Lead Soil Field Test

January 28, 2009

By Ivars Jaunakais
Industrial Test Systems, Inc.
www.sensafe.com

Lead Contaminated Soil

Sources Contaminated Soil:

- Flaking lead paint on exterior structures
  (Lead paint regulated 1978)
- Factory pollution, incinerators, mining
- Tetraethyl lead in gasoline (Regulated 1975)
- Lead arsenate insecticide (Regulated 1988)
- Naturally occurring lead in soil
- Lead in plumbing regulated 1986

Lead Concentrations Contaminated Soil

Wide Variations:

- Natural levels lead in surface soils are 15 to 40 ppm
- Contaminated soils are over 100 ppm
- Soil adjacent to structures with exterior lead-based paints can exceed 10,000 ppm (EPA)
- Soil with 1,000 ppm Lead are legally hazardous
- Lower levels set by some states

Health Effects of Lead – Children

- Childhood lead poisoning is major environmental health problem
- Children with high levels lead suffer from:
    - Damage to brain & nervous system
    - Behavior & learning problems, hyperactivity
    - Slowed growth
    - Hearing problems
    - Headaches

Health Effects of Lead – Adults

- Adults suffer from:
  - Reproductive problems
  - High blood pressure & hypertension
  - Nerve disorders
  - Memory & concentration problems
  - Muscle & joint pain
  - Violent Actions
- Good News: Average blood Lead levels in America have declined during the last 30 years

Lab Testing for Lead in Soil

- Requires expensive analytical equipment, i.e., AA, AES, ICP Spectroscopy, voltametry, etc.,
- Trained technical operator
- Reliable but costly
- Inconvenient – sampling, transport, delays
- Requires sample digestion - dry or wet ashing decomposition method
- Dry ashing requires high temperature
- Wet digestion methods are common

Onsite Testing for Lead in Soils

Previous Field Tests:

- Inexpensive
- Fast
- Rarely recommended because:
  - Reliability of results in question
  - Lacking sensitivity
  - Yes/No answer, or semi-quantitative results
  - Use hazardous reagents
  - Complicated procedures
  - Interferences

Chemistry of Lead

- Oxidation states 0, +2, & +4
- Pb +2 Organic & inorganic salt compounds
- Inorganic salts slightly soluble in water
  - at or less 0.1 gram per Liter
- Below pH 6, Pb^{2+} major ion
- At higher pH values polymeric hydroxocomplexes predominate

