Environmental Lead Proficiency Analytical Testing (ELPAT) Program March and June 1999

H. Amy Feng and Paul Schlecht

Ms. Feng and Mr. Schlecht are with the Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health, Division of Physical Sciences and Engineering. They can be reached at HHS/PHS/CDC/NIOSH, Robert A. Taft Laboratories, 4676 Columbia Parkway (MS/R8), Cincinnati, OH 45226; (513) 841-4128.

ROUND 26 MARCH 1999, and ROUND 27 JUNE 1999

Paint samples were prepared from paint chips collected from a variety of sites in North Carolina and Ohio, including a school, a hospital, and a warehouse. The chips were ground to a maximum particle size of 120 micrometers (μm).

Soil samples came from driplines around North Carolina residences. Soil samples were dried and then sterilized by heating the soil to 325°F for a minimum of 2 hours, and finally sieved to a maximum particle size of 150 μm.

Dust wipes were prepared from dust collected from households in North Carolina and Milwaukee, Wis. Following sterilization by gamma-irradiation, the household and post-abatement dust was sieved to 150 μm and gravimetrically loaded on a premoistened PaceWipe™, which has been shown to meet ASTM E 1792[1] specifications. The loaded wipes were stored under refrigeration until shipment as an antimicrobial measure. Also it was recommended that dust wipe samples be refrigerated until the laboratory analyses were performed, as an additional precaution to reduce/prevent the growth of mold.

For ELPAT Round 26, a total of 332 laboratories was enrolled with 305 laboratories (92%) submitting results. For Round 27, 328 laboratories enrolled with 307 laboratories (94%) submitted the results. Tables I and II list summary statistics of reference laboratories for each matrix and sample number. Agreement among reference laboratories using a variety of sample preparation techniques and analytical methods is demonstrated by relative standard deviations (RSDs). For Round 26, the ranged from 6.5 to 8% for paint chips, 5.4 to 9.4% for soils, and 6.3 to 9.2% for dust wipes. For Round 27, the RSDs ranged from 6.1 to 11.9% for paint chips, 5 to 12.1% for soils, and 7.5 to 9.1% for dust wipes. The RSDs are similar to the findings on previous ELPAT rounds.

Tables III and IV show the number of all participating laboratory analyses that were identified as outliers. For Round 26, the percentage of all participating laboratory analyses that were identified as outliers was less than 10.3% (5.8 to 7.1% for paint chips, 5.8 to 11.1% for soils, and 4 to 10.3% for dust wipes). For Round 27, the percentage of outliers was less than 8.8% (5.7 to 7.4% for paint chips, 5.3 to 8.8% for soils, and 6.2 to 7.6% for dust wipes). This is similar to the frequency of outliers reported on the earlier rounds of ELPAT for each matrix.

Tables V and VI show a summary of acceptable results for the three lead matrices by sample preparation technique and instrumental method used by participating laboratories. Analytical methods that were not identified by laboratories were omitted from the table. Sample extraction techniques are grouped into hotplate, microwave, and "other" techniques reported by participants. Hotplate digestion categories are: NIOSH 7082/7105 (a nitric acid/hydrogen peroxide digestion method modified from the NIOSH *Manual of Analytical Methods*, Method 7082)[2], EPA SW846-3050A[3] (an EPA nitric acid/hydrogen peroxide method), American Society for Testing Materials (ASTM) hotplate methods,[4,5] and other hotplate techniques. Microwave digestion categories are: EPA SW846-3051[6] (a nitric acid digestion method), ASTM microwave methods,[7,8] and other microwave techniques. The "other" category includes nonmicrowave and nonhotplate techniques, such as X-ray fluorescence sample preparation, leaching techniques, ultrasonic extraction, and Parr bomb. Instrumental methods are categorized into flame atomic absorption (FAA), inductively coupled plasma–atomic emission spectroscopy (ICP-AES), graphite furnace atomic absorption (GFAA), laboratory X-ray fluorescence (LAB-XRF), anodic stripping voltammetry (ASV), and others, which includes inductively coupled plasma–mass spectroscopy (ICP-MS).

