Lead in Potable Waters
by Atomic Absorption Spectrophotometry
1976

Methods for the Examination of Waters and Associated Materials
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Methods for the Examination of Waters and Associated Materials

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London Her Majesty's Stationery Office
Warning to users

The analytical procedures given in this booklet should only be carried out by competent trained persons, with adequate supervision when necessary. Local Safety Regulations must be observed. Laboratory procedures should be carried out only in a properly equipped laboratory. Field operations should be conducted with due regard to possible local hazards, and portable safety equipment should be carried. Care should be taken against creating hazards for others. Lone working, whether in the laboratory or field, should be discouraged. Reagents of adequate purity must be used, along with properly maintained apparatus and equipment of correct specification. Specification for reagents, apparatus and equipment are given in manufacturers' catalogues and various published standards. If contamination is suspected, reagent purity should be checked before use.

There are numerous handbooks on first aid and laboratory safety. One such publication is 'Code of Practice for Chemical Laboratories' issued by the Royal Institute of Chemistry, London. Where the Committee have considered that a special unusual hazard exists, attention has been drawn to this in the text so that additional care might be taken beyond that which should be exercised at all times when carrying out analytical procedures. It cannot be too strongly emphasised that prompt first aid, decontamination, or administration of the correct antidote can save life, but that incorrect treatment can make matters worse. It is suggested that both supervisors and operators be familiar with emergency procedures before starting even a slightly hazardous operation, and that doctors consulted after any accident involving chemical contamination, ingestion, or inhalation, be made familiar with the chemical nature of the injury, as some chemical injuries require specialist treatment not normally encountered by most doctors. Similar warning should be given if a biological or radiochemical injury is suspected. Some very unusual parasites, viruses and other micro-organisms are occasionally encountered in samples and when sampling in the field. In the latter case, all equipment including footwear should be disinfected by appropriate methods if contamination is suspected.

The best safeguard is a thorough consideration of hazards and the consequent safety precautions and remedies well in advance. Without intending to give a complete checklist, points that experience has shown are often forgotten include: laboratory tidiness, stray radiation leaks (including ultra violet), use of the correct protective clothing or goggles, removal of toxic fumes and wastes, contamination in the event of breakage, access to taps, escape routes, and the accessibility of the correct and properly maintained first aid, fire-fighting, and rescue equipment. If in doubt it is safer to assume that a hazard may exist and take reasonable precautions than to assume that no hazard exists until proved otherwise.

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About this series

This booklet is one of a series intended to provide recommended methods for the determination of water quality. In the past, the Department of the Environment and its predecessors, in collaboration with various learned societies, has issued volumes of methods for the analysis of water and sewage culminating in ‘Analysis of Raw, Potable and Waste Waters’. These volumes, inevitably, took some years to prepare, so that they were often partially out of date before they appeared in print. The present series will be published as individual methods, thus allowing for the replacement of methods as quickly as possible without need of waiting for the next edition. The rate of publication will also be related to the urgency of requirement for that particular method, tentative methods being used when necessary. The ultimate aim is to provide as complete and up to date a collection of methods and reviews as is practicable, which will, as far as possible, take into account the analytical facilities available in different parts of the Kingdom, and the quality criteria of interest to those responsible for the various aspects of the water cycle. Because both needs and equipment vary widely, where necessary, a selection of methods may be recommended for a single determinand. It will be the responsibility of the users – the senior analytical chemist, biologist, bacteriologist etc, to decide which of these methods to use for the determination in hand. Whilst attention of the user is drawn to any special known hazards which may occur with the use of any particular method, responsibility for proper supervision and the provision of safe working conditions must remain with the user.

The preparation of this series and its continuous revision is the responsibility of the Standing Committee of Analysts (to review Standard Methods for Quality Control of the Water Cycle). The Standing Committee of Analysts is one of the joint technical committees of the Department of the Environment and the National Water Council. It has eight Working Groups, each responsible for one section or aspect of water cycle quality analysis. They are as follows:

1.0 General principles of sampling and accuracy of results
2.0 Instrumentation and on-line analysis
3.0 Empirical and physical methods
4.0 Metals and metalloids
5.0 General non-metallic substances
6.0 Organic impurities
7.0 Biological methods
8.0 Sludge and other solids analysis

The actual methods etc are produced by smaller panels of experts in the appropriate field, under the overall supervision of the appropriate working group and the main committee. The names of those associated with this method are listed inside the back cover.