Pb^{2+} Species Distribution

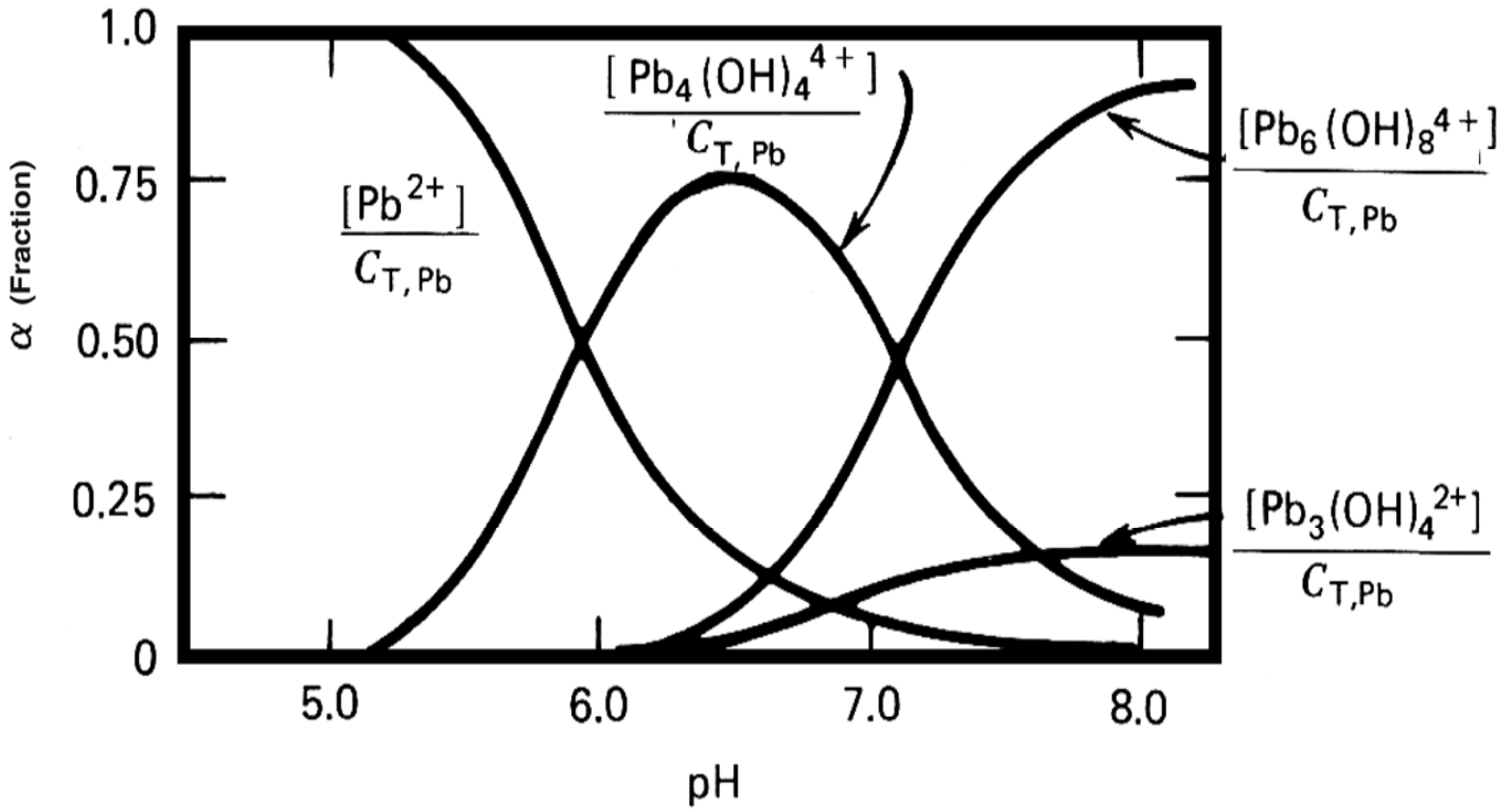


Fig. 1. Species distribution of lead in an aqueous Pb(II) solution $C_{T,PB}$ 0.04 M.
by Olin A. Svensk Kim Tidskr., 73:482(1961).

LEADQuick™ Test Kit

Ten minute test for lead soil uses four reagents

- Requires Hach® LeadTrak Pocket Colorimeter II
- Convenient, easy onsite procedure
- Uses methodology developed for lead in water & lead in paint tests
- Affordable ($3 per test)
- Small sample (0.14 gm)
- Sensitive
- Extraction & sample preparation modified to minimize interfering ions

Soil Sampling

Collect Soil Sample:

- Weigh 0.14 gm soil sample
- If balance unavailable, collect soil sample using 0.1 ml scoop
  (~0.14 gm weight, found weight soil samples to vary from 0.13 to 0.17 gm, 7% below to 18% above target weight)
- Add to 50 ml plastic graduated conical tube

Soil Extraction

- Add 20 drops (0.8 ml) Pb-1-P reagent (14% HNO_3, 7% KNO_3) to soil sample
- Swirl sample briefly to mix
- Wait 5 minutes for lead to extract

Soil Dilution

- Adjust volume to 50 ml with lead-free water
- Cap tube, wait 1 minute as suspended soil settles
- “Soil Extracted Solution” (SES) solution prepared for testing

Step 1 - Test Procedure

- Transfer 200 µl Soil Extraction Solution (SES) to 10 ml glass sample cell

Step 2 - Test Procedure

- Add 15 drops Pb-2 buffer reagent. This adjusts the pH between 9 and 10.
  [(7% 2-amino-2-Methyl-1,3-Propanediol (AMP),15% Tris(hydroxymethyl) -aminomethane (TRIS)]

- Add lead-free water to glass sample cell to 10 ml line

Step 3 - Test Procedure

- Clean and dry cell and place into the Hach® Colorimeter II (LeadTrak or 476 nm)
- Turn meter on &
  select Absorbance
  Mode

