Lead Analysis Report #1: Soils AEC 3-3, AEC 9-3, and AEC 10-1

Analysis of Pb in Blood, Soil, and Associated Materials
in Support of Haskell Lab Medical Research Project No. MR 9727-001

Author
Keith A. McCleary

Study Completed:
1/26/94

Performed By:
DuPont Specialty Chemicals
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Quality Control Laboratory
Chambers Works
Deepwater, NJ 08023

Signature: Keith A. McCleary Date 6/10/94
Keith A. McCleary, Ph.D.

Lead Analysis Report #1: Soils AEC 3-3, AEC 9-3, and AEC 10-1

Summary

The purpose of this study was to determine the amount of lead that was contained in samples associated with MR9727-001. There were essentially three type of samples: blood (microswine), aqueous (drinking water, dosing solutions), and solid (soil, bedding, etc.). Blood samples were prepared by dilution with a modifier solution (to digest sample and break viscosity) and analyzed using Graphite Furnace Atomic Absorption Spectrometry (GFAAS). Aqueous and solid samples were prepared using microwave assisted digestion and analyzed using Inductively Coupled Plasma Atomic Emission Spectometry (ICP-AES). All samples were run in duplicate and average answers reported. In the case of the blood, each sample and duplicate was spiked and checked for recovery. If the spike recovery was greater than 10% in error, a one point standard additions calculation was performed.

Methodology

The descriptions listed below are summaries of the actual methods used to analyze samples. The actual methods are documented in the GLP Protocols Notebook, archived at QCL and copies are available upon request.

Sample Preparation: Blood

Blood samples were received in small polyethylene vails. Approximate sample volume was 100-300 uL. 100 uL of blood is diluted to 1.0 mL with modifier solution. The modifier solution is a solution of nitric acid (digests sample), Triton X-100 (surfactant, breaks viscosity), methanol (antifoaming agent), and ammonium phosphate (GFAAS modifier) in water.

Sample Analysis: Blood

Blood samples are analyzed using Graphite Furnace Atomic Absorption Spectrometry (GFAAS). In GFAAS, a 20 microliter volume of sample + modifier is injected into a small graphite tube. This tube is electromagnetically heated to dry, char, and atomize the sample. A hollow cathode lamp (HCL) shines light corresponding to the atomic emission of Pb (283.3 nm) through the tube. As free Pb atoms are generated by the sample atomization, they absorb the radiation, attenuating the amount of signal from the HCL. The degree of attenuation of the HCL radiation is proportional to the concentration of Pb in the sample. Calibration standards of 10, 20 ,and 30 ng/mL are prepared by serial dilution of a 1000 ug/mL stock. Each blood sample is analyzed twice. Each sample and duplicate is then spiked with 10ng Pb and reanalyzed to check for recovery. If recovery is less than 90% or greater than 110%, a one point standard addition calculation is

Lead Analysis Report #1: Soils AEC 3-3, AEC 9-3, and AEC 10-1

performed to determine the amount of Pb in the sample. Each result is the average of the two replicates.

Sample Preparation: Aqueous Samples

Samples that are primarily aqueous are prepared by acidifying 10.00 mL of sample with 2.00 mL concentrated HNO3. The sample is then diluted to a final volume of 20.00 mL and analyzed by ICP-AES.

Sample Preparation: Solid Samples

Solid Samples are prepared by microwave assisted digestion. 0.1 - 0.2 g of sample are digested with 5.00 mL concentrated HNO3 in a Teflon lined pressure vessel. The vessels are placed in a microwave digestion apparatus and cooked for 10 minutes at 300@W power. The samples are allowed to cool and are then filtered into a 50 mL volumetric flask and diluted to volume with deionized water. Samples are analyzed for Pb content by ICP-AES.

Sample Analysis : Aqueous and Solid Samples

These samples are analyzed by Inductively Coupled Plasma - Atomic Emission Spectrometry (ICP-AES). The prepared samples are pumped into an sample introduction system where a fine spray of aerosol is generated. This sample aerosol flows along a stream of argon gas into the plasma. The plasma is a highly energetic discharge sustained in argon by applied radio-frequency power in excess of 1000 W. This discharge is energetic enough to desolvate, atomize, and excite the sample species to radiative emission. The intensity of the characteristic emission wavelength of Pb (220.35 nm) is proportional to the amount of Pb in the sample.

Results

The results for all samples submitted under MR9727-001 are listed in Appendix I.