Publication of new or revised methods will be notified to the technical press, whilst a list of Methods in Print is given in the current HMSO Sectional Publication List No 5, and the current status of publication and revision will be given in the biennial reports of the Standing Committee of Analysts.

TA DICK
Chairman

LR PITTWELL
Technical Secretary

4 November 1976
# Lead in Potable Waters by Atomic Absorption Spectrophotometry (1976 version)

**Note:** Throughout this method lead is expressed as the element (Pb).

## 1 Performance Characteristics of the Method

(For further information on the determination and definition of performance characteristics see another publication in this series.)

<table>
<thead>
<tr>
<th>1.1 Substance determined</th>
<th>All forms of lead likely to occur in potable waters (see Sections 2 and 8).</th>
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<tr>
<td>1.2 Type of sample</td>
<td>Potable waters.</td>
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<tr>
<td>1.3 Basis of method</td>
<td>Extraction of pyrrolidine dithiocarbamato-lead into 4-methylpentan-2-one followed by atomic absorption spectrophotometry.</td>
</tr>
<tr>
<td>1.4 Range of application (a)</td>
<td>Up to 100 µg/l (see Section 12).</td>
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<td>1.5 Calibration curve (a)</td>
<td>Linear to 100 µg/l (see Section 11).</td>
</tr>
<tr>
<td>1.6 Standard deviation</td>
<td>Lead concentration Standard deviation (µg/l)</td>
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<tr>
<td></td>
<td>Standard deviation (µg/l)</td>
</tr>
<tr>
<td></td>
<td>0.0 (c) 0.3–2.1 (b)</td>
</tr>
<tr>
<td></td>
<td>50.0 (c) 0.9–3.2 (b)</td>
</tr>
<tr>
<td></td>
<td>50.0 (d) 0.9–3.8 (b)</td>
</tr>
<tr>
<td></td>
<td>100.0 (d) 3.0 (a)</td>
</tr>
<tr>
<td></td>
<td>(Each estimate of standard deviation has approximately 8 degrees of freedom).</td>
</tr>
<tr>
<td>1.7 Limit of detection (b)</td>
<td>1.0–6.0 µg/l.</td>
</tr>
<tr>
<td></td>
<td>(Each estimate of limit of detection has either 5 or 10 degrees of freedom).</td>
</tr>
<tr>
<td>1.8 Sensitivity (a)</td>
<td>100 µg/l gives an absorbance of approximately 0.07.</td>
</tr>
<tr>
<td>1.9 Bias (b)</td>
<td>Average bias +2.6 µg/l on analysing 3 tap waters of lead concentrations ranging between 50.0 and 90.0 µg/l.</td>
</tr>
<tr>
<td>1.10 Interferences</td>
<td>See Section 3.</td>
</tr>
<tr>
<td>1.11 Time required for analysis (a)</td>
<td>The total analytical and operator times are the same. Typical times for 1 and 10 samples are approximately 2.25 and 3.0 hours respectively excluding any pretreatment time (see also step 9.7).</td>
</tr>
</tbody>
</table>

(a) These data were obtained at the Water Research Centre (Medmenham Laboratory)<sup>1</sup> using a procedure essentially the same as this method and a single beam atomic absorption spectrophotometer.

(b) These data were obtained from an interlaboratory calibration exercise in which 11 laboratories took part<sup>2</sup> and from tests at the Water Research Centre (Medmenham Laboratory). The range of standard deviations between individual participating laboratories has been given.

(c) Deionised water spiked with the stated lead concentration.

(d) Tap water spiked with the stated lead concentration; the unspiked tap waters contained between 0.0 and 7.0 µg/l.
2 Principle

2.1 The method described is based on experimental work carried out by the Water Research Centre (Medmenham Laboratory). Cadmium is extracted quantitatively together with lead, and both elements may, if required, be determined in the same solvent extract (see cadmium method in this series).