Step 4 - Test Procedure

- Dip eXact® Strip Pb-3 for 20 seconds with gentle back & forth motion. This releases TMPYP indicator and mixes sample.

meso-Tetra(N-methyl-4-pyridylporphine tetratosylate salt (TMPYP)

Step 5 - Test Procedure

- Wait one minute while TMPYP indicator fully reacts with lead
- Then zero meter

  in “abs” mode

Step 6 - Test Procedure

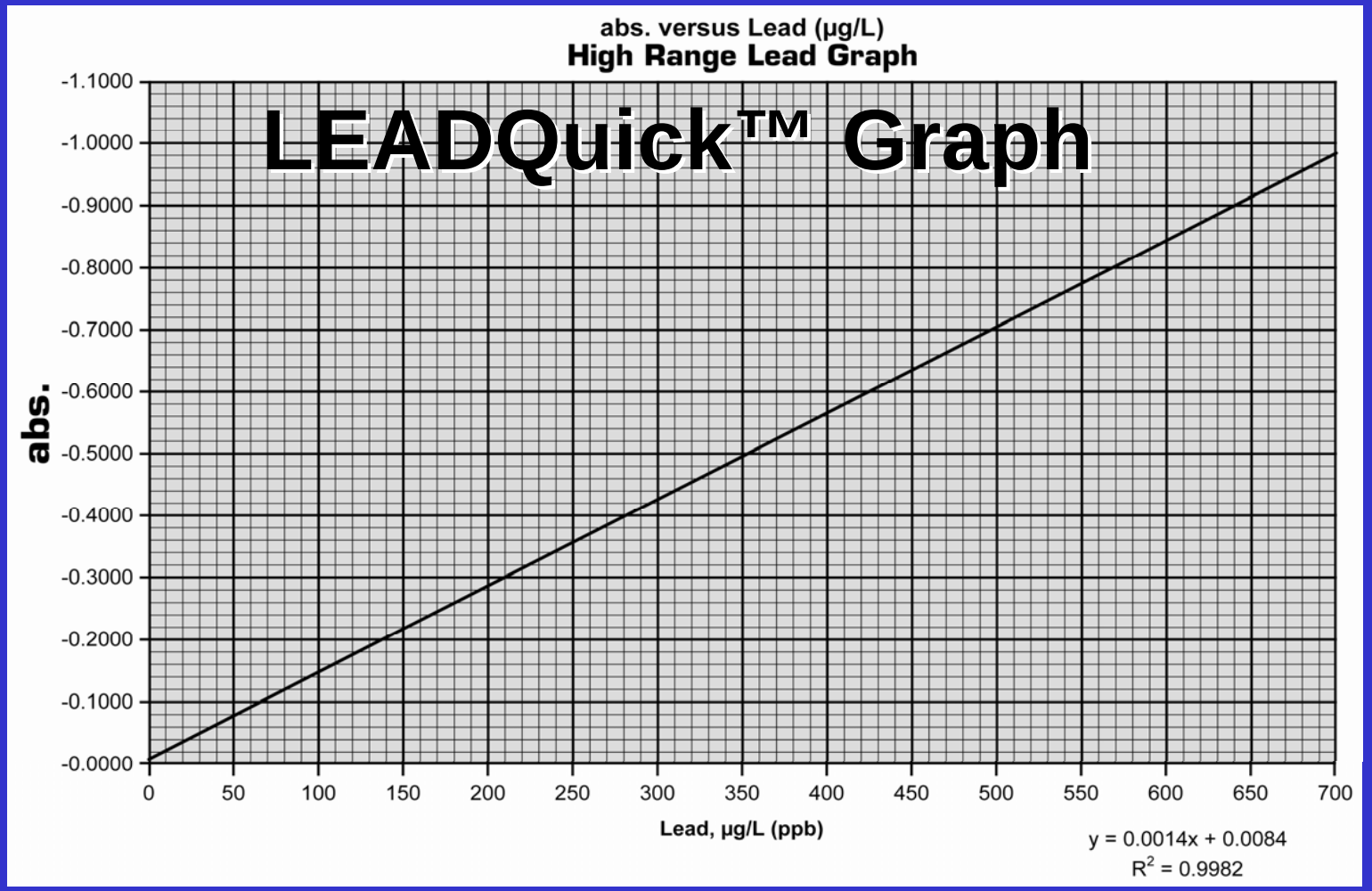
- Dip eXact® Strip Pb-4 (EDTA) into cell sample for 20 seconds with gentle motion
- EDTA destroys colorimetric TMPYP-lead complex

