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Determination of Pb^{2+} in water by isotope dilution gas chromatography–mass spectrometry of tetraethyllead formed by reaction with sodium tetraethylborate

B. J. Feldman, H. Mogadeddi and J. D. Osterloh*

Department of Laboratory Medicine, Center for Occupational and Environmental Health, University of California/San Francisco, San Francisco General Hospital Toxicology Laboratory, Ward 35, 1001 Potrero Ave., San Francisco, CA 94110 (USA)

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ABSTRACT

Aqueous Pb^{2+} samples in the low ng/g concentration range were spiked with stable isotope ^{206}Pb standards and subsequently ethylated by a simple room-temperature reaction with sodium tetraethylborate. After extraction into heptane, samples were analyzed by gas chromatography–mass spectrometry on a bench-top quadrupole instrument. Detection was performed by single ion monitoring at *m/z* 293 (triethyl-^{206}Pb ion) and 295 (triethyl-^{208}Pb ion). The technique is rapid, convenient and has good linearity over the tested concentration range of 0.5–100 ng/g. Internal standardization by stable isotope dilution improves the relative standard deviation (for 20–50 ng/g samples) from 24.9% (for externally calibrated determinations) to 4.2% for intra-assay precision and from 35.0% to 8.7% for inter-assay precision. The ^{206}Pb internal standard also discriminates against interference by other metals. The detection limit (3σ) of 0.3 ng/g (for a 2-ml sample) is due primarily to the large relative standard deviation of the blank. Applications to biological matrices are discussed.

INTRODUCTION

Lead in drinking water is a priority pollutant and the EPA standard is 50 ng/g. Epidemiological evidence has suggested that low levels of blood lead due to environmental exposure (air, water, diet) may be associated with health risks [1–3]. Reassessment of the water standard is being considered [4].

The standard methods for determination of aqueous lead are atomic absorption spectrometry (AAS) and anodic stripping voltammetry (ASV). Although fairly sensitive, these methods can require extensive calibration procedures and, depending on the amount of sample workup required, can be prone to contamination.

Recently, the determination of lead and its ionic alkylated derivatives was performed following ethylation with sodium tetraethylborate (STEB) [5–8]. For Pb^{2+} ion, the derivatization reaction stoichiometry is as follows [9]:

$$2Pb^{2+} + 4B(C_2H_5)_4^- \rightarrow Pb + Pb(C_2H_5)_4 + 4B(C_2H_5)_3 \quad (1)$$

The tetraethyllead (TEL) thus formed was subsequently volatilized and measured by AAS. Rapsomanikis *et al.* [5] pioneered this method for alkyllead ions. Using *in situ*, aqueous STEB ethylation followed by purging, trapping, thermal desorption and subsequent AAS analysis, they were able to obtain an absolute detection limit of 8.7 pg for $(CH_3)_3Pb^+$. Inorganic and organic lead ions have also been determined by liquid chromatography as their tetramethylenedithiocarbamate complexes, followed by postcolumn STEB ethylation [6]. Sturgeon *et al.* [7] were able to determine Pb^{2+} at pg/g levels (in a 10-ml sample) by ethylation and *in situ* concentration in a graphite furnace [7]. Other workers have reported the determination of Sn, Hg, Se, Ge [8] and Cd [10] by ethylation with STEB, although the

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molecular structure of the derivative formed is not clear in all instances.

Ethylation of Pb^{2+} by STEB to form TEL for subsequent analysis is an analytically useful reaction for a number of reasons. Ethylation can be conveniently performed in aqueous solutions, as opposed to alkylation by Grignard reagents, which cannot be performed in water. TEL is volatile, and amenable to gas chromatography, which can be used to remove interfering metals and organics. Also, TEL is virtually absent as a laboratory contaminant, and therefore once the analyte solutions have been ethylated, the chances for subsequent Pb^{2+} contamination are greatly reduced.

To date, lead determinations using STEB ethylation have relied on AAS detection. The mass spectrometer is an attractive, alternative detector. Single ion monitoring (SIM) provides excellent sensitivity, and the isotope dilution method [11–13] is unique in providing an internal standard with chemical reactivity identical with that of the species of interest. In addition, the proliferation of inexpensive bench-top gas chromatographic–mass spectrometric (GC–MS) instruments makes this method of detection attactive for many laboratories.