2.2 Some samples may require pretreatment (see Section 8) by boiling with nitric acid to convert lead to forms capable of reacting with ammonium pyrrolidine dithiocarbamate (APDC). The lead chelate formed by reaction with APDC is extracted into 4-methylpentan-2-one (methyl isobutyl ketone = MIBK) and the amount of lead in the extract is determined by atomic absorption spectrophotometry by aspirating directly into the flame.

3 Interferences

No specific tests have yet been made on the interference effects of known concentrations of other substances. However, lead added to samples has been satisfactorily recovered and it is reported that substances normally present in river waters cause no interference. It is considered, therefore, that no important problems from interference are likely with potable waters.

4 Hazards

The exhaust fumes from the atomic absorption spectrophotometer are toxic and must be ducted away. One of the reagents, 4-methylpentan-2-one (MIBK), is flammable and has a harmful vapour (see Section 5.5). It is irritating to the eyes and mucous membranes and is narcotic in high concentrations. It must not be pipetted by mouth.

5 Reagents

All reagents and standard solutions should be kept in polyethylene bottles unless otherwise stated (see Section 6.3). Analytical reagent grade chemicals are suitable unless otherwise specified.

5.1 Water

The water used for blank determinations and for preparing reagents and standard solutions should have a lead content that is negligible compared with the smallest concentrations to be determined in the samples (see Section 13.2). Deionised water or water distilled from an all glass apparatus is suitable.

5.2 50% V/V Hydrochloric acid

Dilute 500±5 ml of hydrochloric acid (d₂₀ 1.18) with water to 1 litre in a measuring cylinder. Store in a polyethylene bottle.

5.2.1 3% V/V Hydrochloric acid

Dilute 6.0±0.1 ml of 50% V/V hydrochloric acid with water to 100 ml in a measuring cylinder. Store in a polyethylene bottle.

5.3 Nitric acid (d₂₀ 1.42)

5.3.1 10% V/V Nitric acid

Dilute 100±1 ml of nitric acid (d₂₀ 1.42) with water to 1 litre in a measuring cylinder. Store in a polyethylene bottle.

5.4 1% m/V Ammonium pyrrolidine dithiocarbamate (APDC)

Dissolve 1.0±0.1 g of APDC in water and dilute with water to 100 ml in a measuring cylinder. This solution should be freshly prepared before use. Mix thoroughly before use.

5.5 4-Methylpentan-2-one (MIBK)

This reagent is hazardous (see Section 4). It is flammable and has a harmful vapour. A special grade of this solvent for atomic absorption spectrophotometry is preferable. Alternatively other grades may be purified by distillation in an all borosilicate glass apparatus. Adequate precautions must be taken during distillation including carrying it out over a distillation tray. MIBK should be stored in a glass bottle.
5.6 10% m/V Sodium hydroxide
Dissolve 10.0±0.1 g of sodium hydroxide in water in a polyethylene beaker, cool and dilute with water to 100 ml in a polyethylene measuring cylinder. Store in a polyethylene bottle.

5.7 0.1% m/V Bromophenol blue solution
Dissolve 0.10±0.01 g of bromophenol blue in 100±1 ml of 50% V/V aqueous ethanol.

5.8 Standard lead solutions:

5.8.1 Solution A 1 ml is equivalent to 1 mg Pb
Weigh 1.000±0.005 g of lead wire (greater than 99.9% purity) and dissolve with gentle heating in a mixture of 7.0±0.5 ml of nitric acid (d20 1.42) and approximately 20 ml of water. Quantitatively transfer the solution to a 1-litre calibrated flask, dilute with water to the mark and mix well. Store in a polyethylene bottle. This solution is stable for several months.

5.8.2 Solution B 1 ml is equivalent to 2 μg Pb
Dilute 2.00±0.01 ml of solution A with water to 1 litre in a calibrated flask. This solution should be freshly prepared before use.