Lead Analysis Report #1: Soils AEC 3-3, AEC 9-3, and AEC 10-1

PB_PHS2.XLS
MR 9727-001

TIME (hrs)		GROUP (A)				TIME (hrs)		GROUP (B)			
	101	102	103	105			201	202	203	205	
	ng/mL						ng/mL				
Predose	<5	10	7	67		Predose	11	<5	<5	8	
0.5	99	158	231	450		0.6	83	7	51	167	
1	227	120	386	511		1	145	17	122	227	
2	301	823	431	3190		2	346	2740	400	8680	
4	1110	382	603	407		4	266	100	464	386	
8	206	53	61	63		8	344	160	289	470	
12	210	134	32	198		12	173	237	278	357	
24	605	302	1447	396		24	94	240	193	283	
48	348	533	286	167		48	88	148	159	121	
72	143	57	140	214		72	85	91	95	190	
96	108	102	196	93		96	64	99	228	127	
120	58	86	135	45		120	70	67	73	115	
144	141	1073	92	182		144	108	83	111	179	
192	76	67	87	61		192	78	20	17	63	
240	55	50	58	64		240	47	36	45	111	
TIME (hrs)		GROUP (C)				Time (hrs)		GROUP (D)			
	301	302	303	304	305		401	402	403	404	405
	ng/mL						ng/mL				
Predose	8	<5	<5	11	<5	Predose	<5	<5	42	<5	<5
0.5	2460	12400	2700	1030	138	0.5	41	<5	8	111	128
1	1800	11430	4330	218	65	1	75	<5	74	173	251
2	2370	3660	2670	430	175	2	114	<5	95	338	205
4	538	1990	1270	326	205	4	282	95	283	804	624
8	784	821	1780	569	221	8	161	328	260	403	191
12	640	1080	1050	305	180	12	182	300	160	338	196
24	497	946	795	349	133	24	160	391	241	264	263
48	214	680	518	119	50	48	144	193	199	193	210
72	329	512	359	134	78	72	88	170	148	209	173
96	372	479	297	160	74	96	94	135	126	169	122
120	266	388	283	124	68	120	55	93	78	136	63
144	224	259	189	121	97	144	22	76	55	111	62
192	181	272	231	76	43	192	47	54	41	78	75
240	131	298	159	92	21	240	51	55	37	59	50

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PB_PHS2.XLS
MR 9727-001

REPEATS (ng/mL)					NON BLOOD SAMPLES (ug/mL)			
Time	C305 RPT				AEC10-1		516	
predose	<5	C302-0.5	17800				513	
0.5	84	C302-1	12130				686	
1	88	C304-1	670		AEC 9-3		1686	
2	141	C304-2	200				1776	
4	169	C302-4	1870				1411	
8	809	C303-4	1670		AEC 3-3		884	
12	175	C303-8	1070				603	
24	54						596	
48	107				PIG DOSE 1 (AEC 3-3)		753	
72	129				PIG DOSE II (AEC 3-3)		521	
96	128				GRPII 202		1962	
120	58				GRPII 203		1306	
144	91				GRPII 205		1116	
192	30				#401		385	
240	17				#402		536	
					#403		985	
A105-2	488				#404		543	
A101-4	553				#405		696	
A101-8	603				ASPEN LAB SHAVINGS		<1	
A103-8	312				BASAL CHOW		<1	
A105-8	242				dI WATER Rm. 211		<1	
A103-12	322				NITRIC ACID		<1	
A103-12	598				Pb ACETATE DOS. SOLN		19.12%	
A103-24	375							
A103-72	156				Pb SOIL 9-3		4690	5235
A103-120	80						5159	5093
A102-144	73						4868	
					Pb SOIL 10-1		1590	2092
B202-0.5	<5						1869	2012
B202-1	<5						2005	
B202-2	697				Pb SOIL 3-3		1876	2224
B205-2	296						1787	1933
B201-24	<5						1668	
B203-96	74							

DUP040004247

Lead Analysis Report #2: Soils PLW and EC1

Analysis of Pb in Blood, Soil, and Associated Materials
in Support of Haskell Lab Medical Research Project No. MR 9740-001

Author
Keith A. McCleary

Study Completed:
1/26/94

Performed By:
DuPont Specialty Chemicals
Trace Metals Analysis Group
Quality Control Laboratory
Chambers Works
Deepwater, NJ 08023

Signature: Keith A. McCleary Date 8/10/94
Keith A. McCleary, Ph.D.

