	91.0	0
O2	89.8	89.8	1
Y2	89.7	89.7	16
B3	88.6	88.6	13
P1	86.8	86.8	3
Y1	85.2	85.2	0
D1	85.0	85.0	1
C3	84.3	84.3	10
B1	83.7	83.7	15
G2	82.2	82.2	0
Z2	81.5	81.5	0
T1	79.6	79.6	0
C1	47.6	47.6	22
H2	*	*	*
I2	*	*	*
N1	*	*	*
K2	*	*	*

\* NO DATA SUBMITTED AS OF DECEMBER 22, 1987

**CODED SUMMARY OF SCORES**  
**FIRST QUARTER INORGANIC FY 88 NON-CLP SINGLE BLIND**  
**(QB 1 FY 88, CASE NO. 8123)**

---

CODE	POINT SCORE	% SCORE	NO. OF DAYS LATE
G3	99.0	99.0	0
V2	99.5	99.5	1
I1	94.1	94.1	0
A2	90.7	90.7	15
W1	75.1	75.1	1
M2	71.4	71.4	0
R2	70.5	70.5	1
D2	24.4	30.5	27
E1	*	*	*
X1	87.2	87.2	3
F3	*	*	*
H1	*	*	*
N2	*	*	*
S2	*	*	*
V1	*	*	*
O1	*	*	*
L2	*	*	*
L1	71.9	89.9	0

\* NO DATA SUBMITTED AS OF DECEMBER 22, 1987

CODED SUMMARY OF SCORES  
 FIRST QUARTER FY 88 INORGANIC REGIONAL SINGLE BLIND  
 (QB 1 FY 88, CASE NO. 8123)

-----

CODE	POINT SCORE	% SCORE	NO. OF DAYS LATE
-----	-----	-----	-----
T2	*	*	*
E2	*	*	*
J2	*	*	*
U1	*	*	*
P2	74.9	74.9	6
F1	*	*	*
D3	*	*	*
Q1	*	*	*
R1	*	*	*
A3	*	*	*
W2	17.0	17.0	0
M1	47.0	47.0	6
U2	*	*	*

\* NO DATA SUBMITTED AS OF OCTOBER 23, 1987

CODED SUMMARY OF SCORES  
FIRST QUARTER INORGANIC FY 88 DOE SINGLE BLIND  
(QB 1 FY 88, CASE NO. 8123)

-----

CODE	POINT SCORE	X SCORE	NO. OF DAYS LATE
-----	-----	-----	-----
X2	86.5	86.5	1
Q2	82.4	82.4	27
E3	81.9	81.9	1
C2	.	.	.
B2	.	.	.

• NO DATA SUBMITTED AS OF DECEMBER 22, 1987

12/22/1987

ON 1 FT 80 INORGANIC, CASE NO. 8123

WATER SAMPLE (MG/L)

ELEMENT NAME	90 N CI 0 b		95 N CI 01 b		A1	A2	B1	B2	C1	C2	D1	D2	E1	E2
	LOWER	UPPER	LOWER	UPPER										
ALUMINUM	1010	1300	974	1410	1180	1290	1130	950	1400	1220	1170	1300	1250	1180
ANTIMONY	c	c	c	c	53	40	60	30	21	32	1.2	250	5.7	12
ARSENIC	59	83	56	85	36	65.6	72	70	69	71	65	62	76.6	64
BARIUM	409	542	479	592	364	542	570	400	570	554	536	670	560	540
BERYLLIUM	61	75	60	76	73	66.3	67	60	62	69	66	60	73.6	71.3
CADMIUM	207	343	201	349	340	326	300	290	355	320	330	310	323	322.2
CALCIUM	7060	8470	6910	8620	7000	8752	7600	7300	8600	8050	7400	10500	8300	8132
CHROMIUM	53	72	51	74	67	62.6	61	50	55	65	69	10	64.2	67
CORAL	130	152	120	154	144	141	128	140	154	147	135	170	142	147
COPPER	563	689	549	703	696	642	592	600	653	631	600	630	633	652
IRON	664	827	649	845	759	724	740	700	810	720	716	720	569	836
LEAD	27	39	26	40	37	34.6	36	30	25	39	31	50	37.0	36
MAGNESIUM	11300	13000	11100	13200	12700	12350	12900	11800	13700	12100	12800	12700	12300	12320
MANGANESE	107	125	104	127	122	110	115	120	140	124	117	110	125	117
MERCURY	1.5	3.9	1.3	4.2	1.8	2.45	2.8	2.9	3.1	2.5	0.3	2.4	2.4	2.6
NICKEL	220	261	219	264	250	227	253	250	255	243	237	230	255	261
POTASSIUM	7710	11300	7230	11700	10200	10270	10100	8300	10900	9310	9160	10900	9530	9730
SELENIUM	30	50	27	52	30	40.5	41	50	44	37	40	40	37.6	40
SILVER	63	100	59	104	86	83.4	75	90	86	85	71	67	83.5	79.1
SODIUM	13000	16000	12600	16300	15700	15270	11700	12600	16100	15100	14400	19000	14500	14100
THALLIUM	37	51	36	52	42	44.2	44	60	63	52	41	60	44	38
THORIUM	274	304	270	307	290	292	287	280	325	301	292	340	293	291.5
ZINC	89	119	86	122	114	101	87	190	113	102	102	100	99.4	100
TOTAL OUT (ACTION LIMIT)					1	1	2	3	0	0	1	7	1	0
TOTAL OUT (WARNING LIMIT)					1	0	1	3	2	1	0	1	0	1
TOTAL OUT (IDENTIFICATION)					0	0	0	0	0	0	0	1	0	0
TOTAL OUT (FALSE POSITIVE)					0	0	0	0	0	0	0	1	0	0

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12/22/1987

MD 1 FT 00 IDOCARC, CASE NO. 0123

WATER SAMPLE (UG/L)

ELEMENT NAME	90 N CI 0 6		95 N CI 00 6		G1	G2	G3	L1	J1	K1	L1	M2	O2	P1
	LOWER	UPPER	LOWER	UPPER										
ALUMINUM	1010	1300	976	1010	1220	1150	1230	1200	1190	1100	- DT	1260	1260	1250
ANTIMONY	e	e	e	e	52 U	39 U	2	60 U	26 U	50 0	1 <	5 <	27 U	9.6 U
ARSENIC	59	83	36	85	67	76 E	66	72	70	66	73	69 X	70 E	69 E
BARIUM	409	582	479	592	578 ET	554	499	527	510	534	520	515	510	546
BERYLLIUM	61	75	60	76	67	69	65	71	69	67	71	66	67	70
CADMIUM	287	318	281	319	325	304	307	310	325	307	314	299	317	310
CALCIUM	7060	8470	6910	8620	8260	7420	7120	8000	7930	7090	- DE	7930	7630	7630
CHROMIUM	53	72	51	70	61	67	62	54	62	54	62	60	65	60
COBALT	130	152	120	154	141	140	142	134	141	138	145	114 X	145	141
COPPER	563	689	549	703	621	653	602	587	618	609	600	590	627	614
IRON	664	827	649	845	716	693	700	732	689	710	- DE	740	720	792
LEAD	27	39	26	40	33	33 E	35	34	36	28 E	36	62 X	33	34
MAGNESIUM	11300	13000	11100	13100	12900	11600	11900 E	12700	12400	12000	- DE	11730	12200	12200
MANGANESE	107	125	104	127	118 U	113 E	111	112	109 E	113	- DE	110	115	116
MERCURY	1.3	3.0	1.3	4.2	2.5	2.5	2.0	2	2.6	2.7	2	2.7	2.6 U	2.5
NICKEL	270	261	215	264	230 U	235	243	220	237	246	250	205 E	210	235
POTASSIUM	7710	11300	7130	11700	9490	11000 X	10200	9720	9360	10000	- DE	9490	9050	9100
SELENIUM	30	50	27	52	40	40	36	40	35	32 E	40	41	40	35
SILVER	63	100	59	104	66	79	74	76	81	79	76	69 X	76	97
SODIUM	13000	16000	12600	16300	14000	15600	14200	15000	14800 E	15200	- DE	15410	14200	13100 U
THALLIUM	37	51	36	52	47	45	42	40	40	43	47	35 X	30 E	45 E
THORIUM	274	304	270	307	292	287	279	268	279	285	280	280	300	295
ZINC	89	119	86	122	89	113	107	94	109	90	120 0	96	100	105
TOTAL OUT (ACTION LIMIT)					0	1	0	0	0	0	0	6	0	0
TOTAL OUT (WARNING LIMIT)					0	0	0	0	0	0	1	0	0	0
TOTAL OUT (IDENTIFICATION)					0	0	0	0	0	0	0	0	0	0
TOTAL OUT (FALSE POSITIVE)					0	0	0	0	0	0	0	0	0	0

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12/22/1967

NO 1 FY 00 INORGANIC, CASE NO. 0122

WATER SAMPLE (MG/L)

ORNL

ELEMENT NAME	00 R CI 0 6		95 R CI 00 6		02	02	01	01	02	01	01	02	01	02
	LOWER	UPPER	LOWER	UPPER										
ALUMINUM	1010	1300	974	1410	1260	1290 ?	1230	1130	1250	1100 E	1110	1200	1390 0	1130
ANTHRONE	e	e	e	e	07 W	25 W	25 W	24 W	2.3 W	0.9 W	30 W	30 W	30 W	55 W
ARSENIC	50	02	54	05	61	77	63	05 E 0	75	79.6	60	69	03	60
BARIUM	409	502	479	592	505	569	547	531	555	530 E	509	541	530	561
BERYLLIUM	61	75	60	76	66	74	72	63	71	66	66.5 E	73	71	64
CADMIUM	207	303	201	349	312	320	324	293	320	296 E	283 0	341	300	301
CALCIUM	7060	0670	0910	0420	7630	0340	7930	7300	0000	7030 E	7390	7960	0070	7300
CHROMIUM	33	72	51	74	63 E	64	66	55	67	67.5	50.9	75 E E	67	70 E
COBALT	130	152	120	156	105	165 E	155 E	136	152	155 E	130	154 E	134	140
COPPER	563	600	549	703	617	700 0	669	546 E E	611	605	576	619	643	597
IRON	646	027	649	045	704	030 0	735	743 E	723	775 E	715	730	700	755
LEAD	27	30	26	40	41 E	30	29	37	35	31.6	36.2	43 E	30 E	30
MAGNESIUM	11300	13000	11100	13200	12100	11300 E	12200	11400	12900	12300 E	11300	13000	11900	12100
MANGANESE	107	125	104	127	116	125	120	105 E 0	110	110	104 0	123	116	115
MERCURY	1.5	2.9	1.3	4.2	2.0 E	3.5	2.0	2.0	2.4	1.6	2.7	2.3	3.3	3.1
NICKEL	220	261	215	264	245	263 0	254	214 E	250	252	220	257	220	224
POTASSIUM	7710	11300	7330	11700	9040 E	10200	9040	7300 0	9700	7000	8100	9900	9500	9270 E
SELENIUM	30	50	27	52	39	36	30 E	34	39	37.0	42.0	41	39 E	34
SILVER	63	100	59	104	71 E	02	01	77	02	70.0	73.1	07 E	01	77
SODIUM	13000	16000	12600	16300	14700	16100 0	15300	13500	14700	16400 E	13300	14300	14500	13900
THALLIUM	37	51	36	52	49	61	29 E E	65	47	13.2 E	33.4 E E	40	44	52 0
THORIUM	276	304	270	307	290	314 E	290	248 E E	305 0	297 E	291	322 E	310 E E	284
ZINC	09	119	06	122	90 E	101	111	72 E E	103	110 E	91.7	102	91	103

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TOTAL OUT (ACTION LIMIT)	1	3	2	4	0	2	1	0	2	1	0	1	1	1
TOTAL OUT (WARNING LIMIT)	0	4	0	3	1	0	0	0	2	0	0	1	1	1
TOTAL OUT (IDENTIFICATION)	0	0	0	0	0	0	0	0	0	0	0	0	0	0
TOTAL OUT (FALSE POSITIVE)	0	0	0	0	0	0	0	0	0	0	0	0	0	0

0

12/22/1967

NO 1 FT 60 INORGANIC, CASE NO. 0123

WATER SAMPLE (UG/L)

ELEMENT NAME	90 E CI 0 6		95 E CI 00 6		21	22
	LOWER	UPPER	LOWER	UPPER		
ALUMINUM	1010	1300	974	1110	1090	1360
ANTHROPE	c	c	c	c	67 E	60 U
BARIUM	50	83	56	85	67	80
BISMUTH	489	582	479	592	499	563
BERYLLIUM	61	75	60	76	63	76
CADMIUM	207	363	201	349	207	335
CALCIUM	7060	8970	6910	8620	7500	8630 E
CHROMIUM	53	72	51	74	59	66
COBALT	130	152	120	151	131	167 E X
COPPER	343	489	340	503	349	466
IRON	646	827	649	845	685	897
LEAD	27	39	26	40	31	50 SA E
MAGNESIUM	11900	13000	11800	13200	11700	12900
MANGANESE	107	125	104	127	111	128
MERCURY	1.3	3.9	1.3	4.2	3.0	2.3 P
NICKEL	220	261	215	266	229	263
POTASSIUM	7710	8190	7330	8170	7870	8030
SELENIUM	30	50	27	52	46	50 E
SILVER	63	100	59	104	50 E	101 0
SODIUM	13000	16000	12600	16300	14200	15300
THALLIUM	37	51	36	52	43	60 E
ZINC	274	301	270	307	273 0	290
ZINC	89	119	86	122	102 E	107

TOTAL OUT (ACTION LIMIT) 1 6  
 TOTAL OUT (WARNING LIMIT) 1 1  
 TOTAL OUT (IDENTIFICATION) 0 0  
 TOTAL OUT (FALSE POSITIVE) 0 0

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12/22/87

001 TV 00 (MORGAN)C, CASE-NO. 8123

WATER SAMPLE (UG/L)

ELEMENT NAME	90 % CI # b		95 % CI # b		P2	M1	M2
	LOWER	UPPER	LOWER	UPPER			
ALUMINIUM	1010	1300	976	1410	1220	1190	1300
ANTIMONY	c	c	c	c	-2 U	-12.5 U	-50 U
ARSENIC	59	83	56	85	76	72.5	136 X
BARIUM	409	562	479	592	530	561	700 X
BERYLLIUM	61	75	60	76	72.3	76	66
CADMIUM	207	343	201	349	320	312	320
CALCIUM	7040	8470	6910	8620	7700	7490	6500 X
CHROMIUM	53	72	51	76	64.2	-50 M X	100 X
COBALT	130	152	120	154	152	134	150
COPPER	543	609	549	703	639	632	660
IRON	646	827	640	845	814	733	700
LEAD	27	39	26	40	34.4	36.6	-50 M X
MAGNESIUM	11300	13000	11100	13200	12400	13200	0 11800
MANGANESE	107	125	104	127	122	117	120
MERCURY	1.5	3.9	1.3	4.2	0.3 X	2.60	31 X
NICKEL	220	261	210	266	245	230	240
POTASSIUM	7710	11300	7330	11700	13200 X	9000	7300 X
SELENIUM	30	50	27	52	39.9	36.5	50
SILVER	45	100	39	104	89	93	80
SODIUM	13000	16000	12600	16300	14500	14900	13600
THALLIUM	37	51	36	52	46	40	50
VANADIUM	276	306	270	307	303	306	0 -500 M X
ZINC	80	119	86	122	134 X	95	86 0

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TOTAL OUT (ACTION LIMIT)	3	1	0
TOTAL OUT (WARNING LIMIT)	0	2	1
TOTAL OUT (IDENTIFICATION)	0	1	2
TOTAL OUT (FALSE POSITIVE)	0	0	0

12/22/1967

001 BY 00 HUNGARIC, CASE NO. 0123

SOIL SAMPLE (MG/EG)

ELEMENT NAME	90 X CI 0 b		95 X CI 01 b		B1	B2	B1	B3	C1	C3	B1	B2	B3	B2	
	LOWER	UPPER	LOWER	UPPER											
ALUMINUM	9100	16700	8320	17500	13600	15500	16200	30000	13900	16700	6.55 STAR	7730	X	10100	9500
ANTIMONY	c	c	c	c	26 U	32 U	30 U	0.9 LI	11 U	33 U	1.2 U	25		1.1	6 U
ARSENIC	4.5	9.6	3.7	10	0.2 E	5.00	7.5	7	11 X	6.6	5.2	7.2		7.5	3.1 U
BARIUM	173	162	110	166	153	161	145	150	167 X	14	110	170	X	138	130
BERYLLIUM	c	c	c	c	0.5 LI	0.525	1.5 U	0.4 U	1.5 LI	1.1	0.36 LI	0.40		1.1 X	0.6 LI
CAESIUM	c	c	c	c	2.4	2.56	2 U	1 U	0.3	0.01 U	0.53 LI	0.7		0.5 U	1.2 U
CALCIUM	57100	69500	55700	70000	62600	59900	63500	63900	73400 X	65000	66600	157	X	57000	63310
CARBONUM	12	24	11	25	10	19	16	20	15	41 X X	15	13.6		41.1 X	15
CORALIT	4.9	10	0.00	11	11 U	0.19	15 U	7.9 LI	0.4 LI	0.6 LI	7.4 LI	0.2		0.9	7 LI
COPPER	29	43	27	41	40	37.0	36	40	41	36	32	35.5		33.5 E	32
IRON	15700	21000	15100	21500	19900	21600	19200	10500	19100	21500	12500	70 X	X	11500	16420
LEAD	79	106	76	109	97	110	115	100	117 X	92	92	89		97.2	95
MANGANESE	24200	27200	23000	27500	26500	26000	27200	26000	29000 X	24000	25900	22800	X	23700	25010
MANGANESE	513	674	496	682	645	609	587	670	677	615	487	520		576	554
MERCURY	c	u	c	c	0.13	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.2 U	0.12		0.04 U	0.1 U
NICKEL	16	26	13	27	15 LI	22.2	13 LI X	20	22	31 X X	15	16		30.2 X	20 LI
POTASSIUM	1350	2930	1100	3100	2660	2580	2750	2400	2610	2600	1220	0	0	1760	1047 LI
SELENIUM	c	c	c	c	1.5 U	1 U	2.5 U	6.1 U	2.5 U	1 U	0.22 U	0.31		0.2 U	2.5 U
SILVER	c	c	c	c	2.4 U	2 U	5 U	2 U	2 U	6.1 U	1.9 U	1.4		0.01 U	2.1 LI
SODIUM	0	0	0	0	321 LI	217	750 U	260 LI	260 LI	200 LI	11 LI	1920		207	6 U
TUNGSTEN	c	c	c	c	1 U	2 U	5 U	0.4 U	3.4 U	2 U	0.26 U	2.4		0.30 U	5 U
Vanadium	10	00	12	03	27	25.0	31	30	33	43	17	10		25.9	25.3
ZINC	106	127	104	130	126	125	114	130	127	122	110	100	X	114 X	100
TOTAL OUT (ACTION LIMIT)					0	1	2	0	5	2	2	6		3	0
TOTAL OUT (WARNING LIMIT)					0	2	0	1	1	2	3	1		1	0
TOTAL OUT (IDENTIFICATION)					0	0	0	0	0	0	0	0		0	0
TOTAL OUT (FALSE POSITIVE)					0	0	0	0	0	0	0	0		0	0

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12/22/1987

GRI FT 00 INORGANIC, CASE NO. 8123

SOIL SAMPLE (MG/KG)

ELEMENT NAME	90 E CE 0 6		95 E CE 00 6		61	62	63	11	21	61	61	62	62	62
	LOWER	UPPER	LOWER	UPPER										
ALUMINUM	9100	14700	8320	17500	11600	13800	11042	13100	14100	11400	- NR	11530	15900	12300
ANTIMONY	e	e	e	e	26 U	7.7 U	0.0 U	16 U	5.2 U	25 U	0.7	1 <	0.9 117	4.0 U
ARSENIC	4.3	9.6	3.7	10	5.6	6.8 E	7.8 E	15 X	6.6 E	7.0 E	5	3.7	10 64 E	5.0 E
BARITUM	123	162	119	166	125 E	152	135	140	145	153	125	137	153	153
BERYLLIUM	e	e	e	e	2 U	0.4 U	0.3 U	1 U	0.77 11	1 U	0.7	1 <	1 07	1 U
CADMIUM	e	e	e	e	2 U	4 U	0.0 U	0.3 11	1 U	2.5 U	0.7	1.0	3.5 7	2.5
CALCIUM	57100	69500	55700	70000	61500	65100	60700	65500	61100	67000	- NR	57000 0	58000	63000
CHROMIUM	12	24	11	25	19	21	15.9	19	20	10	33 X	12.2	25 0	15
COBALT	4.9	10	0.00	11	4 11 0	9.3 11	0 0	10 U	6.1 112	10 U	7.1	10 <	7.7 111	9.9 11
COPPER	29	43	27	44	29	34	35.9	35	30	32	33	31	39	29
IRON	15700	21000	15100	21500	17600	19400 E	14100	19700	17500	20100	- NR	17130	18300	19700
LEAD	79	106	76	109	95	90	101 U	103	127 11 X	84 U	95	114 X	94 1	75 X
MAGNESIUM	24200	27200	23000	27500	24000	26000	25000	27000	25000	26200	- NR	22625 X	25000	25600
MANGANESE	513	674	496	492	500	543 U X	570	626	540	620	- NR	535	573	606
MERCURY	e	e	e	e	0.1 07	0.1 U	0.3 U	0.1 U	0.1 U	0.1 U	0.2 <	0.05 <	0.1 01	0.1 U
NICKEL	14	26	13	27	25	24	19.5	18	18	26	27 1 0	16.7	22	36 4 X
POTASSIUM	1350	2920	1100	3100	1900 11	2070	2400	2450	2100	1960 11	- NR	2195	3640 11 X	2050 11
SELENIUM	e	e	e	e	0.9 U	3.5 U	0.5 U	1 U	5.0 02	2.5 U	2 <	1 <	0.6 02	0.4 U
SILVER	e	e	e	e	4.9 U	1.4 U	1.4 U	2 U	1.2 U	4 U	0.00	1 <	1.4 01	3.3 11
SODIUM	6	6	6	6	165 11	207 11	221 0	215 11	223 11 11	215 11	- NR	236	361 11	252 11
THALLIUM	e	e	e	e	0.6 U	1.6 U	0.8 U	2 U	0.36 11	5 U	0.1 <	1 <	1.4 02	2.7 U
VANADIUM	14	40	12	43	21 11	29	20.6	29	31	29	30	20	41 1 0	25
ZINC	106	127	104	130	109	114	116	114	110	114	107	100	117	111
TOTAL OUT (ACTION LIMIT)					0	1	0	1	1	0	1	2	2	2
TOTAL OUT (WARNING LIMIT)					1	0	0	0	0	0	1	1	2	0
TOTAL OUT (IDENTIFICATION)					0	1	0	0	0	0	0	0	0	0
TOTAL OUT (FALSE POSITIVE)					0	0	0	0	0	0	0	0	0	0

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12/12/1987

001 FT DE INORGANIC, CASE NO. 0123

SOIL SAMPLE (MG/KG)

ORNL

ELEMENT NAME	90 E CI 0 6		95 E CI 0 6		02	02	01	T1	V2	01	01	02	T1	T2
	LOWER	UPPER	LOWER	UPPER										
ALUMINUM	9100	16700	8320	17500	19700 E	8090 E	8960 0	13500 E	12600	7230 E	6430 0	11800	13400	9000
ANTHRA	e	e	e	e	11 E	2.5 U	12 U	4.0 U	0.46 U	0.17 U	6 U	5 U	7.0 U	12 U
ARSENIC	0.3	9.6	3.7	10	5.0	5.7	10 E 0	9.5	9.3	0.1 0	9.2 U	7	6.8 E	7.1
BARUM	123	162	119	164	155	116 E	139	132	141	131 E	133	137 E	149	131
BERYLLIUM	e	e	e	e	0.73 E	0.35 U	0.5 U	0.3 E	0.0 E	0.71 E	0.5 U	0.71 E	0.50 U	1.1 U
CADMIUM	e	e	e	e	1	0.6	2 U	1.2	0.62	0.75 E	1 U	1.1	1.0	1.6
CALCIUM	57100	89500	55700	70000	61000	63200	64600	66000 E	45000	62000 E	50100	62000	53400 E	59200
CHROMIUM	12	28	11	25	23	11 0	16	16	10	11 0	13.4	19 E	21	30 E
COBALT	0.0	10	0.00	11	0.2 E	6.6 U	0.7 E	6.0 E	0.1	0.0 E	7.0 U	9.2	7.2 E	7.0 E
COPPER	29	43	27	44	35 E	32	40	40	37	34.6 E	31.2	37	32	35
IRON	13700	21000	15100	21500	19200	16900	17000	16900	18400	13000 E	16200	16700	17700	18200
LEAD	79	104	76	109	119 E	110 E	96	99	93	92.4	87.3 U	102	78 E 0	100 0
MAGNESIUM	24200	27200	23000	27500	21900	26200	27000	25200	27500 0	25200	23800 0	24900	23800 0	25600 E
MANGANESE	513	674	496	692	574	594	640	636 E	565	570 E	521	554	544	575
MERCURY	e	e	e	e	0.5 U	0.02 U	0.1 U	0.2	0.05	0.07 U	0.03	0.04	0.4	0.1 U
NICKEL	14	26	13	27	20 E	17.4	20	16	21	16.1	18.0 U	22	19	23
POTASSIUM	1350	2930	1100	3100	4240 E	1610	1730 E	1534	2170	1070 E	1290 0	2370	2350	1890
SELENIUM	e	e	e	e	0.22 E	0.2 U	25 U	1 U	0.21 E	0.19 U	0.4 U	0.30 E	1.1	0.4 U
SILVER	e	e	e	e	0.41 U	0.4 U	2 U	2 U	0.57 E	0.62 U	1 U	1.1 E	1.0	1.0 U
SODIUM	d	d	d	d	276 E	177 U	545 U	261 U	104	146 E	511 U	210 E	258 E	151 E
THALLIUM	e	e	e	e	0.20 E	0.4 U	5 U	2 U	0.67 E	0.19 U	0.6 U	0.14 U	0.59 E	0.4 U
VANADIUM	14	40	12	43	29	10.6	20 E	10	30	22.6 E	20.0 U	20 E	27	26
ZINC	104	127	104	130	124	96.6 E	117	96 E	114	112 E	101 E	115	110	120

TOTAL OUT (ACTION LIMIT)  
 TOTAL OUT (WARNING LIMIT)  
 TOTAL OUT (IDENTIFICATION)  
 TOTAL OUT (FALSE POSITIVE)

0  
 0  
 0  
 0

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12/22/1987

001 FT 00 (ORGANIC, CASE NO. 0123)

SOIL SAMPLE (MG/KG)

ELEMENT NAME	90 E CI 0 b		95 E CI 00 b		21	22
	LOWER	UPPER	LOWER	UPPER		
BARIUM	9100	16700	8320	17500	14100	12200
BETHUNY	0	0	0	0	00	12 W
ARSENIC	4.5	9.6	3.7	10	5	6.6
BARIUM	123	162	119	166	140	113
BERTILLIUM	0	0	0	0	0.01 W	0.6 (1)
CADMIUM	0	0	0	0	0.03 W	1 W
CALCIUM	57100	69500	55700	70000	59500	69700 0
CHROMIUM	12	26	11	25	20	10 7
COBALT	4.9	10	0.00	11	6.9 11	0.9 13
COPPER	29	43	27	44	40	37
IRON	15700	21000	15100	21500	17900	18200
LEAD	79	106	76	109	95	100
MANGANESE	24200	27200	23000	27500	25700	26300
MOLYBDENUM	513	674	496	692	569	612
MERCURY	0	0	0	0	0.25	0.2 W
NICKEL	10	26	13	27	22	21
POTASSIUM	1350	2930	1100	3100	2410	2220
SELENIUM	0	0	0	0	0.35 W	10 W
SILVER	0	0	0	0	1.6 W	2 W
SODIUM	0	0	0	0	241 11E	311 11
THALLIUM	0	0	0	0	0.37 (1)	10 W*
VANADIUM	14	40	12	43	29	27
ZINC	106	127	104	130	117 E	120

TOTAL OUT (ACTION LIMIT) 0 0  
 TOTAL OUT (GRAPHING LIMIT) 0 1  
 TOTAL OUT (IDENTIFICATION) 0 0  
 TOTAL OUT (FALSE POSITIVE) 0 1

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12/23/87

DOT FT 00 INORGANIC, CASE NO. 0123

SOIL SAMPLE (MG/KG)

ELEMENT NAME	96 % CI @ 6		95 % CI @ 6		P2	M1	M2	
	LOWER	UPPER	LOWER	UPPER				
ALUMINIUM	9140	16700	8320	17300	17000	0	11244	12500
ANTIMONY	c	c	c	c	-0.24 U		-2 U	13
ARSENIC	4.3	9.6	3.7	10	6.8		1.19 H	6.33
BARIUM	123	142	119	166	160		130.3	155
BERYLLIUM	c	c	c	c	0.33		0.9	-0.3 U
CADMIUM	c	c	c	c	0.6		3	-0.8 U
CALCIUM	57100	69500	53700	70000	66000		54450	0 43000 H
CHROMIUM	12	24	11	23	31	H	17	32 H
COBALT	4.9	10	0	11	8.8		16.3 H	0
COPPER	29	43	27	44	34		39.3	29.9
IRON	15700	21000	15100	21500	18000		14343 H	13000 H
LEAD	79	104	76	109	100		132.3 H	84
MAGNESIUM	24200	27200	23000	27500	10000	H	28270 H	21300 H
MANGANESE	513	674	496	692	500		550	500 S
MERCURY	c	c	c	c	-0.2 U		-0.2 U	-0.1 U
NICKEL	14	26	13	27	24		37.2 H	17
POTASSIUM	1350	2930	1180	3100	3400	H	1918	1720
SELENIUM	c	c	c	c	-0.3 U		-	0.39
SILVER	c	c	c	c	-0.6 U		1.34	-0.3 U
SODIUM	d	d	d	d	340		370	190
THALLIUM	c	c	c	c	-0.24 U		19.75	9
VANADIUM	14	40	12	43	40		30.3 H	-50 U H
ZINC	106	127	104	130	120		141.3 H	90 H

TOTAL OUT (ACTION LIMIT)	3	0	0
TOTAL OUT (WARNING LIMIT)	1	1	1
TOTAL OUT (IDENTIFICATION)	0	0	1
TOTAL OUT (FALSE POSITIVE)	0	0	0

C-195

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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
OFFICE OF RESEARCH AND DEVELOPMENT  
ENVIRONMENTAL MONITORING SYSTEMS LABORATORY-LAS VEGAS  
P.O. BOX 93478  
LAS VEGAS, NEVADA 89193-3478  
(702/798-2100 - FTS 545-2100)

NOV 06 1987

NOV 11 1987

Mr. W. R. Laing  
Oak Ridge National Laboratory  
P.O. Box X MS 127  
Bethel Valley Rd.  
Oak Ridge, TN 37831-6127

Good Work! Our grade on  
4<sup>th</sup> Quarter PEs was 96.0 vs  
an average grade of 78 for  
23 CLP labs. — Bill Laing

Dear Mr. Laing:

For your information and review, enclosed are the results for your participation in the EMSL-LV Fourth Quarter Inorganic Performance Evaluation Study (QB4 FY-87, Case No. 7761). Your laboratory was coded H1. The samples were prepared by the EMSL-LV and consisted of one soil sample and two water samples. The homogeneous soil sample and one of the water samples were spiked with inorganic parameters. The other water sample was a blank. The samples were to be prepared and analyzed by current IFB procedures as per contract. All laboratories received the samples single blind. Enclosed is more general information about the Superfund Performance Evaluation Program.