ROUNDS 26–27 STATISTICAL ANALYSIS

Statistical tests were performed on ELPAT Round 26 and Round 27 data as previously described in a paper by Schlecht et al.[9] The purpose of these statistical tests are to detect bias differences among analytical methods. Biases on Round 26 were found in the instrumental methods category. Two-way analysis of variance (ANOVA) procedures found statistically significant biases for dust wipes Samples 2, 3, and 4. For dust wipe Samples 2 and 3, FAA has a positive bias over ICP-AES. For dust wipe Sample 4, FAA has a positive bias over ICP-AES and ASV. For Round 27 data, biases were also found in the instrumental methods category. Two-way ANOVA procedures found statistically significant biases for paint Sample 1 and dust wipes Sample 2. For paint Sample 1, FAA has a positive bias over ASV and ICP-AES. For dust wipes Sample 3, ASV has a negative bias compared to FAA and ICP-AES.

NIOSH ELPAT bias studies have found evidence of bias among the principal instrumental methods used by participating laboratories for all three matrices:

Mention of company names or products does not constitute endorsement by the CDC.

TABLE I. ELPAT Round 26 Summary Statistics of Reference Labs

Sample Type	Sample	N	Mean	Minimum	Maximum	STD	RSD (%)	Acceptable Range
Paint chips (%)	1	116	0.0942	0.0829	0.105	0.006	6.5	0.0758–0.1125
	2	116	2.3956	2.09	2.663	0.155	6.5	1.9294–2.8618
	3	116	0.8519	0.7502	0.954	0.060	7.0	0.6733–1.0305
	4	116	5.7041	4.81	6.42	0.458	8.0	4.3304–7.0779
Soil (mg/kg)	1	116	140.5	119	160.1	11.5	8.2	106.1–174.9
	2	116	533.1	477.5	585	29.0	5.4	446.1–620.2
	3	116	408.3	361	443.8	22.7	5.6	340.2–476.4
	4	116	67.6	55.8	78.4	6.34	9.4	48.5–86.6
Dust wipes (μg)	1	116	93.5	79	112	8.62	9.2	67.6–119.4
	2	116	272.5	237	306.1	19.4	7.1	214.1–330.9
	3	116	567.5	504	626.3	35.9	6.3	459.9–675.2
	4	116	119.1	104.3	137	8.89	7.5	92.3–145.8

paint chips, soils, and dust wipes. The biases range from 2 to 26% of the corresponding reference laboratory mean, with the largest biases occurring at low lead levels for dust wipes, generally well below U.S. Department of Housing and Urban Development (HUD) and EPA lead action levels. Although it was expected that differences among sample preparation technique would be found, NIOSH ELPAT bias studies have found no conclusive evidence of bias among the principal sample preparation techniques used by participating laboratories.[9] Another NIOSH study examined the effects of inter- and intralaboratory variabilities on total variability.[10]

The results of NIOSH ELPAT bias studies are consistent with the 3 to 18% bias found by Research Triangle Institute (RTI) in an EPA sponsored collaborative test. In the EPA collaborative test, RTI followed up with participating laboratories and determined that some FAA laboratories failed to perform background corrections one would expect to result in a positive bias. Also, some ICP-AES laboratories failed to take matrix effects into account that one would expect to result in a negative bias. NIOSH does not follow up with participating laboratories to determine if each participating ELPAT laboratory has performed all of the steps of the analytical method reported by the laboratory. However, NIOSH has advised both cooperating accrediting organizations that ELPAT bias could be the result of some ELPAT laboratories not following all steps of the analytical method. NIOSH has recommended that accrediting organizations emphasize FAA background correction and ICP-AES matrix effect minimization procedures when evaluating laboratory accreditation applications and in conducting on-site assessments for EPA National Lead Laboratory Accreditation program (NLLAP) recognition. Laboratories should refer to the RTI collaborative test for a more complete discussion on how bias can be minimized.[11]