Step 7 - Test Procedure

- Wait one minute
- Press read key & record “abs” value
- A negative value indicates lead is present
- A higher negative value indicates higher lead concentration
- Determine lead concentration from “abs” chart or graph

“ABS” Chart

Lead (Pb^{2+}) "abs" vs µg/L or ppb							
“abs”	µg/L	“abs”	µg/L	“abs”	µg/L	“abs”	µg/L
-.001	<3	-.031	22	-.061	40	-.091	56
-.002	<3	-.032	22	-.062	40	-.092	57
-.003	<3	-.033	23	-.063	41	-.093	57
-.004	<3	-.034	23	-.064	41	-.094	58
-.005	3	-.035	24	-.065	42	-.095	59
-.006	4	-.036	25	-.066	42	-.096	60
-.007	5	-.037	25	-.067	43	-.097	60
-.008	5	-.038	26	-.068	43	-.098	61
-.009	6	-.039	27	-.069	44	-.099	62
-.010	7	-.040	27	-.070	45	-.100	62
-.011	8	-.041	28	-.071	45	-.101	63
-.012	9	-.042	28	-.072	46	-.102	63
-.013	10	-.043	29	-.073	46	-.103	64
-.014	11	-.044	30	-.074	47	-.104	65
-.015	12	-.045	30	-.075	47	-.105	65
-.016	13	-.046	31	-.076	48	-.106	66
-.017	14	-.047	31	-.077	48	-.107	67
-.018	15	-.048	32	-.078	49	-.108	67
-.019	15	-.049	33	-.079	49	-.109	68
-.020	16	-.050	33	-.080	50	-.110	68
-.021	16	-.051	34	-.081	50	-.111	69
-.022	17	-.052	34	-.082	51	-.112	69
-.023	17	-.053	35	-.083	51	-.113	70
-.024	18	-.054	36	-.084	52	-.114	70
-.025	18	-.055	37	-.085	53	-.115	71
-.026	19	-.056	37	-.086	53	-.116	72
-.027	20	-.057	38	-.087	54	-.117	73
-.028	20	-.058	38	-.088	54	-.118	74
-.029	21	-.059	39	-.089	55	-.119	75
-.030	21	-.060	39	-.090	56	-.120	75
							Rev. 03/20/08


abs. versus Lead (µg/L)
High Range Lead Graph
LEADQuick™ Graph
abs.
-1.1000
-1.0000
-0.9000
-0.8000
-0.7000
-0.6000
-0.5000
-0.4000
-0.3000
-0.2000
-0.1000
-0.0000
0
50
100
150
200
250
300
350
400
450
500
550
600
650
700
Lead, µg/L (ppb)
y = 0.0014x + 0.0084
R2 = 0.9982

Spiked Recovery Test Method

(Standard Additions Method)

- Add known amount Lead standard solution to 10 mL sample cell
- Standard Solution amount should be above three times the minimum detectable limit of the test (10 ppb). We used 100 ppb.
- This is “spiked sample”

Spiked Recovery Test Method

(Standard Additions Method)

- Test spiked & un-spiked (original) sample using same reagents, instrument & test methods
- “Spiked sample” should show increase equal to amount of standard added
- Value obtained is the % recovery & ideally 100%

Spiked Recovery Test Method

(Standard Additions Method)

- Results acceptable if recovery is

  +/- 20%
- If recovery is below or above this range, interferences may be present
- Use less than 200 µL sample for testing SES to determine if interferences can be eliminated. Dilution is limited by detection limit of test kit.