Thank you for your participation in this study. I trust that this information will be beneficial in your pursuit of excellence as a member of the community of laboratories analyzing hazardous waste samples.

Sincerely,

Larry Butler, Ph.D.  
Supervisor

Performance Evaluation Program  
Quality Assurance Research Branch  
Quality Assurance Methods Development Division

Enclosures

cc: (w/out enclosures)  
Mike Hurd, OERR  
Carla Dempsey, OERR  
William Langley, OERR

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ROUTINE INORGANIC SCORE SHEET

Laboratory: OAK RIDGE NATIONAL (ORNL)  
 Quarter: 4

(D)

Date: 15-Oct-87

Fiscal Year: 87

Maximum Number of Points Possible: 100

I. Sample 1, Case: 7761 - 0  
 A. Identification ( -5 Points X Number of Missed Identifications 0 ) -----  
 B. Quantitation (Points Lost) - 0.0 -----

		1.5	
	Total Number of Elements ( 17 )		X -50
	Number Missed ( 0 )		
1 -	-----		
	Total Number of Elements ( 17 )		

C. False Positives/Unmet CRDL's (-2 Points X False Positives and Unmet CRDL's 0 ) ----- 0

II. Sample 2, Case: 7761 - 0  
 A. Identification ( -5 Points X Number of Missed Identifications 0 ) -----  
 B. Quantitation (Points Lost) - 3.5 -----

		1.5	
	Total Number of Elements ( 21 )		X -50
	Number Missed ( 1 )		
1 -	-----		
	Total Number of Elements ( 21 )		

False Positives/Unmet CRDL's (-2 Points X Positives and Unmet CRDL's 0 ) ----- 0

Imprecision (Maximum of 10 Points Deducted)  
 Number of Duplicate Results Outside of ( 0 ) ----- 0

( 3 Points Deducted )  
 Matrix Spikes Outside of - 0.5 -----

Number of Points Deducted: 4.0 Points  
 Laboratory Point Score: 96.0 Points  
 Laboratory Percent Score: 96.0 %

CODED SUMMARY OF SCORES  
FOURTH QUARTER INORGANIC FY 87 DOE SINGLE BLIND  
(QB 4 FY 87, CASE NO. 7761)

---

<u>CODE</u>	<u>POINT SCORE</u>	<u>% SCORE</u>	<u>NO. OF DAYS LATE</u>	
H1	96.0	96.0	0	ORML
W1	95.5	95.5	16	
D1	81.6	81.6	35	
X1	*			
A2	*			

\* NO DATA SUBMITTED AS OF OCTOBER 23, 1987

10/21/87 Revision  
 00 & FT OF MONROE, CASE NO. 7761

ANALYSIS SHEET (logA)

C-201

PARAMETER	90 S CI 0 0		95 S CI 0 0		M1	C1	S1	E1	F1	M1	M1	J1	L1	M1	M1	M1	F1
	LOWER	UPPER	LOWER	UPPER													
ALUMINUM	1675	2199	1851	2125	1920	1940	2140	1930	1900	1970	2010	1890	1900	2050	1990	1850	1930
ANTHONY	0	76	0	82	25.5 C1	41 C1	-47 U 1	72	46.8	86	47 C1	46	37.8 U	78	0	47	42
ARSENIC	41	54	39	35	47	43	49	48 U	25.7	48	40	44	38	48	44	43	49
BARUM	397	643	584	694	650	622	650	643	630	641	650	614	644	632	621	623	609
BERYLLIUM	0	6.1	0	6.4	5.2	6 C1	4.6 C1	4.7 C1	4.4 C1	5	4.8 C1	-5 U	2.6 U	4.8 C1	6.1 U	-5 C1	2.3 C1
BROMINE	35	54	36	32	43.5	49	47	38	48	43	46	46	44.5	46	47	44	46.3
CALCIUM	20606	21644	20264	23063	22000	22500	22100	22500	21200	21100	23600	22600	22100	22000	22500	22000	22600
CHROMIUM	171	201	171	204	194	186	187	184	180	176	196	179	193	184	215 U 1	162	190
COBALT	0	0	0	0	26.2 C1	25 C1	21 C1	21 C1	20 C1	22 C1	26 C1	-50 U	25.1 U	21 C1	24 C1	-50 C1	28 C1
COPPER	127	135	124	134	140	147	139	140	136	136	140	127	134	143	123	130	133
IRON	3471	4041	3407	4146	3630	3610	3800	3920	3600	3577	3630	3560	3620	3660	3780 U	3500	3900
LEAD	152	223	143	230	153	199	203	202 U	200	171	192	182	178	179	257 U 1	170	200
MAGNESIUM	6730	8115	6394	8261	7390	7190	7230	7530	7300	7150	7200	7440	6400	7450	7600	7100	7730
MANGANESE	704	848	773	898	838	821	838	854	860	799	839	801	821	836	834 U	790	814
MERCURY	0.44	1.6	0.52	1.7	0.7	1.2	0.8	1	0.9 U	0.95	1.1	1.03	0	1.2	1.2	1.3	1.3
NICKEL	0	0	0	0	25.8 C1	25 C1	32 C1	21 C1	20 C1	27 C1	30 C1	-40 U	22.3 U	31 C1	20 C1	36	-24 U
POTASSIUM	0	0	0	0	1930 C1	2100 C1	4090 C1	2190 C1	2100 C1	2140	4200 C1	2150	2820 U	3000 C1	2310 C1	-5000 C1	2190 C1
SELENIUM	0	164	75	173	153 U	114	130 U	120 U	100.4	110	134	120	147	124	116	117 U	
SILVER	0	0	0	0	-7 U	-2 U	-4.5 U	-4 U	-5 U	-3 U	2.2 C1	-10 U	-2 U	-4 U	-9 U 1	-5 C1	-7.7 U
SODIUM	33173	40314	32410	41070	36200	37900	38500	38000	34500	3694	38000	36900	35000	36600	37200 U	35000	34500
TUNGSTEN	0	0	0	0	16.6	-10 U 1	8.1 C1	0 C1	8.3	6.6	8.7 C1	7	5.3 U	0 C1	8.3 C1	0	6.8 C1
VANADIUM	0	0	0	0	24.3 C1	20 C1	27 C1	29 C1	26 C1	27 C1	34 C1	-50 U	30.3 U	22	33 C1	-50 C1	29 C1
ZINC	525	611	516	620	582	571	548 U	606 U	540	584	564	551	592	553	554 U	510 U	538
TOTAL OUT (ACTION LIMIT)	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
TOTAL OUT (DRAWING LIMIT)	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
TOTAL OUT (IDENTIFICATION)	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
TOTAL OUT (FALSE POSITIVE)	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
TOTAL OUT (CR)	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
PARAMETERS MISSED																	
PARAMETERS MISSED																	
PARAMETERS MISSED																	
PARAMETERS MISSED																	
TOTAL OUT (CRP)	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
PARAMETERS MISSED																	
PARAMETERS MISSED																	
PARAMETERS MISSED																	
PARAMETERS MISSED																	

NOTE: A negative value followed by a "U" indicates that the value was not used in the calculation of the CI.

10/23/87 Revision  
 GR & FR BY DORCONIC, CRSE NO. 7761

ANALYSIS SAMPLE (ug/L)

PARAMETER	90 S CI 0 6		95 S CI 0 6		M1	M2	M3	T1	M4	V1	C2	M2	M2	M2	M2									
	LOWER	UPPER	LOWER	UPPER																				
ALUMINUM	1075	2101	1051	2125	1888	1960	2070	1740	X	1920	1903	2010	E	1970	1870	M	1840	X						
ANTHRACENE	0	76	0	82	41	E	-60	M	50	E	75	52	B	36.5	41	E	45.2	E	-34	M	460	E	20	E
ARSENIC	41	54	39	35	45	52	46	44	42.6	30.9	39	40.1	46	460	E	41	460	E	41					
BARITUM	507	683	588	694	645	667	682	620	628	797	X	648	E	660	673	6700	E	566	X					
BERYLLIUM	0	6.1	0	6.4	4	E	-5	M	5.2	6	3.1	E	13.0	E	4.7	E	4.52	3	E	43	E	3	E	
CADMIUM	39	51	38	52	41	51.3	50	47	44.7	49	43	44.9	39	450	E	42	E							
CALCIUM	20506	24644	20268	25063	21200	17990	24300	23400	21200	-	X	22500	22900	22000	MR	20600								
CHROMIUM	174	201	171	204	204	M	163	E	193	130	X	188	188	153	184	1800	E	169	E					
COPPER	d	d	d	d	16	E	-50	M	24	E	25	E	-21.4	M	40	23	E	16.3	16	E	230	+	18	E
COPPER	127	135	124	158	130	120	E	153	130	133	140	138	130	140	1250	E	127							
IRON	3471	4081	3407	4146	3580	3383	E	3660	3810	3640	3743	3760	3780	4010	MR	3400	X							
LEAD	152	223	145	230	183	204	201	170	176	204	154	181	209	1820	E	144	E	X						
MAGNESIUM	6729	8113	6596	8261	7110	6923	8380	E	6700	7010	-	X	7250	7290	7390	MR	7140							
MANGANESE	784	848	773	890	796	833	903	E	830	908	858	850	833	830	MR	787								
MERCURY	0.64	1.6	0.33	1.7	1.4	1	1.2	2.1	E	1.1	1.27	0.71	1.2	11.9	E	0.9								
NICKEL	d	d	d	d	44	-40	M	32	E	26	33	27	15	E	22.2	24	E	2.6	-23	M				
POTASSIUM	d	d	d	d	3080	E	-5000	M	3610	E	3160	3170	B	-	3780	3690	2800	E	MR	3100	E			
SELENIUM	86	164	75	173	122	8	120	103	130	119	126	116	123	122	1300	E	120							
SILVER	e	e	e	e	-0.7	M	-10	M	-5	M	10	E	0.33	B	0.25	-1.3	M	-10	M	-5.6	M	0.3	-5.2	M
SODIUM	33173	40304	32418	41070	33400	34250	39900	37000	34900	-	X	36000	38100	32500	MR	34200								
TALLIUM	e	e	e	e	-3.9	M	-10	M	-10	M	30	E	7	9.1	7.4	E	7	-10	M	32	+	9	E	
VANADIUM	d	d	d	d	30	E	-50	M	23	E	73	E	23.1	34.5	28	E	28.6	23	E	260	+	-39	M	
ZINC	525	611	516	620	530	490	X	612	550	542	557	562	572	555	5600	E	543							

TOTAL BLK ACTION (LMT)	0	5	2	3	0	5	0	1	1	11	5
TOTAL BLK DAMPING (LMT)	1	1	1	1	0	0	1	0	0	0	1
TOTAL BLK IDENTIFICATION	0	0	0	0	0	3	0	0	0	0	0
TOTAL BLK FALSE POSITIVES	0	0	0	0	0	0	0	0	0	1	0

TOTAL BLK CRD	2	0	0	10	2	0	1	0	2	10	0
PARAMETERS MISSED	50, 76				50, 50		50		50, 64		
PARAMETERS MISSED											
PARAMETERS MISSED											
PARAMETERS MISSED											

TOTAL BLK (DPV)	0	0	0	10	0	0	0	0	0	10	0
PARAMETERS MISSED											
PARAMETERS MISSED											
PARAMETERS MISSED											
PARAMETERS MISSED											

NOTE: A negative value followed by a "U" indicates that the value was not used in the calculation of the CI.

C-202

10/23/87 Revision  
 CB 4 FT AT BORGARIC, CASE NO. 7761

MERCURY SAMPLE (ug/L)

PARAMETER	20 F C E 0 0		20 F C E 0 0		L2	M2	M2	M2	M2	M2	M2	M2	M2	M2	M2	M2	
	LOWER	UPPER	LOWER	UPPER													
ALUMINUM	1875	2101	1851	2125	1570 R	2040	1900	1860	1830	2210	2000	2010	2030	2050	1820	2030	1930
ANTHRA	0	76	0	82	-50 U	30	70	-40 U	49 U	57 U	-20 U	50 U	47 U	30 U	36	-60 U	20 U
ARSENIC	41	54	39	35	46	49	27.9	39	32	48	47.2	61 R	48	47	48	48	73 R 1 R
BARIUM	597	685	588	694	600	672	690	680 E	651	737	683	645	644	632	606	650	629
BENTHLE	0	6.1	0	6.4	-3 U	5	5	3 U	3.9 U	5.2	3 U	3.3	1.1 U	-5 U	4	-3 U	4.3 U
CAOXA	30	51	30	52	48	45	45	40	42	57	46	46	45	42	38	45	47
CACTUM	20606	21644	20254	25063	23100	22900	14000	21700	21100	26500	23100	23300	21800	22900	19	23000	20400 ?
CHROMIUM	174	201	171	204	176	191	210	171	186	203	190	191	188	185	177	181	184
CINCL	d	d	d	d	30 U	21	-50 U	30	23 U	30	21 U	25 U	22 U	-50 U	7	-30 U	20 U
COPPER	127	135	124	138	139	134	134	141	139	161	134	144	137	142	128	148	120
LEAD	3471	4081	3487	4146	3740	3600	3400	3920	3470	4330	4060	3860	3910	3940	3300	3630 E	3500 ?
LEAD	152	223	145	230	150	171	190	188	210	186	212	220	180	178	195	170	181
MANGANESE	6730	8115	6594	8261	7190	7370	6100	7300	7270	8430	7730	7160	7160	7730	6.07	7200	6730
MANGANESE	784	848	773	898	800	826	800	832	811	1220	880	852	823	845	773	835	832
MERCURY	0.64	1.6	0.53	1.7	1.2	1	1.2	1.4	0.22	-0.1 U	0.01	1.1	1.3	1.1	1.05	0.7	1.6
NICKEL	d	d	d	d	-30 U	25	-50 U	30 U	20 U	30 U	33 U	26 U	29 U	-40 U	-25 U	27 U	28 U
POTASSIUM	d	d	d	d	3190 U	3300	3500	3680	3300 U	4010 U	3110 U	3640 U	3420 U	3200 U	2.56	3400 U	3410 U
SILICA	84	164	75	173	133	125	109	163	125	163	124	170	112	117	135	130	122
SILICA	c	c	c	c	-4 U	-5 U	-50 U	-9 U	-4 U	-4 U	-4 U	-4 U	-6 U	-10 U	-10 U	-10 U	-10 U
SODIUM	31173	40314	32410	41070	36300	37800	36000	35400	26100	41600	30100	38700	36100	37700	35.3	38700	32000
THALLIUM	c	c	c	c	-10 U	12	-50 U	-9 U	7.6 U	-7.2 U	-10 U	-10 U	-10 U	3.4 U	7.5	-10 U	-10 U
VANADIUM	d	d	d	d	-50 U	31	100	-40 U	28 U	44 U	24 U	30 U	28 U	-50 U	17	28 U	20 U ?
ZINC	325	611	516	620	538	564	530	552	570	631	562	583	564 E	581	502	530	591

C-203

TOTAL BLK (ACTION LIMIT)	1	0	2	0	2	11	1	1	0	0	0	0	0	0	0	1
TOTAL BLK (ACTION LIMIT)	1	0	1	1	1	1	0	1	0	1	0	1	0	1	2	0
TOTAL BLK (IDENTIFICATION)	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
TOTAL BLK (FALSE POSITIVE)	0	0	0	1	0	0	0	0	0	0	0	0	0	0	0	0
TOTAL BLK (AD)	1	0	0	2	0	2	5	0	0	0	0	0	0	0	0	2
PARAMETERS MISSED	Fe			As, Cd		As, Hg	As, Hg									Sb, Se
PARAMETERS MISSED							Hg, Tl									
PARAMETERS MISSED							Zn									
PARAMETERS MISSED																
TOTAL BLK (ADP)	0	0	0	2	0	1	0	0	0	0	0	0	0	0	0	1
PARAMETERS MISSED				EA, Fe		Na										As
PARAMETERS MISSED																
PARAMETERS MISSED																
PARAMETERS MISSED																

NOTE: A negative value followed by a "U" indicates that the value was not used in the calculation of the CL.

ANALYSIS SAMPLE (ug/L)

PARAMETER	30 S CI 0 b		35 S CI 0 b		E3	F3	G3	H3	I3
	LOWER	UPPER	LOWER	UPPER					
ALUMINUM	1875	2101	1851	2125	1970	2020	1777	1910	2110
ANTIMONY	0	76	0	82	-50 U	-53 U	40 CI	40 CI	46 B
ARSENIC	41	34	39	35	38	40	41	31	46
BARIUM	597	625	588	634	639	661	612	631	631
BERYLLIUM	0	6.4	0	6.4	2.3 CI	4 CI	4.4 CI	3.3	3.9 B
BROMINE	39	58	36	52	49 E	44	44.8	43	41
CADMIUM	20486	24644	20268	25063	22000	23000	21340	23800	23200
CAESIUM	174	201	171	204	187	200	173	177	198
COPPER	d	d	d	d	23 CI	-23 U	23 CI	25 CI	26 B
CHLORINE	127	125	124	150	143 E	141	134	140	150
CHROMIUM	3471	3001	3407	4146	3620	3000	3313	3600	3940
COBALT	152	223	145	230	184	180	193	161	183
DIAPHRAGM	6720	8115	6394	8261	7230 E	7100	6805	7200	7940 E
DIAPHRAGM	764	848	773	858	810	825	769	810	866
MERCURY	0.64	3.6	0.53	1.7	1.3	1.2	1.3	1.3	1.3
NICKEL	d	d	d	d	33 CI	28 CI	35 CI	-33 U	37 B
NIOTRISTIN	d	d	d	d	3040 CI	3260 CI	3157 CI	3870 CI	3000
SELENIUM	84	164	75	173	76	121	113	129	110
SILVER	e	e	e	e	-9 U	-7 U	-2.8 U	-4.9 U	-7 U
SODIUM	33173	40314	32418	41070	34300	37300	34330	36800	38100
TUNGSTEN	e	e	e	e	7 CI	9.4 CI B	-10 U	6.7 CI	7.9 B
VANADIUM	d	d	d	d	27 CI	25 CI	27.7 CI	27 CI	25 B
ZINC	525	611	516	620	585	576 E	532	540	526 E
TOTAL OUT (ACTION LIMIT)					0	0	2	0	0
TOTAL OUT (WARNING LIMIT)					2	1	0	0	2
TOTAL OUT (IDENTIFICATION)					0	0	0	0	0
TOTAL OUT (FALSE POSITIVE)					0	0	0	0	1
TOTAL OUT (SD)					0	0	0	0	1
PARAMETERS MISSED									Fe
PARAMETERS MISSED									
PARAMETERS MISSED									
PARAMETERS MISSED									
TOTAL OUT (HPO)					0	0	0	0	0
PARAMETERS MISSED									
PARAMETERS MISSED									
PARAMETERS MISSED									
PARAMETERS MISSED									

NOTE: A negative value followed by a  
 "U" indicates that the value was  
 not used in the calculation of  
 the CI.

C-204

10/23/87 Revision  
 CB 4 FT BT BOREHOLE, CASE NO. 7761

SOIL SAMPLE (mg/kg)

PARAMETER	90 S CI 0 B		90 S CI 0 B		M1	C1	C2	F1	S1	M1	J1	L1	M1	M1	M1	P1	M1
	LOWER	UPPER	LOWER	UPPER													
ALUMINUM	8540	20306	7374	21635	21700	17400	11300	32300	10310	12500	19400	7440	11200	14200	14000	3350	17300
ANTHRA	c		c		23.6	6.7 (U)	50	1.3	2.9 (U)	15	20	9.4 (U)	-23 (U)	-24 (U)	16.9	6 (U)	-4.8 (U)
ARSENIC	104	140	95	197	173	149	132	110	153	158	130	71.7	133	134	175	168 (U)	170
BARIUM	130	185	145	200	193	179	164	130	158	170	135	139	167	166	163	138	186
BERYLLIUM	2.3	4.7	3.4	6.8	4.2	4.2	4.3	3 (U)	2.8	3.9	3.6	2.3	4.1	3.8 (U)	4	3.2	4
CAESIUM	11	21	10	22	18.1	15 (U)	16	18	12.9	13	17.5	13.3	13	14 (U)	17	18	18
CALCIUM	32340	36320	31923	36743	34300	34700	33000	30300 (U)	31520	33600	34700	32100	32300	34500 (U)	33000	34000	34000
CHROMIUM	49	69	47	71	63.1	63	57	58	43.7	57	41	43.3	52	59	47	51	50
COBALT	27	37	26	39	31	37	25	34	27.2	34	30.2	23	30	33 (U)	32	36	33
COPPER	1171	1300	1157	1314	1200	1240	1250	1100 (U)	1105	1130	1130	1100	1230	1210	1090	1010	1110
IRON	20462	26300	19020	27144	24000	25300	24700	21000	18330	21200	24011	15700	21400	23300 (U)	22300	19000	23400
LEAD	62	110	56	124	64.2	91	100 (U)	79.8	79	90	77.5	76.4	86	97	73	69	94
MANGANESE	17949	22640	17443	23193	21100	23200	21300	18200	19330	19300	20900	17200	18000	20500	20000	20100	20500
MERCURY	1022	1130	1006	1173	1090	1080	1100	1100	1008	1090	1070	994	1060	1100 (U)	1015	894	1100
NICKEL	1.1	2.0	0.80	2	1.01	MS (U)	2	1.5	1.9	1.9	1.87	2.1	2	2.2	2.7	2.1	2.9
NIOB	23	39	24	41	41.2	37	30	37	27.2	30	28.9	21.1 (U)	23	35 (U)	31	30	33 (U)
POTASSIUM	480	2237	296	2490	2370	1840 (U)	964 (U)	1200	1093	1370	2064	704 (U)	596 (U)	947 (U)	1140	637 (U)	1310 (U)
SELENIUM	4.6	8.5	4.2	9	-0 (U)	6.9 (U)	6.1 (U)	7.1	4.4 (U)	8.2	12.1	2 (U)	6.0	3.9	1.7	6.4	3.8
SILVER	2.4	0	2.3	0.6	4.6	7.1	6.3	6.2	3.18	7.3	6.2	3	4.6 (U)	-4.3 (U)	-1 (U)	6.9	3.1
SODIUM	c		c		-212 (U)	208 (U)	60 (U)	-200 (U)	54 (U)	70 (U)	64.3	264 (U)	26 (U)	-2060 (U)	-1000 (U)	-211 (U)	67 (U)
THALLIUM	4.3	7.6	4.2	7.9	6.6	5.8 (U)	6.3 (U)	3.4	3.6	3.2	6.3	6.0	3.9	7.9	0	3.6	4.8 (U)
VANADIUM	30	47	20	48	53 (U)	44	36	40	23.5	30	44.2	27.3	33	37 (U)	40	27	34
ZINC	376	450	367	466	442	436	415	330	373	383	427	329	370	412 (U)	375	308	401
TOTAL DUT (ACTION LIMIT)					4	2	0	3	2	1	2	11	0	1	3	7	1
TOTAL DUT (WARNING LIMIT)					2	0	1	0	0	0	0	2	1	1	2	0	0
TOTAL DUT (IDENTIFICATION)					1	1	0	0	0	0	0	0	0	1	0	0	0
TOTAL DUT (FALSE POSITIVE)					0	0	1	0	0	0	0	0	0	0	0	0	0
TOTAL DUT (DQ)					1	2	4	3	1	1	0	4	1	1	MS	1	1
PARAMETERS MISSED					Pb	Hg, Se	Sb, Pb	Br, Hg	Ca	Sb		Sb, Se	Sb	Sb		Ag	Sb
PARAMETERS MISSED						TI	Se, TI	Se				Ag, In					
PARAMETERS MISSED																	
PARAMETERS MISSED																	
TOTAL DUT (DQ)					2	1	0	0	0	0	0	1	0	0	MS	0	0
PARAMETERS MISSED					Pb, Ag	Se						Pb					
PARAMETERS MISSED																	
PARAMETERS MISSED																	
PARAMETERS MISSED																	

C-205

NOTE: 0 negative value followed by a "U" indicates that the value was not used in the calculation of the CI.

BAZZIER Revision  
 CD 4 BY OF BORGWIC, CASE NO. 7761

SOIL SAMPLE (mg/kg)

PARAMETER	90 ± CI 0 b		95 ± CI 0 b		M1	M2	M3	V1	C2	M2	C2	M2	M2
	LOWER	UPPER	LOWER	UPPER									
ALUMINUM	8640	20385	7391	21635	13282	11700	10200	9197	13700	14300	15300		MR 12500
ANTHRA	c				1.19	17	7.9 M	9.8	15	-52 M	-10 M	40	-4.6 M
ARSENIC	104	160	95	197	139.8	128	158	140	109	168	168	174	222 X
BARIUM	130	195	145	290	161.3	163	166	161	166	179	178	186	154
BERYLLIUM	3.5	4.7	3.4	4.8	2.66	3.3	4.9 E X	3.8 X	4 E	3.97	3.8	4.1	1.8 X
CHROMIUM	11	21	10	22	14.48	21	13.8	16.2	14	13.1	18	15	14
CAESIUM	32348	36320	31923	36745	21721	X 34200	33008	-	X 31800	X 36000	35500	MR	31600 X
CADMIUM	49	69	47	71	55.92	52	60	49.2	53	55.2	54	56	52
COPPER	27	37	26	39	32.95	34 E	34.8	24 X	33	32.7	29	25	28
COPPER	1171	1300	1157	1316	1094	X 1250	1190	1259	1190	1250	1260	1100	X 1150 X
IRON	20462	26502	19820	27144	23118	21600 E	23300	22650	21800	23000	25400 E	MR	21300
LEAD	62	110	56	124	90.38	88	88.7	87	66	87.1	83	88	81.5
MANGANESE	17949	22648	17443	23193	20373	21200	19300	-	X 19000	21000	20500	MR	19500
MANGANESE	1022	1150	1008	1173	975	X 1070 E	1080	1120	1030	1090	1160	MR	1030
MERCURY	1.1	2.0	0.88	2	2.63	2.1	2.2	1.62	2.2	0.975	2.4	3.8	2.2
NICKEL	25	38	24	41	37.95	29	30.3	24.7	28	30.4	27	32	26
POTASSIUM	488	2227	256	2190	1453	1060	900 M	-	X 1300	1250	1340	MR	1200
SELENIUM	4.6	8.5	4.2	9	3.94	X 5.7	3.8	6.7	5.2	5.53 E	3.2 E X	8	7.7 M
SILVER	2.4	8	2.9	8.6	3.48	3.4	4.2	4.8	-0.3 M X	2.82 X	4.2	5	6.3
SODIUM	c				118	107 M	58.6 M	-	-9 M	-1000 M	-68 M	MR	313
THALLIUM	4.5	7.6	4.2	7.9	3.19	3.3 M	3.4	3.9	4.4	5.96	4.9	6.2	7.3
Vanadium	30	47	28	48	37.95	33	32.8 E	38	31	34.2	37	53	36
ZINC	376	458	367	466	371	403	384	411	391	398	424 E	398	387
TOTAL BUT (ACTION LIMIT)					4	0	1	5	2	1	1	2	4
TOTAL BUT (WARNING LIMIT)					1	0	0	0	1	0	1	0	0
TOTAL BUT (IDENTIFICATION)					0	0	0	3	1	0	0	0	0
TOTAL BUT (FALSE POSITIVE)					0	0	0	0	0	0	0	0	0
TOTAL BUT (SD)					0	2	0	0	2	7	1	MR	12
PARAMETERS MISSED					Sn, Tl				Hg, Zn	Sb, As			Sb, As, Se
PARAMETERS MISSED									Cd, Ni				Cd, Cr, Co
PARAMETERS MISSED									Sr, Ag				Hg, Ni, Se
PARAMETERS MISSED									Tl				Hg, V, Zn
TOTAL BUT (SD)					0	3	0	0	0	0	0	MR	10
PARAMETERS MISSED					Al, Hg								Al, As, Cd
PARAMETERS MISSED					Tl								Cd, Cr, Co
PARAMETERS MISSED													Fe, Hg, Ni
PARAMETERS MISSED													Zn

C-206

NOTES: A negative value followed by a "U" indicates that the value was not used in the calculation of the CL.

