

Memorandum

To: Hanan Gbantous, H-1
From: Keith A. McCleary, Jackson Lab
Date: July 29, 1994
Subject: Final Report, Lead in Blood Analysis
Cc: C. J. Hensler, Jackson Lab
M. S. Bogdanffy, Haskell Toxicology
R. C. Rhea, Haskell Quality Assurance
R. L. Sobocinski, QCL

Enclosed please find three copies of the final report for the analysis of Pb in blood as conducted by the Du Pont Specialty Chemicals' Trace Metals Analysis Group as part of Medical Research Project No. MR 9727-001: "Absolute Bioavailability of Soil Lead in Microswine: Soils AEC 3-3, AEC 9-3, and AEC 10-1".

A copy of this report is to be kept with those materials relating to the analyses performed (SOP's, methods, and copies of sample logs) under GLP guidelines here at Jackson Lab. If you have any questions, please feel free to call me (Ducom 540-2467).

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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

Trace Metals Analysis Group

Quality Control Laboratory

Chambers Works

Deepwater, NJ 08023

Signature: Keith A. McCleary

Keith A. McCleary, Ph.D.

Date 8/10/94

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 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

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 a 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.

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		0.5	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	286	100	454	386
8	206	53	61	63		8	344	160	289	470
12	210	134	32	199		12	173	237	278	357
24	605	302	1447	395		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	106	102	196	93		96	54	99	226	127
120	58	86	135	45		120	70	67	73	115
144	141	1073	92	182		144	108	93	111	179
192	76	57	87	51		192	78	20	17	53
240	55	50	58	54		240	47	36	45	111
TIME (hrs)	GROUP (C)					Time (hrs)	GROUP (D)			
	301	302	303	304	305		401	402	403	404
	ng/mL						ng/mL			
Predose	8	<5	<5	11	<5	Predose	<5	<5	42	<5
0.5	2460	12400	2700	1030	138	0.5	41	<5	8	111
1	1800	11430	4330	218	65	1	75	<5	74	173
2	2370	3660	2670	430	175	2	114	<5	95	338
4	538	1990	1270	326	205	4	282	95	283	804
8	784	821	1760	569	221	8	161	328	260	403
12	640	1060	1050	305	180	12	182	300	160	338
24	497	946	795	349	133	24	160	391	241	264
48	214	680	518	119	50	48	144	193	199	193
72	329	512	359	134	78	72	88	170	148	209
96	372	479	297	160	74	96	94	136	126	169
120	266	388	283	124	66	120	55	93	76	136
144	224	259	189	121	97	144	22	76	55	111
192	181	272	231	76	43	192	47	54	41	78
240	131	298	159	92	21	240	51	55	37	59

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	52
A103-120	80							5159	50
A102-144	73							4868	
						Pb SOIL 10-1		1590	20
B202-0.5	<5							1869	20
B202-1	<5							2005	
B202-2	697					Pb SOIL 3-3		1876	22
B205-2	296							1787	19
B201-24	<5							1668	
B203-96	74								