ULTRASONIC EXTRACTION AND INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRIC DETERMINATION OF LEAD IN ENVIRONMENTAL REFERENCE MATERIALS[1]

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ABSTRACT

Ultrasonic extraction of lead in environmental reference materials, followed by inductively coupled plasma-atomic emission spectrometric (ICP-AES) determination of lead, was evaluated. Primary and secondary lead-containing reference materials (specifically paints and soils) were subjected to ultrasonic agitation in 5% (v/v) nitric acid for 30 minutes in order to solubilize lead, and the dissolved lead was subsequently determined by ICP-AES. Lead analysis results from using ultrasonic extraction were compared against the known lead contents of the reference materials. Acceptable recoveries (100% ± 10%) of lead are obtained by ultrasonic extraction of performance evaluation materials in diluted HNO_3. The results suggest that this method of sample preparation for lead dissolution may be used in lieu of more rigorous digestion procedures involving concentrated strong acids and elevated temperatures.

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INTRODUCTION

A variety of sample preparation methods for lead in environmental samples are currently in wide use, and these generally entail sample digestion in strong acid mixtures and elevated temperatures in order to solubilize lead and lead compounds. Some examples of these preparation procedures include methods published by the National Institute for Occupational Safety and Health (NIOSH),(1) the U.S. Environmental Protection Agency (EPA),(2) the American Society for Testing and Materials (ASTM),(3) and the International Organization for Standardization (ISO).(4)

These extraction techniques, which are quite rigorous, specify the use of concentrated nitric acid (HNO_3) with (usually) either 30% hydrogen peroxide (H_2O_2) or concentrated hydrochloric acid (HCl), and elevated temperatures by means of a hot plate or microwave oven (which also imparts elevated pressures), to effect efficient extraction of lead. Hydrofluoric acid (HF) is required for the extraction of lead from otherwise insoluble silicates; however, the use of HF is generally not warranted for the determination of lead in industrial hygiene and environmental samples.

Alternative extraction procedures for lead in environmental and industrial hygiene samples, based on the use of ultrasonic energy, are being evaluated in this laboratory and elsewhere. Ultrasonic extraction has been shown to give acceptable recoveries of lead in air particulate samples,(5,6) and forms the basis of an EPA procedure for the determination of lead in ambient air.(7) Also, ultrasonic extraction in dilute nitric acid, followed by anodic stripping voltammetric determination of lead, has been shown to perform equivalently to reference analytical methods employing strong acid hotplate or microwave digestion prior to lead determination by atomic spectrometry.(8,9)

Nevertheless, ultrasonic extraction procedures have not been used extensively for the determination of lead in environmental samples, despite their simplicity, safety and time savings (when compared to the aforementioned rigorous digestion procedures). This may be in part to a relative paucity of published data demonstrating the acceptable performance of ultrasonic extraction procedures for lead. In an effort to fill this knowledge gap, the aim of this work was to evaluate the performance of ultrasonic extraction of lead from primary and secondary reference materials. Subsequently, the analytical technique used to determine solubilized lead in sample extracts was inductively coupled plasma atomic emission spectrometry (ICP-AES).

EXPERIMENTAL

Reference materials. Primary reference materials used herein included National Institute of Standards and Technology (NIST) Standard Reference Materials (SRMs): SRM #1579, lead-based paint (11.87% Pb), SRM #2582, powdered paint (208.8 µg Pb/g); SRM #2711, Montana soil (1163 µg Pb/g); and SRM #1648, urban particulate (655 µg Pb/g). Secondary reference materials consisted of paints and soils from rounds 15 and 16 of the Environmental Lead Proficiency Analytical Testing (ELPAT) program.(10)

Ultrasonic extraction. Performance evaluation materials (0.01 to 0.10 g) were weighed to the nearest 0.0001 g, and were placed in 15-mL borosilicate glass graduated cylinders. 0.5 mL of concentrated HNO_3 (Burdick and Jackson spectroscopic grade) and then 9.5 mL of deionized water (18 M-ohm conductivity) were added to the graduated cylinders containing the samples. The cylinders were then capped, shaken by hand, and placed in an ultrasonic bath (Sonicor model SC-150/H, 100W). Samples were then sonicated for 30 minutes, and allowed to stand overnight.

Analysis. Extracted samples were analyzed directly for lead content by ICP-AES (Jarrell-Ash model 1160) using the 220.4 emission line, following filtration through a 0.45-µm filter.