Spiked Recovery Test Method

(Standard Additions Method)

Percent recovery formula is as follows:

$$\% \text{ Recovery} = \frac{100(Cs-Cu)}{K}$$

Where:

Cs = concentration found testing spiked sample

Cu = concentration found testing un-spiked sample

(NOTE: result should be adjusted for dilution of spike volume if volume change is more then 5%)

K = concentration of spike added to sample

Table 1 - Tested Certified Soil Reference Standards

Ion	Concentrations of Ions in Reference Soil Samples (mg/kg)					
	S1	**S2**	**S3**	**S11**	**S12**	**S13**
Al	2725	7637	1750	4811	10600	5320
As	24.8	339	397.4	13.1	129	148
Ba	586	1839	22.3	56.1	220	61.4
Be	0.18	0.33	NR	5.97	2.55	5.38
B	13.8	17.2	NR	5.29	48.4	67.8
Ca	5426	28320	25584	14184	13500	14300
Cd	1.19	369	20.8	58.4	89.2	254
Co	2.7	4.07	7	NR	95.2	67.1
Cr	10.7	441	13.2	43.8	105	41
Cu	4792	7.76	753.3	5.68	95.5	66.4
CN^-	NR	NR	NR	10.4	NR	NR
Fe	6481	9439	191645	8315	11800	8210
F^-	NR	NR	NR	29.4	NR	NR
Pb	144742	1447	5194.8	7.13	60.6	132
Hg	4.68	99.8	1.2	6.55	6.45	27.9
Mg	2367	4376	2832.3	2466	4180	2590
Mn	174	173	969.4	127	248	138
Mo	NR	<0.8	NR	8.78	58.3	87.4
Ni	12.6	12.2	15.9	6.63	56	119
K	1006	1992	856.5	1476	3140	3558
Se	NR	518	NR	18.5	88.9	16.2
Si	NR	171	NR	169	NR	NR
Ag	6.52	132	34.2	0.04	0.78	0.335
Na	380	313	75.9	997	109.9	1952
Sr	NR	408	24.7	54.4	NR	NR
Sn	304	NR	NR	NR	386	183
Sb	4955	< 3.2	NR	2.32	79	1.59
Th	0.6	< 4.8	NR	NR	33.1	0.347
V	8.66	19.3	NR	29	112	23.3
Zn	546	51.8	3021.7	74.8	227	18.2

Table 2 - Testing Results in Soil Reference Materials

S. No	SES Volume (S), µl	OD (S) Abs.	Calculated Lead found (mg/kg)	Reference value Lead (mg/kg)	SES (µl) + spiked Lead as ppb, (ppb) for Sp	OD (Sp) Abs.	Spiked Lead recovered (µg/l)	% recovery
S1	5	-.614	246340	144742	5+100	-.720	74	74
	10	-.870	175150					
	20	-1.44	High		100+100	-1.46	High	
	100	-1.72	High					
S2	100	-.039	762.7	1447	100+100	-.164	98	98
	200	-.084	734.5		200+80	-.181	98	98
	500	-.231	875.7		500+80	-.330	75	75
	750	-.347	919.1		750+80	-.472	87	87
	1000	-.462	926.6		1000+80	-.588	87	87
S3	100	BDL	BDL	5194.8	100+100	-.123	81	81
	200	BDL	BDL		200+100	-.101	63	63
	500	BDL	BDL		500+100	-.076	48	48
	1000	BDL	BDL		1000+80	-.068	42	42
S11	200	BDL	BDL	7.13	200+100	-.120	75	75
	500	BDL	BDL		500+100	-.104	65	65
	1000	BDL	BDL		1000+100	-.108	67	67
	1500	-.003	BDL		1500+100	-.076	48	48
	2000	-.009	11.5		2000+100	-.090	56	56
S12	200	BDL	BDL	60.6	200+100	-.137	88	88
	500	-.007	33.5		500+100	-.134	81	81
	750	-.016	57.9		750+100	-.139	76	76
	1000	-.022	56.8		1000+100	-.142	74	74
S13	100	BDL	BDL	132	100+100	-.135	88	88
	200	-.008	88.3		200+100	-.136	83	83
	300	-.027	235.4		300+100	-.159	85	85
	500	-.057	268.4		500+100	-.185	90	90

SES= Soil Extracted Solution **OD**= Optical Density **BDL**=Below Detection Level

Calculations Used for Determining Lead Levels

- Use the ABS value in the conversion chart to determine the concentration of the lead (as µg/L). This value is converted to mg/kg as follows:

a) **Conversion factor:** Multiply the µg/L value by 0.01 and this converts µg/L of Lead to µg of Lead).

b) **SES dilution factor:** Divide SES volume with the volume of SES used for analysis (For example: 50 ml/0.2 ml [200 µl] equals 250 dilution factor).

c) **Soil dilution factor:** Divide 1 kg (1000 g) with the weight of the soil sample (For example: 1000/0.14 equals 7,143 soil dilution factor).