SOIL SAMPLE (mg/kg)

PARAMETER	90 5 CI 0 0		93 5 CI 0 0		12	12	12	12	12	12	12	12	12	12	12	12	
	LOWER	UPPER	LOWER	UPPER													
ALUMINUM	6540	20386	7391	21633	10200	11900	12500	18600	13600	13030	13700	10200	12600	16300	15400	17400	10600 ?
ANTHRACENE	a		c		-30 U	8.5	15	-20 U	2.4 U	-11 U	-25 U	20 U	-16 U	10 U	-	-30 U	16
ARSENIC	104	180	95	197	135	167	562	124 U	156	104	139	112	118	140	184	164	95 U ?
BARIUM	150	195	145	200	152	163	190	204 U	172	183	176	150	171	182	176	194	163
BENZ[a]ANTHRAcene	2.5	4.7	2.4	4.8	4.9	4	2.2	4	2.8	2.4	2.52	2.9	6.4	2.8	7	4	4.1 ?
CHLORINE	11	21	10	22	13	14	12.6	13	14	21	16.6	14	16	15	16	21	16
CALCIUM	32140	36320	31923	36743	26100	33800	21000	32300	32800	29100	34400	37300	34100	35700	16350	34100	26000 ?
CADMIUM	49	69	47	71	35	34	58	33 U	35	41	59.4	53	60	57	64	72	53
COPPER	27	37	26	30	23	22	22	50 U	23	34	26.6	24	33	26	40	32	24
CHROMIUM	1171	1300	1157	1314	1200	1130	1090	1260 U	1170	1250	1240	1220	1230	1270	1170	1260	1180
IRON	20462	26502	19020	27144	22700	23200	15200	23300	22000	23400	24300	22500	22800	24400	23400	25500	22200
LEAD	62	110	36	124	70	70	82	86 U	81	111	104 U	87	84	84	110	129	89
MANGANESE	17910	22640	17443	23183	19100	19500	16800	22300 U	19000	19000	20200	21300	20200	21800	17600	20700	18400
MERCURY	1022	1150	1000	1173	1020	1071	1000	1150	1060	1110	1104	1070	1100	1140	1000	1130	1000
NICKEL	1.1	2.0	0.80	2	1.7	2.1	1.9	2.6	1.93	1.8	13.4 U	2	2	1.9	1	2.3	2.2 ?
NITROGEN	23	30	24	41	-15 U	25	20	25	30	29	36.2	31	32	34	47	29	30
POTASSIUM	440	2297	296	2190	1220 U	1020	1120	3660 U	1050	1200 U	1210 U	1120 U	1020 U	1690	-	1740 U	913 U
SELENIUM	4.6	6.5	4.2	9	7 U	2.6	30.1	8	13	8	6.18 U	4.7 U	5.5 U	5.3 U	4.2	8	2.7 U
SILVER	2.4	6	2.9	6.6	-2 U	6	-5 U	-4 U	6.3	-2 U	-4 U	6.5 ?	6.2	6.1	-	7	2.3
SODIUM	a		c		222 U	-200 U	64	306 U	47 U	17 U	-120 U	664 U	-21 U	-620 U	760	-747 U	-159 U ?
THALLIUM	4.5	7.6	4.2	7.9	6.6 U	5.6	12	7	7.3	6.6	6.90	5.6	-2.1 U	2.2	-	7.3	5.4
ZINC	30	47	20	40	37 U	41	40	45	40	43	35.2	35	37	43	-	47	34
ZINC	376	450	367	466	404	375	300	447	390	427	424	301	404	427	426	425	463 U ?

C-207

TOTAL CHL (DICTION LIMIT)	3	3	10	3	1	3	2	1	1	0	1	1	0	10	2	0
TOTAL CHL (RATING LIMIT)	1	1	2	1	1	1	0	0	0	0	0	0	0	3	0	3
TOTAL CHL (IDENTIFICATION)	2	0	1	1	0	1	1	0	1	0	1	0	1	5	0	0
TOTAL CHL (PULSE POSITIVE)	0	0	0	1	0	0	0	0	0	0	1	0	0	0	0	0
TOTAL CHL (SD)	3	0	0	2	1	2	1	2	1	2	2	2	2	0	2	2
PARAMETERS MISSED	Sn, Pb			Sb, Co	Bi	As, Ag	Sr	Sr, Se	Sr	Sr, Se	Sr, Se	Sr, Se	Sr, Se		Sr, Cd	Sr, Se
PARAMETERS MISSED	Cr, Fe															
PARAMETERS MISSED	Zn															
PARAMETERS MISSED																
TOTAL CHL (APD)	0	0	0	3	0	0	2	0	0	2	0	0	0	0	0	3
PARAMETERS MISSED				Cd, Cr			As, Se									As, Se
PARAMETERS MISSED				Hg												Bi
PARAMETERS MISSED																
PARAMETERS MISSED																

NOTE: A negative value followed by a "U" indicates that the value was not used in the calculation of

10/23/87 Revision  
 DD 4 FT BT BARRAGE, CASE NO. 7764

SOIL SAMPLE (mg/kg)

PARAMETER	30 S CT 0 0		35 S CT 0 0		E3	F3	G3	H3	J3
	LOWER	UPPER	LOWER	UPPER					
ALUMINUM	8640	20386	7391	21635	15200	19400	13232	14400	18800
ANTIMONY	c		c		14	-25 B	24 C	43 E	12 S
ARSENIC	104	188	95	197	161	156	123.2	184	157
BARIUM	150	335	145	200	171	189	169	167	186
BERYLLIUM	2.5	4.7	2.4	4.8	4.3	3.9	4.1	4.8	4.1
CADMIUM	11	21	10	22	13	10	13.9	13	14
CALCIUM	22348	36328	31923	36745	33400	36200	31975	33200	35300
CHROMIUM	49	69	47	71	58	68	58	54	63
COBALT	27	37	26	39	30 E	29	35	32	35
COPPER	1171	1308	1157	1314	1190	1270	1203	1160	1270
IRON	20482	26502	19828	27144	22000	23500	22973	21900	25100
LEAD	62	118	56	124	93 E	81 B	83.9	78	88
MANGANESE	17949	22649	17448	23193	20300	21900	19843	19800	21300
MERCURY	1.1	2.8	0.88	3	2.3	1.8	2.1	2.4	0.8 B
NICKEL	23	39	24	41	30	32	29	30	36
POTASSIUM	488	2297	296	2190	1280	2008 C	1318 C	1060 E	2280
SELENIUM	4.6	8.9	4.2	9	2.5 B A X	6.8	3.4	5	3.7 B
SILICON	2.4	8	2.9	8.6	3	8.3	7.6	5.3	7
SODIUM	c		c		-528 U	109 C	64 C E	73 C	71 B
THALLIUM	4.9	7.6	4.2	7.9	6.6	6.6	3	3.3	3.8
ZINC	30	47	28	48	42 E	46	40	32	43
ZINC	376	458	367	466	404	446	412	384	437

C-208

TOTAL DAT ACTION (LMT)	1	0	0	0	1
TOTAL DAT BANNING LIMIT	0	2	0	2	0
TOTAL DAT IDENTIFICATION	0	0	0	0	0
TOTAL DAT FALSE POSITIVE	0	0	0	0	0
TOTAL DAT CND	3	1	2	1	2
PARAMETERS MISSED	Se, Co	Se	Se, Se	Se	Se, Se
PARAMETERS MISSED	Se				
PARAMETERS MISSED					
PARAMETERS MISSED					
TOTAL DAT (DP)	0	1	1	0	0
PARAMETERS MISSED		Pb	Se		
PARAMETERS MISSED					
PARAMETERS MISSED					
PARAMETERS MISSED					

NOTE: A negative value followed by a "U" indicates that the value was



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
OFFICE OF RESEARCH AND DEVELOPMENT  
ENVIRONMENTAL MONITORING SYSTEMS LABORATORY-LAS VEGAS  
P.O. BOX 93478  
LAS VEGAS, NEVADA 89193-3478  
(702/798-2100 - FTS 545-2100)

AUG 08 1988

Mr. William Laing  
Oak Ridge National Laboratory  
P.O. Box 2008, 4500s, MS-127  
Oak Ridge, TN 37831

Dear Mr. Laing:

The Individual Laboratory Summary Report (ILSR) summarizing the results of the participation of your laboratory in the EMSL-LV third quarter organic performance evaluation study (QB3, FY88) is enclosed. In addition, general information concerning the scoring procedure used for QB3 is included.

The score for your laboratory at 78.7 is in the CLP category of acceptable but with a response required regarding any explanations of deficiencies and the changes or actions taken to correct those deficiencies. (Score is less than 90 but 70 or above).

This office will be glad to furnish any counsel and further information regarding this work.

Sincerely,

A handwritten signature in cursive script that reads "Harold A. Vincent".

Harold A. Vincent  
Chemist

Quality Assurance Research Branch, QAD

Enclosures

cc:

D. Karen Knight, DOE HQ

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ORGANIC PERFORMANCE EVALUATION SAMPLE  
INDIVIDUAL LABORATORY SUMMARY REPORT  
FOR Q3 3 FY 88

LABORATORY: Oak Ridge National (TN)  
PERFORMANCE: ACCEPTABLE - Response Explaining Deficiency(ies) Required  
RANK: Above = 42 Same = 0 Below = 24

I SCORE: 78.7  
REPORT DATE: 07/07/88  
MATRIX: WATER

COMPOUND	CONFIDENCE INTERVALS				LABORATORY		#LABS NOT-ID	PROGRAM #LABS MIS-QUANT	DATA #LABS CONTAM	TOTAL #LABS
	WARNING LOWER	UPPER	ACTION LOWER	UPPER	DATA CONC	Q				
<b>TCL VOLATILE</b>										
METHYLENE CHLORIDE	NU	NU	NU	NU	180		0	0	0	66
ACETONE	78	190	62	200	130		1	9	0	66
CARBON DISULFIDE	110	200	100	210	160		0	13	0	66
1,1-DICHLOROETHENE	110	180	100	180	160		0	7	0	66
1,1-DICHLOROETHANE	130	170	120	180	150		1	6	0	66
1,2-DICHLOROETHENE (TOTAL)	110	170	100	180	160		1	3	0	66
CHLOROFORM	120	160	120	170	150		0	7	0	66
1,2-DICHLOROETHANE	130	170	120	170	140		0	4	0	66
2-BUTANONE	85	190	70	200	160		4	5	0	66
1,1,1-TRICHLOROETHANE	120	170	120	180	150		0	7	0	66
CARBON TETRACHLORIDE	110	170	98	180	160		0	5	0	66
VINYL ACETATE	NU	NU	NU	NU	10	U	0	0	0	66
BROMODICHLOROMETHANE	130	170	120	180	150		0	2	0	66
1,2-DICHLOROPROPANE	140	180	140	180	170		0	9	0	66
CIS-1,3-DICHLOROPROPENE	76	140	67	170	190	X	12	5	0	66
TRICHLOROETHENE	120	170	120	170	170		0	8	0	66
DIBROMOCHLOROMETHANE	140	180	130	190	160		0	9	0	66
1,1,2-TRICHLOROETHANE	130	170	120	170	150		0	5	0	66
BENZENE	120	160	120	160	150		0	8	0	66
TRANS-1,3-DICHLOROPROPENE	NU	NU	NU	NU	98		0	0	1	66
BROMOFORM	130	190	120	200	160		0	5	0	66
2-PENTANONE, 4-METHYL-	92	160	82	170	140		1	7	0	66
2-HEXANONE	63	140	52	150	130		1	6	0	66
TETRACHLOROETHENE	100	140	94	160	140		1	5	0	66
TOLUENE	120	160	120	160	160		0	7	0	66
1,1,2,2-TETRACHLOROETHANE	110	160	110	170	140		1	5	0	66
CHLOROBENZENE	130	160	120	170	160		0	3	0	66
ETHYL BENZENE	100	140	97	160	150	S	0	3	0	66
STYRENE	86	150	77	150	150		0	4	0	66
XYLENES (TOTAL)	120	160	110	170	170	S	1	8	0	66
<b>TCL SEMIVOLATILE</b>										
PHENOL	10	42	10	61	7		0	0	0	66
2-CHLOROPHENOL	24	45	21	56	36		0	6	0	66
BENZYL ALCOHOL	NU	NU	NU	NU	20	U	0	0	0	66
2-METHYLPHENOL	22	40	19	50	32		1	0	0	66
4-METHYLPHENOL	20	42	17	53	31		3	4	0	66
2-NITROPHENOL	22	45	19	50	34		0	6	0	66
2,4-DIMETHYLPHENOL	16	30	13	50	26		0	3	0	66
2,4-DICHLOROPHENOL	26	40	23	51	44		1	5	0	66
4-CHLORO-3-METHYL PHENOL	27	48	24	52	38		1	6	0	66
2,4,5-TRICHLOROPHENOL	100	200	89	210	180		1	5	0	66
2-CHLORONAPHTHALENE	25	45	22	55	25		0	4	0	66
3-NITROANILINE	50	120	50	130	100	U	0	4	0	66
4-NITROPHENOL	NU	NU	NU	NU	14		0	0	0	66
4,6-DINITRO-2-METHYLPHENOL	81	160	69	180	170	S	3	7	0	66
N-NITROSDIPHENYLAMINE	52	120	42	140	94		0	5	0	66
HEXACHLOROBENZENE	22	40	18	52	83	X	2	9	0	66
PENTACHLOROPHENOL	NU	NU	NU	NU	51		0	0	0	66
DI-N-BUTYLPHTHALATE	NU	NU	NU	NU	20	U	0	0	0	66
FLUORANTHENE	NU	NU	NU	NU	11		0	0	0	66
BENZO(A)ANTHRACENE	NU	NU	NU	NU	8		0	0	0	66
BENZO(B)FLUORANTHENE	34	110	24	150	110		0	2	0	66
BENZO(K)FLUORANTHENE	40	110	30	120	99		2	3	0	66
BENZO(A)PYRENE	40	110	30	150	99		0	1	0	66
INDENO(1,2,3-CD)PYRENE	28	100	18	140	110	S	1	0	0	66

ORGANIC PERFORMANCE EVALUATION SAMPLE  
INDIVIDUAL LABORATORY SUMMARY REPORT  
FOR Q3 3 FY 88

LABORATORY: Oak Ridge National (TN)  
PERFORMANCE: ACCEPTABLE - Response Explaining Deficiency(ies) Required  
RANK: Above = 42 Same = 0 Below = 24

% SCORE: 78.7  
REPORT DATE: 07/07/88  
MATRIX: WATER

COMPOUND	CONFIDENCE INTERVALS				LABORATORY		#LABS NOT-ID	PROGRAM #LABS MIS-QUANT	DATA #LABS CONTAM	TOTAL #LABS
	WARNING LOWER	UPPER	ACTION LOWER	UPPER	DATA CONC	Q				
DIBENZ(A,H)ANTHRACENE	NU	NU	NU	NU	19		0	0	0	66
BENZO(G,H,I)PERYLENE	NU	NU	NU	NU	21		0	0	0	66
TCL PESTICIDES										
ALPHA-BHC	NU	NU	NU	NU	0.05	U	0	0	1	66
DELTA-BHC	NU	NU	NU	NU	0.05	U	0	0	1	66
HEPTACHLOR EPOXIDE	0.100	0.29	0.071	0.32	0.17		10	4	0	66
4,4'-DDE	NU	NU	NU	NU	0.1	U	0	0	2	66
ENDOSULFAN II	NU	NU	NU	NU	0.1	U	0	0	0	66
METHOXYCHLOR	NU	NU	NU	NU	0.52		0	0	0	66
ALPHA-CHLORDANE	0.82	2.8	0.54	3.1	19	X	7	2	0	66
GAMMA-CHLORDANE	0.53	1.5	0.5	1.7	9.4	X	1	6	0	66
AROCLOR-1016	NU	NU	NU	NU	0.5	U	0	0	0	66
AROCLOR-1260	NU	NU	NU	NU	3		0	0	1	66
NON-TCL VOLATILE										
ETHER, 2-CHLORO-ETHYL-VINYL					55		15	0	0	66
METHANE, TRICHLORO-FLUORO-					0		13	0	0	66
NON-TCL SEMIVOLATILE										
BENZOPHENONE					130		9	0	0	66
META-PICOLINE					19		0	0	0	66
TCL SEMIVOLATILE (Contaminants)										
BIS(2-ETHYLHEXYL)PHTHALATE					2	C0	0	0	1	66
NON-TCL SEMIVOLATILE (Contaminants)										
PHENOL, DICHLORO-METHOXY-					3		0	0	0	66
HEXANONE, METHYL-					64	C	0	0	0	66
PESTICIDE					14	C	0	0	0	66
NON-TCL SEMIVOLATILE (Contaminants)										
UNKNOWN					3		0	0	12	66
UNKNOWN					5		0	0	11	66
UNKNOWN					4		0	0	7	66
UNKNOWN					19	C	0	0	3	66

# OF TCL COMPOUNDS NOT-IDENTIFIED: 0  
 # OF TCL COMPOUNDS MIS-QUANTIFIED: 4  
 # OF TCL CONTAMINANTS: 0  
 # OF NON-TCL COMPOUNDS NOT-IDENTIFIED: 1  
 # OF NON-TCL CONTAMINANTS: 3

**OAK RIDGE NATIONAL LABORATORY**

OPERATED BY MARTIN MARIETTA ENERGY SYSTEMS, INC.

POST OFFICE BOX 2008  
OAK RIDGE, TENNESSEE 37831

November 4, 1988

Vincent Fayne  
USDOE  
Forrestal Bldg, EH-24  
Independence Ave., SW  
Washington, DC 20585

Harold Vincent  
EMSL-LV  
P. O. Box 93478  
Las Vegas, NV 89193-3478

Gentlemen:

Attached is the ORNL response to the QB3 organic performance evaluation report. Please contact John Caton (615/574-4861) if you have any questions.

Sincerely,



W. R. Laing  
ACD Task Leader

WRL:lp

attachment

cc: R. B. Fitts  
W. D. Shults

## Internal Correspondence

MARTIN MARIETTA ENERGY SYSTEMS, INC.

November 2, 1988

W. R. Laing, 4500S, MS-6127

Response to Score for Organic Analyses for 3rd Quarter (FY 1988) PE Samples

Our score for the 3rd quarter organic performance evaluation study (QB3, FY88), was 78.7. Points were deducted because 4 TCL compounds (2 pesticides, 1 volatile, and 1 semivolatile) were mis-quantified (12.5 points); one non-TCL compound was not identified (2.2 points); and 3 non-TCL contaminants were found in the prepared sample (6.6 points). Corrective actions will include the following:

1. Purchase and installation of a high temperature oven to remove all traces of chromatographable organics from preparation glassware. The three contaminants coupled with the fact that all mis-quantified compounds were high indicates "too much" has been recovered. Some parts of the preparation glassware such as continuous extractors, snider columns, etc., contain parts which can be washed only by soaking and rinsing. Therefore, trace residuals might remain especially if the equipment had previously been used for highly contaminated samples; (and we had just completed preparation of a series of samples containing high levels of chlorocarbons immediately preceding receipt of the third quarter PE).
2. Personnel will receive more training. This training will include continuing emphasis on the care, handling, and preparation of both samples and standards. In addition, two staff members were sent to training courses concerning the use and operation of gas chromatograph/mass spectrometers.
3. Special emphasis will be placed on upgrading the capabilities of the pesticide analysis effort. There have been some significant personnel changes in this area. Emphasis will be on careful training; and for the near future, some of the automatic data handling capabilities will be abandoned so that the newer personnel in this effort will gain a better understanding of data interpretation and calculations.



John E. Caton, 4500S, MS-6120 (4-4861)

JEC:llc

cc: M. R. Guerin  
M. P. Maskarinec



MAY 16 1988 Pam  
EJH

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
OFFICE OF RESEARCH AND DEVELOPMENT  
ENVIRONMENTAL MONITORING SYSTEMS LABORATORY-LAS VEGAS  
P.O. BOX 93478  
LAS VEGAS, NEVADA 89193-3478  
(702/798-2100 - FTS 545-2100)

Mr. John E. Caton  
Oak Ridge Nat. Lab  
Bldg 4500-S, MS-120  
Bethel Valley Rd.  
Oak Ridge, TN 37831-6120

Dear Mr. Caton:

For your information and review the results for your participation in the EMSL-LV Second Quarter Organic Performance Evaluation Study (QB2, FY 88) are included here. Enclosed is general information about the Superfund Performance Evaluation Program. The PE portion of the Laboratory Profile Package, called the "Individual Laboratory Summary Report" (ILSR) was described in your letter reports last quarter. Other general information about the PE program is explained on the following pages.

The samples consisted of aqueous materials spiked with Target Compound List (TCL) and non-TCL pollutants at environmentally representative levels. Samples for all laboratories were from the same homogeneous batch. Each sample set was to be prepared and analyzed by current contractually required procedures.

The EMSL-LV thanks you for your participation in this study and wishes to congratulate the laboratories for an overall fine performance. We trust that this information is vital to you as a member of the community of laboratories analyzing hazardous waste samples for Superfund.

Sincerely,

Larry Butler, Ph.D.  
Supervisor, Performance Evaluation Program  
Quality Assurance Research Branch  
Quality Assurance and Methods Development Division

Enclosure

cc: (w/enclosure)  
Carla Dempsey, OERR  
Joan Fisk, OERR  
Emilia Boulos, OERR  
Angelo Carasea, OERR  
Howard Fribush, OERR

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ORGANIC PERFORMANCE EVALUATION SAMPLE  
INDIVIDUAL LABORATORY SUMMARY REPORT  
FOR Q3 2 FY 88

LABORATORY: Oak Ridge National (TN)  
PERFORMANCE: UNACCEPTABLE - Corrective Actions Mandatory  
RANK: Above = 44 Same = 0 Below = 7

X SCORE: 62.3  
REPORT DATE: 4/5/1988  
MATRIX: WATER

COMPOUND	90 % CI		LABORATORY DATA CONC	Q	#LABS NOT-ID	PROGRAM #LABS MIS-QUANT	DATA #LABS CONTAM	TOTAL #LABS
	LOWER	UPPER						
<b>TCL VOLATILE</b>								
BROMOMETHANE	64	240	195		0	2	0	51
METHYLENE CHLORIDE	c	c	79		0	0	0	51
1,1-DICHLOROETHANE	34	55	38		0	3	0	51
2-BUTANONE	38	170	67		3	7	0	51
BROMODICHLOROMETHANE	59	80	63		0	3	0	51
1,1,2-TRICHLOROETHANE	54	76	60		0	8	0	51
BENZENE	12	17	14		1	5	0	51
2-HEXANONE	48	200	84		1	3	0	51
TOLUENE	18	30	20		0	2	0	51
CHLOROBENZENE	85	110	91		0	3	0	51
STYRENE	80	110	90		0	6	0	51
XYLENES (TOTAL)	120	180	131		0	5	0	51
<b>TCL SEMIVOLATILE</b>								
2-CHLOROPHENOL	23	52	30		0	5	0	51
N-NITROSO-DI-N-PROPYLAMINE	45	84	55		0	6	0	51
ISOPHORONE	65	140	66	S	0	5	0	51
2,4-DIMETHYLPHENOL	10	53	21	J	0	2	0	51
BENZOIC ACID	50	200	160		0	7	0	51
HEXACHLOROCYCLOHEPTADIENE	61	160	62	0	0	2	0	51
2-METHYLNAPHTHALENE	20	55	24		0	3	0	51
2,4,6-TRICHLOROPHENOL	55	100	70		0	0	0	51
2-NITROANILINE	50	100	53		0	2	0	51
ACENAPHTHYLENE	59	100	62	0	0	0	0	51
ACENAPHTHENE	61	100	59	X	0	4	0	51
2,4-DINITROPHENOL	81	260	170		3	7	0	51
DIBENZOFURAN	96	160	92	X	0	6	0	51
4-NITROPHENOL	50	200	160		0	1	0	51
FLUORENE	64	100	58	X	0	4	0	51
DIETHYLPHTHALATE	c	c	22	U	0	0	0	51
PENTACHLOROPHENOL	74	230	150		0	6	0	51
PHENANTHRENE	62	100	58	X	0	5	0	51
ANTHRACENE	57	100	55	X	0	4	0	51
PYRENE	42	110	39	X	0	6	0	51
BUTYL BENZYL PHTHALATE	c	c	22	U	0	0	0	51
BENZO(A)ANTHRACENE	31	100	27	X	0	2	0	51
DI-N-OCTYL PHTHALATE	10	100	6	J	0	2	0	51
DIBENZO(A,H)ANTHRACENE	17	140	17	J	0	2	0	51
<b>TCL PESTICIDES</b>								
HEPTACHLOR	0.05	0.43	0.47	X	0	0	0	51
ALDRIN	0.14	0.53	0.15	0	18	5	0	51
ENDRIN	0.16	0.48	0.32		2	11	0	51
TOXAPHENE	c	c	1	U	0	0	1	51
<b>NON-TCL SEMIVOLATILE</b>								
BENZOPHENONE			56		0	0	0	51
DISULFOTON			20	J	0	0	0	51
CHLORPYRIFOS			10	J	0	0	0	51
2-NITRO-P-CRESOL			36		0	0	0	51
<b>TCL SEMIVOLATILE (Contaminants)</b>								
BENZYL ALCOHOL			8	J	0	0	0	51

ORGANIC PERFORMANCE EVALUATION SAMPLE  
 INDIVIDUAL LABORATORY SUMMARY REPORT  
 FOR Q3 2 FY 88

LABORATORY: Oak Ridge National (TN)  
 PERFORMANCE: UNACCEPTABLE - Corrective Actions Mandatory  
 RANK: Above = 44 Same = 0 Below = 7

SCORE: 62.3  
 REPORT DATE: 4/5/1988  
 MATRIX: WATER

COMPOUND	90 % CI		LABORATORY		#LABS NOT-ID	PROGRAM #LABS MIS-QUANT	DATA #LABS CONTAH	TOTAL #LABS
	LOWER	UPPER	CONC	Q				
BIS(2-ETHYLHEXYL)PHTHALATE			12	JB	0	0	1	51
NON-TCL SEMIVOLATILE (Contaminants)								
2-HEXANONE, 5-METHYL-			42	B	0	0	0	51
UNKNOWN			12	JF	0	0	19	51
UNKNOWN			32	F	0	0	10	51

# OF TCL COMPOUNDS NOT-IDENTIFIED: 0  
 # OF TCL COMPOUNDS MIS-QUANTIFIED: 0  
 # OF TCL CONTAMINANTS: 0

# OF NON-TCL COMPOUNDS NOT-IDENTIFIED: 0  
 # OF NON-TCL CONTAMINANTS: 2

OAK RIDGE NATIONAL LABORATORY  
OPERATED BY MARTIN MARIETTA ENERGY SYSTEMS, INC.

POST OFFICE BOX X  
OAK RIDGE, TENNESSEE 37831

May 18, 1988

Harold Vincent  
EMSL-LV  
P. O. Box 93478  
Las Vegas, NV 89193-3478

Dear Harold:

Attached is the letter from Mike Guerin on corrective actions resulting from the QBII performance evaluation sample report. The QBIII sample is almost completed and will be sent to you soon.

Sincerely,



W. R. Laing  
ACD Task Leader

WRL:lp

cc: Karen Knight  
R. B. Fitts

## Internal Correspondence

MARTIN MARIETTA ENERGY SYSTEMS, INC.

May 17, 1988

W. B. Laing

Corrective Action Re OB2 FY88 Performance Evaluation Sample

We are taking the following steps as corrective actions.

1. No DOE Site Survey Samples are currently being analyzed for PCB-pesticides, VOA, or SVO. Samples for these analyses will not be accepted without approval of the ORNL Program Office.
2. The current quarterly Performance Evaluation Sample is being analyzed.
3. Weekly internal quality control samples are being analyzed for PCB-pesticides, VOA, and SVO analytes.

The results will be documented and will be used to design remedial action experiments if the results are found suspect.



M. R. Guerin, 4500-S, MS 120 (4-4862)

MRG:pmt

cc: J. E. Caton  
R. M. Edwards  
G. S. Fleming  
S. H. Harmon  
J. A. Hayden  
G. M. Henderson  
C. A. Treese



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
OFFICE OF RESEARCH AND DEVELOPMENT  
ENVIRONMENTAL MONITORING SYSTEMS LABORATORY-LAS VEGAS  
P.O. BOX 93478  
LAS VEGAS, NEVADA 89193-3478  
(702/798-2100 - FTS 545-2100)

DEC 16 1987

Mr. William R. Laing  
Oak Ridge National Laboratory  
Building 4500 S. MS-131  
Oak Ridge, TN 37831-6107

Dear Bill:

The results of the analyses for the water pollution sample, WP-019, are complete. Comparison sheets are enclosed showing the true values, acceptance limit ranges, warning limit ranges, and the values your laboratory obtained. Values for analytes present in the samples in determined quantities, but not generally determined in this DOE exercise, are also included. These latter values may be ignored or used for whatever purpose your laboratory can find.

Most of the analytical determinations done by the participating DOE laboratories were good. Your laboratory did extremely well and completed determinations for many of the optional analytes. Not all were perfect, and we can still learn from this performance evaluation exercise. Determinations by the ORNL laboratory of the metals on sample vials 1 & 2 were very good. Values your laboratory measured for metals on vials 3 & 4 were off from the true values by a factor of 2 in each case. Values for total dissolved solids were high in each case and should be investigated. Values for non-filterable residue were slightly high, but do not seem to pose a serious problem.

I congratulate you and your laboratory on doing a fine job in this exercise and hope we can continue to rely on your laboratory furnishing the DOE environmental survey with high-quality analytical information.

Sincerely,

A handwritten signature in black ink that reads "Harold A. Vincent".