Lead Analysis Report #2: Soils PLW and EC1

Summary

The purpose of this study was to determine the amount of lead that was contained in samples associated with MR9740-001. There were essentially three type of samples: blood (microswine), aqueous (drinking water, dosing solutions), and solid (soil, bedding, etc.). Blood samples were prepared by dilution with a modifier solution (to digest sample and break viscosity) and analyzed using Graphite Furnace Atomic Absorption Spectrometry (GFAAS). Aqueous and solid samples were prepared using microwave assisted digestion and analyzed using Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES). All samples were run in duplicate and average answers reported. In the case of the blood, each sample and duplicate was spiked and checked for recovery. If the spike recovery was greater than 10% in error, a one point standard additions calculation was performed.

Methodology

The descriptions listed below are summaries of the actual methods used to analyze samples. The actual methods are documented in the GLP Protocols Notebook, archived at QCL and copies are available upon request.

Sample Preparation: Blood

Blood samples were received in small polyethylene vials. Approximate sample volume was 100-300 uL. 100 uL of blood is diluted to 1.0 mL with modifier solution. The modifier solution is a solution of nitric acid (digests sample), Triton X-100 (surfactant, breaks viscosity), methanol (antifoaming agent), and ammonium phosphate (GFAAS modifier) in water.

Sample Analysis: Blood

Blood samples are analyzed using Graphite Furnace Atomic Absorption Spectrometry (GFAAS). In GFAAS, a 20 microliter volume of sample + modifier is injected into a small graphite tube. This tube is electromagnetically heated to dry, char, and atomize the sample. A hollow cathode lamp (HCL) shines light corresponding to the atomic emission of Pb (283.3 nm) through the tube. As free Pb atoms are generated by the sample atomization, they absorb the radiation, attenuating the amount of signal from the HCL. The degree of attenuation of the HCL radiation is proportional to the concentration of Pb in the sample. Calibration standards of 10, 20 ,and 30 ng/mL are prepared by serial dilution of a 1000 ug/mL stock. Each blood sample is analyzed twice. Each sample and duplicate is then spiked with 10ng Pb and reanalyzed to check for recovery. If recovery is less than 90% or greater than 110%, a one point standard addition calculation is

Lead Analysis Report #2: Soils PLW and EC1

performed to determine the amount of Pb in the sample. Each result is the average of the two replicates.

Sample Preparation: Aqueous Samples

Samples that are primarily aqueous are prepared by acidifying 10.00 mL of sample with 2.00 mL concentrated HNO3. The sample is then diluted to a final volume of 20.00 mL and analyzed by ICP-AES.

Sample Preparation: Solid Samples

Solid Samples are prepared by microwave assisted digestion. 0.1 - 0.2 g of sample are digested with 5.00 mL concentrated HNO3 in a Teflon lined pressure vessel. The vessels are placed in a microwave digestion apparatus and cooked for 10 minutes at 300@W power. The samples are allowed to cool and are then filtered into a 50 mL volumetric flask and diluted to volume with deionized water. Samples are analyzed for Pb content by ICP-AES.