We present here a method for the determination of Pb^{2+}, without preconcentration, in the low ng/g range, based on isotope-dilution GC–MS detection of TEL produced by ethylation with an aqueous STEB solution. The method is rapid, accurate and precise, requires less than 1 ml of sample and utilizes commonly available equipment and materials. In addition, the relatively simple sample workup and the nature of the ethylation process render this method less vulnerable to Pb^{2+} contamination, an omnipresent consideration with this ubiquitous metal. Common sources of contamination were studied and are discussed. It is demonstrated that, under our reaction conditions, the isotope dilution technique discriminates against fluctuations in absolute recovery, as the ^{206}Pb internal standard behaves in a chemically identical manner to analyte Pb^{2+}. The result is enhanced precision and accuracy.

EXPERIMENTAL

Materials

Reverse osmosis/deionized (RO/DI) water with a resistivity of 18 MΩ/cm was prepared using a Millipore (Bedford, MA, USA) Milli-Q system. A 1% (w/w) solution of STEB (Alfa, Dandridge, MA, USA) was prepared fresh daily. Special care was taken to protect the integrity of this reagent. Factory-sealed (under argon), the STEB was opened in an argon-filled glove-bag and decanted into preweighed, acid-cleaned, darkened glass vials. The vials were sealed with crimp-tops and septa before being retrieved from the glove-bag, then re-weighed and refrigerated. Aqueous 1% STEB reagent was prepared by adding the appropriate amount of water to a preweighed STEB-containing vial. Considerable variability was observed in the reactivity of factory-fresh STEB.

A lead standard solution of mixed isotopic composition (1015 ng/g) was prepared by dilution of a 1015 μg/g spectroscopic standard (Aldrich, Milwaukee, WI, USA). A ^{206}Pb standard solution (1000 ng/g) was prepared by dissolution of isotopically enriched $PbCO_3$ (99.66% ^{206}Pb) (ORNL, Oak Ridge, TN, USA) in 1% conc. nitric acid (Seastar Chemical, Vancouver, Canada; lead concentration = 12 pg/g). The level of ^{208}Pb contamination in the ^{206}Pb was 0.05%. Heptane (Mallinckrodt, St. Louis, MO, USA) was of analytical-reagent grade. A 0.68 *M* acetate buffer (pH 4.38) was prepared from ultra-clean 2 *M* ammonium acetate solution (Dionex, Sunnyvale, CA, USA; Pb < 0.5 ng/g), concentrated-nitric acid and RO/DI water. Tetraethyllead (TEL) standard was from NIST (Gaithersburg, MD, USA) Standard Reference Material (SRM) 2712 (11.4 ± 0.4 μg/g Pb).

Isotopic abundances for the mixed isotopic lead sample were measured by STEB ethylation of a 92.3 ng/g sample, followed by gas chromatography and SIM detection of *m/z* values 291, 293, 294 and 295, corresponding to the triethyl derivatives (see below) of ^{204}Pb, ^{206}Pb, ^{207}Pb and ^{208}Pb, respectively. The measured abundances of triethyllead isotopes were corrected for mass contributions due to ^{13}C in order to yield true isotopic abundances of 0.514 for ^{208}Pb and 0.256 for ^{206}Pb.

Sample handling

Polyethylene tubes (5 ml) and pipette tips were washed with 10% conc. nitric acid (overnight or longer), then rinsed at least three times with RO/DI water. GC syringes were rinsed with analytical-reagent grade methanol (Mallinckrodt), then at least

three times with sample, prior to sample injection. All samples and reagents were dispensed inside a Sterigard (Baker, Sanford, Me) positive-flow hood with HEPA filter.

Gas chromatography–mass spectrometry

GC of TEL in heptane was performed on a Hewlett-Packard Model 5890 gas chromatograph equipped with a 25 m × 0.2 mm I.D. HP-5 (0.11-μm film thickness) capillary column. The carrier gas (helium) flow-rate was 0.6 ml/min and the injector temperature was 150°C. The initial column temperature was 50°C, which was maintained for 5 min then increased at 20°C/min to a final temperature of 120°C. Under these conditions, TEL eluted at 8.3 minutes (106°C).