Harold A. Vincent

Chemist, Quality Assurance Research Branch  
Quality Assurance and Methods Development Division

Enclosure

cc:(w/o enclosure)  
D. Karen Knight, DOE HQ

LABORATORY: ORNL  
SUMMARY OF DOE SURVEY - LABORATORY SUPPORT (WPO19)

Parameter	Sample Number	True Value	Reported Value	Acceptance Limits	Warning Limits
pH Units	3	4.00	3.97	3.93 - 4.09	3.95 - 4.07
	4	9.19	9.18	8.86 - 9.40	8.93 - 9.33
Spec. Cond.	1	659	675	592. - 732.	610. - 714.
	2	272	279	245. - 302.	252. - 295.
Total Diss. Sol.	1	399	489**	325. - 482.	344. - 462.
	2	158	286**	95.9 - 217.	111. - 202.
Total Hardness	1	159.5	163	151. - 174.	154. - 172.
	2	73.5	74.6	65.1 - 82.9	67.3 - 80.7
Total Alkalinity	1	55.0	56.0	49.0 - 60.4	50.4 - 59.0
	2	7.49	6.50	4.71 - 11.6	5.57 - 10.8
Chloride	1	113	117	106. - 128.	108. - 125.
	2	52.1	52.8	47.1 - 57.1	48.3 - 55.9
Flouride	1	2.01	1.97	1.74 - 2.23	1.80 - 2.17
	2	0.247	0.285	.155 - .337	.178 - .314
Sulfate	1	74.0	73.3	60.7 - 85.5	63.8 - 82.4
	2	33.0	31.6	24.5 - 39.4	26.3 - 37.5
Ammonia <sup>-</sup> N	1	0.800	0.823	.538 - 1.09	.605 - 1.03
	2	3.00	3.19	2.33 - 3.58	2.48 - 3.43
Nitrate <sup>-</sup> N	1	0.500	0.496	.383 - .614	.411 - .586
	2	2.00	2.15	1.59 - 2.38	1.68 - 2.28
Ortho <sup>-</sup> P	1	0.080	0.081	.0454- .108	.0529- .100
	2	0.800	0.816	.682 - .904	.708 - .877
TOC	1	59.2	58.0	46.8 - 74.3	50.4 - 70.7
	2	109	107	86.8 - 128.	92.2 - 122.
Total CN	1	0.124	0.130	.0687- .161	.0805 - .149
	2	0.300	0.307	.174 - .388	.201 - .361
Non-Filt. Res.	1	69.4	73.0*	61.1 - 73.6	62.6 - 72.0
	2	24.7	27.3**	20.5 - 27.2	21.3 - 26.4
Oil and Grease	1	35.3	35.8	20.9 - 43.0	23.7 - 40.3
	2	12.8	12.8	3.99 - 18.1	5.74 - 16.3

NR - Not reported.  
\*Outside warning limits.  
\*\*Outside acceptance limits.

PERFORMANCE EVALUATION REPORT

DATE: 11/16/87

WATER POLLUTION STUDY NUMBER WP019

LABORATORY: ORNL

=====

ANALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
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=====

TRACE METALS IN MICROGRAMS PER LITER:

ALUMINUM	1	87.2	78.0	49.5 - 148.	62.0 - 136.	
	2	828	858	658. - 1050.	707.- 997.	
ARSENIC	1	24.8	26.0	17.3 - 34.1	19.4 - 32.0	
	2	123	130	95.3 - 161.	104. - 153.	
BERYLLIUM	1	94.0	89.9	75.7 - 103.	79.2 - 99.6	
	2	288	270	231. - 306.	241. - 296.	
CADMIUM	1	10.1	10.0	7.22 - 12.8	7.92 - 12.1	
	2	154	150	128. - 170.	133. - 165.	
COBALT	1	47.5	47.5	37.0 - 57.4	39.6 - 54.8	
	2	609	594	506. - 694.	530. - 670.	
CHROMIUM	1	15.4	15.0	8.74 - 20.2	10.2 - 18.8	
	2	245	240	181. - 287.	194. - 274.	
COPPER	1	39.9	40.0	31.6 - 47.6	33.6 - 45.6	
	2	177	176	152. - 195.	157. - 190.	
IRON	1	49.8	50.4	30.4 - 70.0	35.3 - 65.1	
	2	413	420	357. - 471.	371. - 457.	
MERCURY	1	2.24	2.40	1.52 - 3.21	1.73 - 3.00	
	2	15.0	15.6	11.6 - 20.1	12.7 - 19.0	
MANGANESE	1	38.1	37.8	27.8 - 46.1	30.1 - 43.8	
	2	150	147	127. - 164.	132. - 159.	
NICKEL	1	62.6	63.0	46.9 - 78.8	50.9 - 74.8	
	2	282	280	237. - 322.	248. - 311.	
LEAD	1	49.6	50.4	37.2 - 64.4	40.6 - 61.0	
	2	164	168	140. - 197.	147. - 190.	

=====

\*BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

PERFORMANCE EVALUATION REPORT

DATE: 11/16/87

WATER POLLUTION STUDY NUMBER WP019

LABORATORY:

ANALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
----------	---------------	--------------	-------------	-------------------	----------------	------------------------

TRACE METALS IN MICROGRAMS PER LITER:

SELENIUM	1	23.7	20.0	12.4 - 25.8	14.0 - 24.1	
	2	138	120	84.2 - 150.	92.4 - 141.	
VANADIUM	1	62.7	62.0	46.1 - 78.4	50.5 - 74.0	
	2	637	620	520. - 720.	547. - 693.	
ZINC	1	31.3	30.4	22.7- 38.8	24.7 - 36.8	
	2	117	114	90.7 - 134.	96.1 - 129.	
ANTIMONY	3	26.3	13.8	6.04 - 22.6	8.22 - 20.4	
	4	75.1	37.3	21.6 - 54.7	25.9 - 50.4	
SILVER	3	35.2	17.5	13.4 - 21.5	14.4 - 20.4	
	4	6.9	13.43	2.13 - 4.95	2.49 - 4.60	
THALLIUM	3	2.87	3.20	1.58 - 4.82	2.01 - 4.39	
	4	28.6	32.0	21.1 - 43.2	24.1 - 40.2	
MOLYBDENUM	3	8.79	4.40	.352 - 8.85	1.52 - 7.68	
	4	74.7	37.0	19.3 - 49.3	23.2 - 45.4	
STRONTIUM	3	179	91.5	73.7 - 107.	78.3 - 102.	
	4	36.4	18.3	14.3 - 22.2	15.4 - 21.1	
TITANIUM	3	70.6	37.1	19.0 - 52.2	23.6 - 47.6	
	4	303	156	113. - 205.	125. - 192.	

\*BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

SUMMARY OF DOE SURVEY - LABORATORY SUPPORT (WPO19)

Parameter	ORNL		ORGDP		ANL		BCD		INEL	
	Sample 1	Sample 2								
Al	87.2	828	-	-	-	-	-	-	-	-
As	24.8	123	-	-	-	-	-	-	-	-
Be	94.0	288	-	-	-	-	-	-	-	-
Cd	10.1	154	-	-	-	-	-	-	-	-
Co	47.5	609	-	-	-	-	-	-	-	-
Cr	15.4	245	-	-	-	-	-	-	-	-
Cu	39.9	177	-	-	-	-	-	-	-	-
Fe	49.8	413	-	-	-	-	-	-	-	-
Hg	2.24	15.0	-	-	-	-	-	-	-	-
Mn	38.1	150	-	-	-	-	-	-	-	-
Ni	62.6	282	-	-	-	-	-	-	-	-
Pb	49.6	164	-	-	-	-	-	-	-	-
Se	23.7	138	-	-	-	-	-	-	-	-
V	62.7	637	-	-	-	-	-	-	-	-
Zn	31.3	117	-	-	-	-	-	-	-	-
Sb	26.3	75.1	-	-	-	-	-	-	-	-
Ag	35.2	6.91	-	-	-	-	-	-	-	-
Tl	2.87	28.6	-	-	-	-	-	-	-	-
Mo	8.79	74.7	-	-	-	-	-	-	-	-
Sr	179	36.4	-	-	-	-	-	-	-	-
Ti	70.6	303	-	-	-	-	-	-	-	-

C-223

SUMMARY OF DOE SURVEY - LABORATORY SUPPORT (WPO19)

Parameter	ORNL		ORGDP		ANL		BCD		INEL	
	Sample 1	Sample 2								
pH Units	3.97	9.18	4.01	9.20	-	-	-	-	3.90	7.80
Spec. Cond.	675	279	611	254	-	-	-	-	642	298
Total Diss. Sol.	489	286	433	177	384	411	-	-	359	147
Total Hardness	163	74.6	165	76	-	-	-	-	-	-
Total Alkalinity	56.0	6.50	58	9	-	-	-	-	-	-
Chloride	117	52.8	118	51.7	115	74.1	120	48.6	120	51.7
Fluoride	1.97	0.285	1.7	0.2	1.83	0.246	2.12	0.32	2.01	0.305
Sulfate	73.3	31.6	71.1	31.2	67.8	29.7	75.1	34.9	74.4	32.2
Ammonia -N	0.823	3.19	0.83	3.47	-	-	-	-	-	-
Nitrate -N	0.496	2.15	0.51	1.95	0.45	1.87	-	-	.492	2.062
Ortho -P	0.081	0.816	0.08	0.77	0.0743	0.78	-	-	.0729	.765
TOC	58.0	107	-	-	-	-	-	-	57.2	110
Total CN	0.130	0.307	0.13	0.35	0.095	0.283	0.096	0.046	.0933	0.287
Non-Filt. Res.	73.0	27.3	70	25	66.2	23.8	50.6	21.4	65.8	24.7
Oil and Grease	35.8	12.8	31	11	30.8	11.1	16.9	5.4	28.2	-

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SUMMARY OF DOE SURVEY - LABORATORY SUPPORT (WPO19)

Parameter	ORNL		ORGDP		ANL		BCD		INEL	
	Sample 1	Sample 2								
pH Units	3.97	9.18	4.01	9.20	-	-	-	-	3.90	7.80
Spec. Cond.	675	279	611	254	-	-	-	-	642	298
Total Diss. Sol.	489	286	433	177	384	411	-	-	359	147
Total Hardness	163	74.6	165	76	-	-	-	-	-	-
Calcium	61.6	1.25	-	-	-	-	-	-	-	-
Magnesium	0.553	18.0	-	-	-	-	-	-	-	-
Sodium	59.9	18.1	-	-	-	-	-	-	-	-
Potassium	18.6	10.0	-	-	-	-	-	-	-	-
Total Alkalinity	56.0	6.50	58	9	-	-	-	-	-	-
Chloride	117	52.8	118	51.7	115	74.1	120	48.6	120	51.7
Fluoride	1.97	0.285	1.7	0.2	1.83	0.246	2.12	0.32	2.01	0.305
Sulfate	73.3	31.6	71.1	31.2	67.8	29.7	75.1	34.9	74.4	32.2
Ammonia -N	0.823	3.19	0.83	3.47	-	-	-	-	-	-
Nitrate -N	0.496	2.15	0.51	1.95	0.45	1.87	-	-	.492	2.062
Ortho -P	0.081	0.816	0.08	0.77	0.0743	0.78	-	-	.0729	.765
Kjeld. -N	0.527	4.36	-	-	-	-	-	-	-	-
Total -P	0.304	2.19	-	-	-	-	-	-	-	-
COD	166	323	-	-	-	-	-	-	-	-
TOC	58.0	107	-	-	-	-	-	-	57.2	110
5-day BOD	88.0	183	-	-	-	-	-	-	-	-
Total CN	0.130	0.307	0.13	0.35	0.095	0.283	0.096	0.046	.0933	0.287
Non-Filt. Res.	73.0	27.3	70	25	66.2	23.8	50.6	21.4	65.8	24.7
Oil and Grease	35.8	12.8	31	11	30.8	11.1	16.9	5.4	28.2	-
Total Phenolics	0.494	1.35	-	-	-	-	-	-	-	-
Total Res. Chlorine	0.70	1.48	-	-	-	-	-	-	-	-

C-225

PERFORMANCE EVALUATION REPORT

DATE: 11/16/87

WATER POLLUTION STUDY NUMBER WP019

LABORATORY:

ANALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
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TRACE METALS IN MICROGRAMS PER LITER:

ALUMINUM	1		78.0	49.5- 148.	62.0- 136.	
	2		858	658.-1050.	707.- 997.	
ARSENIC	1		26.0	17.3- 34.1	19.4- 32.0	
	2		130	95.3- 161.	104.- 153.	
BERYLLIUM	1		89.9	75.7- 103.	79.2- 99.6	
	2		270	231.- 306.	241.- 296.	
CADMIUM	1		10.0	7.22- 12.8	7.92- 12.1	
	2		150	128.- 170.	133.- 165.	
COBALT	1		47.5	37.0- 57.4	39.6- 54.8	
	2		594	506.- 694.	530.- 670.	
CHROMIUM	1		15.0	8.74- 20.2	10.2- 18.8	
	2		240	181.- 287.	194.- 274.	
COPPER	1		40.0	31.6- 47.6	33.6- 45.6	
	2		176	152.- 195.	157.- 190.	
IRON	1		50.4	30.4- 70.0	35.3- 65.1	
	2		420	357.- 471.	371.- 457.	
MERCURY	1		2.40	1.52- 3.21	1.73- 3.00	
	2		15.6	11.6- 20.1	12.7- 19.0	
MANGANESE	1		37.8	27.8- 46.1	30.1- 43.8	
	2		147	127.- 164.	132.- 159.	
NICKEL	1		63.0	46.9- 78.8	50.9- 74.8	
	2		280	237.- 322.	248.- 311.	
LEAD	1		50.4	37.2- 64.4	40.6- 61.0	
	2		168	140.- 197.	147.- 190.	

\* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

PERFORMANCE EVALUATION REPORT

DATE: 11/16/87

WATER POLLUTION STUDY NUMBER WP019

LABORATORY:

ANALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
TRACE METALS IN MICROGRAMS PER LITER:						
SELENIUM	1		20.0	12.4- 25.8	14.0- 24.1	
	2		120	84.2- 150.	92.4- 141.	
VANADIUM	1		62.0	46.1- 78.4	50.5- 74.0	
	2		620	520.- 720.	547.- 693.	
ZINC	1		30.4	22.7- 38.8	24.7- 36.8	
	2		114	90.7- 134.	96.1- 129.	
ANTIMONY	3		13.8	6.04- 22.6	8.22- 20.4	
	4		37.3	21.6- 54.7	25.9- 50.4	
SILVER	3		17.5	13.4- 21.5	14.4- 20.4	
	4		3.43	2.13- 4.95	2.49- 4.60	
THALLIUM	3		3.20	1.58- 4.82	2.01- 4.39	
	4		32.0	21.1- 43.2	24.1- 40.2	
MOLYBDENUM	3		4.40	.352- 8.85	1.52- 7.68	
	4		37.0	19.3- 49.3	23.2- 45.4	
STRONTIUM	3		91.5	73.7- 107.	78.3- 102.	
	4		18.3	14.3- 22.2	15.4- 21.1	
TITANIUM	3		37.1	19.0- 52.2	23.6- 47.6	
	4		156	113.- 205.	125.- 192.	

\* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

PERFORMANCE EVALUATION REPORT

DATE: 11/16/87

WATER POLLUTION STUDY NUMBER WP019

LABORATORY:

ANALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
MINERALS IN MILLIGRAMS PER LITER: (EXCEPT AS NOTED)						
PH-UNITS	3		4.00	3.93- 4.09	3.95- 4.07	
	4		9.19	8.86- 9.40	8.93- 9.33	
SPEC. COND. (UMHOS/CM AT 25 C)	1		659	592.- 732.	610.- 714.	
	2		272	245.- 302.	252.- 295.	
TDS AT 180 C	1		399	325.- 482.	344.- 462.	
	2		158	95.9- 217.	111.- 202.	
TOTAL HARDNESS (AS CaCO3)	1		159.5	151.- 174.	154.- 172.	
	2		73.5	65.1- 82.9	67.3- 80.7	
CALCIUM	1		63.0	54.7- 74.0	57.1- 71.6	
	2		0.905	.700- 1.78	.835- 1.65	
MAGNESIUM	1		0.520	.424- .635	.451- .608	
	2		17.3	14.8- 19.8	15.4- 19.2	
SODIUM	1		52.6	46.0- 58.4	47.5- 56.8	
	2		13.7	10.8- 16.2	11.4- 15.6	
POTASSIUM	1		18.0	14.9- 21.0	15.6- 20.2	
	2		10.0	8.29- 11.5	8.68- 11.1	
TOTAL ALKALINITY (AS CaCO3)	1		55.0	49.0- 60.4	50.4- 59.0	
	2		7.49	4.71- 11.6	5.57- 10.8	
CHLORIDE	1		113	106.- 128.	108.- 125.	
	2		52.1	47.1- 57.1	48.3- 55.9	
FLUORIDE	1		2.01	1.74- 2.23	1.80- 2.17	
	2		0.247	.155- .337	.178- .314	
SULFATE	1		74.0	60.7- 85.5	63.8- 82.4	
	2		33.0	24.5- 39.4	26.3- 37.5	

\* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

PERFORMANCE EVALUATION REPORT  
 WATER POLLUTION STUDY NUMBER WPO19

DATE: 11/16/87

LABORATORY:

ANALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
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NUTRIENTS IN MILLIGRAMS PER LITER:

AMMONIA-NITROGEN	1	0.800	.538-	1.09	.605-	1.03
	2	3.00	2.33-	3.58	2.48-	3.43
NITRATE-NITROGEN	1	0.500	.383-	.614	.411-	.586
	2	2.00	1.59-	2.38	1.68-	2.28
ORTHOPHOSPHATE	1	0.080	.0454-	.108	.0529-	.100
	2	0.800	.682-	.904	.708-	.877
KJELDAHL-NITROGEN	3	0.500	.0635-	1.02	.179-	.903
	4	4.00	2.78-	5.16	3.07-	4.87
TOTAL PHOSPHORUS	3	0.300	.226-	.394	.246-	.373
	4	2.00	1.63-	2.43	1.73-	2.34

DEMANDS IN MILLIGRAMS PER LITER:

COD	1	150	118.-	168.	124.-	162.
	2	275	213.-	307.	225.-	295.
TOC	1	59.2	46.8-	74.3	50.4-	70.7
	2	109	86.8-	128.	92.2-	122.
5-DAY BOD	1	97.8	61.6-	134.	70.5-	125.
	2	175	108.-	242.	125.-	225.

PCB'S IN MICROGRAMS PER LITER:

PCB-AROCLOR 1016/1242	1	4.57	2.01-	6.61	2.60-	6.02
PCB-AROCLOR 1250	2	1.86	.733-	2.54	.996-	2.28
PCB-AROCLOR 1262	2	1.86	1.18-	2.25	1.32-	2.11

\* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

PERFORMANCE EVALUATION REPORT

DATE: 11/16/87

WATER POLLUTION STUDY NUMBER WPO19

LABORATORY:

ANALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
PESTICIDES IN MICROGRAMS PER LITER:						
ALDRIN	1	0.851		.225- 1.16	.344- 1.04	
	2	0.334		.0833- .460	.131- .412	
DIELDRIN	1	0.829		.453- 1.12	.538- 1.03	
	2	0.290		.134- .405	.168- .370	
DDD	1	0.390		.135- .565	.189- .511	
	2	0.975		.419- 1.31	.533- 1.20	
DOE	1	0.676		.285- .920	.365- .840	
	2	0.169		.0926- .255	.113- .234	
DDT	1	0.297		.0879- .477	.137- .428	
	2	0.742		.330- 1.07	.424- .973	
HEPTACHLOR	1	0.540		.203- .745	.272- .676	
	2	0.166		.0595- .239	.0824- .216	
HEPTACHLOR EPOXIDE	1	0.105		.0550- .144	.0664- .132	
	2	0.456		.262- .603	.305- .560	
CHLORDANE	3	7.73		3.56- 9.39	4.31- 8.65	
	4	0.620		.240- .919	.327- .833	

\* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

PERFORMANCE EVALUATION REPORT

DATE: 11/16/87

WATER POLLUTION STUDY NUMBER WP019

LABORATORY:

ANALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
VOLATILE HALOCARBONS IN MICROGRAMS PER LITER:						
1,2 DICHLOROETHANE	1		54.8	37.3- 72.9	41.9- 68.3	
	2		3.65	.694- 7.74	1.60- 6.83	
CHLOROFORM	1		92.9	52.8- 129.	62.6- 120.	
	2		14.7	8.21- 21.7	9.93- 20.0	
1,1,1 TRICHLOROETHANE	1		32.6	18.4- 52.7	22.8- 48.3	
	2		9.38	4.84- 15.5	6.20- 14.1	
TRICHLOROETHENE	1		48.2	30.3- 67.6	35.0- 62.8	
	2		2.41	1.02- 3.74	1.37- 3.39	
CARBONTETRACHLORIDE	1		27.2	16.7- 38.7	19.5- 35.9	
	2		6.81	3.31- 11.0	4.29- 9.99	
TETRACHLOROETHENE	1		28.9	15.7- 42.0	19.0- 38.6	
	2		5.36	1.65- 9.06	2.59- 8.11	
BROMODICHLOROMETHANE	1		32.2	24.5- 45.4	27.1- 42.7	
	2		7.24	4.11- 11.5	5.05- 10.5	
DIBROMOCHLOROMETHANE	1		67.7	37.7- 108.	46.6- 98.7	
	2		2.26	.643- 4.15	1.09- 3.70	
BROMOFORM	1		32.9	21.8- 48.8	25.2- 45.3	
	2		4.93	2.23- 7.22	2.87- 6.58	
METHYLENE CHLORIDE	1		42.6	25.8- 67.3	31.1- 62.0	
	2		2.13	D.L.- 5.51	.608- 4.79	
CHLOROBENZENE	1		30.8	18.7- 43.8	21.9- 40.6	
	2		3.85	1.48- 6.07	2.07- 5.48	

\* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.  
 D.L. STANDS FOR DETECTION LIMIT

PERFORMANCE EVALUATION REPORT

DATE: 11/16/87

WATER POLLUTION STUDY NUMBER WPO19

LABORATORY:

ANALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
VOLATILE AROMATICS IN MICROGRAMS PER LITER:						
BENZENE	1	9.89	6.29-	14.0	7.29-	13.0
	2	42.9	29.4-	57.7	33.0-	54.0
ETHYLBENZENE	1	8.47	4.52-	11.6	5.44-	10.7
	2	26.1	16.3-	35.5	18.8-	33.1
TOLUENE	1	5.95	3.24-	8.80	3.97-	8.07
	2	29.7	20.8-	39.4	23.2-	37.0
1,2-DICHLOROBENZENE	1	5.42	1.20-	9.58	2.37-	8.41
	2	61.4	36.0-	89.4	43.0-	82.4
1,3-DICHLOROBENZENE	1	3.46	.773-	5.89	1.44-	5.22
	2	26.0	10.7-	38.1	14.5-	34.3
1,4-DICHLOROBENZENE	1	4.47	1.15-	8.26	2.13-	7.28
	2	35.8	18.8-	55.0	23.6-	50.2
MISCELLANEOUS PARAMETERS:						
TOTAL CYANIDE (IN MG/L)	1	0.124	.0687-	.161	.0805-	.149
	2	0.300	.174-	.388	.201-	.361
NON-FILTERABLE RESIDUE (IN MG/L)	1	69.4	61.1-	73.6	62.6-	72.0
	2	24.7	20.5-	27.2	21.3-	26.4
OIL AND GREASE (IN MG/L)	1	35.3	20.9-	43.0	23.7-	40.3
	2	12.8	3.99-	18.1	5.74-	16.3
TOTAL PHENOLICS (IN MG/L)	1	0.505	.229-	.775	.298-	.706
	2	1.29	.588-	1.96	.762-	1.79
TOTAL RESIDUAL CHLORINE (IN MG/L)	1	0.654	.401-	.848	.459-	.790
	2	1.31	.920-	1.56	1.0-	1.48

\* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
OFFICE OF RESEARCH AND DEVELOPMENT  
ENVIRONMENTAL MONITORING SYSTEMS LABORATORY-LAS VEGAS  
P.O. BOX 93478  
LAS VEGAS, NEVADA 89193-3478  
(702/798-2100 - FTS 545-2100)

JUN 20 1988

Mr. William R. Laing  
Oak Ridge National Laboratory  
Building 4500 S. MS-131  
Oak Ridge, TN 37831-6107

Dear Bill:

The multi-laboratory study of the analyses for the water pollution sample, WP-020, is complete. Comparison sheets are enclosed showing the true values, acceptance limit ranges, and warning limit ranges. Values for some analytes present in the samples in determined quantities, but not generally determined in this DOE exercise, are also provided. The ORNL laboratories provided values for many of the optional analytes, and comparison with the true values should yield helpful information. A good general agreement is apparent.

Comparison of the shorter list of analytes, used for the DOE laboratories in this study, shows only one value outside the acceptance range. That is the one for fluoride at a true value of 0.123 milligrams per liter. It can be noted from the comparison sheets that a larger fraction of the participating laboratories had difficulty with that determination than for most others.

The enclosed information should be reviewed by your laboratory staff with regard to installing any corrective action which would improve analytical quality. I congratulate your laboratory on the completion of a large group of analytical determinations of high quality and thank you for your participation in the study. We remain ready to provide counsel regarding any portion of this study.

Sincerely,

A handwritten signature in cursive script that reads "Harold A. Vincent".

Harold A. Vincent  
Chemist, Quality Assurance Research Branch  
Quality Assurance and Methods Development Division

Enclosure

cc: (w/ enclosure)  
D. Karen Knight, DOE HQ



METALS	TRUF	DEWL
Aluminum	115	120
	626	652
Arsenic	391	371
	111	99
Beryllium	70.4	71.6
	571	584
Cadmium	100	195
	270	276
Cobalt	75.3	79.9
	382	391
Chromium	832	894
	89.2	90.1
Copper	291	296
	100	100
Iron	1410	1480
	763	766
Mercury	2.24	2.29
	0.936	0.94
Manganese	211	221
	860	858
Nickel	571	613
	171	170
Lead	171	158
	914	923
Selenium	32	33.4
	82.1	80.4
Vanadium	1310	1360
	360	363
Zinc	650	698
	1270	1320
Antimony	82.8	87.8
	124	128
Silver	14.6	15
	5.48	7.29
Thallium	5.76	6.23
	54.4	57.6
Molybdenum	61.6	60.1
	26.4	40
Strontium	61	62.4
	15.3	17.2
Titanium	48.2	45.8
	278	273

DCE ENVIRONMENTAL SURVEY LABORATORIES, WISCONSIN

3/23/77

MOE LAB RESULTS ; WP020

Analyte	TRUE	ORNL	ACPT LMTS	WARN LMTS
Chloride	69.6 218.	74.6 234.	69.6-77.0 209.-237.	68.3-75.7 212.-234.
Fluoride	1.11 0.123**	1.09 0.377**	.953-1.25 .0601-.198	.990-1.21 .0775-.181
Sulfate	5.01 120.	4.41 119.	2.61-7.11 101.-137.	3.17-6.55 105.-132.
Ammonia	2.10 10.3	2.58* 10.7	1.59-2.63 8.42-12.0	1.71-2.50 8.84-11.6
Nitrate	5.50 0.950	5.81 0.87	4.50-6.48 .750-1.16	4.74-6.24 .799-1.11
Ortho-P	1.10 4.80	1.20 5.13	.919-1.27 4.14-5.46	.961-1.23 4.30-5.30
Kjeld-N	8.10 14.5	8.48 14.0	5.98-10.0 11.0-17.6	6.47-9.53 11.8-16.8
Total-P	9.50 4.40	9.57 4.16	7.45-11.0 3.52-5.11	7.87-10.6 3.71-4.92
Cyanide	0.460 0.155	0.444 0.141	.308-.587 .0845-.207	.343-.552 .0999-.192
Non-F Res	56.3 34.8	53. 38.	44.9-67.7 24.7-45.0	47.7-64.9 27.2-42.5
Oil/Greas	14.0 21.0	13.4 18.6	6.52-18.7 10.1-27.4	8.04-17.2 12.3-25.2

\*\* EXCEEDS ACCEPTANCE LIMITS    \* EXCEEDS WARNING LIMITS

C-235

JUL 14 1988 *PSH*

OAK RIDGE NATIONAL LABORATORY  
OPERATED BY MARTIN MARIETTA ENERGY SYSTEMS, INC

POST OFFICE BOX 2008  
OAK RIDGE, TENNESSEE 37831

July 14, 1988

Harold Vincent  
EPA-LV  
P. O. Box 93478  
Las Vegas, NV 89193-3478

Dear Harold:

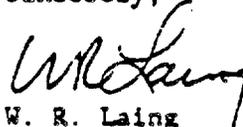
I have checked the fluoride value that we obtained on the last water pollution sample, WP-20. The measurement was made using ion chromatography. The sample was run on triplicate, with no dilution, using two ion chromatographs. The results were as follows:

	<u>System 1</u>		<u>System 2</u>	
	<u>Seq.</u>	<u>F. mg/L</u>	<u>Seq.</u>	<u>F. mg/L</u>
QC	1	0.56	1	0.55
Calib.	2	OK	2	OK
WP-20	4	0.313	3	0.415
WP-20			13	0.403

Sample QC is an internal QC sample whose value is unknown to the analyst. The value for this control is 0.58 mg/L. Calibration is the daily calibration standard. Sequence is the sequence number within the sample data group. The three values obtained (0.313, 0.415 and 0.403) were averaged to obtain the 0.377 value reported. Although the scatter in the 3 results is greater than I would expect, I can find no problems with the measurement itself. It may be, as you noted, that there was not good precision between laboratories on this measurement of this sample.

Please call me if you have any questions.

Sincerely,



W. R. Laing  
Section Head  
Analytical Chemistry Division

WRL:lp

cc: Karen Knight  
Susan Holladay

Internal Correspondence

JAN 06 1989 OSH

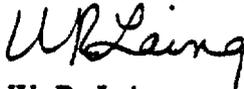
MARTIN MARIETTA ENERGY SYSTEMS, INC.

December 28, 1988

## Distribution

Results of Water Pollution Sample. WP-021

Attached are the results on EPA WP-21 for inorganics. All results were satisfactory. Note the large number of results that are very close to the true value. This is really good work!



