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ELPAT Program Report

Background and Current Status

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ELPAT Program Report:

Background and Current Status

Paul C. Schlecht 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 (OPPT) 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 to 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, and laboratories that do not intend to seek laboratory accreditation. 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] NLLAP requirements include successful participation in the ELPAT Program for EPA recognition of accreditation. Two organizations, the American Association for Laboratory Accreditation (A2LA)[4] and the AIHA,[5] have announced environmental lead laboratory accreditation systems that are EPA NLLAP recognized. 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.

Reference Laboratories

Reference laboratories are preselected to provide the performance limits for each sample. Initially, reference laboratories are those ELPAT Program laboratories that:

1. have met the reference laboratory requirements of an industrial hygiene proficiency test program, the Proficiency Analytical Testing (PAT) Program. (PAT reference laboratories must be proficient in analyzing a wide variety of industrial hygiene air samples including airborne lead, and industrial hygiene laboratory operations must be accredited by AIHA); or
2. have performed well in an earlier EPA-sponsored interlaboratory evaluation conducted by RTI of sample digestion techniques (microwave and hotplate) used to analyze paint chip and soil samples.[6]

Once a history of ELPAT performance is available, reference laboratories will include all participating laboratories in the ELPAT Program that have previously demonstrated proficient performance in analyzing all matrices of the ELPAT Program. Eventually, a requirement will be added that ELPAT reference laboratories must be accredited by an EPA NLLAP recognized accrediting organization.

ELPAT Performance Evaluation

Laboratories are evaluated at NIOSH each quarter (designated a round) for each sample analyzed by comparing the laboratory's reported result against an acceptable performance range. The acceptable performance range 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 PAT Program.[7]

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 becomes 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 the 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.

Initial criteria for proficient performance are similar to the procedure used in the PAT Program.[7] However,

the ELPAT statistical protocol and related computer programs have been designed to permit future change to harmonize these proficiency test requirements with internationally harmonized proficiency test protocols. An international protocol for consensus values from reference laboratories using Z-Scores is being developed by the International Organization for Standardization (ISO), the Association of Official Analytical Chemists International (AOAC), and the International Union of Pure and Applied Chemists (IUPAC).[8]

ELPAT Round 5, November 1993

Paint samples for Round 5 were prepared from paint chips collected from a variety of sites. The chips were ground to a maximum particle size of 120 micrometers (μm).

Soil samples came from lead-contaminated soils. Soil samples were dried, 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.

Round 5 dust wipes were prepared by gravimetrically loading Whatman 40 filter paper with sterilized (gamma-irradiated) household and postabatement dust, sieved to a maximum particle size of 250 μm. 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. In the future, the wipe medium may be changed from the Whatman filter to a commercially available wipe that more closely represents field sample media, if a single sample medium is recommended by various lead methods.

A total of 242 laboratories were enrolled for the fifth round of the ELPAT Program, with 229 (94%) laboratories submitting results either by paper or the ELPAT Automated Data Entry System. Table I lists summary statistics of reference laboratories for each matrix and sample number.

TABLE I. Environmental Lead Proficiency Analytical Testing (ELPAT) Program Summary Statistics of Reference Laboratories for Round 005

Sample Type	Sample	N	Mean	Minimum	Maximum	STD	RSD(%)	Acceptable Range
Paint chips (%)	1	36	1.761	1.4	1.992	.170	9.6	1.2524-2.2695
	2	36	0.0222	0.0186	0.0271	.003	12.3	0.014-0.0304
	3	36	4.4022	2.91	5.0212	.577	13.1	2.6708-6.1337
	4	36	0.5568	0.464	0.6603	.055	9.8	0.3923-0.7212
Soil (mg/kg)	1	27	361.9	308	427.5	33.7	9.3	260.7-463
	2	27	359.6	319.3	418.4	27.9	7.8	276-443.3
	3	27	580.9	515.4	636	37.8	6.5	467.4-694.3
	4	27	1597.7	1304	1760	120	7.5	1237.1-1958.3
Dust wipes (μg)	1	34	21	13	26.2	3.92	18.7	9.3-32.8
	2	34	39.1	29.2	47.3	5.37	13.7	23-55.2
	3	34	69.7	56.3	84.2	7.95	11.4	45.8-93.6
	4	34	77.8	64.6	94.5	9.04	11.6	50.7-104.9