Sample Analysis : Aqueous and Solid Samples

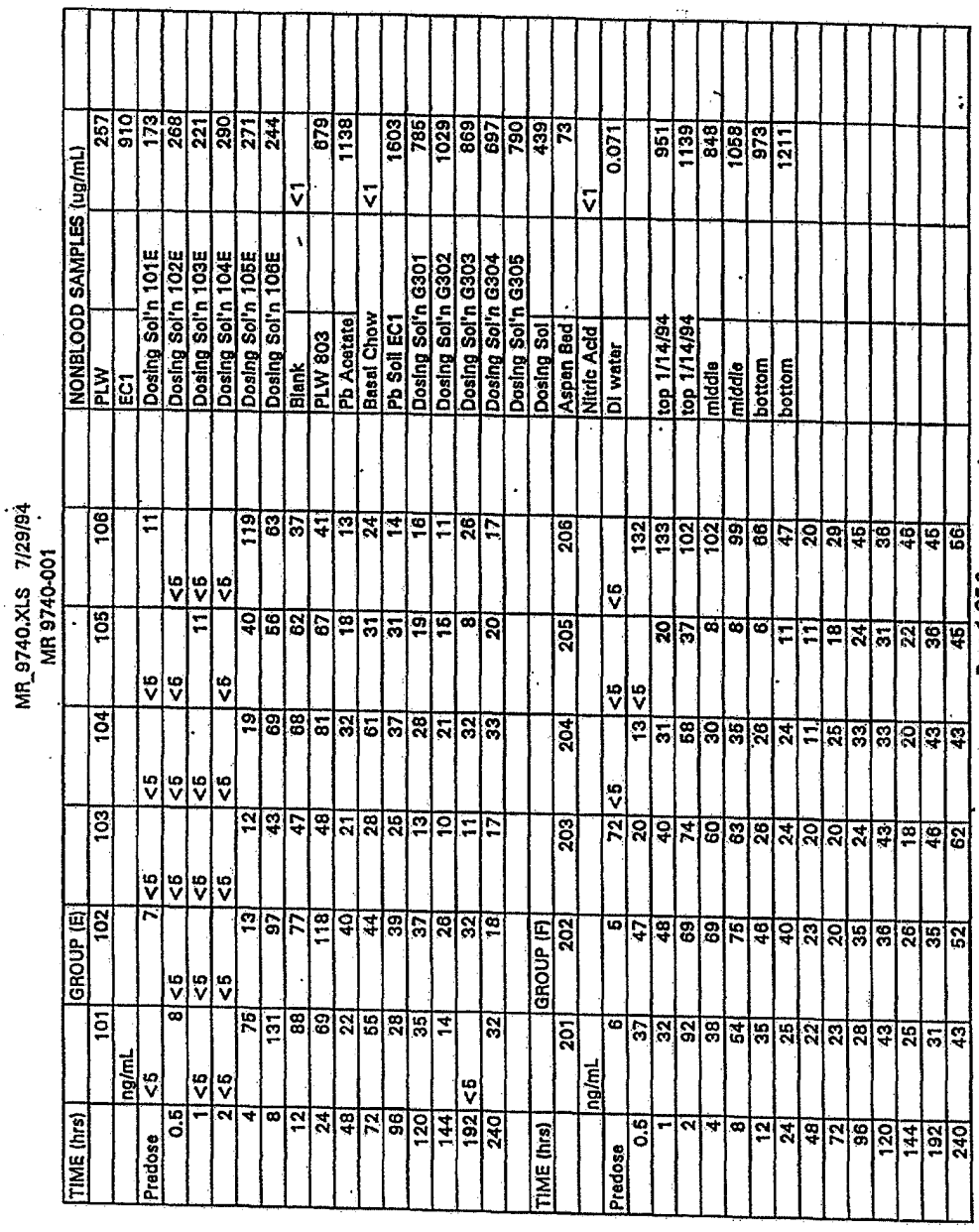
These samples are analyzed by Inductively Coupled Plasma - Atomic Emission Spectrometry (ICP-AES). The prepared samples are pumped into an sample introduction system where a fine spray of aerosol is generated. This sample aerosol flows along a stream of argon gas into the plasma. The plasma is a highly energetic discharge sustained in argon by applied radio-frequency power in excess of 1000 W. This discharge is energetic enough to desolvate, atomize, and excite the sample species to radiative emission. The intensity of the characteristic emission wavelength of Pb (220.35 nm) is proportional to the amount of Pb in the sample.

Results

The results for all samples submitted under MR9740-001 are listed in Appendix L

DUP040004250

Lead Analysis Report #2: Soils PLW and EC1



MR_9740.XLS 7/29/94
MR 9740-001

TIME (hrs)		GROUP (E)						NONBLOOD SAMPLES (ug/mL)	
	101	102	103	104	105	106	PLW		257
	ng/mL						EC1		910
Predose	<5	7	<5	<5	<5	11	Dosing Sol'n 101E		173
0.5	8	<5	<5	<5	<5	<5	Dosing Sol'n 102E		268
1	<5	<5	<5	<5	11	<5	Dosing Sol'n 103E		221
2	<5	<5	<5	<5	<5	<5	Dosing Sol'n 104E		290
4	76	13	12	19	40	119	Dosing Sol'n 105E		271
8	131	97	43	69	56	63	Dosing Sol'n 106E		244
12	88	77	47	68	62	37	Blank	<1	
24	69	118	48	81	67	41	PLW 803		679
48	22	40	21	32	18	13	Pb Acetate		1138
72	55	44	28	61	31	24	Basal Chow	<1	
96	28	39	25	37	31	14	Pb Soil EC1		1603
120	35	37	13	28	19	16	Dosing Sol'n G301		786
144	14	28	10	21	15	11	Dosing Sol'n G302		1029
192	<5	32	11	32	8	26	Dosing Sol'n G303		869
240	32	18	17	33	20	17	Dosing Sol'n G304		697
							Dosing Sol'n G305		790
TIME (hrs)		GROUP (F)					Dosing Sol		439
	201	202	203	204	205	206	Aspen Bed		73
	ng/mL						Nitric Acid	<1	
Predose	6	5	72	<5	<5		DI water		0.071
0.6	37	47	20	13	<5	132			
1	32	48	40	31	20	133	top 1/14/94		951
2	92	69	74	58	37	102	top 1/14/94		1139
4	38	69	60	30	8	102	middle		848
8	54	75	63	35	8	99	middle		1058
12	35	46	26	26	6	66	bottom		973
24	25	40	24	24	11	47	bottom		1211
48	22	23	20	11	11	20			
72	23	20	20	25	18	29			
96	28	35	24	33	24	46			
120	43	36	43	33	31	38			
144	25	26	18	20	22	46			
192	31	35	46	43	36	45			
240	43	52	62	43	45	56			