Laboratory studies of field portable methods such as ultrasonic extraction and anodic stripping voltammetry of lead from environmental samples show promise as viable techniques. For a more complete discussion, laboratories can refer to a NIOSH study comparing ultrasonic extraction to hotplate and microwave digestion and field portable anodic stripping voltammetry to laboratory based FAA on a series of laboratory generated air samples and National Institute of Standards and Technology Standard Reference Materials.[12]

BACKGROUND

The Environmental Lead Proficiency Analytical Testing (ELPAT) Program is administered by AIHA in cooperation with researchers at the Centers for Disease Control and Prevention (CDC), National Institute for Occupational Safety and Health (NIOSH), and the U.S. Environmental Protection Agency (EPA), Office of Pollution Prevention and Toxics to evaluate and improve the performance of laboratories conducting analyses associated with lead abatement.[13,14] Proficiency test samples are prepared by an AIHA contractor, RTI, using real-world paint chips, dusts, and soils. Quarterly samples are sent to participating laboratories by RTI, and the performance of the laboratories is evaluated at AIHA with sufficient time for laboratories to obtain repeat samples and to

TABLE II. ELPAT Round 27 Summary Statistics of Reference Labs

Sample Type	Sample	N	Mean	Minimum	Maximum	STD	RSD (%)	Acceptable Range
Paint chips (%)	1	109	0.0347	0.027	0.0434	0.004	11.9	0.0223–0.0472
	2	109	0.3744	0.33	0.4202	0.025	6.8	0.2985–0.4503
	3	109	1.4793	1.28	1.635	0.090	6.1	1.2084–1.7501
	4	109	7.4625	5.59	8.539	0.722	9.7	5.2979–9.6271
Soil (mg/kg)	1	109	413.3	363.8	458.8	26.2	6.3	334.6–491.9
	2	109	87.9	68.4	107.6	10.7	12.1	55.8–119.9
	3	109	3216.5	2883.9	3510	160	5.0	2737.7–3695.4
	4	109	711.9	643.9	791	40.9	5.7	589.2–834.6
Dust wipes (μg)	1	109	275.8	237	313	20.7	7.5	213.5–338
	2	109	565.4	461.5	637.5	44.8	7.9	431–699.8
	3	109	120.1	98.1	139.2	11.0	9.1	87.1–153.1
	4	109	1119.1	896.2	1252	89.3	8.0	851.1–1387.1

TABLE III. ELPAT Round 26 Outliers (All Labs)

Sample Type	Sample No.	N of Labs Rated	Acceptable Labs	Low Outlier	High Outlier
Paint chips (%)	1	295	278	11	6
	2	295	275	11	9
	3	295	278	7	10
	4	295	274	8	13
Soil (mg/kg)	1	261	234	8	19
	2	261	246	10	5
	3	261	245	12	4
	4	261	232	8	21
Dust wipes (μg)	1	273	262	5	6
	2	273	257	15	1
	3	273	245	21	7
	4	273	252	15	6

correct analytical problems before the next round of samples is sent.

The ELPAT Program is open to all interested laboratories, including laboratories outside the United States, laboratories seeking accreditation by various private or state laboratory accreditation systems, laboratories that do not intend to seek laboratory accreditation, and laboratories conducting analyses at permanent fixed locations, in self-contained mobile facilities, and at temporary locations (e.g., abatement sites). The ELPAT Program is part of an EPA program, NLLAP, to recognize private and state laboratory accreditation systems.[15] HUD *Guidelines for the Evaluation and Control of Lead-Based Paint Hazards in Housing*[16] requires the use of NLLAP recognized laboratories to ensure the consistency and quality of measurements of lead in paints, soils, and dusts. NLLAP requirements include successful participation in the ELPAT Program for EPA recognition of accreditation. Two organizations, the American Association for Laboratory Accreditation (A2LA)[17] and AIHA,[18] are recognized as accrediting organizations under NLLAP and have in place environmental lead laboratory accreditation systems. Each of these accreditation systems requires participation in ELPAT for environmental lead analysis of paint chips, dusts, and soils. Information on specific A2LA or AIHA laboratory accreditation requirements can be obtained from A2LA and AIHA at the addresses listed at the end of this column.