W. R. Laing

Distribution  
CAPA Group  
EAL Group  
W. Shults  
S. Holladay  
P. Howell ✓  
D. Bostick  
B. Fitts  
K. Owenby  
K. Daniels



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
OFFICE OF RESEARCH AND DEVELOPMENT  
ENVIRONMENTAL MONITORING SYSTEMS LABORATORY-LAS VEGAS  
P.O. BOX 93478  
LAS VEGAS, NEVADA 89193-3478  
(702/798-2100 - FTS 545-2100)

DEC 19 1988

Mr. William R. Laing  
Oak Ridge National Laboratory  
P.O. Box 2008  
Building 4500 S. MS-131  
Oak Ridge, TN 37831-6107

Dear Bill:

Results of the multi-laboratory study of the analyses for the water pollution sample, WP-021, are attached. The attachments include comparison sheets showing the true values, values determined in your laboratory, acceptance limit ranges, and warning limit ranges. Explanations of these terms are given on one attachment.

The laboratories participating in the DOE environmental survey were instructed to use the WP performance evaluation samples to augment available PE materials by providing analytical determinations for survey-requested analytes which were not available as components in those other PE samples. The laboratories could option to determine other WP sample components for their own QA/QC purposes. The comparison of the survey list of analytes, shows no ORNL values out of range. No response regarding corrective action is required.

Thank you for your participation in the study. We remain ready to counsel regarding any portion of this work.

Sincerely,

A handwritten signature in cursive script that reads "Harold A. Vincent".

Harold A. Vincent  
Chemist, Quality Assurance Research Branch  
Quality Assurance and Methods Development Division

Enclosure

cc: (w/Enclosure)  
Vincent Fayne, DOE HQ  
Alan Crockett, INEL

DOE LAB RESULTS ; WFO21

12/7/88

Analyte	EPA	ORNL
pH-units	5.61	
	8.35	
Spec cond.	642	
	670	
TDS	370	
	377	
Tot Hrdns	235	
	92.2	
Sodium	11.0	14.5
	95.0	105
Potassium	11.0	12.0
	21.5	24.0
Total Alk	13.9	
	104	
Chloride	172	172
	65.4	66.0
Fluoride	0.320	0.40
	3.70	3.71
Sulfate	15.1	14.8
	116	123
AmmoniaN	0.270	
	1.20	
NitrateN	0.250	
	1.90	
Ortho-P	0.065	
	0.900	
Kjeld-N	0.380	
	5.71	
Total-P	0.150	
	3.50	
COD	43.5	
	229	
TOC	17.2	
	90.5	
BOD	27.9	
	146	
Cyanide	0.150	0.154
	0.225	0.226
Non-F res	81.1	
	43.0	
Oil/Greas	5.2	
	29.5	
Tot-Phen	0.557	
	2.82	
TotRC1	0.301	
	1.91	

WPO21 Continued --

METALS

Aluminum	624	627
	234	234
Arsenic	390	383
	54.2	51.8
Beryllium	135	139
	8.99	8.91
Cadmium	222	221
	24.0	24.3
Cobalt	509	510
	17.0	18.2
Chromium	125	128
	41.7	42.7
Copper	96.0	102
	8.00	10.2
Iron	210	216
	42.0	42.7
Mercury	10.7	9.79
	1.47	1.31
Manganese	315	323
	70.0	70.4
Nickel	350	372
	140	145
Lead	126	116
	21.0	19.5
Selenium	180	181
	40.0	40.3
Vanadium	124	133
	43.1	45.7
Zinc	190	198
	63.3	70.5
Antimony	149	153
	179	170
Silver	0.95	0.93
	11.7	11.1
Thallium	8.00	7.91
	72.0	66.3
Molybdenum	47.5	45.8
	18.5	
Strontium	42.7	36.7*
	8.54	8.25
Titanium	100	98.6
	63.1	62.4

\*\* EXCEEDS ACCEPTANCE LIMITS ..

\* EXCEEDS WARNING LIMITS

DOE LAB RESULTS ; WPO21

12/7/88

Analyte	TRUE	ORNL	ACPT LMTS	WARN LMTS
Chloride	172 65.4	172 66.0	157 - 179 58.6- 71.7	159 - 176 60.3- 70.1
Fluoride	0.320 3.70	0.40 3.71	.242- .403 3.06- 4.12	.263- .383 3.20- 3.99
Sulfate	15.1 116	14.8 123	11.5- 18.2 96.1- 133	12.3- 17.4 101- 128
AmmoniaN	0.270 1.20		.0896-.517 .856- 1.56	.141-.466 .942- 1.48
NitrateN	0.250 1.90		.163- .334 1.51- 2.26	.183- .313 1.60- 2.17
Ortho-P	0.065 0.900		.0380-.0922 .762- 1.04	.0445-.0857 .796- 1.01
Kjeld-N	0.380 5.71		D.L.- .900 4.07- 7.22	.0680- .785 4.45- 6.84
Total-P	0.150 3.50		.0960- .216 2.85- 4.33	.110- .202 3.03- 4.15
Cyanide	0.150 0.225	0.154 0.226	.0844- .196 .128- .297	.0986- .182 .150- .276
Non-F Res	81.1 43.0		74.9- 84.6 36.7- 45.7	76.1- 83.4 37.9- 44.6
Oil/Greas	5.2 29.5		1.37- 9.14 16.8- 36.7	2.33- 8.17 19.3- 34.2

\*\* EXCEEDS ACCEPTANCE LIMITS

\* EXCEEDS WARNING LIMITS

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**Draft-Do Not Cite**  
**ANL Data Document**  
**Issue Date: June 1989**  
**Revision: 01**

**BCD Results of Inorganic and Organic Performance Evaluation Studies**

**Draft-Do Not Cite**  
**ANL Data Document**  
**Issue Date: June 1989**  
**Revision: 01**

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**PERFORMANCE EVALUATION SCORES FOR BCD**

<b>Code</b>	<b>Score</b>
QB2FY89 Inorganic	•
QB1FY89 Inorganic	•
QB4FY88 Inorganic	•
QB3FY88 Inorganic	89.0
QB2FY88 Inorganic (JA61 ICP Lab)	90.1
QB2FY88 Inorganic (JA70 ICP Lab)	66.3
QB3FY88 Organic	95.6
QB2FY88 Organic	47.3
QB1FY88 Organic	47.2

\* Information was requested and will be distributed upon receipt.

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**ANL Data Document**  
**Issue Date: June 1989**  
**Revision: 01**

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ANL Data Document  
Issue Date: June 1989  
Revision: 01

NOTE

Documentation to support Battelle-Columbus' participation in the EMSL-LV's Inorganic QB2, FY89 has been requested. ORNL will attach this documentation upon receipt from Battelle.

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**Revision: 01**

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ANL Data Document  
Issue Date: June 1989  
Revision: 01

NOTE

Documentation to support Battelle-Columbus' participation in the EMSL-LV's Inorganic QB1, FY89 has been requested. ORNL will attach this documentation upon receipt from Battelle.

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**Issue Date: June 1989**  
**Revision: 01**

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NOTE

Documentation to support Battelle-Columbus' participation in the EMSL-LV's Inorganic QB4, FY88 has been requested. ORNL will attach this documentation upon receipt from Battelle.

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**Revision: 01**

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P.O. BOX 93478  
LAS VEGAS, NEVADA 89193-3478  
(702/798-2100 - FTS 545-2100)

JUL 15 1986

Dr. Judith Gebhart  
Battelle-Columbus Division  
505 King Avenue  
Columbus, Ohio 43201-2693

REC. JUL 21 1986

Dear Dr. Gebhart:

The results of the participation of your laboratory in the EMSL-LV ~~third quarter inorganic~~ performance evaluation study (OB3, FY88, Case Number 9302) are enclosed. This includes copies of the analysis reports for inorganics in soil and water samples. The reports also present statistical information on the numbers of laboratories having difficulties with specific analytes.

The score for your laboratory is 89 and is acceptable but since it is less than 90, a formal response is required describing any changes or corrective actions taken to improve the performance evaluation score.

This office will be glad to furnish any council and further information regarding this work.

Sincerely,

Harold A. Vincent

Chemist, Quality Assurance Research Branch  
Quality Assurance and Methods Development Division

Enclosures

cc: (w/enclosure)  
D. K. Knight, DOE HQ

xc VICTOR FISHMAN  
Dennis Raichart  
Mark Ross

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INORGANIC PERFORMANCE EVALUATION SAMPLE  
 INDIVIDUAL LABORATORY SUMMARY REPORT  
 FOR QB 3 FY 88

LABORATORY NAME: Battelle Columbus (OH) (Z1)  
 PERFORMANCE LEVEL: ACCEPTABLE - Corrective Actions Necessary  
 LABORATORY RANK: Above = 27 Same = 0 Below = 19

I Score: 89  
 REPORT DATE: 6/28/1988  
 MATRIX: WATER

ELEMENT NAME	95 % CI		LAB RESULTS		#LABS NOT-ID	#LABS NIS-QUANT	PROGRAM DATA			TOTAL #LABS
	LOWER	UPPER	REPORTED VALUE	QUALIFIER CODE			#LABS FALSE POS	#LABS NSPK OUT	#LABS DUP OUT	
ALUMINUM	1790	2190	2000		0	4	0	0	0	47
ANTIMONY	86	156	117		2	5	0	3	0	47
ARSENIC	40	58	55.7		0	4	0	5	3	47
BARIUM	265	331	301		0	4	0	1	0	47
BERYLLIUM	5.0	6.7	5.2		2	2	0	0	0	47
CADMIUM	65	82	69.7		0	4	0	2	0	47
CALCIUM	8970	11000	10000		0	4	0	0	0	47
CHROMIUM	90	117	123	X	0	7	0	0	0	47
COBALT	61	87	76.7		0	2	0	0	0	47
COPPER	126	170	146		0	4	0	2	0	47
IRON	492	621	567		0	1	0	0	1	47
LEAD	5.0	7.5	6.8		5	11	0	5	2	47
MAGNESIUM	5740	6770	6460		0	5	0	0	0	47
MANGANESE	35	50	45.7		0	2	0	0	0	47
MERCURY	2.8	5.2	3.8		0	1	0	4	1	47
NICKEL	40	85	65.4		0	5	0	1	0	47
POTASSIUM	6700	8220	8510	X	1	6	0	0	0	47
SELENIUM	39	62	46.5		0	3	0	0	2	47
SILVER	10.0	15	8.1	U	16	2	0	4	3	47
SODIUM	8970	10900	11400	X	0	6	0	0	0	47
THALLIUM	17	31	27.0		2	6	0	7	0	47
VANADIUM	64	93	82.0		0	2	0	0	0	47
ZINC	124	170	152		0	3	0	0	0	47

# OF ELEMENTS NOT-IDENTIFIED: 0  
 # OF ELEMENTS NIS-QUANTIFIED: 3  
 # OF FALSE POSITIVES: 0

# OF MATRIX SPIKES OUT: 2  
 WATER : Cd, Pb

# OF DUPLICATES OUT: 0  
 WATER :

INORGANIC PERFORMANCE EVALUATION SAMPLE  
 INDIVIDUAL LABORATORY SUMMARY REPORT  
 FOR Q3 3 FY 88

LABORATORY NAME: Battelle Columbus (OH) (Z1)  
 PERFORMANCE LEVEL: ACCEPTABLE - Corrective Actions Necessary  
 LABORATORY RANK: Above = 27 Same = 0 Below = 19

% Score: 89  
 REPORT DATE: 6/28/1988  
 MATRIX: SOIL

ELEMENT NAME	95 % CI		LAB RESULTS		#LABS NOT-ID	#LABS NIS-QUANT	PROGRAM DATA			TOTAL #LABS
	LOWER	UPPER	REPORTED VALUE	QUALIFIER CODE			#LABS FALSE POS	#LABS NSPK OUT	#LABS DUP OUT	
ALUMINUM	8310	16200	14100		0	3	0	0	0	47
ANTIMONY	c	c	2.6	U	0	0	0	30	1	47
ARSENIC	2.0	2.3	1.4	B	9	10	0	5	2	47
BARIUM	40.0	57	50.3		0	1	0	3	0	47
BERYLLIUM	d	d	0.3	B	0	0	1	1	0	47
CADMIUM	c	c	0.3	-B	0	0	1	0	1	47
CALCIUM	1000.0	4150	3160		0	1	0	0	0	47
CHROMIUM	13	34	30		0	2	0	2	0	47
COBALT	d	d	8.5	B	0	0	1	0	0	47
COPPER	8.9	22	15.5		0	1	0	1	0	47
IRON	8720	19000	14800		0	1	0	0	0	47
LEAD	3.2	7.1	4.7		1	5	0	0	5	47
MAGNESIUM	3340	5550	4830		0	3	0	0	0	47
MANGANESE	171	282	251		0	3	0	3	1	47
MERCURY	c	c	0.1	U	0	0	2	2	2	47
NICKEL	24	45	36.4		0	4	0	1	0	47
POTASSIUM	d	d	430		0	0	1	0	0	47
SELENIUM	c	c	0.7	U	0	0	0	13	0	47
SILVER	c	c	0.8	U	0	0	1	9	1	47
SODIUM	d	d	27.8		0	0	0	0	0	47
THALLIUM	c	c	1	U	0	0	1	3	1	47
VANADIUM	17	53	37.4		0	4	0	0	0	47
ZINC	31	59	56		0	0	0	1	3	47

# OF ELEMENTS NOT-IDENTIFIED: 0  
 # OF ELEMENTS NIS-QUANTIFIED: 0  
 # OF FALSE POSITIVES: 0

# OF MATRIX SPIKES OUT: 1  
 OIL : Sb

# OF DUPLICATES OUT: 0  
 OIL :

No. 111-8224(822)

Name	Initials	Date
Originator MS Ross	MSR	10/18/88
Concurrence		
Approved VA Fishman	Yef	10/18/88

Internal Distribution  
RL Joiner/VA Fishman  
JW McDonald  
GA Dus Sault  
DW Raichart ←  
RA Mayer  
MS Ross

October 18, 1988

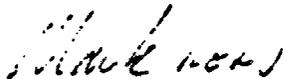
Dr. Harold Vincent  
U.S. EPA Environmental Monitoring  
Systems Laboratory (EMSL-LV)  
944 East Harmon  
Las Vegas, Nevada 89109

Dear Dr. Vincent:

Please find enclosed for your review a ~~corrective action~~ response regarding our participation in the EMSL-LV Third Quarter FY 88 ~~Inorganic Performance~~ Evaluation Study (~~QB3 FY 88~~).

If you have any questions or comments concerning this response, please contact me at 614-424-3326.

Sincerely,



Mark Ross  
Inorganic Chemistry Group Leader  
Chemistry and Spectroscopy Section

MSR:d1m

Enclosure

## REVIEW AND ACTIONS ON QB-3 FY 88 REPORT

### INTRODUCTION

Twenty-three elements were determined in QB-3 FY 88 water and soil samples following SOW-787 methodology by inductively coupled plasma emission spectrometry (ICP) or graphite furnace atomic absorption (GFAA). The Battelle Columbus Division score of 89 is acceptable, but since it is less than 90, a formal response is required to state corrective actions to improve the performance evaluation.

### PERFORMANCE PROBLEMS

Chromium, potassium and sodium in water samples are out of the 95 percent confidence interval (CI) by only 5.1 percent, 3.5 percent, and 4.6 percent, respectively. The spiked recoveries of 50 µg/L cadmium and 20 µg/L lead in the water sample are reported as 150 percent. The recovery of 128 percent on antimony spiked with 10.1 mg/kg (50.5 µg/L in the final test solution) in the soil sample is also beyond the acceptable 100 ± 15 percent range.

### CORRECTIVE ACTIONS

The excellent recoveries of 97.7-103.8 percent for all six elements in EPA initial calibration verification samples indicate no problems with the method at those concentration levels. In fact, even at lower concentration the deviations of the reported data in the water sample (regular) from 95 percent confidence interval for chromium, potassium and sodium are only 6 µg/L (123 vs 117), 290 µg/L (8510 vs 8220), and 500 µg/L (11400 vs 10900), respectively. All of the above deviations are less than the contract-required detection limit and are within or near the standard deviation of the corresponding elements.

Based on the SOW 787 requirement for spike recovery calculation, the recovery for antimony was 128.2 percent. This requirement for recovery calculation does not allow for subtraction of the analyte value in the sample if that value is less than the contract required detection limit. If

the calculation had been performed by subtracting the sample analyte value of 2.60 mg/kg from the spiked sample result (SSR-SR) of 12.94 mg/kg, based on a spike of 10.1 mg/kg, the recovery would be 102 percent.

The recoveries of antimony are  $103.3 \pm 1.7$  percent for ICV-3,  $102.2 \pm 0.6$  percent, and  $105.7 \pm 0.6$  percent for CCV (pages 0020, 0021 June 23 report), and 97.5 percent for ICS (page 0035), respectively. These results lead to the reasonable conclusion that the sample concentration should be subtracted for calculating the spike recovery.

After careful review, suspected contamination could be spotted through blank analyses for cadmium and lead. The migration of trace level elements in plastic and glass containers during storage has been found to bias trace analyses for these elements, especially in the  $\mu\text{g/L}$  range.

#### CONCLUSIONS

It is suspected that trace level contamination has played a role in the high bias regarding analysis of chromium, potassium, sodium, cadmium, and lead in the water sample.

A thorough review of glassware preparation and cleaning, as well as a review of associated causes of contamination, will be undertaken by the staff of the Battelle Columbus Division Inorganic Analytical Laboratory.

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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
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LAS VEGAS, NEVADA 89193-3478  
(702/798-2100 - FTS 545-2100)

9/19

SEP 14 1988

XC VICTOR FISMAN  
Dennis Raichart ✓  
Mark Ross  
REC'D SEP 19 1988

Dr. Judith Gebhart  
Battelle-Columbus Division  
505 King Avenue  
Columbus, Ohio 43201-2693

Dear Dr. Gebhart:

The data from your submittal for the ~~inorganic performance~~ sample, QB2FY88, has been rescored at my request to correspond to the ICP instrument situation at the Battelle Laboratory when these samples were being analyzed. There are two scores; one for each instrumental lab. One of the scores is good and the other is not. Copies of the Individual Laboratory Summary Reports along with scoring procedure information sheets are attached. An explanation of what this special scoring means and how they should be used is in order.

It is my understanding from conversations with Ray Siery and later, Mark Ross, that your laboratory intends to replace the older ICP unit with the newly acquired Jarrel Ash unit for the CLP type determinations. I gave advice for your laboratory to submit data for the QB2FY88 from both ICP units, so that the quality assurance of analyses for the DOE environmental survey would be covered for samples analyzed by either instrument. Any data produced for the survey would then be bracketed by appropriate performance evaluation sample information.

The scores are given as if for two laboratories with the Atomic Absorption Furnace analyses common to the two. The scores are 66.3% for the JY70 ICP lab. and 90.1% for the JA61 ICP laboratory. A response from your laboratory detailing problems and corrective actions to solve them, is required by the DOE Environmental Survey program. A separate response for each ICP laboratory is advised.

These reports and responses become part of the quality assurance record for analytical work done with these instruments and may be added to documents for DOE survey sites. Since the QB2 submittal was considerably late and a further delay was occasioned by this special scoring effort, we would appreciate receiving your responses at your earliest convenience.

This office will be glad to furnish any counsel and further information regarding this work.

Sincerely,



Harold A. Vincent

Chemist, Quality Assurance Research Branch  
Quality Assurance and Methods Development Division

Enclosures

cc:

Randal Scott, DOE HQ  
Alan Crockett, INEL  
Duane Hilmas, BCD  
Mark Ross, BCD  
Greg Du Sault, BCD

INORGANIC PERFORMANCE EVALUATION SAMPLE  
 INDIVIDUAL LABORATORY SUMMARY REPORT  
 FOR QB 2 FY 88

LABORATORY NAME: Battelle Columbus (OH) JA61 ICP  
 PERFORMANCE LEVEL: ACCEPTABLE  
 LABORATORY RANK: Above = 17 Same = 0 Below = 19

Z Score: 90.1  
 REPORT DATE: 8/12/1988  
 MATRIX: WATER

ELEMENT NAME	95 % CI		LAB RESULTS		PROGRAM DATA					TOT. #LAB
	LOWER	UPPER	REPORTED VALUE	QUALIFIER CODE	#LABS NOT-ID	#LABS MIS-QUANT	#LABS FALSE POS	#LABS MSPK OUT	#LABS DUP OUT	
ALUMINUM	2560	3270	2790		0	1	0	0	1	37
ANTIMONY	60.0	112		NR	4	2	0	1	3	37
ARSENIC	60	105		NR	0	1	0	2	0	37
BARIUM	374	448	412		0	5	0	0	1	37
BERYLLIUM	30	51	42.5		0	1	0	0	1	37
CADMIUM	20	32	24.7		0	1	0	0	1	37
CALCIUM	12300	15600	13100		0	2	0	0	0	37
CHROMIUM	14	41	26.2		0	1	0	0	1	37
COBALT	65	112	91.2		0	1	0	0	0	37
COPPER	182	243	209		0	2	0	1	3	37
IRON	349	446	394	E	0	6	0	0	0	37
LEAD	12	25		NR	0	0	0	5	3	37
MAGNESIUM	7040	9640	8000		0	2	0	0	0	37
MANGANESE	61	82	70.3		0	1	0	0	1	37
MERCURY	10	20		NR	0	3	0	1	1	37
NICKEL	86	124	102		0	3	0	0	1	37
POTASSIUM	8000	12500	12400		0	2	0	0	0	37
SELENIUM	18	28		NR	0	3	0	3	1	37
SILVER	c	c	2.8	B	0	0	1	6	0	37
SODIUM	6000	8200	8150		0	0	0	0	0	37
THALLIUM	50	88		NR	0	2	0	9	1	37
THORIUM	118	154	135	E	1	2	0	1	0	37
ZINC	46	66	56.7		0	7	0	1	2	37

# OF ELEMENTS NOT-IDENTIFIED: 0  
 # OF ELEMENTS MIS-QUANTIFIED: 0  
 # OF FALSE POSITIVES: 0

# OF MATRIX SPIKES OUT: 0  
 WATER :

# OF DUPLICATES OUT: 0  
 WATER :

INORGANIC PERFORMANCE EVALUATION SAMPLE  
 INDIVIDUAL LABORATORY SUMMARY REPORT  
 FOR Q8 2 FY 88

LABORATORY NAME: Battelle Columbus (OH)  
 PERFORMANCE LEVEL: ACCEPTABLE  
 LABORATORY RANK: Above = 17 Same = 0 Below = 19

Z Score: 90.1  
 REPORT DATE: 8/12/1988  
 MATRIX: SOIL

ELEMENT NAME	95 % CI		LAB RESULTS		#LABS NOT-ID	#LABS MIS-QUANT	PROGRAM DATA			TOTAL #L
	LOWER	UPPER	REPORTED VALUE	QUALIFIER CODE			#LABS FALSE POS	#LABS MSPK OUT	#LABS DUP OUT	
ALUMINUM	4890	11700	11200	#	0	3	0	0	0	3
ANTIMONY	12.0	53		NR	4	4	0	25	0	3
ARSENIC	17	28		NR	0	6	0	7	1	3
BARIUM	156	189	196	X	0	6	0	1	0	3
BERYLLIUM	16	21	19.9		0	3	0	1	0	3
CADMIUM	9.9	17	14.3	E	0	2	0	3	0	3
CALCIUM	75800	104000	94300		0	3	0	0	0	3
CHROMIUM	16	51	57.6	X	0	4	0	0	1	3
COBALT	71	94	92.9	#	0	4	0	0	0	3
COPPER	88	111	103		0	5	0	1	0	3
IRON	12400	17500	17000		0	4	0	0	1	3
LEAD	165	224		NR	0	6	0	2	0	3
MAGNESIUM	41300	56500	51600		0	3	0	0	1	3
MANGANESE	2760	3570	3540	#	0	5	0	1	0	3
MERCURY	13	24		NR	0	5	0	2	1	3
NICKEL	26	54	52.3	#	0	4	0	3	1	3
POTASSIUM	1000.0	1930	1720	E	0	5	0	0	0	3
SELENIUM	6.7	20		NR	0	3	0	4	5	3
SILVER	32	52	43.8		0	3	0	5	2	3
SODIUM	d	d	221	BE	0	0	0	0	0	3
THALLIUM	21	41		NR	0	1	0	9	3	3
VANADIUM	41	70	62.8		0	2	0	0	0	3
ZINC	150	211	212	X	0	3	0	3	0	3

# OF ELEMENTS NOT-IDENTIFIED: 0  
 # OF ELEMENTS MIS-QUANTIFIED: 3  
 # OF FALSE POSITIVES: 0

# OF MATRIX SPIKES OUT: 0  
 SOIL :

# OF DUPLICATES OUT: 0  
 SOIL :

INORGANIC PERFORMANCE EVALUATION SAMPLE  
 INDIVIDUAL LABORATORY SUMMARY REPORT  
 FOR QB 2 FY 88

LABORATORY NAME: Battelle Columbus (OH) [F1]  
 PERFORMANCE LEVEL: UNACCEPTABLE, Corrective Actions Mandatory  
 LABORATORY RANK: Above = 34 Same = 0 Below = 2

Y Score: 66.3  
 REPORT DATE: 9/8/1988  
 MATRIX: WATER

ELEMENT NAME	95 % CI		LAB RESULTS		#LABS NOT-ID	#LABS NIS-QUANT	PROGRAM DATA			TOTAL #L
	LOWER	UPPER	REPORTED VALUE	QUALIFIER CODE			#LABS FALSE POS	#LABS NSPK OUT	#LABS DUP OUT	
ALUMINUM	2560	3270	2950		0	1	0	0	1	1
ANTIMONY	60.0	112	92		4	2	0	1	3	3
ARSENIC	68	105	89.8		0	1	0	2	0	1
BARIUM	374	440	395		0	5	0	0	1	1
BERYLLIUM	38	51	50		0	1	0	0	1	1
CADMIUM	20	32	24.9		0	1	0	0	1	1
CALCIUM	12300	15600	13600		0	2	0	0	0	2
CHROMIUM	14	41	25.9		0	1	0	0	1	1
COBALT	65	112	90.2		0	1	0	0	0	1
COPPER	182	243	220		0	2	0	1	3	3
IRON	349	446	429		0	6	0	0	0	6
LEAD	12	25	17.8		0	0	0	5	3	3
MAGNESIUM	7840	9640	8990		0	2	0	0	0	2
MANGANESE	61	82	66.1		0	1	0	0	1	1
MERCURY	10	20	16.1		0	3	0	1	1	3
NICKEL	86	124	112		0	3	0	0	1	3
POTASSIUM	8880	12500	10900		0	2	0	0	0	2
SELENIUM	18	28	24.5		0	3	0	3	1	3
SILVER	c	c	9.3	B	0	0	1	6	0	3
SODIUM	6080	8280	8380	X	0	8	0	0	0	8
THALLIUM	50	88	69.4		0	2	0	9	1	3
VANADIUM	118	154	139		1	2	0	1	0	3
ZINC	46	66	49.9		0	7	0	1	2	7

# OF ELEMENTS NOT-IDENTIFIED: 0  
 # OF ELEMENTS NIS-QUANTIFIED: 1  
 # OF FALSE POSITIVES: 0

# OF MATRIX SPIKES OUT: 0  
 WATER :

# OF DUPLICATES OUT: 0  
 WATER :

INORGANIC PERFORMANCE EVALUATION SAMPLE  
 INDIVIDUAL LABORATORY SUMMARY REPORT  
 FOR QB 2 FY 88

LABORATORY NAME: Battelle Columbus (OH) (F1)  
 PERFORMANCE LEVEL: UNACCEPTABLE, Corrective Actions Mandatory  
 LABORATORY RANK: Above = 34 Same = 0 Below = 2

Z Score: 66.3  
 REPORT DATE: 8/8/1988  
 MATRIX: SOIL

ELEMENT NAME	95 % CI		LAB RESULTS		PROGRAM DATA				
	LOWER	UPPER	REPORTED VALUE	QUALIFIER CODE	#LABS NOT-ID	#LABS NIS-QUANT	#LABS FALSE POS	#LABS NSPK OUT	#LABS DUP OUT
ALUMINUM	4890	11700	11800	X	0	3	0	0	0
ANTIMONY	12.0	53	9.9	B	4	4	0	25	0
ARSENIC	17	28	25.5		0	6	0	7	1
BARIUM	156	189	192	X	0	6	0	1	0
BERYLLIUM	16	21	22.3	X	0	3	0	1	0
CADMIUM	9.9	17	16.1	*	0	2	0	3	0
CALCIUM	75800	104000	88200		0	3	0	0	0
CHROMIUM	16	51	52.8	X	0	4	0	8	1
COBALT	71	94	94.8	X	0	4	0	0	0
COPPER	88	111	113.6	X	0	5	0	1	0
IRON	12400	17500	17100	*	0	4	0	0	1
LEAD	165	224	197		0	6	0	2	0
MAGNESIUM	41300	56500	51700		0	3	0	0	1
MANGANESE	2760	3570	3630	X	0	5	0	1	0
MERCURY	13	24	16.8		0	5	0	2	1
NICKEL	25	54	54.6	X	0	4	0	3	1
POTASSIUM	1000.0	1930	2220	X	0	5	0	0	0
SELENIUM	6.7	20	15.3		0	3	0	4	5
SILVER	32	52	42		0	3	0	5	2
SODIUM	d	d	773	B	0	0	0	0	0
THALLIUM	21	41	32.3		0	1	0	9	3
VANADIUM	41	70	65.7		0	2	0	0	0
ZINC	158	211	217	X	0	3	0	3	0

# OF ELEMENTS NOT-IDENTIFIED: 0  
 # OF ELEMENTS NIS-QUANTIFIED: 10  
 # OF FALSE POSITIVES: 0

# OF MATRIX SPIKES OUT: 1  
 SOIL : 50

# OF DUPLICATES OUT: 0  
 SOIL :

No. 101-8221(822)

Name	Initials	Date
Originator MS Ross	MSR	9/27/88
Concurrence		
Approved VA Fishman	VA Fishman	9-27-88

Internal Distribution  
RL Joiner JE Gebhart  
VA Fishman DA Raichart  
JH McDonald RA Mayer  
GA Dus Sault  
MS Ross

September 27, 1988

Dr. Harold Vincent  
U.S. EPA Environmental Monitoring  
Systems Laboratory (EMSL-LV)  
944 East Harmon  
Las Vegas, Nevada 89109

Dear Dr. Vincent:

Please find enclosed for your review a corrective action response regarding our participation in the EMSL-LV Second Quarter FY 88 Inorganic Performance Evaluation Study (QB2-FY-88).