6 Apparatus

6.1 An atomic absorption spectrophotometer equipped for an air/acetylene flame and with a lead hollow cathode lamp. A chart recorder is the most desirable form of read out. Scale expansion should be used to ensure that adequate recorder response is achieved with the highest calibration standard used.

6.2 Special apparatus
Glass tubes 20×50 mm for the collection of the organic phases after the solvent extraction of the samples. These tubes should be fitted with snap-on polyethylene lids.
400-ml graduated borosilicate glass beakers.
250-ml glass separating funnels fitted with ground glass stoppers and taps.

6.3 Cleanliness
Cleanliness is essential for this determination. If possible, apparatus should be reserved solely for lead determinations: all residual lead from previous lead determinations must be removed. Clean all new glass and polyethylene ware by filling with or soaking in 10% V/V nitric acid for 2 days. Rinse thoroughly with water. Thereafter a thorough rinse in 10% V/V nitric acid followed by a thorough rinse with water after each determination should suffice.

7 Sample Collection and Preservation
Clean a polyethylene bottle by the procedure described in Section 6.3, add 2.00±0.05 ml of 50% V/V hydrochloric acid per litre of sample to be collected and then collect the sample. The acidification minimises the adsorption of lead onto the walls of the bottle. Under certain circumstances (eg sampling by a householder) it may be necessary to modify the sampling procedure. When it is known that pretreatment will not be necessary (see Section 8) it is satisfactory to add to the empty bottle sufficient 50% V/V hydrochloric acid to bring the collected sample to pH 2.5±0.3. It is then necessary to start the analytical procedure at step 9.5 by placing 200±1 ml of the sample in the separating funnel.

8 Sample Pretreatment
Samples containing suspended and/or colloidal material may require pretreatment to convert lead to an extractable form. A few organic lead compounds may not be converted by this pretreatment procedure. Experience will indicate to analysts whether pretreatment is necessary for certain waters. The pretreatment procedure is given in steps 9.1 and 9.2.
9 Analytical Procedure

READ SECTION 4 ON HAZARDS BEFORE STARTING THIS PROCEDURE

<table>
<thead>
<tr>
<th>Step</th>
<th>Experimental Procedure</th>
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<td></td>
<td>Analysis of samples</td>
<td></td>
</tr>
<tr>
<td></td>
<td><em>Pretreatment stage</em> (note a)</td>
<td></td>
</tr>
<tr>
<td>9.1</td>
<td>Add 200±1 ml of the sample (note b) to a 400-ml graduated borosilicate glass beaker. Add 1.0±0.1 ml of nitric acid (d_20 = 1.42). Cover the beaker with a watch glass and simmer on a hot plate until the solution volume is reduced to 20±5 ml (note c).</td>
<td>(a) If pretreatment is not required (see Section 8) add 200±1 ml of sample to a 400-ml graduated borosilicate glass beaker and start at step 9.3, but omit step 9.4. This will result in a volume slightly greater than 200 ml, but it will not significantly affect the final result.</td>
</tr>
<tr>
<td>9.2</td>
<td>Cautiously wash down the watch glass and the sides of the beaker with water until the total volume in the beaker is 150±5 ml. Replace the watch glass and allow the solution to cool to ambient temperature.</td>
<td>(b) See Section 12 for the concentration range of the method.</td>
</tr>
<tr>
<td></td>
<td><em>Solvent extraction stage</em></td>
<td></td>
</tr>
<tr>
<td>9.3</td>
<td>Add 3 drops of 0.1 % m/V bromophenol blue solution and, whilst swirling, slowly add 10 % m/V sodium hydroxide until a blue colour persists. Whilst swirling, add 3 % V/V hydrochloric acid dropwise until the blue colour is just discharged. Then add 2.0±0.1 ml of 3 % V/V hydrochloric acid (note d).</td>
<td>(c) Great care must be taken during this step to minimize contamination (see Section 13).</td>
</tr>
<tr>
<td>9.4</td>
<td>Transfer the solution to a measuring cylinder and dilute with water to 200±1 ml (note c).</td>
<td>(d) Experience shows that the pH value at the end of step 9.3 should be 2.5±0.3. Very occasionally solutions may require readjustment to this value.</td>
</tr>
<tr>
<td>9.5</td>
<td>Transfer the solution to a separating funnel. Add 4.00±0.05 ml of APDC solution and shake to mix. Add 10.00±0.05 ml of MIBK (notes f and g) and stopper the funnel.</td>
<td>(e) The aqueous volume affects the final result. A constant 200 ml is therefore used.</td>
</tr>
<tr>
<td>9.6</td>
<td>Shake the funnel vigorously for 2 min±15 s. Allow to stand for 5 min±30 s and then separate and discard the aqueous phase.</td>
<td>(f) MIBK has a harmful vapour and must not be pipetted by mouth.</td>
</tr>
<tr>
<td>9.7</td>
<td>Run the organic phase into a sample tube and fit the lid (note h). Complete the atomic absorption stage during the same working day.</td>
<td>(g) If other elements, eg cadmium, are to be determined on the same aliquot of the sample, up to 25 ml of MIBK may be used throughout. However, there will be considerable loss of sensitivity and possibly also of precision.</td>
</tr>
<tr>
<td></td>
<td><strong>Blank determination</strong></td>
<td></td>
</tr>
<tr>
<td>9.8</td>
<td>A blank must be run with each batch (eg up to 10 samples) of determinations using the same batch of reagents as for the samples. To a 400-ml graduated borosilicate glass beaker add 0.40±0.05 ml of 50 % V/V hydrochloric acid and 200±1 ml of water.</td>
<td>(h) All samples, blanks and standards should be processed to this stage before proceeding to the atomic absorption stage.</td>
</tr>
</tbody>
</table>
9.9 If the pretreatment stage was used for the samples, carry out steps 9.1 to 9.7 inclusive. If not, carry out steps 9.3 and 9.5 to 9.7 inclusive.

