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Curtis A. Esche and Jensen H. Groff, Column Editors

Introduction

The Environmental Lead Proficiency Analytical Testing (ELPAT) Program is administered by the American Industrial Hygiene Association (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.[1,2] Proficiency test samples are prepared by an AIHA contractor, Research Triangle Institute (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 NIOSH with sufficient time for laboratories to obtain repeat samples and 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, the National Lead Laboratory Accreditation Program (NLLAP), to recognize private and state laboratory accreditation systems.[3] U.S. Department of Housing and Urban Development (HUD) Guidelines for the Evaluation and Control of Lead-Based Paint Hazards in Housing[4] require 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)[5] and AIHA,[6] 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 upon consensus values from reference laboratories and is modeled after the evaluation procedures currently used in an industrial hygiene proficiency testing program, the Proficiency Analytical Testing (PAT) Program.[7] 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. This is the same criterion used by NIOSH to establish acceptable and outlier performance of industrial hygiene laboratories in the PAT Program.[7]

Laboratories are rated based upon 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.

ELPAT Round 16, August 1996

Paint samples for round 16 were prepared from paint chips collected from a variety of sites in North Carolina and Ohio, including a school, a warehouse, and a hospital. The chips were ground to a maximum particle size of 120 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 and abatement sites in North Carolina, and Milwaukee, Wisconsin. After sterilization by gamma-irradiation, the dust was sieved to a maximum particle size of 150 μm and then gravimetrically loaded onto Whatman 40 filter paper. The loaded filters were moistened with 0.5 ml of 3 percent hydrogen peroxide solution. The blank wipe was prepared from a Whatman filter moistened with the same hydrogen peroxide solution. Whatman filters are easier to digest than other wipe media (e.g., baby wipes, hand wipes) used by many laboratories.

A total of 384 laboratories were enrolled for round 16 of the ELPAT Program, with 365 (95%) laboratories submitting results. Table 1 lists summary statistics of reference laboratories for each matrix and sample number. Agreement among reference laboratories is demonstrated by relative standard deviations (RSDs) ranging from 6.3 to 11.8 percent for paint chips, 4.2 to 16.2 percent for soils, and 6.0 to 12.8 percent for dust wipes. The highest RSDs for paint chips, soil, and dust were obtained for some of the lowest lead concentrations used in the ELPAT Program. However, these RSDs are lower than those obtained in the past at the lowest lead concentrations.

Table 2 shows the number of all participating laboratory analyses that were identified as outliers. The percentage of outliers for all analyses was less than 10.9

TABLE 1. ELPAT Program Summary Statistics of Reference Laboratories for Round 016

Sample Type	Sample	N	Mean	Minimum	Maximum	STD	RSD (%)	Acceptable Range
Paint chips (%)	1	92	8.1281	7.08	8.95	0.514	6.3	6.587–9.6704
	2	92	0.8782	0.7676	0.968	0.057	6.5	0.708–1.0484
	3	92	0.0306	0.0232	0.0377	0.004	11.8	0.0197–0.0415
	4	92	0.5232	0.455	0.571	0.033	6.4	0.423–0.6234
Soil (mg/kg)	1	92	687.8	620	757.6	38.3	5.6	573–802.6
	2	92	329.3	290	369.5	20.9	6.3	266.5–392.1
	3	92	1234.3	1140	1317	52.3	4.2	1077.5–1391.2
	4	92	49.4	36.1	66.6	7.99	16.2	25.4–73.4
Dust wipes (μg)	1	92	715.5	623.5	798	49.9	7.0	565.7–865.2
	2	92	56.9	41.8	70	7.29	12.8	35–78.8
	3	92	1110.8	979	1215.5	67.2	6.0	909.2–1312.5
	4	92	329.7	278	361.7	22.7	6.9	261.7–397.8

percent (2.5 to 10.9% for paint chips, 4.3 to 8.9% for soils, and 4.2 to 8.2% for dust wipes). This is also similar to the frequency of outliers reported on the earlier rounds of ELPAT for each matrix.

Sample digestion techniques are grouped into hotplate, microwave, ultrasonic, X-ray fluorescence sample preparation, leaching, and all 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[8]), EPA SW846-3050A[9] (an EPA nitric acid/hydrogen peroxide method), American Society for Testing Methods (ASTM) hotplate methods, and other hotplate techniques. ASTM hotplate and microwave methods may be obtained by contacting ASTM at the address at the end of this column. Microwave digestion categories are: EPA SW846-3051[10] (a nitric acid digestion method), ASTM microwave methods, and other microwave techniques. The remaining sample preparation techniques include ultrasonic digestion, X-ray fluorescence sample preparation, and leaching. The "other" category includes techniques such as muffle furnace and Parr bomb.