ELPAT PERFORMANCE EVALUATION

The evaluation of the individual laboratories in the ELPAT Program is based on consensus values from reference laboratories and is modeled after the evaluation procedures previously used in an industrial hygiene proficiency testing program, the Proficiency Analytical Testing (PAT) Program.[19] Reference laboratories are preselected to provide the performance limits for each sample. These laboratories must meet the following criteria: the laboratory was proficient in the previous ELPAT round for paint chips, soils, and dust wipes, and the laboratory must be accredited by an EPA NLLAP recognized accrediting organization.

After data from reference laboratories are collected and extreme reference laboratory data have been statistically treated, the mean ±3 standard deviations of the treated reference laboratory data become the acceptable performance range. Laboratory results are acceptable if they fall within the performance limits. Results falling outside the performance limits are designated as outliers.

Laboratories are rated based on performance in the ELPAT Program over the last year (i.e., four rounds) for each lead matrix—paint chips, soil, and dust wipes. The laboratory is proficient for each lead matrix if the following occurs:

(1) all four results have been reported and all are designated as acceptable for the last two consecutive rounds; or

(2) three-fourths or more of the results reported in the last four consecutive rounds are designated as acceptable.

However, if a laboratory does not report values for the lead matrix on the round being evaluated, the laboratory is not rated.

TABLE IV. ELPAT Round 27 Outliers (All Labs)

Sample Type	Sample No.	N of Labs Rated	Acceptable Labs	Low Outlier	High Outlier
Paint chips (%)	1	299	282	5	12
	2	299	282	10	7
	3	299	277	10	12
	4	299	279	13	7
Soil (mg/kg)	1	262	245	7	10
	2	262	248	5	9
	3	262	239	15	8
	4	262	248	7	7
Dust wipes (μg)	1	276	259	12	5
	2	276	259	12	5
	3	276	255	12	9
	4	276	258	13	5

LEAD REFERENCE MATERIALS

The ELPAT Program is designed to supplement, but not replace, a laboratory's internal quality control program. Use of materials of known lead content in suitable matrices is important in obtaining accurate and reliable lead results. Such materials should be used to validate methods when sample preparation techniques or instrumental methods are adopted or modified. In addition, the materials should be used for daily quality control charting of laboratory/analyst performance. A2LA and AIHA should be contacted about potential sources of primary and secondary reference materials.

EPA NLLAP

Under Title X of the Housing and Community Development Act of 1992, EPA, in consultation with the Department

TABLE V. ELPAT Round 26 Lab Performance Summary

Instrument	Digestion	Method	Paint Chips (%) Acceptable %	Paint Chips (%) Failures %	Soil (mg/kg) Acceptable %	Soil (mg/kg) Failures %	Dust Wipes (μg) Acceptable %	Dust Wipes (μg) Failures %
FAA	hotplate	NIOSH-7082/7105	97	3	88	12	93	7
		EPA-SW846-3050A	94	6	89	11	97	3
	microwave	EPA-SW846-3051	90	10	95	5	96	4
	ultrasonic	—	100	0	92	8	100	0
GFAA	hotplate	NIOSH-7082/7105	25	75	0	0	100	0
		EPA-SW846-3050A	100	0	75	25	75	25
	microwave	EPA-SW846-3051	0	0	0	0	100	0
	ultrasonic	—	0	0	100	0	0	0
ICP-AES	hotplate	NIOSH-7082/7105	90	10	96	4	84	16
		EPA-SW846-3050A	93	7	95	5	92	8
	microwave	EPA-SW846-3051	91	9	95	5	72	28
	ultrasonic	—	100	0	100	0	0	0
LAB-XRF	XRF sample prep	—	81	19	83	17	0	0
ASV	ultrasonic	—	92	8	81	19	90	10
Total			93	7	92	8	92	8

of Health and Human Services, has the responsibility to review and determine if effective voluntary laboratory accreditation systems are in place. If EPA determines effective voluntary laboratory accreditation systems are not in place, EPA is responsible to establish a federal laboratory certification system.[20]