Agreement among laboratories is demonstrated by relative standard deviations (RSD) ranging from 9.6 to 13.1 percent for paint chips, 6.5 to 9.3 percent for soils, and 11.4 to 18.7 percent for dust wipes for reference laboratories. This is similar to the agreement among reference laboratories on previous ELPAT rounds for each matrix.

Table II shows the number of all participating laboratory analyses that were identified as outliers. The percentage of outliers for all analyses was under 9.9 percent (6.0 to 8.5 percent for paint chips, 5.5 to 9.9 percent for soils, and 4.8 to 8.1 percent 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 the most common hotplate, microwave, and all other techniques reported by participants. Hotplate digestion categories are NIOSH 7082/7105 [a nitric acid/hydrogen peroxide digestion method modified from NIOSH Manual of Analytical Methods (NMAM) method 7082[9]], EPA SW846-3050A[10] (an EPA nitric acid/hydrogen peroxide method), and other hotplate techniques. Microwave digestion categories are EPA SW846-3051[11] (a nitric acid digestion method), EPA AREAL[6] [a nitric/hydrochloric acid digestion method from AREAL (RTP-MRDD-037) standard operating procedure], and other microwave techniques. The "all other" category includes nonmicrowave and nonhotplate techniques such a X-ray fluorescence sample preparation, leaching techniques, 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), laboratory X-ray fluorescence (lab XRF), and "others" which includes ICP-mass spectrometry (ICP-MS).

Table III shows a summary of failures (outliers) for the three lead matrices by digestion technique and analytical method used by participating laboratories. A series of Fischer's exact tests (nonparametric tests) were used to compare the various combinations of digestion techniques (hotplate and microwave) and analytical methods (FAA, GFAA, ICP-AES) for statistically significant differences in the ability of the digestion techniques/analytical method combinations to meet ELPAT performance limits.[12] To detect differences in performance, a criterion was

TABLE II. Summary of Performance—All Laboratories Participated

Sample Type	Sample No.	No. of Labs Rated	Acceptable Labs	Low Outlier	High Outlier
Paint chips (%)	1	222	208	10	4
	2	222	202	4	16
	3	222	207	12	3
	4	222	206	5	11
Soil (mg/kg)	1	192	177	8	7
	2	192	172	10	10
	3	192	180	6	6
	4	192	178	7	7
Dust wipes (μg)	1	200	190	1	9
	2	200	186	6	8
	3	200	183	11	6
	4	200	188	7	5

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 were detected among the different methods.

Inspection of Table III shows that the predominant analytical methods were FAA and ICP-AES. One laboratory used ICP-MS and was able to meet acceptable performance limits for all paint chip, soil, and dust wipe samples. Two laboratories have used laboratory XRF, but they have been inconsistent in meeting acceptable performance limits.

A more complete comparison of biases and interlaboratory precision differences among digestion techniques and instrumental methods is being undertaken at NIOSH.

ELPAT Round 4 Revisited

Further analysis of ELPAT Round 4 results were performed to confirm if there were any statistically significant differences among sample preparation techniques and instrumental methods. Analysis of variance (ANOVA) with Scheffe's multiple comparison procedure was utilized to identify differences in the mean of the reported lead values among sample preparation techniques and instrumental methods for each matrix.[13] The analyses compared the following principal sample preparation techniques: NIOSH 7082/7105[9], EPA SW846-3050A[10], EPA SW846-3051[11], and EPA AREAL[6], and the following principal instrumental methods: FAA, GFAA, and ICP-AES. Grubb's statistics were used for eliminating outliers. The necessary assumptions for the ANOVA procedure were tested. (Levin's statistics were used to test for homogeneity of variance and Shapiro-Wilk Statistics used to test for normality of data.)[14,15]