RESULTS AND DISCUSSION

Observed recoveries of lead from ultrasonic extraction/ ICP-AES analysis of primary reference materials (NIST SRMs) are given in Table I. Lead recoveries from ultrasonic extraction are essentially quantitative, as demonstrated by recovery values of 92% to 102%. For the NIST SRMs examined here, these recoveries are similar to those obtained previously by confirmatory analytical methods.(11,12) Hence, ultrasonic extraction with 5% (v/v) HNO_3 appears to be equivalent to hotplate digestion in concentrated HNO_3 and 30% H_2O_2(11) or microwave digestion in concentrated HNO_3 and HCl.(12)

Analytical results from ultrasonic extraction/ ICP-AES determination of lead in secondary reference material paints and soils (ELPAT samples) are shown in Tables II and III, respectively. The data show that the results from ultrasonic extraction compare extremely well with the results from interlaboratory analysis by ELPAT reference laboratories. In all cases, t-tests of the results (95% confidence limits) indicate that there is no statistically significant difference between the data for ultrasonic extraction and the reference values. The vast majority of ELPAT reference laboratories employ rigorous extraction procedures using concentrated strong acid hotplate or microwave digestion.(10) Hence, it is shown by the data of Tables II and III that the ultrasonic procedure in diluted acid performs equivalently to the more rigorous sample preparation procedures for extracting lead from ELPAT samples.

Ultrasonic extraction may be applicable to the extraction of lead in industrial hygiene samples for subsequent field-portable analysis.(8) Ultrasonication as a sample preparation method could also replace laboratory-based rigorous digestion procedures, due to its simplicity, cost and time savings, and safety considerations. Ultrasonic sample preparation methods are presently not employed extensively by industrial hygiene laboratories, but as their performance is demonstrated their use may increase dramatically.

CONCLUSION

It is shown that ultrasonic energy, when imparted to a diluted nitric acid solution, offers adequate conditions for acceptable recoveries of lead in the performance evaluation materials investigated here. This sample preparation procedure may be applicable to the extraction of lead from industrial hygiene samples. Ultrasonic extraction of lead may be preferable to more rigorous digestion procedures, owing to its simplicity, time savings, reduced costs, and safety considerations.

ACKNOWLEDGMENT

We thank Jensen Groff (NIOSH) and Sharon Harper (EPA) for their critical review of the draft manuscript.

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Table I. Recoveries of lead from NIST SRMs using ultrasonic extraction in diluted nitric acid, with subsequent analysis by ICP-AES. ± values are standard deviations.

NIST SRM	No. of samples	Recovery (%)
1579, Lead-based paint	6	102.5 ± 3.6
2581, Powdered paint	4	98.5 ± 3.7
1648, Urban dust	6	92.7 ± 3.0
2711, Montana soil	4	92.2 ± 0.8

Table II. Comparison of analytical results from ultrasonic extraction/ ICP-AES of ELPAT paint samples vs. reference values determined from interlaboratory analysis. ± values are standard deviations.

ELPAT Sample	Reference [Pb], %	Measured [Pb], %
Round 15, Paint 1	5.76 ± 0.36 (n = 80)	5.80 ± 0.21 (n = 10)
Round 15, Paint 2	0.163 ± 0.01 (n = 80)	0.156 ± 0.003 (n = 10)
Round 15, Paint 3	0.417 ± 0.03 (n = 80)	0.410 ± 0.007 (n = 10)
Round 15, Paint 4	2.13 ± 0.15 (n = 80)	2.08 ± 0.03 (n = 10)
Round 16, Paint 1	8.13 ± 0.51 (n = 92)	7.86 ± 0.10 (n = 10)
Round 16, Paint 2	0.878 ± 0.057 (n = 92)	0.853 ± 0.045 (n = 10)
Round 16, Paint 3	0.0232 ± 0.0038 (n = 92)	0.0260 ± 0.0014 (n = 10)
Round 16, Paint 4	0.455 ± 0.033 (n = 92)	0.474 ± 0.027 (n = 10)

Table III. Comparison of analytical results from ultrasonic extraction/ ICP-AES of ELPAT soil samples vs. reference values determined from interlaboratory analysis. ± values are standard deviations.

ELPAT Sample	Reference [Pb], µg/g	Measured [Pb], µg/g
Round 15, Soil 1	867 ± 47 (n = 80)	853 ± 22 (n = 9)
Round 15, Soil 2	525 ± 30 (n = 80)	532 ± 14 (n = 9)
Round 15, Soil 3	3191 ± 187 (n = 80)	3366 ± 52 (n = 9)
Round 15, Soil 4	82.6 ± 7.3 (n = 80)	81.2 ± 4.0 (n = 9)
Round 16, Soil 1	688 ± 38 (n = 92)	677 ± 35 (n = 9)
Round 16, Soil 2	329 ± 21 (n = 92)	315 ± 31 (n = 9)
Round 16, Soil 3	1234 ± 52 (n = 92)	1229 ± 17 (n = 9)
Round 16, Soil 4	49.4 ± 8.0 (n = 92)	41.3 ± 3.9 (n = 9)

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VOLUME I

Proceedings of a Specialty Conference
April 29-May 1, 1997
Research Triangle Park, NC

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