Instrumental methods are categorized into flame atomic absorption (FAA), graphite furnace atomic absorption (GFAA), inductively coupled plasma–atomic emission spectroscopy (ICP–AES), anodic stripping voltammetry (ASV), laboratory X-ray fluorescence (Lab XRF), and "other," which includes ICP-mass spectroscopy.

Table 3 shows a summary of acceptable results for the three lead matrices by sample preparation technique and instrumental method used by participating laboratories. A series of Fischer's exact tests (nonparametric tests) were used to compare the various combinations of sample preparation techniques (hotplate, microwave, etc.) and instrumental methods (FAA, GFAA, ICP–AES, etc.) for statistically significant differences in the ability of the sample preparation/instrumental method combinations to meet ELPAT performance limits.[11] To detect differences in performance, a criterion was then used where participating laboratories are classified into two groups: those that had no outliers on the four ELPAT samples of the matrix and those that had one or more outliers. Fischer's exact test was then repeated for each ELPAT matrix. No statistically significant differences in the ability of methods to measure lead used by participating laboratories were detected for paint chips, soils, or dust wipes. Analytical methods that were not identified by laboratories were omitted from the table.

ELPAT Round 16 Bias Analysis

Statistical significance tests are performed for investigating differences in bias

TABLE 2. ELPAT Round Program Summary of Performance—All Laboratories Participated for Round 016

Sample Type	Sample No.	No. of Labs Rated	Acceptable Labs	Low Outlier	High Outlier
Paint chips (%)	1	358	319	31	8
	2	358	349	5	4
	3	358	331	2	25
	4	358	341	10	7
Soil (mg/kg)	1	305	290	8	7
	2	305	292	5	8
	3	305	278	17	10
	4	305	278	6	21
Dust wipes (μg)	1	331	308	22	1
	2	331	317	8	6
	3	331	304	23	4
	4	331	308	13	10

TABLE 3. ELPAT Program Labs Performance Summary for Round 016

			Paint Chips (%)		Soil (mg/kg)		Dust Wipes (μg)	
Instrument	Digestion	Method	Acceptable (%)	Failures (%)	Acceptable (%)	Failures (%)	Acceptable (%)	Failures (%)
FAA	Hotplate	NIOSH-7082/7105	94	6	92	8	96	4
		EPA-SW846-3050A	95	5	96	4	97	3
	Microwave	EPA-SW846-3051	98	2	94	6	92	8
	Ultrasonic		88	13	100	0	100	0
GFAA	Hotplate	NIOSH-7082/7105	100	0	0	0	100	0
		EPA-SW846-3050A	81	19	100	0	94	6
	Microwave	EPA-SW846-3051	100	0	100	0	75	25
ICP–AES	Hotplate	NIOSH-7082/7105	94	6	94	6	96	4
		EPA-SW846-3050A	97	3	91	9	94	6
	Microwave	EPA-SW846-3051	88	13	86	14	93	7
	Ultrasonic		100	0	100	0	100	0
Lab-XRF	XRF Sample Prep		83	17	100	0	0	0
ASV	Ultrasonic		86	14	81	19	69	31
Total			94	6	93	7	94	6

among the principal sample preparation and instrumental methods and among the combinations of these two factors. The tests are performed for each matrix (paint chips, soils, and dust wipes) and ELPAT sample (sample numbers 1, 2, 3, and 4) whenever at least three laboratories pass the Grubb's outlier test.[12]

Analysis of variance (ANOVA) is used if the data meet the general assumptions of the ANOVA procedure, homogeneity of variances, and normality. Bartlett's test is used for testing homogeneity of variances and the Shapiro–Wilk test is used for testing normality.[13,14] If the ANOVA assumptions are violated, the Box–Cox transformation procedure is used to examine the data for possible transformations to correct the problem.[13] If the transformed data meet the ANOVA assumptions, then the ANOVA tests are performed on the transformed data. If homogeneity of variance and normality are not achieved by transformation of the data, then a nonparametric approach is used.

In instances where variances are homogeneous and data are normally distributed (either before or after transformation), a one-way ANOVA followed by the Scheffe's multiple comparison test procedure is performed to test for differences in bias among the combinations of the principal sample preparation techniques and instrumental methods.[15] A two-way ANOVA, followed by Scheffe's multiple comparison test procedure to test for any difference among principal sample preparation techniques and principal instrumental methods, is also performed. Two-way ANOVAs separate bias that may be the result of sample preparation, instrumental method, or interaction of these two factors.