For ELPAT Round 4 samples, a one-way ANOVA procedure with Scheffe's comparison was used, the same test used on previous rounds to test for differences in mean lead levels among combinations of sample preparation techniques and instrumental methods whenever there were more than three laboratories using the sample preparation/instrumental method. Statistically significant differences were found for paint chip samples 1($p = 0.0006$) and 2($p = 0.009$). A subsequent multiple comparison Scheffe's Test found EPA SW846-3051 digestion (a nitric acid digestion) with ICP-AES analysis to give lower results than nitric acid/hydrogen peroxide digestion (EPA SW846-3050A or NIOSH 7082/7150) with ICP-AES analysis. A multiple comparison Scheffe's test on paint

TABLE III. Environmental Lead Proficiency Analytical Testing Round 005 All Labs Performance Summary

			Paint Chips (%)				Soil (mg/kg)				Dust Wipes (μg)			
			Acceptable		Failures		Acceptable		Failures		Acceptable		Failures	
Method	Sample	Preparation	N	%	N	%	N	%	N	%	N	%	N	%
FAA	Hotplate	NIOSH-7082/7105	142	93	10	7	86	98	2	2	187	97	5	3
		EPA-SW846-3050A	207	91	21	9	261	95	15	5	135	89	17	11
		Other-Hotplate	72	95	4	5	24	100	0	0	34	94	2	6
	Microwave	EPA AREAL	4	100	0	0	4	100	0	0	4	100	0	0
		EPA-SW846-3051	32	100	0	0	26	93	2	7	24	100	0	0
		Other-Microwave	4	100	0	0	0	0	0	0	4	100	0	0
	Other	All Others	22	92	2	8	5	63	3	38	24	100	0	0
	—	—	0	0	0	0	0	0	0	0	2	50	2	50
GFAA	Hotplate	NIOSH-7082/7105	4	100	0	0	4	100	0	0	20	100	0	0
		EPA-SW846-3050A	10	63	6	38	14	70	6	30	30	83	6	17
	Microwave	EPA-SW846-3051	0	0	0	0	4	100	0	0	4	100	0	0
ICP	Hotplate	NIOSH-7082/7105	47	98	1	2	27	84	5	16	46	88	6	12
		EPA-SW846-3050A	190	93	14	7	188	90	20	10	148	95	8	5
		Other-Hotplate	11	92	1	8	4	100	0	0	24	100	0	0
	Microwave	EPA AREAL	12	100	0	0	8	100	0	0	8	100	0	0
		EPA-SW846-3051	24	100	0	0	23	96	1	4	20	100	0	0
		Other-Microwave	19	95	1	5	20	100	0	0	17	85	3	15
	Other	All Others	4	100	0	0	0	0	0	0	0	0	0	0
Lab-XRF	Other	All others	4	100	0	0	3	75	1	25	0	0	0	0
	—	—	3	38	5	63	3	38	5	63	2	50	2	50
Others	Hotplate	NIOSH-7082/7105	0	0	0	0	0	0	0	0	4	100	0	0
	Microwave	EPA-SW846-3051	4	100	0	0	3	75	1	25	0	0	0	0
		Other-Microwave	8	100	0	0	0	0	0	0	0	0	0	0
—	—	—	0	0	0	0	0	0	0	0	10	83	2	17
Total			823	93	65	7	707	92	61	8	747	93	53	7

—: NOT REPORTED.

chip 2 could not identify differences among sample preparation and instrumental techniques.

Two-way ANOVA found that there is a statistically significant difference in biases among sample preparation and instrumental methods for paint chip sample 1 ($p = 0.013$) and dust wipe sample 3 ($p = 0.01$). Subsequent multiple comparison Scheffe's test found that the differences were due to ICP–AES instrumental techniques giving lower results than FAA instrumental technique. For paint chip sample 1, the bias between ICP–AES and FAA results was 0.013 percent or about 6½ percent of the reference value (0.20%) reported to participating laboratories and 6.9 μg or about 8.6 percent of the reference value (80.4 μg) for dust wipe sample 3.