If you have any questions or comments concerning this response, please contact me at 614-424-3326.

Sincerely,



Mark Ross  
Inorganic Chemistry Group Leader  
Chemistry and Spectroscopy Section

MSR:d1m

Enclosure

## Performance Problems (QB2 FY 88) JY70ICP

Ten elements in the QB2 FY 88 soil sample analyzed by the Jobin-Yvon 70 Inductively Coupled Plasma Spectrometer were detected and reported at levels which exceeded the 90 percent confidence interval (CI) for these analytes, resulting in an overall score of 66.3. Further investigation of these out-of-range results showed that they all exceeded the upper limit of their 90 percent CI, but, with the exception of potassium, were very close to acceptability by these standards. By contrast, the same QB2 FY 88 soil sample was also analyzed with the Jarrell Ash ICAP 61 Spectrometer, the overall score for these results being an acceptable 90.1.

## Corrective Actions

The three most likely causes for the consistently high bias in our JY-70 reported values were investigated. First, the analytical balance used to weigh the sample was checked against class-s weights. If the balance used had been in error in recording the sample weight (specifically, biased low), this would indicate the use of more sample than necessary, thereby causing higher than expected results. However, this was not the case, as the analytical balance was well within tolerance.

Second, the Eppendorf pipets used in the preparation of calibration standards were checked for accuracy by weighing, on a calibrated analytical balance, the portions of water drawn from ten different injections. Accuracy was found to be within  $\pm 1$  percent, indicating that the Eppendorf pipets are an unlikely cause of high bias.

Third, the standards used for instrument calibration undergo an ongoing check against standards of another source, as well as analysis by two different instrumentation systems. In addition, the accuracy of the calibration standards was indicated by the fact that the EPA-supplied initial calibration verification solutions analyzed during this performance evaluation were well within tolerance.

The fact that none of these three possibilities apparently proved relevant to the out-of-range JY-70 soil results led us to consider the possibility that our soil extraction efficiency might be higher than in other laboratories. While we have no conclusive proof that our high results

for ten analytes are attributable to this effect, we favor this explanation because of two pieces of circumstantial evidence:

- (1) The QB 2 FY 88 JY-70 results for water (where the digestion matrix remains homogeneous and complete extraction is likely in all cases) were completely acceptable. In addition, several analytes (e.g., Be and Mn) in the solid laboratory control sample results (where a heterogeneous digestion could lead to variable extraction efficiency as in an actual soil sample) were very close to the high side of the 90 percent CI.
- (2) Our acceptable soil results on the JY-70 for QB4 FY87 and QB1 FY88 and for the JA-61 for QB2 FY88 were consistently on the high side of the acceptable range, again in keeping with high extraction efficiency in our laboratory. Indeed, with the exception of potassium, the agreement between the JY-70 and JA-61 soil results for QB2 FY88 is good, the out-of-range former readings being only slightly higher than the acceptable latter values which were just within the 90 percent CI on the high end.

Based on this circumstantial evidence, our corrective action with regard to the high soil values will be to reevaluate our solid sample digestion procedure, making sure that we are not "overdigesting" with regard to the method outlined in the Statement of Work. We feel that this is the most likely explanation for our small deviations near the upper limit 90 percent CI for the ten analytes listed above using the JY-70 system. In addition, it is also possible that small interelement effects might come into play in the higher-solids soil matrix. This could make our choice of background correction points more critical than we realized and could require the use of additional interelement corrections. (For QB2 FY88 we only used IECs when an ICS AB or LCS was out of tolerance.) We will also investigate these possibilities which are obviously specific to the JY-70 instrument.

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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
 OFFICE OF RESEARCH AND DEVELOPMENT  
 ENVIRONMENTAL MONITORING SYSTEMS LABORATORY-LAS VEGAS  
 P.O. BOX 93478  
 LAS VEGAS, NEVADA 89193-3478  
 (702/798-2100 - FTS 545-2100)

xc RL Jenner  
 Dennis Richard  
 Bruce Kelly  
 Sue Hetzel  
 Ramona Meyer  
 Greg Das Sant

AUG 08 1988

REC'D 15 1988

Dr. Judith Gebhart  
 Battelle-Columbus Division  
 505 King Avenue  
 Columbus, Ohio 43201-2693

Dear Dr. Gebhart:

+ with attachment

The Individual Laboratory Summary Report (ILSR) summarizing the results of the participation of your laboratory in the EMSL-LV third quarters ~~organics~~ performance evaluation study ~~Q3 1988~~ is enclosed. In addition, general information concerning the scoring procedure used for QB3 is included.

The score for your laboratory at 95.6 is in the CLP category of acceptable (score--90 or above), with no response required regarding any changes or corrective actions. Even with the good score, it would be wise to examine the report for information which would be helpful to your laboratory in this kind of analysis.

Congratulations on the good score! This office will be glad to furnish any counsel and further information regarding this work.

Sincerely,

Harold A. Vincent  
 Chemist  
 Quality Assurance Research Branch, QAD

Enclosures

cc:  
 D. Karen Knight, DOE HQ

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NOTE

Documentation to support Battelle's 95.6 score for EMSL-LV's Organic QB3 FY88 evaluation has been requested. ORNL will attach this documentation upon receipt from Battelle.

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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
 OFFICE OF RESEARCH AND DEVELOPMENT  
 ENVIRONMENTAL MONITORING SYSTEMS LABORATORY-LAS VEGAS  
 P.O. BOX 93478  
 LAS VEGAS, NEVADA 89193-3478  
 (702/798-2100 - FTS 545-2100)

*Revised 4/29/88  
by*

*Copied: J.E. GERHART 4/29/88  
D.W. ROICHNER 4/29/88*

Mr. Gregory A. DusSault  
 Battelle Columbus Division  
 Anal & Struct. Chem. Center  
 505 King Avenue  
 Columbus, OH 43201-2693

Dear Mr. DusSault:

For your information and review the results for your participation in the EMSL-LV ~~Second-Quarter-Organic~~ Performance Evaluation Study (OB2-~~44-38~~) are included here. Enclosed is general information about the Superfund Performance Evaluation Program. The PE portion of the Laboratory Profile Package, called the "Individual Laboratory Summary Report" (ILSR) was described in your letter reports last quarter. Other general information about the PE program is explained on the following pages.

The samples consisted of aqueous materials spiked with Target Compound List (TCL) and non-TCL pollutants at environmentally representative levels. Samples for all laboratories were from the same homogeneous batch. Each sample set was to be prepared and analyzed by current contractually required procedures.

The EMSL-LV thanks you for your participation in this study and wishes to congratulate the laboratories for an overall fine performance. We trust that this information is vital to you as a member of the community of laboratories analyzing hazardous waste samples for Superfund.

Sincerely,

Larry Butler, Ph.D.

Supervisor, Performance Evaluation Program  
 Quality Assurance Research Branch  
 Quality Assurance and Methods Development Division

Enclosure

cc: (w/enclosure)  
 Carla Dempsey, OERR  
 Joan Fisk, OERR  
 Emile Boulos, OERR  
 Angelo Carasea, OERR  
 Howard Fribush, OERR

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ORGANIC PERFORMANCE EVALUATION SAMPLE  
 INDIVIDUAL LABORATORY SUMMARY REPORT  
 FOR Q3 2 FY 88

LABORATORY: Battelle Columbus (OH)  
 PERFORMANCE: UNACCEPTABLE - Corrective Actions Mandatory  
 RANK: Above = 47 Same = 0 Below = 5

SCORE: 47.3  
 REPORT DATE: 4/13/1988  
 MATRIX: WATER

COMPOUND	90 % CI		LABORATORY DATA		#LABS NOT-ID	PROGRAM #LABS MIS-QUART	DATA #LABS CONTAM	TOTAL #LABS
	LOWER	UPPER	CONC	Q				
<b>TCL VOLATILE</b>								
BROMOMETHANE	64	240	120		0	3	0	52
METHYLENE CHLORIDE	0	0	04		0	0	0	52
1,1-DICHLOROETHANE	34	55	44		0	3	0	52
2-BUTANONE	38	170	10	U	0	7	0	52
BROMODICHLOROMETHANE	59	80	73		0	4	0	52
1,1,2-TRICHLOROETHANE	54	76	62		0	8	0	52
BENZENE	12	17	15		1	5	0	52
2-HEXANONE	48	200	99		1	4	0	52
TOLUENE	19	30	23	B	0	2	0	52
CHLOROBENZENE	85	110	100		0	3	0	52
STYRENE	80	110	100		0	6	0	52
XYLENES (TOTAL)	120	180	150		0	6	0	52

**TCL SEMIVOLATILE**

2-CHLOROPHENOL	23	52	46	44	0	6	0	52
N-NITROSO-DI-N-PROPYLAMINE	45	84	32	88	X	7	0	52
ISOPHORONE	65	140	122	130	0	6	0	52
2,4-DIETHYLPHENOL	10	53	57	59	X X	2	0	52
BENZOIC ACID	50	200	190	220	X	7	0	52
HEXACHLOROCYCLOHEPTADIENE	61	160	160	150	0	3	0	52
2-METHYLNAPHTHALENE	20	55	48	52	0	3	0	52
2,4,6-TRICHLOROPHENOL	55	100	44	92	0	9	0	52
2-NITROANILINE	50	100	67	77	0	2	0	52
ACENAPHTHYLENE	59	100	120	120	X X	9	0	52
ACENAPHTHENE	61	100	100	110	X	5	0	52
2,4-DINITROPHENOL	81	260	140	210	0	7	0	52
DIBENZOFURAN	96	160	140	150	0	7	0	52
4-NITROPHENOL	50	200	140	170	0	1	0	52
FLUORENE	64	100	110	120	X X	5	0	52
BIS(2-ETHYL)PHTHALATE	0	0	0	0	0	0	0	52
PENTACHLOROPHENOL	74	230	180	140	0	6	0	52
PHENANTHRENE	62	100	100	100	0	5	0	52
ANTHRACENE	57	100	100	96	0	5	0	52
PYRENE	42	110	110	100	0	6	0	52
BUTYL BENZYL PHTHALATE	0	0	0	0	0	0	0	52
BENZO(A)ANTHRACENE	31	100	85	92	0	2	0	52
DI-N-OCTYL PHTHALATE	10	100	45	45	0	2	0	52
DIBENZ(A,H)ANTHRACENE	17	140	60	61	1	2	0	52

**TCL PESTICIDES**

HEPTACHLOR	0.05	0.43	0.29		0	6	0	52
ALDRIN	0.14	0.53	0.38		18	5	0	52
ENDRIN	0.16	0.48	0.36	X	2	11	0	52
TOXAPHEN					0	0	0	52

**NON-TCL SEMIVOLATILE**

BENZOPHENONE	4+ 995	per 870	97	J	0	0	0	52
DISULFOTON			0	0	0	0	0	52
CHLORPYRIFOS	4+ 963	per 540	19	J	0	0	0	52
2-NITRO-P-CRESOL	4+ 999	per 827	77	J	0	0	0	52

**TCL SEMIVOLATILE (Contaminants)**

BENZYL ALCOHOL			14	MS MSB	13	11	0	0	0	52
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ORGANIC PERFORMANCE EVALUATION SAMPLE  
 INDIVIDUAL LABORATORY SUMMARY REPORT  
 FOR QB 2 FY 88

LABORATORY: Battelle Columbus (OH)  
 PERFORMANCE: UNACCEPTABLE - Corrective Actions Mandatory  
 RANK: Above = 47 Same = 0 Below = 5

SCORE: 47.3  
 REPORT DATE: 4/13/1988  
 MATRIX: WATER

COMPOUND	90 % CI		LABORATORY DATA CONC	#LABS NOT-ID	PROGRAM #LABS HIS-QUANT	DATA #LABS CONTAM	TOTAL #LABS
	LOWER	UPPER					
BIS(2-ETHYLHEXYL)PHTHALATE			0.51	0	0	1	52
TCL PESTICIDES (Contaminants)							
DIELDRIN			0.51	0	0	1	52
HEPTACHLOR EPOXIDE			0.012	0	0	0	52
ALPHA-CHLORDANE			0.04	0	0	0	52
NON-TCL SEMI-VOLATILE (Contaminants)							
2H-INDOL-2-ONE, 1,3-DIHYDRO-	ft 967	ppm 600	21	0	0	2	52
BORANE, DIMETHOXY-	ft 954	ppm 529	15	0	0	0	52
BENZENE, POSS C2 NITRO-	ft 906	ppm 452	48	0	0	0	52
FURANONE, BENZO-3(2H)-	ft 931	ppm 277	12	0	0	0	52

- A # OF TCL COMPOUNDS NOT-IDENTIFIED: 1
  - B # OF TCL COMPOUNDS HIS-QUANTIFIED: 7
  - C # OF TCL CONTAMINANTS: 1
  - D # OF NON-TCL COMPOUNDS NOT-IDENTIFIED: 1
  - E # OF NON-TCL CONTAMINANTS: 4
- K = ~~36~~ # of Total TCLs spiked  
 36

$$\text{SCORE} = 100 - \left[ \frac{150}{X} * (2A+B+C) + 2.2 * (D+E) \right]$$

$\frac{2+7+1}{4.17}$

For Review and Approval

No. G1271-2260 (826)

	Name	Initials	Date
Originator	BJ Hidy	BH	6/2/88
Concurrence			
	JE Gebhart	JEG	6/2/88
	RA Mayer	RM	6/2/88
Approved			

Internal Distribution

RL Joiner/JE Gebhart  
 DW Raichart  
 LH Kenny  
 SS Hetzel  
 RA Mayer  
 RMO  
 File

June 2, 1988

Dr. Harold Vincent  
 U.S. EPA Environmental Monitoring  
 Systems Laboratory (EMSL-LV)  
 944 E. Harmon  
 Las Vegas, NV 89109

Dear Dr. Vincent:

Please find enclosed for your review and approval, a listing of the ~~corrective actions~~ taken in response to our participation in the EMSL-LV ~~Second Quarter FY-88~~ ~~Quarterly~~ Performance Evaluation Study ~~(Q2 FY-88)~~ [Case No. 8783].

The information provided by the Superfund Performance Evaluation Program has been of great use to Battelle by indicating areas in which we can improve the performance of our analytical and quality assurance programs.

If you have any questions or comments concerning the corrective actions we have taken, please contact me at (614-424-4605) or Bruce Hidy at (614-424-4591).

Sincerely,



J. E. Gebhart, Ph. D.  
 Section Manager  
 Analytical Chemistry Section

JEG:gp

cc: Karen Knight (DOE)

Enclosure

## CORRECTIVE ACTIONS FOR QB2, FY 88

### TCL VOLATILE

#### Performance Problems

One TCL volatile compound, 2-Butanone, was not detected. This compound is difficult to detect due to its poor purging efficiency, poor chromatography (broad peak shape), and poor response (low response factor). Careful inspection of the sample file showed this compound to be present at the expected retention time.

#### Corrective Actions

We are currently trying to improve the purging efficiency of this compound by increasing the purge flow from 30 mL/min to 40 mL/min. We have also increased the sensitivity of the automated search procedure and will continue to manually search all samples for this compound until we are certain that the automated procedure is reliable.

### TCL SEMIVOLATILE

#### Performance Problems

Six TCL semivolatile compounds were detected and reported at levels which exceeded the 90% confidence interval (CI) for each compound. Additionally, three TCL semivolatile compounds were flagged as exceeding their upper warning limit. Further investigation of this fraction showed that the majority of the compounds detected and reported were near the upper limit of their 90% CI.

#### Corrective Actions

The two most likely causes for this consistent high bias in our reported values were investigated. First, the volume calibration for the sample extract vials was checked. If the samples extracts had been concentrated to a volume less than 1.0 mL then the analyte concentrations would appear to be higher than expected. Each sample vial was clearly and accurately marked for 1.0 mL. The second likely cause was that the concentration of our internal standard solution had changed such that the concentration of the internal standard analytes was less than the 40 ng/ $\mu$ L specified by the SOW. A fresh internal standard solution was prepared from a new ampule of the same Lot number used for the QB analyses. A comparison of the response of the two solution showed very good agreement for all of the compounds. At this point a third, less likely, cause was investigated. A fresh calibration curve was prepared from materials obtained from the EPA QAMB. The 50  $\mu$ g/L standard used for the daily CCC used during the analysis of the QB samples was compared to the 50  $\mu$ g/L standard from QAMB materials. Again, all analytes were found to be in good agreement between the two standards. None of the above items would appear to be the source of the consistent high bias in our data. At this point we have been unable to identify any additional possibilities likely or unlikely which we can evaluate. The only other possibility we have considered is based on the fact that we prepared these samples using continuous liquid-

liquid extraction and normally achieve high extraction efficiency and high recoveries of the analytes. If the majority of the reporting laboratories used separatory funnel extractions, which may have yielded lower recoveries, then the 90% CI may be bias toward the lower recovery values.

### TCL PESTICIDES

#### Performance Problems

One TCL pesticide compound, Endrin, was reported above the 90% CI established for that compound. This compound was confirmed using the secondary column. However, confirmation of the quantification was not investigated prior to the submission of this QB. Further investigation showed that the endrin standard used for calibration for this data had degraded significantly resulting in a lower than expected response for that standard. This caused the reported value for the sample to be higher than it should have been. No other standards were found to have degraded.

#### Corrective Actions

We will carefully evaluate the performance of all of our standards for each of the compounds based on their historical performance prior to the analysis of all samples. Any significant change (as specified by the SOW) in the response of any analyte will be addressed by preparation of a new standard for that analyte.

### NON-TCL VOLATILES

#### Performance Problems

None indicated.

#### Corrective Actions

None required.

### NON-TCL SEMIVOLATILE

#### Performance Problems

One Non-TCL semivolatle compound, Disulfoton, was not detected. This compound was found to be totally unresolved chromatographically from phenanthrene-d10, an internal standard present at a relatively high level in the sample.

#### Corrective Actions

Additional attention will be paid to the symmetry of the TCL compound peaks, internal standard and surrogate compound peaks for indications of partial coelution of Non-TCL compounds. Also, additional attention will be paid to the mass spectra of the TCL compounds detected and the mass spectra of all

internal standard and surrogate standard peaks to determine the presents of "extra" ions which would indicate complete coelution of a Non-TCL compound with these other standard peaks.

#### TCL VOLATILE (Contaminants)

##### Performance Problems

None indicated.

##### Corrective Actions

None required.

#### TCL SEMIVOLATILE (Contaminants)

##### Performance Problems

One TCL semivolatile compound, Benzyl alcohol, was reported as detected at 14 µg/L, just above the CRQL of 10 µg/L. Confirmation of the mass spectra for benzyl alcohol was made against that days CCC standard. This compound was also detected and report in the matrix spike and matrix spike duplicate analyses at 13 µg/L and 11 µg/L respectively. Benzyl alcohol was not detected or reported in the sample blank analysis.

##### Corrective Actions

Based on the above data we believe that the detection and reporting of this compound was valid and no corrective actions are justified.

#### TCL PESTICIDE (Contaminants)

##### Performance Problems

One TCL pesticide, Dieldrin, was detected and reported as 0.051 µg/L (Form I PEST, page 0270) which is below the CRQL of 0.10 µg/L. The value was incorrectly entered as 0.51 µg/L on the EPA Individual Laboratory Summary Report Form.

##### Corrective Actions

Because the value was incorrectly entered by EPA no corrective actions are justified.

#### NON-TCL SEMIVOLATILE (Contaminants)

##### Performance Problems

Four Non-TCL semivolatile compounds (TICs) detected and reported were scored as contaminants. In the judgement of the experienced analysts who generated

and reviewed the data, all of the criteria required to report these compounds as TICs were met. Additional review of the matrix spike and matrix spike duplicate analyses showed the presence of these compounds in both samples. None of these compounds were detected in the sample blank or the standards analyzed for this QB. The results of the forward library search gave FIT values of >900 and PURITY values of >300 for each compound. However, the three correctly identified TICs all had FIT values >950 and PURITY values >500.

#### Corrective Actions

In the future, the analysts who generate and review the TIC data will use as an additional guideline that the expected FIT values should be >950 and the expected PURITY values should be >500. However, we will continue to report all TIC compounds which in the judgement of an experienced analyst meet the criteria required for reporting the compound.

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ORGANIC PERFORMANCE EVALUATION SAMPLE  
INDIVIDUAL LABORATORY SUMMARY REPORT  
FOR Q8 1 FY 88

LABORATORY: Battelle Columbus (OH)  
PERFORMANCE: UNACCEPTABLE - Corrective Action Mandatory  
RANK: Above = 30 Same = 0 Below = 8

SCORE: 47.2  
REPORT DATE: 5/2/1988  
MATRIX: WATER

COMPOUND NAME	90 % CI		LABORATORY DATA CONC	0	#LABS MISS ID	#LABS MISS QUAN	#LABS ID CONT	TOTAL #LABS
	LOWER	UPPER						
<b>TCL VOLATILE</b>								
VINYL CHLORIDE	99	250	180		0	0	0	39
ACETONE	0	0	130		0	0	0	39
CHLOROFORM	41	58	51	B	0	5	0	39
CARBON TETRACHLORIDE	14	28	18		0	4	0	39
CIS-1,3-DICHLOROPROPENE	40	100	62		5	7	0	39
BROMOFORM	100	170	120		0	5	0	39
1,1,2,2-TETRACHLOROETHANE	55	84	62		0	6	0	39
CHLOROBENZENE	98	138	120		0	4	0	39
<b>TCL SEMIVOLATILE</b>								
PHENOL	0	110	110	③	0	2	0	39
1,2-DICHLOROBENZENE	40	92	74		0	5	0	39
4-METHYLPHENOL	12	59	43		0	3	0	39
HEXACHLOROETHANE	44	120	84		0	3	0	39
2-NITROPHENOL	29	78	56		0	3	0	39
1,2,4-TRICHLOROBENZENE	34	82	73		0	4	0	39
4-CHLORO-3-METHYL PHENOL	35	88	76		0	4	0	39
HEXACHLOROCYCLOPENTADIENE	0	100	18	④	0	2	0	39
2,4,5-TRICHLOROPHENOL	0	0	63		0	0	0	39
2-CHLORONAPHTHALENE	19	49	40		0	3	0	39
DIMETHYL PHTHALATE	0	100	8	J	0	1	0	39
4-CHLOROPHENYL PHENYL ETHER	27	62	28 10	U E	0	4	0	39
HEXACHLOROBTZENE	48	100	46 10	U E	0	2	0	39
3,3'-DICHLOROBENZIDINE	0	170	20	U ①	0	3	0	39
CHRYSENE	58	140	62 10	U E	1	5	0	39
BENZO(a)PYRENE	31	170	55 10	U E	0	4	0	39
INDENO(1,2,3-cd)PYRENE	39	130	77		1	4	0	39
<b>TCL PESTICIDES</b>								
CANNA-BUC (LINDANE)	0	0	0.17		0	0	1	39
DIELDRIN	0.44	0.68	0.52		0	8	0	39
ENDOSULFAN SULFATE	0.17	0.46	0.28		9	2	0	39
ENDRIN KETONE	0.42	0.78	0.61		5	5	0	39
<b>NON-TCL VOLATILE</b>								
DISBROMOMETHANE			63	J	0	0	0	39
<b>NON-TCL SEMIVOLATILE</b>								
ANILINE			0	ND	0	0	0	39
1,2,3,4-TETRACHLOROBENZENE			110	J	0	0	0	39
2,4-D			0	NA	0	0	0	39
ATRAZINE			140	J	0	0	0	39
TRIFLURALIN			140	J	0	0	0	39
2,3,4,6-TETRACHLOROPHENOL			53	J	0	0	0	39
<b>TCL VOLATILE (Contaminants)</b>								
METHYLENE CHLORIDE			3	J				
1,2-DICHLOROPROPANE			10					
TRICHLOROETHENE			2	J				
TRANS-1,3-DICHLOROPROPENE			3	J				
TOLUENE			3	BJ				
<b>TCL SEMIVOLATILE (Contaminants)</b>								
2,4,6-TRICHLOROPHENOL			66	F				

*False positive from 2,4,5. Noted on Q888 Report and included in Formmaster.*

ORGANIC PERFORMANCE EVALUATION SAMPLE  
 INDIVIDUAL LABORATORY SUMMARY REPORT  
 FOR Q3 1 FY 88

LABORATORY: Battelle Columbus (OH)  
 PERFORMANCE: UNACCEPTABLE - Corrective Action Mandatory  
 RANK: Above = 30 Same = 0 Below = 8

SCORE: 47.2  
 REPORT DATE: 5/2/1988  
 MATRIX: WATER

COMPOUND NAME	90 % CI		LABORATORY DATA	CONC	Q	#LABS MISS ID	#LABS MISS QUAN	#LABS ID CONT	TOTAL #LABS
	LOWER	UPPER							
TCL PESTICIDE (Contaminants)									
ALPHA-BHC			0.053		F				
HEPTACHLOR			0.015						
GAMMA-CHLORDANE			0.087		J				

# OF TCL COMPOUNDS NOT IDENTIFIED: 4  
 # OF TCL COMPOUNDS MISQUANTIFIED: 0  
 # OF TCL CONTAMINANTS: 0

# OF TIC COMPOUNDS NOT IDENTIFIED: 1  
 # OF TIC CONTAMINANTS: 2

CODED SUMMARY OF LABORATORY SCORES  
 QB 1 FY 88  
 5/2/1988

CODE	SCORE	TCLS NOT ID	TCLS MISQUANT	TCLS CONTAM	TCLS CPDS	TICS NOT ID	TICS CONTAM	TICS CPDS
J1	100	0	0	0	7	0	0	1
U1	100	0	0	0	26	0	0	6
C6	100	0	0	0	25	0	0	6
V2	100	0	0	0	7	0	0	1
Z3	100	0	0	0	7	0	0	1
T2	100	0	0	0	7	0	0	1
O3	97.0	0	0	0	7	1	0	1
T1	97.0	0	0	0	7	1	0	1
P2	94.2	0	1	0	26	0	0	6
C4	94.2	0	1	0	26	0	0	6
C5	94.2	0	1	0	26	0	0	6
H3	93.4	0	0	0	26	3	0	6
H2	93.4	0	0	0	26	1	2	6
P3	92.0	0	0	1	26	1	0	6
E2	91.2	0	0	0	26	1	3	6
J2	89.8	0	1	0	26	1	1	6
R1	89.0	0	0	0	26	2	3	7
T3	89.0	0	0	0	26	2	10	7
Y3	88.5	0	1	1	26	0	0	6
A1	88.5	1	0	0	26	0	0	6
S3	87.6	0	0	1	26	3	0	6
E1	86.3	0	2	0	26	0	1	6
U2	85.7	0	1	0	7	0	0	1
C2	85.7	0	1	0	7	0	0	1
H1	85.7	0	1	0	7	0	0	1
I1	83.2	0	1	0	26	2	3	6
D2	81.9	0	2	0	26	0	3	6
A4	81.9	0	2	0	26	1	2	6
X1	80.5	0	2	1	26	1	0	6
D6	80.5	0	3	0	26	0	1	6
I2	80.5	0	3	0	26	0	1	6
A3	77.5	0	1	1	26	2	3	6
C1	76.1	0	3	0	26	0	3	6
D5	76.1	0	3	0	26	0	3	6
R2	74.7	0	3	1	26	0	1	6
F3	74.7	0	5	0	26	0	1	6
J3	72.5	0	2	2	26	0	2	6
B1	71.7	1	1	1	26	1	4	6
V3	69.4	1	1	0	23	2	7	6
D4	69.0	0	5	0	26	0	1	6
Z2	68.4	2	0	0	19	0	0	5
Y2	66.8	1	2	1	26	0	2	6
K3	65.9	1	1	1	26	2	3	6
L2	59.9	0	6	1	26	0	0	6
G2	59.6	2	1	1	26	0	0	6
K2	58.8	1	2	2	26	2	1	6
E3	58.8	1	3	1	26	1	2	6
S1	57.4	1	5	0	26	1	0	6
B2	57.1	1	0	1	7	0	0	1
Y1	56.6	2	0	2	26	1	3	6
D3	54.4	1	3	1	26	2	4	6
Q3	51.5	0	4	0	16	2	4	5
D1	48.1	1	6	1	26	0	0	6
V2	45.9	1	7	0	26	1	0	6
H2	42.8	2	2	2	26	1	6	6
X3	42.8	1	5	1	26	4	1	6
F1	41.5	1	4	3	26	1	2	6
K1	35.7	2	6	0	26	1	2	6
F2	35.7	4	1	1	26	1	2	6
R3	28.6	4	0	4	26	1	0	6
U3	25.5	2	2	5	26	5	0	6
A2	25.5	0	9	2	26	1	6	6
O1	0	2	11	1	26	3	7	6
B4	0	0	15	1	26	5	4	6

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	Name	Initials	Date
Originator	BJ Hidy	<i>BJH</i>	6/2/88
Concurrence	JE Gebhart	<i>JEG</i>	6/2/88
	RA Mayer	<i>RAM</i>	6/2/88
Approved			

## Internal Distribution

RL Joiner/JE Gebhart  
 DW Raichart  
 LH Kenny  
 SS Hetzel  
 RA Mayer  
 RMO  
 File

June 2, 1988

Dr. Harold Vincent  
 U.S. EPA Environmental Monitoring  
 Systems Laboratory (EMSL-LV)  
 944 E. Harmon  
 Las Vegas, NV 89109

Dear Dr. Vincent:

Please find enclosed for your review and approval, a listing of the corrective actions taken in response to our participation in the EMSL-LV First Quarter FY 88 ~~Organic~~ Performance Evaluation Study ~~6/1/88-6/30/88~~ [Case No. 8124].

The information provided by the Superfund Performance Evaluation Program has been of great use to Battelle by indicating areas in which we can improve the performance of our analytical and quality assurance programs.