In instances where ANOVA cannot be performed on either the original data or transformed data, one of two nonparametric tests is performed. If transformed data meet the homogeneity of variances but not the normality assumptions, then the Kruskal–Wallis rank sums test, followed by the Mann–Whitney–Wilcoxon test with a Bonferroni adjustment, is used.[16] If no transformation can equalize the variances, then the median scores test followed by the sign test with Bonferroni adjustment is used.[17]

Sufficient data were reported to make comparisons among four sample preparation techniques: NIOSH 7082/7105 (a nitric/hydrogen peroxide hotplate digestion), EPA SW846-3050A (a nitric/hydrogen peroxide hotplate digestion), EPA SW846-3051 (a nitric/hydrochloric microwave digestion), and ultrasonic digestion, and five instrumental methods: ASV, FAA, GFAA, ICP–AES, and Lab XRF.

For paint chip sample 2 data, a statistically significant difference was found in the mean concentrations among analytical method groups. FAA (mean = 0.887% lead) had a higher mean concentration than ICP–AES (mean = 0.855% lead). The maximum difference among these methods when compared with the reference value was 6 percent. For paint chip sample 3, a statistically significant difference was found between FAA (mean = 0.0323% lead) and ICP–AES (mean = 0.0295% lead). The maximum difference between these methods compared with the reference value was 11 percent. For paint chip sample 4, ASV (mean = 0.5405% lead) and FAA (mean = 0.5262% lead) had higher mean concentrations than GFAA (0.4557% lead) and ICP–AES (mean = 0.5083% lead). The maximum difference among these methods compared with the reference value was 16 percent. For dust wipe sample 4, FAA (mean = 334.5 μg lead) had higher mean concentrations than ICP–AES (mean = 319.8 μg lead). The maximum difference between these methods compared with the reference value was 13 percent.

Over the first 16 rounds, NIOSH ELPAT bias studies have found evidence of bias among the principal instrumental methods used by participating laboratories for all three matrices: paint chips, soils, and dust wipes. The biases range from 2 to 26 percent of the corresponding reference laboratory mean, with the largest biases occurring at low lead levels for dust wipes, generally well below HUD and EPA lead standards. 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.

The results of NIOSH ELPAT bias studies are consistent with the 3 to 18 percent bias found by 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, which one would expect to result in a positive bias, and some ICP–AES laboratories failed to take matrix effects into account, which 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 NLLAP recognition. Laboratories should refer to the RTI collaborative test for a more complete discussion on how bias can be minimized.[18] Laboratory studies of field-portable methods such as ultrasonic extraction and ASV 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 ASV to laboratory-based FAA on a series of laboratory-generated air samples and National Institute of Standards and Technology standard reference materials.

EPA NLLAP

Under Title X of the Housing and Community Development Act of 1992, EPA, in consultation with the Department of Health and Human Services, has the responsibility to review and determine if effective voluntary laboratory accreditation systems are in place. If EPA determines that effective voluntary laboratory accreditation systems are not in place, EPA is responsible to establish a federal laboratory certification system.[20]

The EPA has established NLLAP to recognize laboratories performing analysis associated with lead abatement. Published HUD Guidelines for the Evaluation and Control of Lead-Based Paint Hazards in Housing require the use of NLLAP-recognized laboratories to ensure the consistency and quality of measurements of lead in paints, soils, and dusts.[4] 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 upon the recommendations of a Federal Interagency Taskforce on Lead Based Paint, a group of 17 federal agencies involved with lead issues, that recognition should be based upon 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 cost, should contact the recognized laboratory accreditation organizations. If other laboratory accreditation organizations are recognized, this information will be included in subsequent ELPAT columns.

Lists of laboratories that have performed successfully and are accredited in the ELPAT Program are provided upon request to the public by the Lead Information Clearinghouse (1-800-424-LEAD). 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 NLLAP list provided by the Lead Information Clearinghouse.

Upcoming ELPAT Round Information

Round 17 ELPAT samples were sent to participants on November 1, 1996. The reporting date of the laboratories was December 2, 1996. Round 17 was the first round for the new dust wipe media, the PaceWipe™. The PaceWipe replaced the Whatman filter paper used in previous ELPAT rounds and has been treated with benzalkonium chloride instead of hydrogen peroxide to retard fungal growth.

Disclaimer

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

Information

A2LA laboratory accreditation, certified reference materials, and seminars on environmental lead laboratory accreditation:

American Association for Laboratory Accreditation (A2LA)
656 Quince Orchard Road
Gaithersburg, MD 20878
Phone: (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
American Industrial Hygiene Association (AIHA)
2700 Prosperity Avenue, Suite #250
Fairfax, VA 22031
Phone: (703) 849-8888
FAX: (703) 207-3561

Orders for the ASTM Standards on Lead-Based Paint Abatement in Buildings publication:

ASTM Customer Service
1916 Race Street
Philadelphia, PA 19103
Phone: (215) 299-5585
FAX: (215) 977-9679

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