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Lead Analysis Report #2: Soils PLW and EC1

MR_9740.XLS 7/29/94
MR 9740-001

TIME (hrs)		GROUP (G)					
	301	302	303	304	305	306
	ng/mL					
Predose	45	48	38	34	40	33
0.5	42	46	52	52	67	26
1	44	21	48	50	55	60
2	46	54	55	48	31	50
4	51	53	39	57	70	62
8	58	97	49	60	46	52
12	102	120	107	87	77	79
24	83	191	187	176	128	155
48	112	106	124	98	67	82
72	61	67	82	102	55	61
96	61	61	63	94	59	81
120	42	79	74	97	77	69
144	42	56	65	82	28	55
192	33	57	63	65	61	54
240	64	49	76	84	84	66

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Lead Analysis Report #3: Soils FM (Fort Madison) and PLW1

Analysis of Pb in Blood, Soil, and Associated Materials
in Support of Haskell Lab Medical Research Project No. MR-9905

Author
Keith A. McCleary

Study Completed:
8/22/94

Performed By:
DuPont Specialty Chemicals
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Deepwater, NJ 08023

Signature: Keith A McCleary Date 10/19/1994
Keith A. McCleary, Ph.D.

Lead Analysis Report #3: Soils FM (Fort Madison) and PLW1

Summary

The purpose of this study was to determine the amount of lead that was contained in samples associated with MR9905. There were essentially three type of samples: blood (microswine), aqueous (drinking water, dosing solutions), and solid (soil, bedding, etc.). Blood samples were prepared by dilution with a modifier solution (to digest sample and break viscosity) and analyzed using Graphite Furnace Atomic Absorption Spectrometry (GFAAS). Aqueous and solid samples were prepared using microwave assisted digestion and analyzed using Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES). All samples were run in duplicate and average answers reported. In the case of the blood, each sample and duplicate was spiked and checked for recovery. If the spike recovery was greater than 10% in error, a one point standard additions calculation was performed.

Methodology

The descriptions listed below are summaries of the actual methods used to analyze samples. The actual methods are documented in the GLP Protocols Notebook, archived at QCL and copies are available upon request.

Sample Preparation: Blood

Blood samples were received in small polyethylene vials. Approximate sample volume was 100-300 uL. 100 uL of blood is diluted to 1.0 mL with modifier solution. The modifier solution is a solution of nitric acid (digests sample), Triton X-100 (surfactant, breaks viscosity), methanol (antifoaming agent), and ammonium phosphate (GFAAS modifier) in water.

Sample Analysis: Blood

Blood samples are analyzed using Graphite Furnace Atomic Absorption Spectrometry (GFAAS). In GFAAS, a 15 microliter volume of sample + 5 microliters of modifier is injected into a small graphite tube. This tube is electromagnetically heated to dry, char, and atomize the sample. A hollow cathode lamp (HCL) shines light corresponding to the atomic emission of Pb (283.3 nm) through the tube. As free Pb atoms are generated by the sample atomization, they absorb the radiation, attenuating the amount of signal from the HCL. The degree of attenuation of the HCL radiation is proportional to the concentration of Pb in the sample. Calibration standards of 10, 20 ,and 30 ng/mL are prepared by serial dilution of a 1000 ug/mL stock. Each blood sample is analyzed twice. Each sample and duplicate is then spiked with 10ng Pb and reanalyzed to check for recovery. If recovery is less than 90% or greater than 110%, a one point standard

Lead Analysis Report #3: Soils FM (Fort Madison) and PLW1

addition calculation is performed to determine the amount of Pb in the sample. Each result is the average of the two replicates.