Calibration standards

9.10 Duplicate calibration standards must be run with each batch (eg up to 10 samples) of determinations (see Section 13.4). To a 500-ml calibrated flask add 1.00±0.05 ml of 50% V/V hydrochloric acid. Pipette into the flask 25.0 ml of standard lead solution B, dilute with water to the mark and mix well. Place 200±1 ml of this solution in a 400-ml graduated borosilicate glass beaker.

9.11 If the pretreatment stage was used for the samples, carry out steps 9.1 to 9.7 inclusive. If not, carry out steps 9.3 and 9.5 to 9.7 inclusive.

Atomic absorption stage

9.12 Set up the instrument according to the manufacturer's instructions for aspirating organic solvents into an air/acetylene flame. The wavelength required is 283.3 nm.

9.13 Aspirate pure MIBK and adjust the zero. Aspirate one of the calibration standards (notes i and j) and adjust the instrument to give a suitable response, eg approximately 80% of full scale deflection.

(j) Keep the aspiration tube above the bottom of the sample tube to avoid aspiration of water which may have collected in the bottom of the sample tube.

(i) Do not aspirate more than one-third of the organic phase at this stage as it is required for 2 further aspirations.

9.14 Aspirate pure MIBK and readjust the zero if necessary. Re-aspirate both the calibration standards with an aspiration of pure MIBK after each and measure the maximum instrument responses $C_1$ and $C_2$ (eg peak height).

9.15 Aspirate the blank (note i) and then pure MIBK and measure the maximum instrument response $B_1$. Aspirate the samples (note i) with an aspiration of pure MIBK after each. Measure the maximum instrument response of the sample, $S$.

9.16 To check for any instrument drift aspirate both calibration standards and the blank with an aspiration of pure MIBK after each and measure the maximum instrument responses (eg peak height) $C_3$, $C_4$ and $B_3$ respectively. If $C_1$, $C_2$, $C_3$ and $C_4$ and also $B_1$ and $B_3$ are in satisfactory agreement calculate the means $\bar{C}$ and $\bar{B}$. 
<table>
<thead>
<tr>
<th>Step</th>
<th>Experimental Procedure</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>9.17</td>
<td>Calculate the concentration, $A$, of lead in the sample from</td>
<td></td>
</tr>
<tr>
<td></td>
<td>$A = \frac{S - \bar{B}}{C - \underline{B}} \times 100 \mu g/l$</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Where $C = C_1 + C_2 + C_3 + C_4$</td>
<td></td>
</tr>
<tr>
<td></td>
<td>$\bar{B} = \frac{B_1 + B_2}{2}$</td>
<td></td>
</tr>
<tr>
<td></td>
<td>This calculation assumes a linear calibration curve.</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Linearity must be checked (see Section 11).</td>
<td></td>
</tr>
</tbody>
</table>