TEL detection was performed with an HP 5971A mass-selective detector in the electron impact ionization mode. The transfer line and mass spectrometer ionization source were maintained at 280°C. Single ion monitoring was performed simultaneously at m/z ratios of 293 (triethyl-$^{206}Pb^+$) and 295 (triethyl-$^{208}Pb^+$) at dwell times of 250 ms for each ion. The instrument was calibrated for masses of 69, 219 and 502 with perfluorotributylamine. This detector is capable of a resolution of 1 u. The ionization voltage was 70 eV.

Standard procedure

A lead-containing aqueous sample (0.5–2 ml) was placed in a clean polyethylene tube, 10 μl of 0.68 *M* acetate buffer (pH 4.38) and 10 μl of 1000 ng/g ^{206}Pb standard were added and the tube was vortex-mixed. A 300-μl volume of heptane and 10 μl of a 1% STEB solution were added and the tube was capped and shaken for 30 min at room temperature. A 100-μl volume of the (TEL-containing) heptane phase was withdrawn and placed in a 2-ml glass vial, which was then septa-sealed. The heptane extracts TEL, but not STEB. Therefore, after reaction with STEB, the TEL-containing heptane phase is relatively invulnerable to contamination as background environmental levels of TEL are low. A 1-μl volume of the heptane phase was injected onto the GC–MS column for analysis.

Sample lead concentrations were calculated as follows (all lead concentrations are in ng/g):

$$\text{total Pb} = \frac{C_{208}}{0.514} = \frac{P_{208}\, C_{206}\, MR}{0.514(P_{206} - P_{208} \cdot 0.256/0.514)} \quad (2)$$

where C_{208} is the concentration of ^{208}Pb in the unspiked sample, C_{206} is the concentration of ^{206}Pb added to the sample (after correction for volume change), P_{206} and P_{208} are the natural isotopic peak areas (corrected for effects due to the presence of ^{13}C) from SIM–GC–MS, 0.514 and 0.256 are the measured abundances of ^{208}Pb and ^{206}Pb in the sample, respectively, and MR is the mass ratio of $^{208}Pb/^{206}Pb$ (1.0097). The contribution of ^{208}Pb as an impurity (0.05%) in the ^{206}Pb standard was ignored.

Methods validation

The accuracy was assessed by addition of premeasured amounts of 1015 or 101.5 ng/g lead standard solution (prepared by dilution of the 1015 μg/g standard) to RO/DI water to give solutions of known lead concentrations. The standard procedure (see above) as applied to these solutions gave a measured lead concentration (Pb_{meas}) which was compared with the calculated amount of Pb (Pb_{added}) added to the solution. Each concentration was determined in triplicate.

Single-sample (instrumental) precision was determined by repeated injection ($n = 10$) of a single preparation from a 40.6 ng/g sample. Intra-assay precision was determined by replicate preparations ($n = 10$), of a single concentration, performed on a single day, of samples containing either 5.0 or 49.8 ng/g of lead. Interassay precision was evaluated by analyzing a 19.9 ng/g sample in triplicate on five successive days.

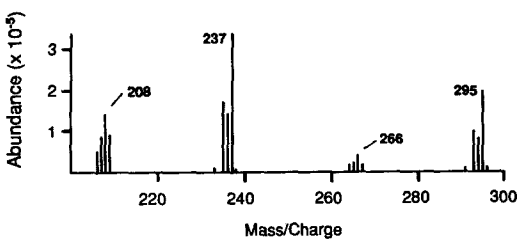

Fig. 1. Mass spectrum (m/z scan range = 200–500) of 11.4 μg/g TEL in fuel (NIST SRM 2712). Injection volume, 1.5 μl. TEL eluted at 8.276 min.

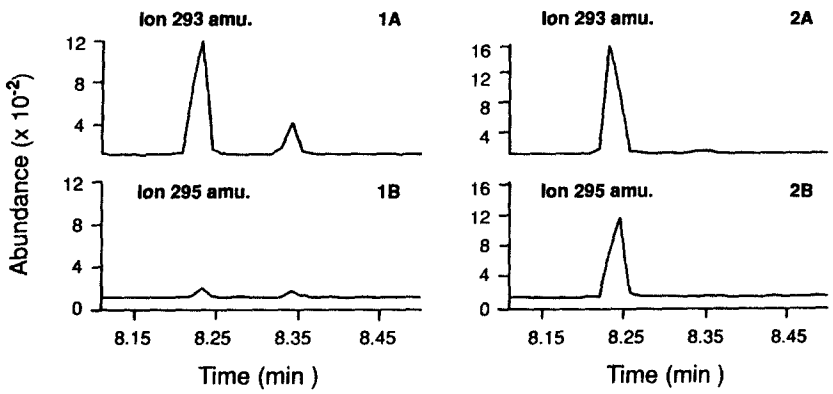

Fig. 2. SIM ion chromatograms for m/z = 293 (triethyl-^{206}Pb ion, A) and 295 (triethyl-^{208}Pb ion, B). Nominal Pb concentrations: sample 1, ^{206}Pb = 19.6 ng/g; sample 2, ^{206}Pb = 28.6 ng/g, ^{208}Pb = 20.8 ng/g.