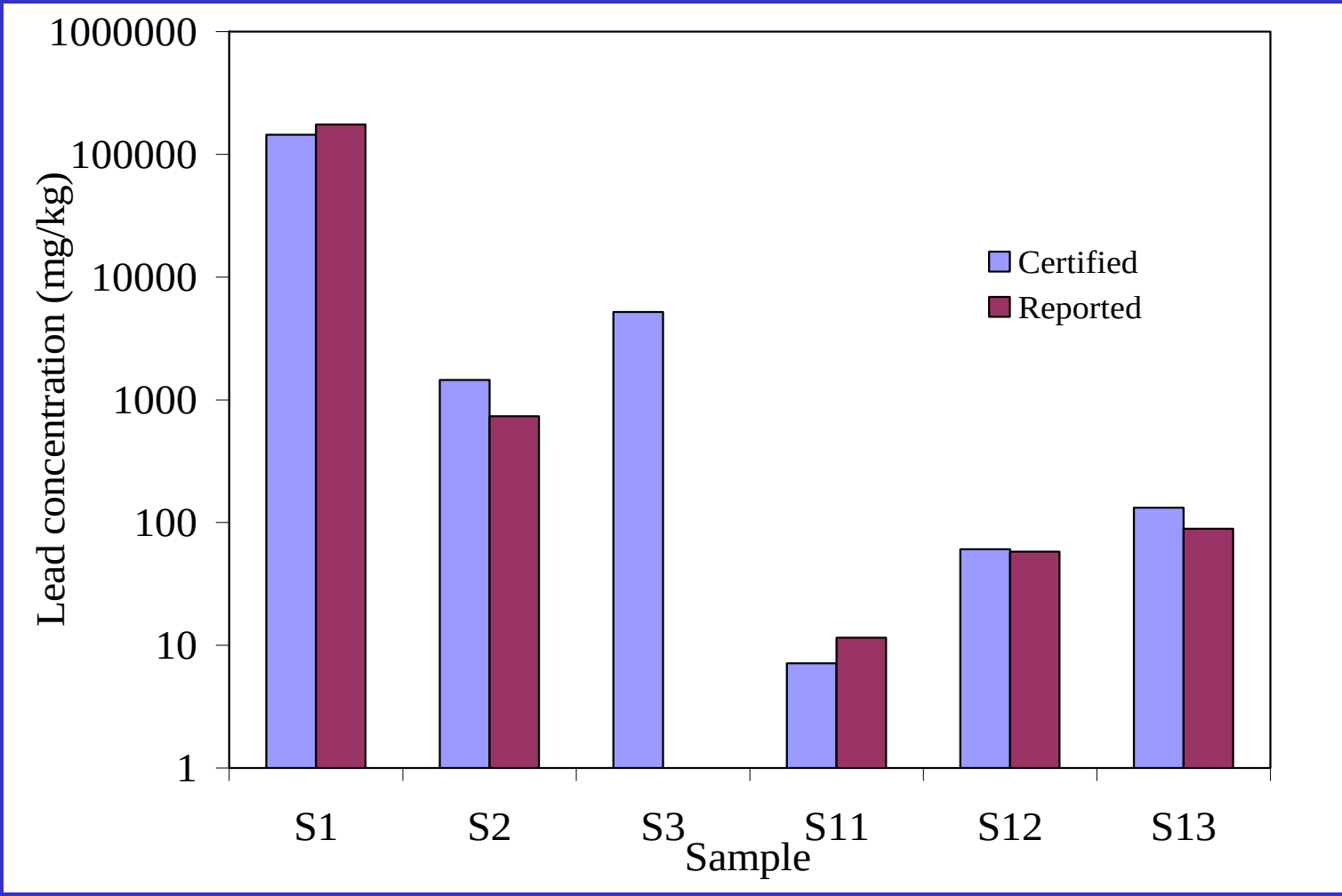
- Multiply 'a' x 'b' x 'c' to get the Lead concentration in mg/kg.
  (0.01 x 250 x 7,143 = 17,858)

Note: If no Lead is found at 200 µl then larger samples of SES are used (such as 2,000 µl for lower detection). If the Lead level is very high, then the sample is retested using a sample of 100 µl or less. The appropriate SES dilution factor is used for the above calculation.