If you have any questions or comments concerning the corrective actions we have taken, please contact me at (614-424-4605) or Bruce Hidy at (614-424-4591).

Sincerely,

*J. E. Gebhart*

J. E. Gebhart, Ph. D.  
 Section Manager  
 Analytical Chemistry Section

JEG:gp

Enclosure

cc: Karen Knight

CORRECTIVE ACTIONS FOR OBI, FY 88

TCL VOLATILE

Performance Problems

None indicated.

Corrective Actions

None required.

TCL SEMIVOLATILE

Performance Problems

Four (4) TCL compounds, 4-Chlorophenyl phenyl ether, Hexachlorobenzene, Chrysene, and Benzo(a)pyrene were reported on Form I SV-2 (page 0125) as not detected. However, data from our QUAN report (pages 0130 and 0131) show clearly that all of these compounds were detected and quantified. Therefore, an error occurred during the transfer of the data between Finnigan INCOS<sup>TM</sup> data system QUAN program and the Finnigan PC based QA Formaster<sup>TM</sup> II software. This error was not detected during our review of the data because only compounds reported as detected on Form I are checked against the QUAN report.

Corrective Actions

We have revised our data review procedures such that the reviewer will check from the QUAN report to the Form I to ensure the all of the compounds verified and reported on the QUAN report have been transferred and correctly reported on Form I.

TCL PESTICIDES

Performance Problems

None indicated.

Corrective Actions

None required.

NON-TCL VOLATILES

Performance Problems

None indicated.

Corrective Actions

None required.

## NON-TCL SEMIVOLATILE

### Performance Problems

One (1) Non-TCL semivolatile compound, aniline, was not detected. This compound appears to be only partially resolved chromatographically from phenol, a TCL compound present at a relatively high level in this sample.

### Corrective Actions

Additional attention will be paid to the symmetry of the TCL compound peaks for indications of partial coelution of Non-TCL compounds. Also, additional attention will be paid to the mass spectra of the TCL compounds detected to determine the presents of "extra" ions which would indicate complete coelution of a Non-TCL compound with a TCL compound.

## TCL VOLATILE (Contaminants)

### Performance Problems

None indicated.

### Corrective Actions

None required.

## TCL SEMIVOLATILE (Contaminants)

### Performance Problems

One (1) TCL semivolatile compound, 2,4,6-Trichlorophenol, was reported as detected on Form I SV-1 (page 0124). This contaminant is actually a false positive caused by the close elution of 2,4,5-Trichlorophenol, another of the TCL semivolatile compounds. The false positive status of this compound was detected and indicated on the QUAN report (page 0129) during the initial review of the data, however the entry for this compound was not edited from the QUAN report prior to transfer of the data to Form I.

### Corrective Actions

We have added an additional review of the QUAN report for each sample just prior to the transfer of the data to the QA Formaster<sup>TM</sup> II system. This will ensure that the QUAN report is free of false positive entries or that an adequate notation is made to that samples review file so that any incorrect entries can be edited from the final Form I.

## TCL PESTICIDE (Contaminants)

### Performance Problems

One (1) TCL pesticide, alpha-BHC, was detected and reported at 0.053 µg/L which is just above the CRQL of 0.05 µg/L. The retention time for this compound was confirmed on the secondary column. However, the quantification for this compound above the CRQL was not confirmed on the secondary column.

### Corrective Actions

We are now using the quantitative information provided by the secondary column as well as the data from matrix spike and matrix spike duplicate analyses (when available) to more carefully evaluate how compounds at or near their CRQL will be reported.

From a technical standpoint, the considerations are:

*Deficient*



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
OFFICE OF RESEARCH AND DEVELOPMENT  
ENVIRONMENTAL MONITORING SYSTEMS LABORATORY-LAS VEGAS  
P.O. BOX 93478  
LAS VEGAS, NEVADA 89193-3478  
(702/798-2100 - FTS 345-2100)

NOV 2 1987

Mr John B. Murphy  
Oak Ridge National Laboratory  
P. O. Box 2008, 4500S, MS-102  
Oak Ridge, TN 37831-6102

Dear Mr. Murphy,

Final reports describing the on-site evaluation audit for the field sampling effort by your ORNL team at the Argonne National Laboratory are enclosed. Included are the completed check list and comments by LEMSCO, and the field evidence report from CEAT-Techlaw. Our records do not show that these reports have been issued and this is to make up that deficiency.

Kevin Cabble and Lewis Todechiney from LEMSCO were present during sampling activities on 11/13 and 11/16 through 11/18, 1987. Troy Sanders of Techlaw and I were present during the activities from 11/16 through 11/18. Kevin and Lewis held an informal debriefing with the ORNL crew before I arrived because some members were leaving the Argonne site. A formal debriefing, with all auditors and available sample crew members present, was held on the afternoon of 11/17. Handwritten copies of the auditors comments were provided by Techlaw and LEMSCO audit team members.

Preliminary reports of the on-site audit were sent from EMSL-LV by facsimile to D. Karen Knight at DOE Headquarters on 11-20 and 11-24, 1987.

The comments provided in this final report should be identical to those items used for comments during the November 17th debriefing by the LEMSCO and Techlaw team members and to those provided to DOE HQ by facsimile. Some change in the text from preliminary to final draft may have been introduced for clarity.

Some major technical items in the critique included comments describing a single sampler collecting liquid samples from a container in an atmosphere that could have been hazardous. A minimum of one additional person should have been there to act as the "clean" person and for personal safety reasons. A response agreeing with the comment was noted during debriefing. The absence of field instruments that were functioning could be critical to both safety and the handling of the sample and sample

R.D. P...  
11/24/87

containers. Possible contamination of soil samples from liquids kept in a vehicle and from exhaust from one vehicle were strongly emphasized by the auditors.

The issue of acceptance of field logsheets was discussed. Keith Owenby described the uses of the field logsheets which are bound temporarily with stiff paper covers and three roundhead paper fasteners, [(CID)A-A-247], placed through punched holes along one side of each sheet. One preprinted copy set is made for each environmental problem. Standard practice for logbook notation is observed. The set is kept intact until sampling for that environmental problem is finished. Data are extracted from each set for entry into computer memory, the pages of the set are removed from the temporary binder, stapled together on the fastener side, and then placed as a set in a binder as part of the case file record.

In an opinion from techlaw, these sheets are acceptable as technical evidence when the qualifications of signing, dating, serialization, and error notation are handled in the same manner as for bound logbooks.

From a technical standpoint, the considerations are:

- + more complete information for the field team.
- + more complete information for the case file.
- + same QA parameters as for a logbook.
- + saving of time in the field.
- + more accuracy in the field.
- + readable by all persons.
- risk of loss or misplacement of a sheet(s).
- temporary binding is not as durable under field conditions as a bound logbook would be.

The conclusion from this is that the gains from using the logsheets outweigh the risks and that the use of logsheets is good when they are used as designed.

A written response to the audit report is required for the record. The sampling audit reports and the response by the ORNL sampling team, with reports of corrective actions instituted as a result of the audit, will become part of the quality assurance record for the DOE environmental survey for the ANL site. In order that we may meet scheduling requirements, please respond to the comments by the auditing teams within 30 days of your receipt of these reports so that a review and assembly of a package to be included in the data document report for the ANL site can be completed. The response should be addressed to this office (Attn: H. A. Vincent) with copies to DOE headquarters (Attn: V. Fayne) and the ORNL program manager (R. B. Fitts).

If you have any questions regarding this matter, you can call me at FTS 545-2129 or (702)798-2129.

Sincerely,



Harold A. Vincent  
Analytical Chemist  
Quality Assurance Research Branch  
Quality Assurance Methods and Development Division

Enclosures

cc: (w/enclosures)  
Vincent Fayne, DOE HQ  
Robert B. Fitts, ORNL

 **Lockheed Engineering and Management  
Services Company, Inc.**

Environmental Programs Office  
1050 E. Flamingo Road, Suite 120, Las Vegas, Nevada 89119  
(702) 734-3200

November 20, 1987

United States Environmental  
Protection Agency  
P.O. Box 93478  
Las Vegas, Nevada 89193-3478

ATTENTION: MR. W. L. KINNEY

VIA: *NAH* M. T. HOMSHER  
*12/2/87*

SUBJECT: DOE ENVIRONMENTAL SURVEY FIELD SAMPLING ACTIVITY AUDIT  
OF ORNL PERSONNEL AT ARGONNE NATIONAL LABORATORY,  
ARGONNE, ILLINOIS

Dear Mr. Kinney:

On November 13, 16, and 17, a field sampling activity audit of Oak Ridge National Laboratory personnel was conducted at Argonne National Laboratory in Argonne, Illinois. The audit was conducted in support of the DOE Environmental Survey by Lewis Todechiney and Kevin Cabble (Lockheed-EMSCO), Troy Sanders (TechLaw) and Harold Vincent (U.S. EPA). T. Sanders and H. Vincent participated on November 16-17 only. The following comments and checklist are those of Lewis Todechiney and Kevin Cabble only. Comments concerning documentation and chain-of-custody will be forwarded by Troy Sanders under separate cover.

If you have any questions, I can be contacted at 734-3268.

Very truly yours,

*Kevin J. Cabble*

K. J. Cabble  
Senior Scientist  
DOE Environmental Survey

KJC/ahh

cc: H. A. Vincent ✓	L. R. Todechiney
C. S. Soong	G. D. Merritt
E. D. Flotard	D. W. Bottrell
K. Asbury	J. O. 70.23
QA 11-13	WP-2043C

ATTACHMENTS: (Comments)

C-296

COMMENTS FOR FIELD SAMPLING ACTIVITY AUDIT OF ORNL  
PERSONNEL AT ARGONNE NATIONAL LABORATORY

Comments are listed in the order of importance with one being most important to the integrity of the sample.

1. Exhaust from the trailer mounted power auger was directed through the open rear doors of the "clean" equipment van. Although the rear doors were only occasionally opened, the side doors were open the entire time with the distinct exhaust odor present where the sample bottles were filled. Sample aliquots at the sites where this occurred include hydrocarbons, volatiles and semi-volatiles. In addition, two 5-gallon gasoline cans were stored in the van next to the sample cooler. A recommendation was made to use two vehicles, one for "clean" sampling equipment and the other for gasoline, oil, etc., and to remain hitched to the auger trailer to stabilize it.
2. At some of the sample sites where volatile organics aliquots were to be collected, a post hole digger was used to remove the soil and then all soil composited prior to filling the volatile organics aliquot. Suggest using a zero contamination sampler to collect VOA samples prior to post hole digging and compositing the rest of the aliquots.
3. Only one employee was sent to collect sample numbers 568, 579, 682 and 693 of request AR500. Not having a second "clean" person on the team resulted in gloved hands contaminated with chemical wastewater coming in contact with the notebook, pen, Horiba Instrument, Beta-Gamma Meter, cooler handles, sample bottles, Kimwipe<sup>®</sup> box etc.
4. Between two different samples for the same sample request, post hole diggers and compositing utensils were not decontaminated. Sampling equipment must be decontaminated between each sample.
5. In one case, soil was being collected from surface to three feet instead of one foot to three feet. This was corrected by the audit team prior to the sample being mixed. Suggest reading sample depths carefully.
6. In several cases, sampling equipment such as radiation meters, aluminum foil, mixing equipment, etc., were being stored with the samples in the same cooler. Recommend that samples be stored only with freezer packs and packing material.
7. In two instances, samplers were observed collecting soil samples for the same sample request inconsistently. Always use identical sampling technique for samples under the same sample request number.
8. Although liquid VOA samples were poured slowly, it was recommended that both bottle and dipper be tilted to complete a smooth transaction of the liquid.

9. In one case, VOA samples were not placed in a cooler at 4°C immediately after collection. This was recommended.
10. Keep all instruments charged. In two cases, the radiation monitors gave false positive readings before discovering that the instruments were malfunctioning.
12. It was recommended that foil only be removed from sampling equipment immediately prior to sampling. This did not happen on several occasions resulting in equipment being exposed to the ground prior to sampling. The recommendation was to keep post hole diggers, trowels, auger bits, etc., on clean plastic prior to sampling if foil is removed or contains no foil.
13. Liquid samples were spilled while filling narrow mouth bottles. A funnel was recommended.
14. A gloved hand came in contact with a 40 mL vial teflon liner which fell out onto plastic. Recommend that sampler use a clean spare bottle.
15. 125 mL bottles replaced 250 mL bottles in all cases as 250 mL bottles were not available. It was recommended that this be documented in the field notebook each time that this is done.
16. Prior to collection of soil, an area was not cleared large enough to prevent leaves and twigs from entering the hole in a few cases. Recommend a minimum of two feet clearance from center of hole.
17. Liquid samples requiring preservation are not preserved until the team returns to the logistics room. It was recommended that a few drops of preservative be placed in the aliquot bottle prior to sampling and pH checked and adjusted if necessary upon return.
18. In some cases, sample bottles, full and empty, were placed on the ground, van floor etc. Recommend that these bottles be placed on clean aluminum foil or plastic.
19. Post hole diggers were not marked with depth gradients which required samplers to use a tape measure each time depth was checked. The tape measure is a possible source of contamination.
20. When filling sampling aliquots with a dipper from a chemical wastewater tap, three dipper volumes were required. This needs to be documented in the remote case that the three dipper volumes are not homogenous.

#### SUMMARY

Sampling teams worked well together and exhibited good collection techniques in general. However, concern for contamination of samples needs major improvement. It does not appear that all personnel were trained together. Recommend a single training course for consistency in sample handling. Concern was also voiced by employees about safety. Items mentioned by employees or noticed by the audit team regarding safety were; no organic monitoring devices used during sampling, Tyvek coveralls not available for samplers (some sampled in street clothes), respirator not worn during collection of AR500 samples when obvious odors were present in an enclosed area at the tap, three post size holes three feet deep not filled in upon completion of sample collection, radiation instruments not well maintained, and a sampler allowed to collect a hazardous sample in an enclosed area alone. Sample packaging procedures as well as notebook documentation was good. Sample grids, depths, etc., had to be modified on several occasions and there is good documentation showing this.

U.S. DEPARTMENT OF ENERGY  
Environmental Survey

FIELD AUDIT REPORT

ARGONNE NATIONAL LABORATORY SITE  
Argonne, Illinois

November 16-17, 1987

National Enforcement Investigations Center  
Contract Evidence Audit Team  
12600 West Colfax Avenue, Suite C-310  
Lakewood, Colorado 80215

This work was conducted on behalf of the Environmental Protection Agency's (EPA) National Enforcement Investigations Center (NEIC) under EPA Contract #68-01-7369.

FIELD AUDIT REPORT

U.S. Department of Energy  
Office of Environmental Audit & Compliance

Argonne National Laboratory Site  
Argonne, Illinois

November 16-17, 1987

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Kevin Cabble  
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12600 West Colfax Avenue  
Suite C310  
Lakewood, Colorado 80215  
(303) 233-1248

Troy Sanders

C-301

## INTRODUCTION

On November 16-17, 1987, NEIC's Contract Evidence Audit Team (CEAT-TechLaw) personnel conducted a field audit of the document control, chain-of-custody, and sample handling procedures followed by the Oak Ridge National Laboratory (ORNL) during sampling conducted at the Argonne National Laboratory (ANL) site in Argonne, Illinois. Present were those personnel listed on the cover page of this report.

The Argonne Sampling and Analysis Plan was provided to the CEAT personnel by ORNL prior to the audit. Field sampling activities were reviewed for conformance to the Sampling and Analysis Plan and NEIC's Policies and Procedures Manual.

This field evidence audit report contains a description of the audit activities conducted by the CEAT during the November 16-17, 1987 sampling episode. The report of these activities is arranged into the following sections: Audit Sequence, Sampling Plan, Accountable Field Documents, Field Observations, and Summary.

## AUDIT SEQUENCE

On November 16-17, 1987, sampling of soils and surface water took place. Sampling-related documents were examined in the field and at the DOE Environmental Survey on-site office. On the afternoon of November 16, 1987, packing of samples for shipment was observed by the CEAT auditor.

A debriefing was held at the conclusion of the audit on November 17, 1987. Recommendations and comments concerning the overall performance of the sampling teams were made at this time by the EMSL and CEAT auditors.

## SAMPLING PLAN

A Sampling and Analysis Plan was prepared for the sample collection effort by the DOE Environmental Survey and ORNL. The Sampling and Analysis Plan was reviewed and approved by the DOE, ORNL, and EPA/EMSL-LV.

The sampling plan included the following:

- o Introduction describing purpose and goals of the DOE Environmental Survey.
- o ANL site background information.
- o Sampling and analysis strategy.

- o Field sampling guidelines and sample control.
- o Quality assurance/quality control.
- o Data management and analysis.
- o Logistics, schedule, and cost.
- o Health, safety, and security.

Applicable sections of this Sampling and Analysis Plan were used by the auditor as part of the basis for the field evidence audit.

#### ACCOUNTABLE DOCUMENTS

An accountable document system was used. As specified in the sampling plan, field logbooks, custody records, and sample tags contained document control numbers. Custody of the documents was maintained by C. Wear. Control numbers and disposition of the documents were recorded in a logbook, which remained in the on-site survey office. Entries in this logbook were recorded clearly and neatly; however, the logbook cover was unlabeled.

(The CEAT auditor recommended that an appropriate title (e.g., Document Control Logbook), site name, and sampling organization be added to the front cover of this logbook.)

#### Project Logbook

A Project Logbook was maintained by F. Taylor and K. Owenby, the sampling team leaders. The following information was recorded in this logbook:

- o Table of Contents
- o Name and Signature of Sample Team Members
- o Sample Team Assignments
- o Shipment Log
- o Unusual Weather Conditions
- o Sample Deviations
- o Visitors
- o Chronology of Samples
- o Auditors Comments
- o Daily Meeting Notes

Entries in the logbook were legible and clearly recorded.

The logbook appeared to contain all required information, with the following exceptions:

- o Errors were not consistently corrected properly.
- o Information present in four sections of the logbook (weather conditions, visitors, sample chronology, and sample team signatures) was not current at the time of the audit.

(The CEAT auditor recommended that errors be corrected by drawing a single line through the errors, then initialing and dating the correction, and that all sections of the logbook be updated daily.)

#### Field Logbooks

General Field Logbooks were used by sampling team members to record information and data pertaining to the collection of soil and surface water. Each field logbook contained pertinent sample collection information for specific request numbers. The logbook consisted of the following forms:

- o Table of Contents
- o Task Team Activity Log Sheet
- o Sample Log Sheet

After sample collection, the logbooks were grouped by media (water, soil, vegetation, and air) into "three-ring" binders. The auditor observed that the various logbook forms were not identified with unique page numbers. Thus, document control of all logbook pages completed in the field was not maintained.

(The auditor recommended that all Field Logbook pages containing sample information be assigned a unique page number and that this number be tracked, thereby accounting for all logbook pages involved in field activities.)

All of the information in the General Field Logbooks was entered neatly and clearly and appeared to contain all required information, with the following exceptions, which occurred infrequently in one or two of the notebooks:

- o Field sampling information was not recorded directly into the Field Logbook, but on "note pads" to be transcribed into the logbooks at a later time.
- o Unused logbook sheets were not consistently voided.
- o Errors were occasionally written over or obliterated.

(The CEAT auditor recommended that all required information be recorded directly into Field Logbooks, unused Field Logbook pages be consistently marked through with a slash and labeled "Void," and that errors be corrected by drawing a single line through the error and initialing and dating the correction.)

An Instrument Calibration Logbook was used to record instrument model numbers and calibration data. The logbook contained a Table of Contents and Instrument Calibration Log Sheets for each field measurement.

The Instrument Calibration Logbook data was neatly recorded and appeared to contain all required information. All forms were completed, signed, and dated. It was observed that occasionally corrections to errors were not signed and dated.

(The CEAT auditor recommended that all errors be corrected by drawing a line through the error, then initialing and dating the correction.)

#### Sample Tag Labels

Sample tags were affixed to each sample jar as specified in the sampling plan. Tags were pre-printed with "DOE Environmental Survey" and contained document control numbers to uniquely identify each sample.

Sample tags were examined by the CEAT auditor prior to the packing of samples into coolers. The tags were completed correctly and included the following:

- o Sample Identification Number
- o Date and Time of Collection
- o Sampling Location
- o Sampler Name
- o Analysis Required
- o Concentration
- o Radiation Screening Results

Tag numbers were also recorded on the Chain-of-Custody Records.

Several sample labels were examined by the CEAT auditor at the time of sample collection and prior to the packing of samples into the cooler. The majority of sample labels were completed correctly; however, one or two labels examined had errors which were not corrected properly. Sample labels contained the following information:

- o Sample Identification Number
- o Collection Date and Time
- o Analysis Required
- o Preservative

(The CEAT auditor recommended that errors should be corrected by drawing a single line through the error, then initialing and dating the correction.)

#### Chain-of-Custody Records

EPA-type Chain-of-Custody records were used during the sampling episode. All sample numbers were listed on the Chain-of-Custody records and samples were arranged in coolers either as Organics or Inorganics, indicating their destination upon receipt at ORNL.

All Chain-of-Custody records examined by the auditor were completed and appeared to be consistent, with the following exceptions:

- o Blank areas of Chain-of-Custody Records were not consistently slashed through to prevent further entries.
- o Errors were not corrected properly.
- o Unused Chain-of-Custody Records were not consistently voided.

(The CEAT auditor recommended that blank portions of custody records be slashed or "z'd" through; that errors be corrected by drawing a single line through errors, then initialing and dating the correction; and that unused Chain-of-Custody Records be slashed or "z'd" through and then be labeled "Void.")

#### FIELD OBSERVATIONS

Soil and surface water samples were collected during the two-day audit period. Collection of soil samples was observed by the CEAT auditor on November 16, 1987. Samples were obtained by F. Taylor, W. Parsons, and B. Hensley. Documentation related to the collection of samples was completed by B. Hensley.

Soil samples were collected from boreholes augered with a portable drilling rig. These samples were spooned into pre-labeled sample bottles by B. Hensley. Sample tags were attached to the bottles at the on-site survey office after the radiation screen was completed.

Procedures for preparing samples for shipment were observed by the CEAT auditor. Samples were prepared by K. Owenby and C. Wear. Labels, tags, and Chain-of-Custody Records accompanying several coolers were examined for completeness. Tags and custody records were compared to ensure that tag numbers and sample information were consistent.

Samples were prepared for shipment according to specifications in the sampling plan, with the following exceptions:

- o Drain plugs were not consistently taped shut on the inside and outside of the coolers.
- o The coolers were not lined with a heavy duty trash bag prior to the addition of vermiculite and samples.

(The CEAT auditor recommended that drain plugs on coolers be consistently taped shut on both the inside and outside surfaces; and that coolers be lined with a heavy duty plastic bag before packing samples, as specified in Appendix I of the DOE Environmental Survey Manual.)

The CEAT auditor observed the collection of surface water samples by S. Hall on November 17, 1987. Water samples were collected from several acid retention tanks and placed into pre-labeled sample containers. Field measurements, including sample temperature, pH, and conductivity, were obtained and recorded in the Field Logbook by S. Hall. The samples were returned to the on-site survey office and prepared for shipment.

Several logbooks were examined by the CEAT auditor on the afternoon of November 17, 1987. These included the Project Logbook, Document Control Logbook, Instrument Calibration Logbook, and numerous Field Logbooks.

#### SUMMARY

A debriefing was held on November 17, 1987 at the on-site survey office. Present were all personnel listed on the cover of this report.

The following comments and recommendations were made regarding logbooks, sample labels, Chain-of-Custody Records, and shipping procedures:

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### Project and Document Control Logbooks

- o All sections of the logbooks should be updated promptly when applicable information becomes available.
- o All logbook covers should contain, as a minimum, the title, site name, and sampling organization.
- o Errors should be corrected by drawing a single line through the error, then initialing and dating the correction.

### Field and Instrument Calibration Logbooks

- o All field sampling information should be recorded directly into Field Logbooks.
- o Unused Field Logbook pages should be consistently marked through with a slash and labeled "Void."
- o Assign unique page numbers to each Field Logbook page which contains sample information, and track this page number.
- o Errors should be corrected by drawing a single line through the error, then initialing and dating the correction.

### Sample Labels

- o Errors should be corrected by drawing a single line through the error, then initialing and dating the correction.

### Chain-of-Custody Records

- o Blank portions of the Chain-of-Custody Records should be marked through with a slash or "z'd" out.
- o Unused Chain-of-Custody Forms should be consistently marked through with a slash and labeled "Void."
- o Errors should be corrected by drawing a single line through the error, then initialing and dating the correction.

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Sample Shipment Procedures

- o The drain plug on coolers should be taped shut on the inside and outside surfaces.
- o A plastic liner bag should be placed in each cooler prior to the addition of vermiculite and packing of samples.

The field evidence audit of ORNL's sampling team at the Argonne National Laboratory site was concluded on November 17, 1987.

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ON-SITE SAMPLING EVALUATION FOR  
ARGONNE NATIONAL LABORATORY  
ARGONNE, ILLINOIS

This checklist was compiled utilizing the  
Sampling Plan for the Argonne National Laboratory  
Dated October 28, 1987

by

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SAMPLING FIELD AUDIT

I. GENERAL INFORMATION

Purpose: The purpose of this sampling evaluation is to document the extent to which procedures identified in the sampling protocol and/or quality assurance plan are being followed with respect to implementing specified field tests, chain-of-custody, record keeping, quality assurance, and sampling procedures and techniques, and sample handling methods.

Audit Dates: 11/13/87 to 11/17/87

Facility/Site Information

Facility/Site Name: Argonne National Laboratory

Facility/Site Address or Location: 9700 South Cass Avenue

Argonne National Laboratory, Argonne, Illinois 60439

Facility/Site Telephone No.: (312) 971-3311  N/A

Facility Contact (Name/Title): Lyle Cheever DOE-ANL Contact

Jim Specht DOE-ANL 971-4000

N/A

Function/Description of Facility/Site: RESEARCH IN THE BASIC ENERGY AND RELATED SCIENCES (PHYSICAL, CHEMICAL, MATERIAL, NUCLEAR, BIOMEDICAL AND ENVIRONMENTAL) AND SERVES AS AN IMPORTANT ENGINEERING CENTER FOR THE STUDY OF NUCLEAR AND NON-NUCLEAR ENERGY SOURCES.

Media Being Sampled:

- |  |  |
|--|--|
| <input checked="" type="checkbox"/> Soils          | <input type="checkbox"/> Containerized Liquids         |
| <input type="checkbox"/> Sludges/Sediments         | <input type="checkbox"/> Ambient Gases                 |
| <input type="checkbox"/> Bulk Materials            | <input type="checkbox"/> Soil Gases                    |
| <input checked="" type="checkbox"/> Surface Waters | <input checked="" type="checkbox"/> Ionizing Radiation |
| <input type="checkbox"/> Ground Water              |  |

Sampling Team Information

Team Contact (Name/Title/Affiliation): John Murphv - ORNL (Y-10)

Keith Owenby - ORNL (Y-10)

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Team Members (Name/Title/Affiliation):

1. Betty Hensley ORNL

2. Wayne Parsons ORNL

3. Fred Taylor ORNL

4. Steve Hall Grand Junction

5. Lisa Lesperance Grand Junction

6. Marv Smuin Grand Junction

7. John Zutman Grand Junction

8. Chris Muhr Grand Junction

9. J. B. Watson ORNL

10. Cindy Wear ORNL

Team Contact Telephone No.: (615) 574 - 5971 FTS 624 - 5971.

Team Contact Address: Oak Ridge National Laboratory

Bethel Valley Road

Oak Ridge, TN 37831 c/o K. Owenby BLDG 4500 South

Audit Team Information

Team Leader (Name/Title/Affiliation): Kevin Cabbie, Sr. Scientist.

Lockheed - EMSCO

Team Members (Name/Title/Affiliation):

1. Lewis Todechinev, Sr. Engineer, Lockheed - EMSC
2. Harold Vincent, Project Manager, U.S. EPA
3. Trov Sanders, Consultant Associate, Tech Law
4. \_\_\_\_\_
5. \_\_\_\_\_
6. \_\_\_\_\_
7. \_\_\_\_\_
8. \_\_\_\_\_
9. \_\_\_\_\_
10. \_\_\_\_\_

Team Contact Telephone No.: (702) 734 - 3268 FTS 595 - 2129.

Team Contact Address: U.S. EPA c/o Harold Vincent  
944 E. Harmon  
Las Vegas, NV89119

Debriefing

A debriefing will be conducted onsite with sampling personnel.

Date/time and location of debriefing: LOGISTICS ROOM FOR ORNL PERSONNEL  
(RM 138H IN BLDG 203) FRIDAY NOVEMBER 13, AND TUESDAY NOVEMBER 17 AT 5:00 P.M.

Names of those attending debriefing:

1. Kevin Cabble November 13 and 17
2. Lewis Todechinev November 13 and 17
3. John Murphy November 13

- ↓
4. Gindy Wear November 13 and 17
  5. Wayne Parsons November 13 and 17
  6. Betty Hensley November 13 and 17
  7. Fred Taylor November 13 and 17
  8. Lisa Lesperance November 13
  9. Marv Smuin November 13
  10. John Zutman November 13
  11. Chris Muhr November 13
  12. J. B. Watson November 13
  13. Steve Hall November 13
  14. Kieth Owenby November 17
  15. \_\_\_\_\_
  16. \_\_\_\_\_
  17. \_\_\_\_\_
  18. \_\_\_\_\_
  19. \_\_\_\_\_
  20. \_\_\_\_\_

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II. ORGANIZATION AND PERSONNEL - MANAGEMENT STRUCTURE

Project Manager: Bob Fitts

Sample Team Leaders: John Murphv, K. Owenbv, F. Tavlör

QA Officer: Keith Owenbv

Data Management: Cindy Wear

CERCLA Sampling: Fred Tavlör

RCRA Sampling: Fred Tavlör

Radiation Sampling: N/A

Surface Water Sampling: Steve Hall, Keith Owenbv

Ground Water Sampling: Wayne Parsons

Sample Control Officer: John Murphv, Keith Owenbv

Health and Safety Officer: Fred Tavlör

Project Director: (Individual responsible for overall technical effort):

Bob Fitts

1. Sample Preparation: (Individual(s) responsible for preparing samples for analysis). Name, Media, and Experience.

J. B. Watson - 11/13

Keith Owenbv - 11/16 - 11/17

2. Do personnel assigned to this project have the appropriate education and/or experience to successfully accomplish the objectives of this program?