Sample Preparation: Aqueous Samples

Samples that are primarily aqueous are prepared by acidifying 10.00 mL of sample with 2.00 mL concentrated HNO3. The sample is then diluted to a final volume of 20.00 mL and analyzed by ICP-AES. Concentrated samples may require further dilution, or a higher initial dilution to prepare the sample to be analyzed within the standardized range of the instrument.

Sample Preparation: Solid Samples

Solid Samples are prepared by microwave assisted digestion. 0.1 - 0.2 g of sample are digested with 5.00 mL concentrated HNO3 in a Teflon lined pressure vessel. The vessels are placed in a microwave digestion apparatus and cooked for 10 minutes at 300@W power. The samples are allowed to cool and are then filtered into a 50 mL volumetric flask and diluted to volume with deionized water. Samples are analyzed for Pb content by ICP-AES.

Sample Analysis : Aqueous and Solid Samples

These samples are analyzed by Inductively Coupled Plasma - Atomic Emission Spectrometry (ICP-AES). The prepared samples are pumped into an sample introduction system where a fine spray of aerosol is generated. This sample aerosol flows along a stream of argon gas into the plasma. The plasma is a highly energetic discharge sustained in argon by applied radio-frequency power in excess of 1000 W. This discharge is energetic enough to desolvate, atomize, and excite the sample species to radiative emission. The intensity of the characteristic emission wavelength of Pb (220.35 nm) is proportional to the amount of Pb in the sample.

Results

The results for all samples submitted under MR9905 are listed in Appendix I.

DUP040004255

Lead Analysis Report #3: Soils FM (Fort Madison) and PLW1

APPENDIX 1
MR9905.XLS

UNITS OF ALL BLOOD SAMPLES ARE ppb (ng/ml)																
	PRE	0.5 HR	1.0 HR	2.0 HR	4.0 HR	8.0 HR	12 HR	24 HR	48 HR	72 HR	96 HR	120 HR	144 HR	192 HR	240 HR	
101H	<5	9	27	60	247	130	176	152	80	55	61	45	48	35	101	
102H	11	10	9	<5	29	70	88	68	35	15	15	14	11	8	81	
103H	<5	10	20	34	130	113	89	72	28	21	28	17	24	21	57	
104H	10	8	<5	<5	43	45	91	64	30	13	13	21	21	15	49	
105H	<5	11	33	59	174	156	103	68	45	18	22	17	26	7	55	
201I	13	441	529	609	167	209	224	164	113	96	74	64	56	40	70	
202I	14	37	75	177	46	52	35	60	37	42	41	27	16	26	24	
203I	10	162	133	258	127	162	112	93	74	59	45	58	41	44	53	
204I	13	529	469	336	302	232	148	98	83	108	70	43	64	41	35	
205I	6	223	168	344	225	190	139	142	108	112	72	52	34	46	50	
301J	<5	19	29	132	79	214	154	261	340	159	43	36	39	17	7	
302J	<5	780	1758	1439	493	366	412	614	421	200	71	64	69	65	67	
303J	<5	195	123	308	296	282	219	328	841	83	66	41	64	47	40	
304J	<5	88	64	597	208	685	357	459	418	135	43	34	26	30	23	
305J	<5	443	1167	979	647	376	328	388	861	98	70	38	71	48	61	
													Pb Acetate 6/17/94		1.76%	
NOTE: ALL READINGS BELOW ARE GIVEN IN UNITS OF ppm (mg/kg)													Pb Acetate 9/20/94		13.60%	
SOIL #1 FT MADISON 6/13					354		ASPEN SHAVINGS				15					
SOIL #1 FT MADISON DOS SOLN					174		OREO COOKIES				<15		101H-240 RERUN			
duplicate					179		BLANK				15		21ng/mL			
													102H-240 RERUN			
SOIL #1 GRP I 101-H 6-14-94					158		SOIL2 PLW1				3079		15ng/mL			
SOIL #1 GRP I 102-H 6-14-94					128		SOIL2 PLW1 DOS SOLN				788		103H-240 RERUN			
SOIL #1 GRP I 103-H 6-14-94					159								38 ng/mL			
SOIL #1 GRP I 104-H 6-14-94					109		GROUP III 301J PLW1				1644		104H-240 RERUN			
SOIL #1 GRP I 105-H 6-14-94					139		GROUP III 302J PLW1				1729		15ng/mL			
							GROUP III 303J PLW1				1897		106H-240 RERUN			
							GROUP III 304J PLW1				619		27ng/mL			
							GROUP III 305J PLW1				1296					

10/19/94