Table 3 - Interfering Ions for Lead Detection

	Soil S2 testing (200µL)		Drinking water testing	
Ion	Interference level (mg/kg)	Type of Interference	Interference level (mg/L)	Type of Interference
Al(III)	2	Negative	6	negative
Cd(II)	0.006	Positive	0.02	positive
Co(II)	5	Negative	13	negative
Cr(VI)	0.2	Negative	0.6	negative
Cu(II)	5	Positive	10	positive
Cl^-	>500	None	>500	None
Fe(II)	0.1	Negative	0.5	negative
Fe(III)	0.2	Negative	0.2	negative
Hg(II)	0.05	Positive	0.02	positive
Mg(II)	150	Negative	200	negative
Mn(II)	0.35	Negative	0.45	negative
Mo(VI)	20	Negative	80	negative
Ni(II)	1	Negative	35	negative
PO_4^{3-}	0.5	Negative	16	negative
Sn(II)	0.5	Negative	0.8	negative
SO^{2-}_4	> 750	None	>750	None
V(V)	2	Negative	6	negative
Zn(II)	3	Negative	8.5	negative

Figure 1: Certified Versus Our Reported Lead Concentration using 200 µl of SES (Soil Extracted Solution)



Conclusion

- Using 200 µl SES, we can detect 132 mg/kg lead with average 83% recovery
- Using 500 & 2000 µl of SES, we can detect 60 & 7 mg/kg of lead, respectively
- LeadQuick™ test kit can determine lead soil concentrations onsite, with minimal interference & well below USEPA levels (400 mg/kg)
- Multiple sample testing confirmed good precision

References

- Chrastny. V., Komarek. M., Jrovcova. E., and Stichova. J. 2008. International Journal of Environmental Analytical Chemistry, 88(6), 385-396.
- Environews: Focus–The Apple Bites Back: Claiming Old Orchards for Residential Development, 2006. Environmental Health Perspectives, 114(8), A470-A478.
- Gryschko. R., Kuhnle. R., Terytze. K., Breuer. J., and Stahr. K. 2005. J. Soils and Sediments, 5(2), 101-106.
- Hoenig, M., and De Kersabiec, A.M. 1996. Spectrochim Acta, B51, 1297.
- Hoenig, M., and Thomas, P. 2002. Techniques De l'ingénieur, P5(8), 1450.
- Jaunakais, I., and Anand, S.M. 2008. US Patent No. 7,333,194 B2.
- Jones, R.A., and Szutka, A. 1966. Anal. Chem., 38(6), 779-781.
- Krasnodebska-Ostrega, B., and Piekarska, J. 2005. Electroanalysis, 17(9), 815- 818.
- Li, Z.J., Tang, J.A., and Pan, J.M. 2004. Food Control, 15(7), 565-570.
- Liu, J., and Lu, Y. 2004. J. Am. Chem. Soc. 126, 12298-12305.
- Melaku, S., Dams, R., and Moens, L. 2005. Anal. Chim. Acta, 543, 117-123.
- Metivier, R., Leray, I., and Valeur, B. 2004. Chem. Eur. J. 10, 4480-4490.
- Palomares, E., Vilar, R., and Durrant, J.R. 2004. Chem. Commun., 362-363.
- Prasad, K., Gopikrishna, P., Kala, R., Prasada Rao, T., and Naidu, G.R.K. 2006. Talanta, 69, 938-945.
- Tarley, C.R.T., and Arruda, M.A.Z. 2005. Analytical Letters, 38 (9), 1427-1443.
- Thompson, K.C., and Nathanail, C.P. 2003. Analytical Chemistry Series: Chemical Analysis of Contaminated Land, CRC Press.
- Tuzen, M., 2003. J. Trace Microprobe Tech., 21, 513.
- Wang, X., Drew, C., Lee, S.H., Senecal, K.J., Kumar, J., and Samuelson, L.A. 2002. Nano Letters, 2(11), 1273-1275.