Memorandum

To: Hanan Ghantous, H-1

From: Keith A. McCleary, Jackson Lab

Date: October 19, 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 9905.

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).

Diana Lead Data Entry Coding Form:

DecControl#: 235.01

BatesBegin:	DUP040004277	BatesEnd:	DUP040004280	TotalPages	4	Attmnt?#	0
Doc Type:	Research Report	Year Date:	1994	Month Date:	10	Day Date:	19

Title Reference:	Analysis of Pb in Blood, Soil, and Associated Materials in Support of Haskell Lab. Medical Research Project No. MR 9905
Primary Author Last Name:	McCleary
First Name, Middle Initial:	Keith A.
Secondary Authors:	
Publication:	
Primary Recipient (s):	Hanan Ghantous
CC's:	C.J. Hensler, M.S. Bogdanffy, R.C. Rhea, R.L. Sobosinski
Names Stated:	DuPont Specialty Chemicals Trace Metals Analysis Group, Quality Control Laboratory, Chambers Works Plant, Deepwater, NJ, Graphite Furnace Atomic Absorption Spectrometry (GFAAS), Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)
Doc Problems:	

Case Produced In	Steven Thomas	Produced By:	DPT	Source:	Responsive to Plaintiff's 1st Re
Dup/Cross Ref #'s:					
Doc Control Comments:	Disk, Box 1, January 2002, SH001001 - SH001002				

Obj. Coder Nam	D. Green	Obj. Data Entry Nam	D. Green	ObjDataDate	8/21/2002

N 26885.01

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
Trace Metals Analysis Group
Quality Control Laboratory
Chambers Works
Deepwater, NJ 08023

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

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

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.

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
101H	<5	9	27	60	247	130	175	152	80	55	61	45	48
102H	11	10	9	<5	29	70	86	68	35	15	15	14	11
103H	<5	10	20	34	130	113	89	72	28	21	28	17	24
104H	10	8	<5	<5	43	45	91	64	30	13	13	21	21
105H	<5	11	33	59	174	156	103	68	45	18	22	17	26
201I	13	441	529	509	167	209	224	164	113	96	74	64	56
202I	14	37	75	177	46	52	35	60	37	42	41	27	16
203I	10	162	133	258	127	152	112	93	74	59	45	58	41
204I	13	529	469	336	302	232	148	98	83	108	70	43	64
205I	6	223	166	344	225	190	139	142	108	112	72	52	34
301J	<5	19	29	132	79	214	154	251	340	159	43	36	39
302J	<5	780	1758	1439	493	366	412	614	421	200	71	54	69
303J	<5	195	123	308	296	282	219	328	641	83	56	41	54
304J	<5	88	64	597	288	685	357	459	418	135	43	34	25
305J	<5	443	1167	979	547	375	328	388	861	96	70	38	71

			Pb Acetate 6/17/94
NOTE: ALL READINGS BELOW ARE GIVEN IN UNITS OF ppm (mg/kg)			Pb Acetate 9/20/94
SOIL #1 FT MADISON 6/13	354	ASPEN SHAVINGS 15	
SOIL #1 FT MADISON DOS SOLN	174	OREO COOKIES <15	101H-240
duplicate	179	BLANK 15	21ng/mL
			102H-240
SOIL #1 GRP I 101-H 6-14-94	156	SOIL2 PLW1 3079	15ng/mL
SOIL #1 GRP I 102-H 6-14-94	126	SOIL2 PLW1 DOS SOLN 768	103H-240
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
SOIL #1 GRP I 105-H 6-14-94	139	GROUP III 302J PLW1 1729	15ng/mL
		GROUP III 303J PLW1 1897	105H-240
		GROUP III 304J PLW1 619	27ng/mL
		GROUP III 305J PLW1 1296	