### 10 Measurement of Maximum Instrument Responses

The maximum instrument responses for samples, standards and blanks are measured with respect to the response of pure MIBK aspirated on either side. The following example is for the measurement of peak height when a chart recorder is in use.

Draw a line on the chart through the traces for the pure MIBK bracketing the peak height to be measured. Draw a line through the trace corresponding to the equilibrium response of that peak height, and measure the perpendicular separation of the two constructed lines using a ruler. When the recorder traces show short-term variations it is suggested that the lines be drawn through the visually estimated mid-points of the equilibrium portions of the traces.

### 11 Checking the Linearity of the Calibration Curve

The procedure given in this section must be carried out on at least two independent occasions before application of this method to any samples and regularly thereafter.

To each of a series of 500-ml calibrated flasks add $1.00 \pm 0.05$ ml of 50% V/V hydrochloric acid. Pipette respectively to these flasks 0.0, 5.0, 10.0, 15.0, 20.0 and 25.0 ml of standard lead solution $B$ and dilute with water to the mark. These flasks contain respectively 0, 20, 40, 60, 80 and 100 $\mu g/l$ lead. Place $200 \pm 1$ ml of these solutions in a series of 400-ml graduated borosilicate glass beakers and carry out the procedure given in steps 9.3, 9.5 to 9.7 inclusive and steps 9.12 to 9.16 inclusive. Plot the maximum instrument response (eg peak height) against $\mu g/l$ lead.

The calibration curve is normally linear to 100 $\mu g/l$ lead; however, the linearity of the curve may depend on the type of instrumentation used and therefore linearity must be checked. If the calibration curve departs from linearity, the calibration standard in step 9.10 is not appropriate, nor is the range given in Section 1.4. In such a case the calibration standard chosen for step 9.10 should be the highest concentration on the linear portion of the calibration curve and the concentration range of the method should be adjusted accordingly.

### 12 Change of Concentration Range of the Method

If the lead concentration in the sample is likely to exceed 100 $\mu g/l$ an appropriately smaller aliquot of the sample must be taken for analysis. To this volume of sample, V ml, add sufficient 50% V/V hydrochloric acid so that there is the same total volume of 50% V/V hydrochloric acid present as there would be in 200 ml of sample. Dilute with water to 200 ml and proceed as in step 9.1 onwards. It is necessary to alter the calculation of the result, step 9.17, as follows:

$$A = \frac{S - \bar{B}}{C - \underline{B}} \times 100 \times \frac{200}{V} \mu g/l \text{ lead}$$
13 Sources of Error

The attention which it is necessary to pay to sources of error depends on the accuracy required of the analytical results. The following sub-sections summarize the main sources of error.

13.1 Contamination

It is desirable to carry out the analysis in a laboratory in which no appreciable amounts of lead or its compounds are handled. The technique and working conditions should be critically examined and any sources of contamination eliminated or minimized. In particular, it is desirable to reserve the glass apparatus used for the lead determinations solely for this purpose and to carry out a preliminary series of blank determinations to ensure low blank values before analysing any samples.

13.2 Lead content of the water used for blank determinations

If the water used for the blank determinations contains lead the results will be falsely low. The importance of this error depends on the lead concentration of the blank water and the concentrations of interest in the samples. Ideally the lead content of the water used for each blank determination should be measured and an appropriate correction made. An upper limit for the lead content of the water can be calculated by converting the maximum instrument response (eg peak height) to concentration units. If the concentration obtained is negligible compared with the concentrations of interest in the samples no further action is required. If the concentration obtained is not negligible then the procedure which follows should be used to determine the lead content of the water:

(a) To each of two 500-ml borosilicate glass beakers add 200±5 ml of water and 0.40±0.05 ml of 50% V/V hydrochloric acid.