The finding that FAA can sometimes give higher results than ICP–AES for paint chips and wipes is consistent with ANOVA findings on previous ELPAT rounds, and it is also consistent with the findings of an EPA-sponsored collaborative test of microwave and hotplate digestion techniques conducted by RTI. The RTI study found that FAA consistently gave 3 to 18 percent higher results than ICP–AES. The RTI study did show that failure to use background correction causes a positive bias in results for laboratories using FAA. The RTI study also found suppression of the ICP signal for lead in paint and dust samples believed to be associated with matrix effects that could be minimized by either 1) diluting final solutions to a lead concentration to 10 μg of lead/ml, 2) use of standard additions, or 3) internal standardization. Laboratories that do not do FAA background corrections or take ICP signal suppression into account may find it more difficult to consistently meet ELPAT proficiency requirements.

Although a few instances have been identified on the first four ELPAT rounds (48 samples) of statistically significant differences in sample preparation techniques, no consistent pattern was identified among sample preparation techniques.

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.

ELPAT paint chip, soil, and dust wipe samples from completed ELPAT rounds are available from AIHA at the address listed at the end of this column. ELPAT materials differ from the certified reference materials listed below. ELPAT materials are destroyed either in one analysis (dust wipes), or the amount of material in bottles is limited to reduce the number of times that analyses can be repeated by laboratories reporting in the proficiency test round. National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) values report lead as total lead, whereas ELPAT- and EPA-certified reference materials report extractable lead.

Certified reference materials are commercially available from NIST and from commercial reference material suppliers participating in the U.S. EPA/A2LA environmental reference material certification program.[16] The materials listed in Table IV are useful for daily quality control of analyses and initial evaluation of methods associated with residential or steel structure lead abatement. Since work continues on developing additional reference materials, this list of certified reference materials is subject to significant change. Updated lists of available certified reference materials are available from NIST, EPA-EMSL Cincinnati, and A2LA at the addresses listed at the end of this column.

TABLE IV. Certified Reference Materials

NIST Standard Reference Materials (SRMs)	Lead
SRM 1579a Powdered Lead Based Paint	11.995 ± 0.031%
SRM 2580 Powdered Lead Based Paint to be released 06/94	(nominal value 4%)
SRM 2581 Powdered Lead Based Paint to be released 06/94	(nominal value 0.5%)
SRM 2582 Powdered Lead Based Paint to be released 06/94 total lead by weight	(nominal value 500 ppm)
SRM 2709 Lead in Soil	18.9 ± 0.5 ppm
SRM 2710 Lead in Soil	5532 ± 80 ppm
SRM 2711 Lead in Soil	1162 ± 31 ppm
SRM 2583 Lead in Household Dust to be released 10/94	(nominal value 100–200 ppm)
SRM 2579 Lead Paint Film on Mylar (Set of 5)	3.53 ± 0.24 mg/cm²
	1.63 ± 0.08 mg/cm²
	1.02 ± 0.04 mg/cm²
	0.29 ± 0.01 mg/cm²
	less than 0.001 mg/cm²
(Intended for checking the calibration of portable, hand-held, X-ray fluorescence analyzers when testing for lead in paint coatings on interior and exterior building surfaces in the field.)	
SRM 1648 Urban Particulate Matter	0.655% ± 0.008%
SRM 2704 Buffalo River Sediment total lead by weight	161 ± 17 ppm

EPA-A2LA Certified Reference Materials	
Commercial Supplier	
RT Corp. thru Fischer Scientific	**Lead**
SRS014-50 Bag House Dust	1914 ± 180 ppm*
SRS013-50 Paint Blasting Waste	643 ± 56 ppm*
SRS006-50 Paint Sludge	753 ± 51 ppm*

*The concentrations of lead determined in a sample following digestion by EPA Methods 3010, 3020, or 3050. All concentrations expressed on dry weight basis. The 50-gram samples should be mixed well before removing subsamples.