RESULTS

Fig. 1 shows a mass spectrum of TEL. Peaks clustered about m/z 208, 237, 266 and 295 are due to the fragmentation products Pb^+, ethyl-Pb^+, diethyl-Pb^+ and tri-ethyl Pb^+, respectively. Isotopic abundances of ethylated fragments are those expected for Pb-containing ions. Peaks at m/z 293 (triethyl-$^{206}Pb^+$) and 295 (triethyl-$^{208}Pb^+$) were chosen for quantification of lead. Although these peaks had a lower total abundance than peaks at m/z 235 and 237 (ethyllead isotopes), the smaller background counts at the higher mass were judged to yield a greater signal-to-noise ratio.

Fig. 2 shows typical ion chromatograms at m/z 293 and 295 of two lead-containing samples. Nominal lead concentrations were as follows: sample 1 contained a 19.6 ng/g spike of ^{206}Pb, while sample 2 contained 28.6 ng/g of ^{206}Pb and 20.9 ng/g of ^{208}Pb (the latter added as a 40.6 ng/g spike of mixed-isotope lead). The peak at m/z 295 in sample 1 is due to lead contamination, from atmospheric dust, sample containers or reagents (see below). Comparison of the two sample 1 peak areas gives a value for lead contamination of 0.5 ng/g (0.26 ng) in this particular sample (0.51 ml). In sample 2, a ratio of the two peak areas gives a measured total lead concentration (exclusive of added ^{206}Pb spike) of 41.6 ng/g, close to the nominal value of 40.6 ng/g. Note that there are no interfering peaks which co-elute with the analytes, facilitating quantification.

In order to determine the accuracy and linearity of this technique, ion chromatograms such as those shown in Fig. 2 were obtained for a range of nominal lead concentrations (exclusive of added ^{206}Pb spikes) varying from 0 to 91.5 ng/g. Fig. 3 shows a comparison of the measured lead concentration (Pb_{meas}) plotted against the nominal, added lead concentration (Pb_{added}), which varies from 0 to 91.5 ng/g. The regression line calculated through the data

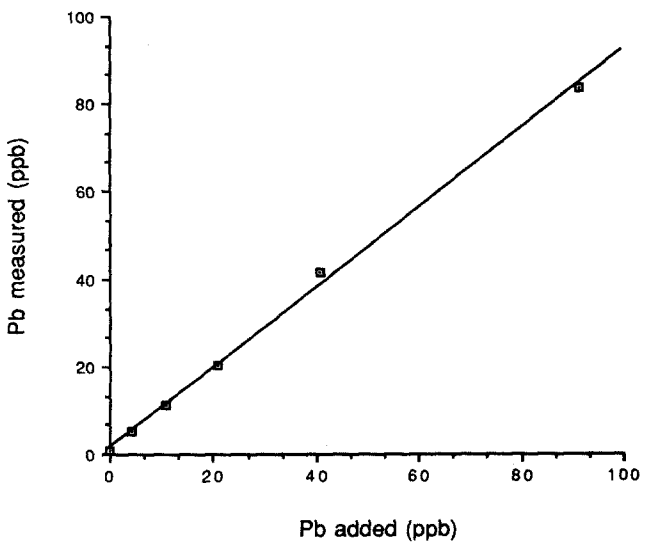

Fig. 3. Calibration graph for intermediate Pb^{2+} concentrations. "Pb added" is the calculated amount of Pb^{2+} in the analyte sample; "Pb measured" is the Pb^{2+} concentration determined by isotope dilution mass spectral analysis (ppb = ng/g). See Experimental for other details.