The EPA has established an NLLAP to recognize laboratories performing analysis associated with lead abatement. HUD *Guidelines for the Evaluation and Control of Lead-Based Paint Hazards in Housing* requires the use of NLLAP recognized laboratories to ensure the consistency and quality of measurements of lead in paints, soils, and dusts.[16] NLLAP recognition of laboratories analyzing lead in paint chips, soils, and dusts has two requirements: (1) successful participation in proficiency testing using real-world matrices; and (2) laboratory accreditation including on-site assessment of laboratory operations. NLLAP requirements are based on the recommendations of a federal interagency task force on lead based paint, a group of 17 federal agencies involved with lead issues, that recognition should be based on both proficiency testing and laboratory accreditation.[21] Similarly, proficiency testing and laboratory accreditation requirements were also part of the recommendations for environmental laboratories of a 1991 National Conference on Laboratory Issues in Childhood Lead Poisoning Prevention sponsored by the Association of State and Territorial Public Health Laboratory Directors, the CDC, and EPA.

Laboratory accreditation takes some time to achieve. Laboratory accreditation involves submittal of a description of a laboratory's quality system and manual to the accrediting organization and the on-site evaluation by NLLAP qualified assessors of laboratory operations including equipment, facilities, analytical methods, staff, and internal quality control. Laboratories interested in obtaining accreditation information such as the program requirements, time needed to complete the process, and

TABLE VI. ELPAT Round 27 Lab Performance Summary

Instrument	Digestion	Method	Paint Chips (%) Acceptable %	Paint Chips (%) Failures %	Soil (mg/kg) Acceptable %	Soil (mg/kg) Failures %	Dust Wipes (μg) Acceptable %	Dust Wipes (μg) Failures %
FAA	hotplate	NIOSH-7082/7105	90	10	93	7	96	4
		EPA-SW846-3050A	95	5	93	7	94	6
	microwave	EPA-SW846-3051	94	6	89	11	92	8
	ultrasonic	—	95	5	95	5	94	6
GFAA	hotplate	EPA-SW846-3050A	100	0	100	0	75	25
	microwave	EPA-SW846-3051	0	0	0	0	100	0
ICP-AES	hotplate	NIOSH-7082/7105	93	7	100	0	88	13
		EPA-SW846-3050A	95	5	94	6	95	5
	microwave	EPA-SW846-3051	93	7	91	9	82	18
	ultrasonic	—	0	0	0	0	100	0
LAB-XRF	XRF sample prep	—	88	13	100	0	0	0
ASV	ultrasonic	—	68	32	100	0	75	25
Total			93	7	93	7	93	7

cost should contact the recognized laboratory accreditation organizations. If other laboratory accreditation organizations are recognized, this information will be included in subsequent ELPAT articles.

Lists of laboratories that have performed successfully and are accredited in the ELPAT Program are provided on request to the public on "The Lead Listing" Internet site (www.leadlisting.org). The ELPAT proficiency testing program is open to all interested laboratories. This means laboratories outside the United States and laboratories that do not wish to be accredited can continue to participate in ELPAT. However, only accredited laboratories will appear on "The Lead Listing" web site.

INFORMATION

A2LA laboratory accreditation and seminars on environmental lead laboratory accreditation: American Association for Laboratory Accreditation (A2LA), 656 Quince Orchard Road, Gaithersburg, MD 20878; tel (301) 670-1377; fax (301) 869-1495.

AIHA laboratory accreditation, ELPAT Program information, ELPAT sample orders, and seminars on environmental lead laboratory accreditation: ELPAT Coordinator, AIHA, 2700 Prosperity Avenue, Suite #250, Fairfax, VA 22031, tel (703) 849-8888; fax (703) 207-3561.

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Volume 61, Number 1
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See the following issues for

Accredited Laboratories
March/April, September/October
Annual Author/Subject Index
November/December
Committees
November/December
Consultants Listing
January/February, July/August
Organizational Members
January/February, May/June, July/August, November/December
Publishing Policy
January/February

TECHNICAL ARTICLES