Yes  No Comments: ALL TEAM LEADERS PARTICIPATED AT PAST DOE

SAMPLING SITES (PANTEX, LLNL, SNL)

3. Are resumes available for all sampling personnel?

Yes  No Comments: RECOMMEND THAT RESUMES BE PROVIDED TO  
VERIFY EXPERIENCE OF SAMPLING PERSONNEL.

4. Is the sampling organization adequately staffed to meet project commitments in a timely manner?

Yes  No Comments: SAMPLING IS ONE DAY AHEAD OF SCHEDULE.  
ADEQUATE NUMBER OF PERSONNEL ON EACH SAMPLING TEAM (2 OR 3).

5. Was the Project Director and/or Manager available during the evaluation?

Yes  No Comments: JOHN MURPHY 11/13 AND KEITH OWENBY 11/16-17  
WERE AVAILABLE AS TEAM LEADERS. THIS WAS SUFFICIENT AS THESE PERSONNEL WERE  
COMPLETELY KNOWLEDGEABLE ABOUT THE SITE OPERATIONS.

6. Are the same personnel performing on-site sampling procedures as those described in the Sampling Plan and/or QA plan?

Yes  No Comments: \_\_\_\_\_

III. GENERAL FACILITIES

The sampling field work is headquartered in ARGONNE, ILLINOIS (city & state)  
Sample team personnel and the on-scene manager work out of this facility.

1. Do the sampling and/or sample preparation facilities have adequate workspace?

Yes     No    Comments: LARGE LOGISTICS/PREPARATION ROOM. LARGE  
STORAGE AREA IN BASEMENT.

2. Is the sampling and/or sample preparation facility maintained in a clean and organized manner?

Yes     No    Comments: ORGANIZED WELL. ROOM WAS CLEAN EXCEPT FOR  
HOOD.

3. Are hoods provided for work with dusty, volatile or radioactive materials?

Yes     No    Comments: HOOD WAS NOT VERY CLEAN.

4. Are adequate facilities (including cold storage) provided for storage of samples?

Yes     No    Comments: REFRIGERATORS (ROOM G150) AND FREEZERS FOR  
BLUE ICE IN BASEMENT.

5. Are the temperatures of the cold storage units recorded daily in logbooks?

Yes     No    Comments: \_\_\_\_\_

6. Are contingency plans available if freezers malfunction?

Yes     No    Comments: FREEZERS WERE RENTED. WOULD SWITCH THEM  
OUT IF THEY MALFUNCTIONED.

7a. ASTM Type II water is produced by distillation or deionization so that its conductivity is less than  $1 \mu\text{mho/cm}$ . Is the sampling facility utilizing ASTM Type II water?

Yes     No    Comments: MILLI-O SYSTEM WATER SHIPPED TO SITE FROM  
ORNL (Y-10 FACILITY)

7b. If yes, is the conductivity of the ASTM Type II water routinely checked and recorded?

Yes     No    Comments: WATER CHECKED AT 18 MEGA OHMS PRIOR TO  
COLLECTION.

7c. Can the sampling supervisor document that ASTM Type II water is available for preparation of standards and blanks?

Yes     No    Comments: GENERAL LOGBOOK.

7d. What is the source of the ASTM Type II water? ORNL (Y-10 LAB)

Comments: \_\_\_\_\_

8. Are waste disposal policies/procedures adequate?

Yes     No    Comments: \_\_\_\_\_

9. Is the sampling and/or sample preparation facility secure?

Yes     No    Comments: SPARE EQUIPMENT IN BASEMENT WAS NOT SECURE.

IV. QUALITY ASSURANCE/QUALITY CONTROL (QA/QC) PLAN-SAMPLING PROTOCOLS

1. Is a QA/QC Plan available for review?

Yes       No      Comments: OCTOBER 1987

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2. Does the QA/QC Plan and/or sampling protocol discuss the objectives of the sampling program and how the sampling approach(es) will satisfy program requirements?

Yes       No      Comments: \_\_\_\_\_

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3. Are levels of precision and confidence levels identified in the QA/QC Plan?

Yes       No      Comments: \_\_\_\_\_

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4. Does the QA/QC Plan and/or sampling protocol describe documentation and sample control procedures, i.e. the system to be used for chain-of-custody identifying, logging and tracking all samples?

Yes       No      Comments: \_\_\_\_\_

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5. Are sampling methods, and sampling equipment discussed in the QA/QC Plan and/or sampling protocol?

Yes       No      Comments: \_\_\_\_\_

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6. Does the QA/QC Plan and/or sampling protocol identify criteria used for selecting the media (e.g., soil, etc.) to be sampled?

Yes       No      Comments: \_\_\_\_\_

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7. Does the sampling protocol identify criteria for selecting sampling sites for each media?

Yes       No      Comments: \_\_\_\_\_

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8. Does the QA/QC Plan and/or sampling protocol identify the size, number, locations, and types of samples to be collected?

Yes       No      Comments: \_\_\_\_\_

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9. Does the QA/QC Plan and/or the sampling protocol describe procedures, for compositing or other sample reduction methods?

Yes     No    Comments: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

10. Are the type of sample containers identified in the sampling plan?

Yes     No    Comments: 125 ML CONTAINERS USED INSTEAD OF 250 ML.  
\_\_\_\_\_  
\_\_\_\_\_

11. Are methods and materials used to clean sample containers identified in the sampling plan?

Yes     No    Comments: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

12. Are procedures and materials for field decontamination of sampling equipment discussed in the sampling plan?

Yes     No    Comments: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

13. Has a Health and Safety Project Plan been prepared?

Yes     No    Comments: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

14a. For all instruments, is the date of each calibration or inspection recorded in the instrument's logbook?

Yes     No    Comments: LAB CALIBRATION PERFORMED AT BEGINNING OF SURVEY. FIELD CALIBRATIONS PERFORMED ON THE SAME DAY OF INSTRUMENTS USE. (NO ADJUSTMENTS MADE DURING OBSERVED CALIBRATION).

14b. If yes, does the information include date, person performing the activity, type of inspection, and a list of any discovered defects?

Yes     No    Comments: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

15. Are the results of routine calibration checks recorded in the field sampling logbook?

Yes     No    Comments: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

16. Are the date, time, standards used, and the name of the person conducting the calibration recorded in the calibration logbook?

Yes     No    Comments: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

17. Are direct radiation instruments only used by personnel trained in their use?

Yes     No    Comments: RADIATION INSTRUMENTS NOT TROUBLE SHOT OR MAINTAINED PROPERLY. SAMPLERS ON TWO CASES ENTERED ERRONEOUS READINGS IN LOGBOOK NOT REALIZING INSTRUMENT WAS MALFUNCTIONING.

18. Are blanks prepared and packaged by the appropriate personnel, at the appropriate time?

Yes       No      Comments: \_\_\_\_\_

\_\_\_\_\_  
\_\_\_\_\_

V. SAMPLING PROCEDURES

1. Have any changes (additions or deletions) to the following listed media been made?

Yes       No      Comments: SOIL SAMPLE DEPTHS, LOCATIONS AND NUMBERS  
OF SAMPLES COLLECTED WERE CHANGED IN SOME CASES.

Are these changes noted in the program's logbook?

Yes       No      Comments: CHANGES WERE NOTED IN THE FIELD NOTEBOOKS.

2. The number of subsamples collected for a composite should be recorded in the field logbook; is this being done?

Yes       No      Comments: .

3. Are sampling depths being documented?

Yes       No      Comments: DEPTHS WERE DIFFERENT FROM THOSE IN THE  
SAMPLING PLAN IN SEVERAL CASES, BUT THIS WAS DOCUMENTED WITH REASONS.

4. Are samples being preserved and stored in ice chests?

Yes       No      Comments: SAMPLES SHOULD BE AT LEAST PARTIALLY  
PRESERVED (i.e. 2 DROPS IN 150 ML BOTTLE) IN THE FIELD PRIOR TO RETURN TO  
THE LAB.

Table V-1. The following soil samples will be collected during the sampling at Argonne National Laboratory.

AR Number	Location	Sampling Method	No. of Samples	Other
404016	West of bldg 6.	Auger, shovels	1 G	60 segments Collect samples at 3 to 8 ft and 15 to 20 ft.
404027	Adjacent to existing			
404038	tanks			
404049				
404050				
404061				
405017	South end of	Auger, shovels	1 G	60 segments Collect samples at 3 to 8 ft and 18 to 23 ft.
405028	bldg 212			
405039	Adjacent to			
405040	existing tanks			
405051				
405062				
806012	Earthen lagoon	Auger	1 G	pH 80 segments Auger to approx 10 ft in vegetated area Collect samples at 0-5 and 3-10 ft
806023	at Wastewater			
806034	Treatment Plant			
806045	Area 570			
806056				
806067				
806067				
806078				
806089				

(continued)

Table V-1. (Continued).

AR Number	Location	Sampling Method	No. of Samples	Other
810018	Former transfer line between bldgs 19 and 34 and tank attached to building 34	Scoop, corer	1 G	pH Collect samples below trans- ition fill
810029				
810030				
810041				
810052				
810063				
810074				
810085				
810096				
815013	Suspected location of former Site A Landfill	Shovel, Post hole, Digger	1 G	pH Max 10 ft deep
815024				
815035				
815046				
815057				
815068				
815079				
815080				
815091				
815104				
815115				
815126				
807013	Abandoned NIKE Site samples	Auger	1 G	pH See page 3-180
807024				
807035				
807046				
807057				
807068				
807079				
807080				

Table V-1. (Continued).

AR Number	Location	Sampling Method	No. of Samples	Other
808014	Underground Fuel	Auger	1 G	See page 3-133
808025	Storage Tank at			
808036	Abandoned NIKE			
808047	Site			
-----				
801017	South Base of	Hand corer	1 G	pH
801028	ENE 319			See page 3-157
801039	Landfill			
801040				
801051				
801062				
-----				

VI. Soils

1. For AR404, is the area gridded into 60 segments?

Yes       No    Comments: NOT OBSERVED

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2. For AR404, is the auger to a maximum depth of 20 ft.?

Yes       No    Comments: NOT OBSERVED

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3. For AR404, are soil samples collected from each of 2 intervals (3 to 8 ft. and 15 to 20 ft.)?

Yes       No    Comments: NOT OBSERVED

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4. For AR405, are soil samples collected from each of 2 intervals (3 to 8 ft. and 18 to 23 ft.)?

Yes       No    Comments: \_\_\_\_\_

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5. For AR404, 405 is the presence of water in the borehole recorded?

Yes       No    Comments: \_\_\_\_\_

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6. For AR806 is the lagoon bottom gridded into 80 segments, four of which are randomly selected for sampling?

Yes       No      Comments: \_\_\_\_\_

\_\_\_\_\_

7. For AR806, is augering to a maximum depth of 10 ft. in the vegetated area?

Yes       No      Comments: KEEP AUGER BITS ON PLASTIC INSTEAD OF GRASS. ALSO KEEP GAS CANS OUT OF VAN WITH SAMPLES.

\_\_\_\_\_

8. For AR 806, are soil samples from 0-5 ft. and 5-10 ft. intervals?

Yes       No      Comments: \_\_\_\_\_

\_\_\_\_\_

9. For AR810, is each area (A, B, and C) gridded into 60 segments?

Yes       No      Comments: NOT OBSERVED

\_\_\_\_\_

10. For AR810, is the first sample location in each area randomly chosen from segments 1-20?

Yes       No      Comments: NOT OBSERVED

\_\_\_\_\_

11. For AR810, is each soil grab sample collected from below the transition from fill to native soil (A: approx. 8-10 ft. below surface; B: approx. 10 ft. below surface; C: approx. 0-1 ft. below surface)?

Yes  No Comments: NOT OBSERVED

12. For AR815, are samples collected from within the 20 ft. radius of the sampling point?

Yes  No Comments: \_\_\_\_\_

13. For AR815, is the area gridded into 80 segments from which 4 are randomly selected for sampling?

Yes  No Comments: THIS WAS AMENDED TO 9 PRE-SHAPED PIECES BECAUSE OF THICK BRUSH.

14. For AR815, is augering to a maximum depth of 10 ft.?

Yes  No Comments: AUGER RIG UNABLE TO GET INTO AREA. HOLE WAS DUG TO 4 FEET WITH A POST HOLE DIGGER. LEAVES SHOULD BE BRUSHED AWAY FROM HOLE (AT LEAST A 2 FT. RADIUS).

15. For AR807 is area A gridded into 50 segments?

Yes  No Comments: \_\_\_\_\_

16. For AR807, are the 2 Area A samples collected downgradient, but near the pipe discharge?

Yes       No      Comments: \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

17. For AR807, is Area B a 5 yd. radius semicircle which is located about 30 ft. south of the distribution boxes?

Yes       No      Comments: \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

18. For AR808, is augering to a depth of 10 ft.?

Yes       No      Comments: NOT OBSERVED

\_\_\_\_\_

\_\_\_\_\_

19. For AR808, is an 8-10ft. interval composite soil sample collected at each location?

Yes       No      Comments: NOT OBSERVED

\_\_\_\_\_

\_\_\_\_\_

20. For AR808, is the area gridded into 80 segments?

Yes       No      Comments: NOT OBSERVED

\_\_\_\_\_

\_\_\_\_\_

21. For AR801, is the area 20 ft north from the south end of the landfill mound and west of the stream?

Yes       No      Comments: \_\_\_\_\_

\_\_\_\_\_  
\_\_\_\_\_

22. For AR801, is augering to a maximum depth of 21 ft.?

Yes       No      Comments: TRUCK MOUNTED AUGER NOT ABLE TO ACCESS

LANDFILL SAMPLE COLLECTED USING POST HOLE DIGGER.

\_\_\_\_\_

23. For AR801, are composite soil samples collected from intervals of 1-11 ft. and 11-21 ft.?

Yes       No      Comments: ONLY ABLE TO DIG 3 FEET DOWN.

\_\_\_\_\_  
\_\_\_\_\_

V.1 Soils (Generic)

1. In the sampling log, are site location, depth, and soil type recorded?

Yes     No    Comments: THERE WERE FREQUENT DEVIATIONS  
FROM SAMPLING PLAN BUT THESE WERE WELL DOCUMENTED.

2. If an auger is used:

2a. Are accumulated soils periodically removed to prevent loose material from falling back into the bore hole?

Yes     No    Comments: \_\_\_\_\_

2b. After reaching the desired depth, is the auger removed slowly and carefully from the bore hole?

Yes     No    Comments: \_\_\_\_\_

2c. Is the surface area cleared of debris?

Yes     No    Comments: LEAVES FREQUENTLY ENTERED HOLE.

3. If a thin-wall tube sampler is used:

3a. Is the sampler carefully lowered to avoid hitting the sides of the bore hole?

Yes     No    Comments: N/A

3b. Is the sampler gradually forced into the soil?

Yes       No      Comments: N/A

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4. If a hand corer is used:

4a. Is the corer forced into the media with a smooth continuous motion?

Yes       No      Comments: N/A

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4b. Is the hand corer twisted and withdrawn in a single smooth motion?

Yes       No      Comments: N/A

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5. If a spade or scoop is used, is it non-plated?

Yes       No      Comments: ALUMINUM TROWELS. SCOOPS USED.

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6. Is the sampling equipment decontaminated as described?

Yes       No      Comments: POST HOLE DIGGERS AND TROWELS NOT DECONTAMINATED BETWEEN DIFFERENT SAMPLES OF THE SAME REQUEST NUMBER.

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7. Are the decontamination liquids contained for disposal?

Yes       No      Comments: \_\_\_\_\_

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8. Do sample labels include date, time of collection, and preservative?

Yes       No      Comments: \_\_\_\_\_

9. For volatile organic samples:

9a. Are volatile organic samples collected first?

Yes       No      Comments: SAMPLES COLLECTED BY POST HOLE  
DIGGER WERE MIXED THOROUGHLY PRIOR TO FILLING VOA SAMPLE CAUSING MUCH  
OF THE VOLATILES TO BE LOST.

9b. Is the headspace in the sample containers minimized?

Yes       No      Comments: \_\_\_\_\_

9c. Are the samples immediately placed in a 4°C environment?

Yes       No      Comments: NOT IN ALL CASES

10. Are sample jar lids retightened after initial cool down or immediately prior to shipping?

Yes       No      Comments: \_\_\_\_\_

Table V-4. The following surface water samples will be collected during the sampling at Argonne National Laboratory.

AR Number	Location	Sampling Method	No. of Samples	Other
309010	Drainage from buildings	Dipper, Horiba	1 G	pH, cond., temp.
309021	815 and 817.	Dipper, Horiba	1 G	Volatiles
309032		Dipper, Horiba		
-----				
500013	Laboratory Sewer	Horiba, tap	36 G	ph, cond., temp.
to	Wastewater Treatment		33 VOA	Volatiles
500693	Facilities			
-----				
503016	Unlined Impoundment	Dipper	1 G	pH, cond., temp.
503027	adjacent to building		1 G	Volatiles
503038	145		1 G	
-----				
508011	Sumps located near	Dipper	1 G	ph, Volatiles
508022	building 108		1 G	
508033			1 G	
-----				
818016	Seep at Plot M Site	Dipper, Horiba	1 G	pH, cond., temp.
818027			1 G	Volatiles
818038			1 G	
-----				
400012	Well #1	Dipper	1 G	ph, cond., temp.
401013	Well #2	Dipper	1 G	Volatiles, run
402014	Well #3	Dipper	1 G	tap for 10 min-
403015	Well #4	Dipper	1 G	utes
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V.2 Surface Water

1. Is oil and grease collected if a sheen is present for AR309?

Yes       No      Comments: NOT OBSERVED

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2. Are samples collected as close to culvert as possible for AR309?

Yes       No      Comments: NOT OBSERVED

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3. Are volatile organic components collected before and after circulating the contents of the tank for AR500?

Yes       No      Comments: VOLATILES COLLECTED, CIRCULATED EACH TANK FOR 20-30 MINUTES, THEN COLLECTED SECOND SET OF VOLATILES.

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4. Are samples collected on 3 non-consecutive days for AR500?

Yes       No      Comments: SAMPLE OBSERVED WAS LAST OF 3 SAMPLES COLLECTED ON NON-CONSECUTIVE DAYS COLLECTED NOVEMBER 11,13,17.

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5. Are water samples collected at the same location as the silt samples (Request 507) for AR508?

Yes       No      Comments: NOT OBSERVED

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6. For requests 400 through 403, is the tap allowed to run for 10 minutes before collection of samples?

Yes       No      Comments: NOT OBSERVED

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V.2 Surface Water (Generic)

1. Did each sampling device used have a volume of at least 500 mL?

Yes       No      Comments: 1000 ML DIPPER

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2. If a dipper or pond sampler was used:

2a. Was the sample container tilted properly to fill with the least amount of disturbance?

Yes       No      Comments: POURED INTO CONTAINER AT 90° FOR

ALL ALIQUATS INCLUDING VOA ALIQUAT.

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2b. Was the dipper/pond sampler emptied slowly with minimal entry disturbance?

Yes       No      Comments: \_\_\_\_\_

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2c. Was the dipper/pond sampler allowed to fill slowly and continuously?

Yes       No      Comments: TOP EMPTIED RAPIDLY INTO DIPPER.

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3. If sample container immersion was used:

3a. Did the sampling personnel wear gloves?

Yes       No      Comments: N/A

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3b. Was the sample container tilted to fill with the least amount of entry disturbance?

Yes       No      Comments: N/A

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4. If a peristaltic pump was used:

4a. Was sampling for parameters other than volatile organics, oil or grease?

Yes       No      Comments: N/A

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4b. Was clean medical-grade silicon tubing attached to the pump head?

Yes       No      Comments: N/A

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4c. Was heavy-wall Teflon<sup>®</sup> connected to the intake side of the pump tubing?

Yes       No      Comments: N/A

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4d. Prior to sample collection, were several liters of sample allowed to pass through the system as a purge?

Yes       No      Comments: N/A

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5. If a Kemmerer bottle was used:

5a. Did sampling personnel wear gloves:

Yes       No      Comments: N/A

5b. Was the sample allowed to flow slowly down the side of the sample bottle with minimal disturbance?

Yes       No      Comments: N/A

6. Was the sample equipment decontaminated as described?

Yes       No      Comments: HCL AND RINSE.

7. Were decontamination liquids contained for disposal?

Yes       No      Comments: DISPOSED OF PROPERLY IN WASTE TANK.

8. Did the sample labels include date, time of collection, and the preservative?

Yes       No      Comments: TIMES WERE RECORDED AS SAMPLING FOR ALL ALIQUOTS WAS PERFORMED.

9. Was the sample bottle placed in an appropriate carrying container maintained at 4°C throughout the sampling & transportation period?

Yes       No      Comments: VOLATILES NOT PLACED IN COOLER IMMEDIATELY IN ONE CASE. NO THERMOMETERS TO VERIFY TEMPERATURE. SOME COOLERS NEEDED MORE BLUE ICE.

10. For volatile organic samples:

10a. Are volatile organic samples collected first?

Yes       No      Comments: \_\_\_\_\_

\_\_\_\_\_  
\_\_\_\_\_

10b. Is the headspace in the sample container minimized?

Yes       No      Comments: 40 ML VIALS COMPLETELY FULL.

\_\_\_\_\_  
\_\_\_\_\_

11. Were sample jar lids retightened after initial cool down or immediately prior to shipping?

Yes       No      Comments: \_\_\_\_\_

\_\_\_\_\_  
\_\_\_\_\_

V.3 Ionizing Radiation

1. Are sample locations surveyed for radiation hazards using portable instruments?

Yes       No      Comments: SAMPLES DID NOT SEEM FAMILIAR WITH  
THE RADIATION METERS IN A FEW CASES.

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2. Is the sample media surveyed for radiation hazards?

Yes       No      Comments: ONLY ON OCCASION. THIS SITE IS  
NOT CONSIDERED A RADIATION PROBLEM WITH THE EXCEPTION OF TRITIUM IN  
A FEW PLACES.

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3. Are the sample containers surveyed with a portable radiation detector?

Yes       No      Comments: \_\_\_\_\_

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VI. SAMPLE PREPARATION FIELD PROCEDURES

1. Sample size, container, preservatives, holding times and other comments are identified in the Sampling Plan. Are these procedures being followed?

Yes       No    Comments: 125 ML BOTTLES WERE BEING SUBSTITUTED FOR  
250 ML BOTTLES BECAUSE THEY DIDN'T HAVE 250 ML BOTTLES. THIS WAS NOT  
DOCUMENTED IN THE LOGBOOK.

2. If no, are different procedures identified and documented?

Yes       No    Comments: SEE #1

3. Are all liquid volatiles stored in 40 ml septum capped glass bottles?

Yes       No    Comments: \_\_\_\_\_

4. Are aqueous rad analysis samples collected in a 125 ml HDPE container for gross alpha and beta?

Yes       No    Comments: N/A

5. Are solid rad samples collected in 125 ml polyethylene or glass jars?

Yes       No    Comments: N/A

6. Are solid uranium samples collected in 1-liter polyethylene or glass jars?  
 Yes  No Comments: N/A

7. Are iodine-129 and technetium-89 collected in 1-liter glass containers?  
 Yes  No Comments: N/A

8. Are semi-volatile aqueous samples collected in amber glass 1-liter bottles with Teflon<sup>®</sup> lined caps?  
 Yes  No Comments: \_\_\_\_\_

9. If a sample requires refrigeration, is a sufficient quantity of freezer packs being used to maintain the sample at 4°C?  
 Yes  No Comments: SAMPLES NOT ALWAYS PLACED IN COOLER IMMEDIATELY AFTER SAMPLING (THOSE REQUIRING 4° C PRESERVATION).

10. Are all low-concentration environmental samples sealed in plastic bags?  
 Yes  No Comments: SAMPLES NOT BAGGED SEPARATELY UNTIL JUST PRIOR TO SHIPPING.

11. Are all samples placed in a plastic bag lined ice chest and packed in vermiculite?  
 Yes  No Comments: GOOD PACKING TECHNIQUE.

12. Are solid pesticide/PCB samples collected in 250 ml glass wide mouth jars with Teflon<sup>®</sup> lined caps?

Yes  No Comments: N/A

13. Are all sample preparation procedures filled out and up-to-date in the sample logbook?

Yes  No Comments: LOGBOOKS WERE NEAT AND COMPLETE IN MOST CASES.

14. Are sample preparation equipment being stored in a secure, non-contaminatory environment?

Yes  No Comments: SOME EQUIPMENT (RE COOLERS, BAGS, VERMICULITE, ACIDS ETC.) STORED IN BASEMENT WHICH WAS NOT SECURE.

15. Are all disposable sample preparation equipment being properly disposed of?

Yes  No Comments: \_\_\_\_\_

16. Are swipes being conducted to check for contaminated equipment in the sample preparation area?

Yes  No Comments: \_\_\_\_\_

17. Are all concentrated acids used for preserving the samples stored in a vented storage cabinet?

Yes  No Comments: NOT OBSERVED

18. Are any food, drink, tobacco or lotions being used in the sample preparation area?

Yes       No      Comments: \_\_\_\_\_

\_\_\_\_\_

19. Are the TOC, volatile organics, Ag, TOH, extractable organics, organochlorine pesticides, and PCB samples shielded from light.

Yes       No      Comments: \_\_\_\_\_

\_\_\_\_\_

20. Are the appropriate number of shipping blanks packed in each cooler?

Yes       No      Comments: \_\_\_\_\_

\_\_\_\_\_

VII. HEALTH AND SAFETY

1. Is a Health and Safety Coordinator (HSC) on site during the entire Survey?

Yes       No      Comments: FRED TAYLOR

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2. Is appropriate protective clothing and equipment made available to the sampling teams by the site contractor?

Yes       No      Comments: ORNL PERSONNEL HAVE THEIR OWN EQUIPMENT.  
SAFETY EQUIPMENT NOT COMPLETE (RE: TYVEK COVERALLS NOT AVAILABLE TO  
SAMPLERS).

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3. Are all members of the sampling team formally trained in appropriate health and safety considerations?

Yes       No      Comments: TRAINED IN GRAND JUNCTION CO AND ORNL, TN

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4. For sampling sites where routine operations do not occur and there is no established protocol, are the principal hazards and the protective measures taken determined by document review by the team leader, and the contractor H&S representative?

Yes       No      Comments: DID NOT OBSERVE

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5. Are acid/base spill kits and eye wash kits available in each sampling vehicle?

Yes       No      Comments: EYE WASH KITS WERE. NO ACIDS/BASES USED  
IN THE FIELD TO REQUIRE SPILL KITS.

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6. Are all normal on-site field sampling activities conducted in at least Level-F-protection? (Coveralls, steel toed boots, latex surgical gloves, safety glasses, and hard hats where required).

Yes  No Comments: STREET CLOTHES IN SOME CASES.

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7. Are any food, drink, tobacco or lotions being used during sampling activities?

Yes  No Comments: \_\_\_\_\_

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8. Are sampling personnel fit-tested, and trained in the use of respiratory protection?

Yes  No Comments: THIS COULD NOT BE VERIFIED.

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9. Are any members of the sampling team trained in First Aid/CPR?

Yes  No Comments: \_\_\_\_\_

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10. Have all sampling personnel undergone medical examination?

Yes  No Comments: \_\_\_\_\_

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11. Do all sampling personnel have their radiation exposure histories completed prior to beginning sampling?

Yes       No      Comments: NOT OBSERVED

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12. Is the HSC a professional Industrial Hygienist?

Yes       No      Comments: \_\_\_\_\_

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13. Are Material Safety Data Sheets available at all times for inspection by the Field Sampling Team?

Yes       No      Comments: \_\_\_\_\_

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VIII. ON-SITE WORK PERFORMANCE

1. Indicate sampling team performance in the following areas observed during the on-site audit. (NOTE: Identify poor work practices and violations of protocol under comments.)

<u>Work Practice</u>	<u>Good</u>	<u>Fair</u>	<u>Poor</u>
Sampling technique	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>
Safety procedures	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>
Forbidden personal practices (e.g., smoking, eating in forbidden areas)	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Equipment use/maintenance/calibration	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>

Comments: SAMPLING TECHNIQUE WOULD HAVE BEEN RATED GOOD IF IT WASN'T FOR ALL OF THE CROSS CONTAMINATION POSSIBILITIES. SAMPLE INTEGRITY NEEDS IMPROVEMENT. AN OVA, HNU OF SIMILAR SHOULD BE USED AT ALL SAMPLING LOCATIONS. COVERALLS SHOULD ALWAYS BE WORN. SOME SAMPLERS WERE NOT FAMILIAR WITH THE RADIATION INSTRUMENT. NO ACTION TAKEN IN R0500 AREA WHEN ODOR AND PEGGED RADIATION INSTRUMENT INDICATED A PROBLEM.

2. Indicate sample preparation performance in the following areas observed during the on-site audit. (NOTE: Identify poor work practices and violations of protocol under comments.)

<u>Work Practice</u>	<u>Good</u>	<u>Fair</u>	<u>Poor</u>
Sampling preparation technique	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>
Safety procedures	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>
Forbidden personal practices (e.g., smoking, eating in forbidden areas)	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Equipment use/maintenance/calibration	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

Comments: MORE ATTENTION IS NEEDED TO MAINTAINING SAMPLES AT 4°C AND PREPARING THE VOA ALIQUOTS. THE HOOD WAS NOT VERY CLEAN. PRESERVE BOTTLES PRIOR TO SAMPLING. GOOD COOLER PACKAGING TECHNIQUE.

**Draft - Do Not Cite  
ANL Data Document  
Issue Date: June 1989  
Revision: 01**

**NOTE:**

Because the sampling phase of this work was completed one year prior to the receipt of this audit by the ORNL Field Team Leader, no response was prepared.