TABLE I

RELATIVE STANDARD DEVIATIONS FOR EXPERIMENTAL PRECISION

Precision	n	R.S.D. (%)[a]	R.S.D. (%) (A_{295})[b]
Instrumental (40.6 ng/g Pb)	10	2.8	6.7
Intra-assay (49.8 ng/g Pb)	10	4.2	24.9
Intra-assay (5.0 ng/g Pb)	10	9.3	10.0
Inter-assay (19.9 ng/g Pb)	5	8.7	35.0

[a] R.S.D. of the standard procedure (see Experimental).
[b] R.S.D. for area of peak at m/z 295 (triethyl-$^{208}Pb^{+}$), assuming a constant injection volume of 1 μl.

points has an intercept of 1.66 ng/g and a slope of 0.91 (the theoretical value is 1). The correlation coefficient was 0.9988.

Fig. 4 shows a graph similar to that in Fig. 3, but which encompasses a lower range of concentrations, with nominal lead values varying from 0 to 2.5 ng/g. This graph also shows excellent linearity and precision (correlation coefficient = 0.9955), with an intercept of 0.45 ng/g and a slope of 0.70. The reasons for this low slope are not clear; however, it is possible that the polyethylene tubes absorb small amounts of Pb^{2+} and make it unavailable for ethylation. As the samples are exposed to the

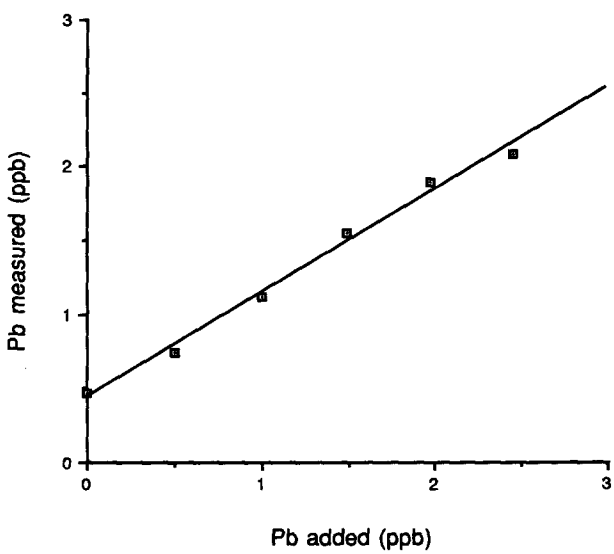

Fig. 4. Calibration graph for low Pb^{2+} concentrations. Axis labels as in Fig. 3. See Experimental for other details.

polyethylene tubes for longer than the ^{206}Pb spikes, a disproportionate amount of the sample lead may gave been absorbed. This phenomenon is important only at low concentrations.

Relative standard deviations (R.S.D.s) for instrumental, intra-assay (at two concentrations, 5.0 and 49.8 ng/g) and inter-assay precision are given in Table I. For purposes of comparison, the R.S.D.s for the areas of the analytical peaks (m/z = 295), *not* ratioed to the internal standard, at a constant 1-μl injection volume, were also calculated.

As the yield of TEL is believed to be dependent on reaction stoichiometry [9], and as STEB may be an important source of contaminating lead, the effect of varying reagent (1% STEB) volumes on this procedure was investigated. ^{206}Pb (due to a standard spike of 10 μl of 1 μg/g ^{206}Pb solution) peak area and contaminating lead were determined for volumes of 1% STEB ranging from 10 to 500 μl (all solutions contained 200 μl of 0.68 M acetate buffer to insure pH control when using larger volumes of 1% STEB reagent). Fig. 5 shows a plot of nanograms of contaminating lead *vs.* amount of STEB reagent. For STEB volumes of 10–50 μl, the amounts of contaminating lead appear constant and within experimental error. At these volumes, STEB is apparently not the major source of lead contamination. At higher volumes (100–500 μl), lead contamination varies linearly with STEB volume. The slope of the line drawn through the data points in Fig. 5 allows a calculation of lead contamination within our 1% STEB reagent of ca. 10.5 ng/ml. This is an important consideration as previously published methods utilizing STEB reported reagent volumes as high as 2 ml of 0.5% reagent [7] and 3 ml of 0.43% reagent [5].

We also studied the effect of different sample tube types on residual lead contamination and the results are given in Table II (measurements are averages of three determinations.) Little difference was observed between the various tube types, suggesting that the tubes are not the source of the observed lead blank contamination, which is typically 0.2–0.6 ng.