(b) To each of two 500-ml borosilicate glass beakers add 400±10 ml of water and 0.40±0.05 ml of 50% V/V hydrochloric acid.

(c) Cover all beakers with clean watch glasses and heat those from (b) on a hot plate until the volumes in them have been reduced to approximately 200 ml. Add a further 200±5 ml of water to each beaker from (b) and continue heating until the volumes are reduced to 200±5 ml. Cool the solution to room temperature.

(d) Analyse the contents of all four beakers as described in Section 9 and let the measured maximum instrument responses be \( W_1 \) and \( W_2 \) for the two unheated beakers and \( W_{11} \) and \( W_{21} \) for the two heated beakers.

(e) The lead content of the blank water is equivalent to a maximum instrument response of

\[
W = \frac{(W_{11} + W_{21}) - (W_1 + W_2)}{4}.
\]

(f) The concentration of lead, \( A_w \), in the blank water is then given by

\[
A_w = \frac{W}{C - B} \times 100 \mu g/l \text{ lead}
\]

(See step 9.17).

13.3 Interfering substances

See Section 3. The effect of possible interfering substances may be determined by analysing samples spiked with lead and various concentrations of the potential interfering substance.

13.4 Calibration standards

The calibration curve for this method has been found to be linear though its slope may vary from one set of determinations to another. Such variations are caused by changes in the sensitivity of the atomic absorption spectrophotometer. Therefore a calibration standard must be run for each batch of analyses and steps 9.10 onwards give the necessary procedure. This procedure assumes a linear calibration curve and the linearity must be checked (see Section 11).
Once the methods have been put into normal routine operation many factors may subsequently adversely affect the accuracy of the analytical results. It is recommended that experimental tests to check certain sources of inaccuracy should be made regularly. Many types of tests are possible and they should be used as appropriate. As a minimum, however, it is suggested that a standard solution of lead of suitable concentration be analysed at the same time and in exactly the same way as normal samples (see Section 5.8.2 and step 9.10). The results obtained should then be plotted on a quality control chart which will facilitate detection of inadequate accuracy, and will also allow the standard deviation of routine analytical results to be estimated.

15 References


Address for Correspondence

However thoroughly a method may be tested, there is always the possibility of a user discovering a hitherto unknown problem. Users with information on this method are requested to write to:

The Technical Secretary
The Standing Committee of Analysts
The Department of the Environment
2 Marsham Street
LONDON SW1P 3EB
England
Standing Committee of Analysts

Members of the Committee Responsible for this Method:

Mr DE Bond until January 1974*
Mr J Borland after June 1975*
Dr JM Carter after June 1975*
Dr GW Clayfield*
Dr V Collins after June 1975*
Mr EC Conchie†
Dr RL Cooper after June 1975*
Dr BT Croll after June 1975*
Mr TA Dick after February 1975*
Mr GE Eden until June 1975*
Mr B Gubbins until September 1975†
Dr N Harkness until June 1975*
Mr PJ Hewitt†
Mr E Hodges after June 1975*
Mr GJ Holland after June 1975*
Dr AJ Howard after June 1975*
Mr OD Hydes after January 1974†
Mr WM Lewis *†
Mr PJ Long after June 1975*
Mr GF Lowden†
Mr D Mercer until June 1974*
Mr P Morries after June 1975*
Mr D Myles after June 1975*
Mr AH Nield after January 1976*
Dr HA Painter after June 1975*
Mr JF Palframan†
Dr AT Palin until June 1975*
Dr SJ Patterson *
Mr LR Pittwell *
Dr JE Portmann after June 1975*
Mr LD Purdie after June 1975*
Dr L Ranson after February 1975†
Mr BD Ravenscroft after June 1975*
Prof JP Riley*†
Mr R Sinar *
Mr PAH Sperring until January 1976*
Mr BT Whitham after June 1975*
Mr AL Wilson *†
Dr R Wood after June 1975*

main committee member*

working group member†