EPA National Lead Laboratory Accreditation Program (NLLAP)

Under Title X of the Housing and Community Development Act of 1992, EPA, in consultation with the Department of Health and Human Services (DHHS), 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.[17]

The EPA has established the NLLAP to recognize laboratories performing analysis associated with lead abatement. 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.[18] 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. NLLAP requirements for laboratories are based upon Guide 25-1990, "General Requirements for the Competence of Calibration and Testing Laboratories," a guide already in use by many national laboratory accreditation systems worldwide.[19]

The ELPAT Program began providing paint chip, soil, and dust audit samples to evaluate laboratory performance in the fall of 1992 and has grown to over 260 participating laboratories. Lists of laboratories that have performed successfully (rated proficient) in the ELPAT Program are prepared at NIOSH and are provided upon request to the public via toll-free numbers by the Lead Information Clearinghouse (1-800-424-LEAD) and NIOSH (1-800-35-NIOSH).

In December 1993, the first two laboratory accreditation organizations, A2LA and AIHA, have been recognized by NLLAP. 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.

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. As a result of discussions among representatives of A2LA, AIHA, EPA, and NIOSH, we expect that information on the accreditation status of laboratories will be included on the list of NLLAP recognized laboratories in March or April 1994. (At that time, both laboratories that are accredited and those that are not will be listed and provided to the public.)

Once a sufficient number of laboratories (several hundred) geographically dispersed across the United States have received accreditation, only accredited laboratories which are ELPAT proficient will be NLLAP recognized and included on the list of laboratories provided to the public by the Lead Clearinghouse and NIOSH. Given the capacity of cooperating laboratory accreditation organizations to perform on-site assessments and initial laboratory demand for accreditation, it is projected that this will occur in the first quarter of 1995.

Participation in the ELPAT proficiency testing program would continue to be open to all interested laboratories. That means laboratories outside the United States and laboratories that do not wish to be accredited can continue to participate in ELPAT. However starting in 1995, laboratories that are not accredited by a laboratory accrediting organization recognized by NLLAP will not be recognized by the EPA NLLAP Program or included on lists of NLLAP recognized laboratories that will be provided to the public by the federal government.

Upcoming ELPAT Round Information

Round 6 ELPAT samples were sent to participants on February 1, 1994. The reporting date of the laboratories was March 10, 1994. Starting with the new year, all laboratories were required to use the new 5-digit laboratory identification numbers instead of the old 8-digit numbers. The dust wipes were preserved with 0.5 ml of 3 percent hydrogen peroxide solution. This is to retard the formation of any fungal growth in the samples and should not have any effect on the digestion and analysis of them.

An entirely new Automated Data Entry System (ADES) has been developed recently and is now in use with the ELPAT program. This system was developed to provide a faster and less error prone means to receive ELPAT data from laboratories. All that is required by the laboratory is a computer, a modem, a copy of communications software, and a phone line.

Disclaimer: Mention of company names or products does not constitute endorsement by the Centers for Disease Control and Prevention.

Information

A2LA Laboratory Accreditation, A2LA Certified Reference Materials, and A2LA/AIHA 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 A2LA/AIHA 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 NIST Standard Reference Materials (SRMs):

National Institute of Standards and Technology
Standards Reference Materials Program
Room 204, Building 202
Gaithersburg, MD 20899
Phone: (301) 975-6776
FAX: (301) 948-3730

Orders for RT Corporation commercial reference materials:

RT Corporation
2931 Soldier Springs
P.O. Box 1346
Laramie, WY 82070
Phone: (307) 742-5452
FAX: (307) 745-7936
or your local Fischer Scientific representative at (800) 766-7000.

Information on other EPA Certified Reference Materials:

Jim Longbottom
EPA-EMSL
Quality Assurance Research Division
26 West Martin Luther King Drive
Cincinnati, OH 45268
Phone: (513) 569-7308
FAX: (513) 569-7115

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