Other workers have reported some interference by non-lead metal ions (*e.g.*, a 15% signal suppression by a 1000-fold excess of Cu^{2+} [7]). Table III shows the effect on the standard procedure of one- and tenfold excesses of Fe^{2+}, Zn^{2+} and Cu^{2+} ions. A tenfold excess of Fe^{2+} results in some suppression of

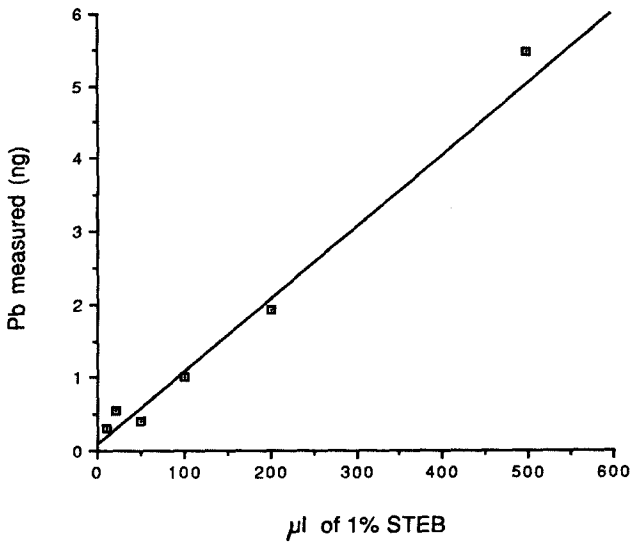

Fig. 5. Pb^{2+} contamination resulting in solutions containing no added ^{208}Pb and derivatized with the indicated amount of 1% STEB solution. Solutions were buffered with 200 μl of 0.68 *M* acetate buffer and spiked with 10 μl of 1 μg/g ^{206}Pb solution.

TEL formation (A_{295}), Zn^{2+} has little effect on the signal and both one- and tenfold excesses of Cu^{2+} result in a *ca.* tenfold decrease in TEL formation. Whereas Fe^{2+} and Cu^{2+} reduce TEL formation, internal standardization with ^{206}Pb reduces the error due to decreased TEL formation. This compensation is only partial in cases of severe interference (Cu^{2+}), where Pb_{meas} values vary more substantially (10–30%) from the nominal values.

Finally, the amount of lead present was measured in delayed- and first-draw San Francisco city water. Compared with our blank solution, which contained 0.1 ng/g contamination, first- and delayed-draw San Francisco city water contained 3.9 and 1.4 ng/g of lead, respectively. This finding confirms the relatively low concentration of lead attributed to San Francisco city water [14], and reaffirms the expected result that water stagnation in pipes is a source of lead contamination.

TABLE II

EFFECT OF TUBE TYPE ON LEAD CONTAMINATION

Tube type	Pb contamination (ng)
Polyethylene[a]	0.28
Polyethylene[b]	0.25
Polyethylene[c]	0.44
Glass, Pyrex[a]	0.24
Glass, borosilicate[a]	0.37

[a] Tube freshly acid cleaned.
[b] Tube kept open in clean hood for 5 days.
[c] Tube kept open in room air for 5 days.

TABLE III

EFFECT OF CONTAMINATING METALS ON TEL PRODUCTION

Contaminant	Concentration (ng/g)	*n*	A_{295}[a]	Pb_{added} (ng/g)[b]	Pb_{meas} (ng/g)[c]
None	–	2	19 345	19.5	21.2
Fe	18.9	3	22 206	19.5	20.5
	161.0	3	9976	16.6	17.9
Zn	18.9	3	22 813	19.5	21.0
	161.0	3	20 775	16.6	17.8
Cu	18.9	3	2220	19.5	23.2
	161.0	3	1808	16.6	22.1

[a] Average area (arbitrary units) of peak at *m/z* 295 (triethyl-$^{208}Pb^{+}$).
[b] Nominal Pb concentration calculated from amount of added standard.
[c] Pb concentration determined by standard procedure (see Experimental).

DISCUSSION

This procedure for the determination of lead in aqueous samples at low-ng/g concentrations is based on aqueous ethylation of a ^{206}Pb-spiked sample by STEB. The TEL thus formed is extracted into heptane and determined by GC–MS. It is an extension of the work of Rapsomanikis *et al.* [5], who first applied the STEB derivatization procedure to the determination of alkyllead cations, and of Sturgeon *et al.* [7], who first applied STEB ethylation for the determination of Pb^{2+}. We have added the use of a ^{206}Pb internal standard and have detected the product TEL by SIM–GC–MS. The technique is rapid and convenient; all the required reagents are inexpensive and the equipment is commercially available. In particular, the reaction vessel is a simple polyethylene tube, as opposed to the complicated reaction vessels [6,7] and cold traps [5] required in previous procedures.

The use of a ^{206}Pb internal standard improves

both precision and accuracy. Although Sturgeon *et al.* [3] reported an excellent R.S.D. of 4% with their method, we observed up to an eight-fold larger variation in the absolute amounts of TEL formed (see Table I). Variability in the extent of ethylation may result, as the $B(C_2H_5)_3$ produced in eqn. 1 is itself capable of ethylating Pb^{2+} [6]; therefore, the recovery of Pb^{2+} (theoretically 50%) may vary with the relative ratios of STEB and Pb^{2+} in solution. Sturgeon *et al.* [7] found a recovery of 58%. We observed that ^{206}Pb-derived peak areas actually decreased with increasing volumes of STEB reagent. Variability in the extent of ethylation is compensated for by the stable isotope internal standard, as both ^{206}Pb and ^{208}Pb can be expected to undergo ethylation to the same extent. Also, the extent of the ethylation reaction is dependent on the freshness of the reagent (1-week-old STEB solution was observed to produce 75% of the response given by freshly prepared STEB) [7]. Second, other metals (especially Cu^{2+}, which may be expected to be relatively common in aqueous samples) may attack TEL [15], or interfere with the ethylation reaction. The results in Table III show that, whereas other metals can decrease the yield of TEL, the isotope dilution procedure still yields accurate Pb^{2+} concentration values. Lastly, the use of a ^{206}Pb internal standard corrects for variabilities in injection volume, injection port volatility and ionization or collection efficiency, in addition to recovery losses due to evaporation of TEL or heptane.

Note that for intermediate concentrations (40–50 ng/g), the instrumental (2.8%) and intra-assay precision (4.2%) are very similar, suggesting that at these concentrations, preparative imprecision contributes little to the total procedural imprecision, *i.e.*, errors are dominated by noise in the mass-sensing detector.

The blank ($n = 9$) for the standard procedure is calculated to be 0.40 ± 0.20 ng of lead. The blank due to a 10-μl aliquot of 1% STEB is calculated (from the slope of Fig. 5) as 0.10 ng of lead. Hence the STEB contributes substantially, but does not dominate the blank. The relatively constant blank from different tube types suggests that our RO/DI water may be the source of much of the Pb^{2+} blank.

The detection limit (3σ) is calculated to be 0.6 ng, or 0.3 ng/g for a 2-ml sample (note that the concentration detection limit could be improved by using a larger sample volume). Sturgeon *et al.* (7) achieved a detection limit of 14 pg (1 pg/g for a 10-ml sample). Our relatively higher detection limit is caused by a higher level of background lead contamination and a greater R.S.D. in the blank. The former problem could probably be obviated by obtaining cleaner water and by invoking more stringent clean room procedures, and the latter is probably due to the mass spectrometer instrumental noise. In any event, this procedure retains advantages of convenience for the analysis of low ng/g lead-containing aqueous samples.

This method is applicable to more complicated matrices (*e.g.*, blood, urine, soft drinks), but will require some modification. For example, the standard procedure utilizes a fairly low buffering capacity (about 10 m*M*), and hence might be overwhelmed by a concentrated biological fluid. Although published data [7] show that a wide pH range can support ethylation by STEB, pH values <2 or >9 should be avoided. We used a modified procedure (with an acid digestion and increased buffering capacity) to analyze 100-μl aliquots ($n = 6$) of human blood with a nominal lead level of 620 ng/g (from atomic absorption spectrometry). The modified isotope dilution procedure gave 798 ± 146 ng/g. Relatively low peak areas (reduced by nearly a factor of 100 compared with aqueous samples with comparable concentrations) were observed for $m/z = 295$; this resulted in a fairly large R.S.D. (18.3%), which may have been increased by the interference of Cu^{2+} and/or Fe^{2+} ions. Extension of this method to more complicated media will require strategies (*e.g.*, complexation of non-lead metal ions) for discriminating against interferents which can greatly reduce the yield of TEL.

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