Magnesium in Waters and Sewage Effluents by Atomic Absorption Spectrophotometry 1977

Methods for the Examination of Waters and Associated Materials





The British Library

LENDING DIVISION

Boston Spa Wetherby West Yorkshire United Kingdom LS23 7BQ

Best available copy

Magnesium in Waters and Sewage Eff



by Atomic Absorption **Spectrophotometry** 1977

Tentative Method

Otherwise follow lending library's instructions as given below. 30.DEC 91 3

I declare that this item is required only for purposes of private study or research.

If date only given this is BLDSC return date.

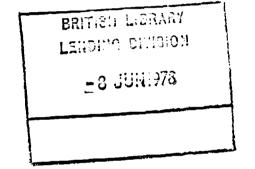
ENCLOSE WITH ITEM

Date

Methods for the Examination of Waters and Associated Materials

Contents

| Wai | rning to users | 2 |
|-----|---|----------------|
| Abo | out this series | 3 |
| 1 | Performance Characteristics of the Method | 4 |
| 2 | Principle | 5 |
| 3 | Interferences | 5 |
| 4 | Hazards | 5 |
| 5 | Reagents | 5 |
| 6 | Apparatus | 5 |
| 7 | Sample Collection and Preservation | 6 |
| 8 | Sample Pretreatment | 6 |
| 9 | Analytical Procedure | 6 |
| 10 | Measurement of Maximum Instrument Respons | es 8 |
| 11 | Checking the Linearity of the Calibration Curve | 8 |
| 12 | Change in Concentration Range of the Method | 8 |
| 13 | Sources of Error | 9 |
| 14 | Checking the Accuracy of Analytical Results | 9 |
| 15 | References | 9 |
| 16 | Appendix—Estimation of the Accuracy of Analytical Results | 10 |
| Ad | dress for Correspondence | 12 |
| Me | embership responsible for this method is back of | nside cover |



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. Specifications 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 has 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 microorganisms 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, containment 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.

© Crown copyright 1978 First published 1978

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 or addition 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 issued when necessary. The 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 nine 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
- 9.0 Radiochemical methods

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 Secretary

20 July 1977

Magnesium in Waters and Sewage Effluents by Atomic Absorption Spectrophotometry Tentative Method (1977 version)

Note: Throughout this method magnesium is expressed as the element (Mg).

1 Performance Characteristics of the Method

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

| 1.1 | Substance determined | All forms of magnesium (see Sections 2 and 8) | | |
|------|---|---|--|--|
| 1.2 | Type of sample | Waters and sewage effluents. | | |
| 1.3 | Basis of method | The acidified sample is treated with a lanthanum salt and aspirated into the flame of an atomic absorption spectrophotometer. | | |
| 1.4 | Range of application (a) | Up to 5 mg/l (see Section 12). | | |
| 1.5 | Calibration curve (a) (b) | Linear to 5 mg/l (see | Section 11). | |
| 1.6 | Standard deviation (a) (d) (within-batch) | Magnesium concentration (mg/l) | Within-batch standard deviation (mg/l) | |
| | | 0.0 0.0 0.1 5.0 | 0.0158 (b) 0.0184 (c) 0.0215 (b) 0.0366 (b) | |
| | | (Each estimate has 9 degrees of freedom). | | |
| 1.7 | Limit of detection (a) (d) | 0.06 mg/l (with 9 degrees of freedom) (b) (c). | | |
| 1.8 | Sensitivity (a) (c) | 1.0 mg/l gives an absorbance of approximately 0.067. | | |
| 1.9 | Bias | Not known. | | |
| 1.10 | Interferences | See Section 3. | | |
| 1.11 | Time required for analysis (b) (c) | _ | and operator times are the for 1 and 10 samples are | |

(a) These performance characteristics are based on taking the equivalent of a 5 ml sample.

30 and 45 minutes respectively excluding any pretreatment time. Typical total analytical time for 10 samples including pretreatment time is

- (b) These data were obtained at the Water Research Centre (Medmenham Laboratory)⁽¹⁾ using single beam atomic absorption spectrophotometer.
- (c) These data were obtained at the Water Research Centre (Stevenage Laboratory)⁽²⁾ using a double beam atomic absorption spectrophotometer.
- (d) These data were obtained using distilled water spiked with the stated magnesium concentration. Pretreatment was not carried out.

2 Principle

- 2.1 The method is based on experimental work carried out at the Water Research Centre (Medmenham⁽¹⁾ and Stevenage⁽²⁾ Laboratories). The amount of magnesium in the treated sample is measured by atomic absorption spectrophotometry by aspirating directly into the flame. Lanthanum^(3,4) is present in the solution which is aspirated in order to release magnesium from refractory compounds.
- 2.2 Normally collection of the sample into acid or acidification of the sample on receipt in the laboratory is sufficient to ensure that all forms of magnesium are dissolved. However, for certain types of sample, pretreatment (see Section 8) with nitric acid may be necessary.

3 Interference

The atomic absorption procedure is considered to be specific for magnesium. Interferences ^(5,6, and 7) caused by substances which produce refractory magnesium compounds such as aluminium compounds, phosphates, sulphates and silicates, can be minimized by the addition of a lanthanum salt.

4 Hazards

The exhaust fumes from the atomic absorption spectrophotometer are toxic and must be ducted away.

5 Reagents

All reagents and standard solutions should be kept in polyethylene bottles unless otherwise stated (see Section 6.2). 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 magnesium content that is negligible compared with the smallest concentration to be determined in the samples. Deionized water or water distilled from an all-glass apparatus is suitable.

5.2 5M Hydrochloric acid (approximately)

Dilute 445 ± 5 ml of hydrochloric acid (d₂₀ 1.18) with water to 1 litre in a measuring cylinder.

5.3 1% m/V Lanthanum/5M hydrochloric acid solution

Dissolve 11.70 ± 0.05 g of oven dried $(120 \pm 10$ °C) lanthanum oxide in 1 litre of 5M hydrochloric acid.

5.4 Standard magnesium solutions

5.4.1 Solution A 1 ml contains 100 µg magnesium

Dissolve 0.166 ± 0.001 g of oven-dried $(120 \pm 10$ °C) magnesium oxide in approximately 20 ml of 5M hydrochloric acid in a 1-litre calibrated flask and dilute with water to the mark.

5.4.2 Solution B 1 ml contains 1 µg magnesium

Dilute 10.00 ± 0.02 ml of solution A above to 1 litre in a calibrated flask with water. This solution should be prepared freshly before use.

5.5 Nitric acid (d₂₀ 1.42)

5.5.1 10% V/V Nitric acid

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

6 Apparatus

6.1 An atomic absorption spectrophotometer equipped for an air/acetylene flame and with a magnesium hollow cathode lamp. A chart recorder is the most desirable form of read-out.

6.2 Cleanliness of glass and polyethylene ware

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 rinse with 10% V/V nitric acid followed by thorough rinsing with water prior to analysis should be sufficient.

7 Sample Collection and Preservation

Clean a polyethylene bottle by the procedure described in Section 6.2, add 2.0 ± 0.1 ml of 5M hydrochloric acid per litre of sample to be collected and then collect the sample. This acidification, which is preferable but not essential, is to prevent precipitation of magnesium salts and to assist in the dissolution-of-colloidal and particulate forms of magnesium.

8 Sample Pretreatment

Potable waters should not require pretreatment as the collection of the samples into acid or the subsequent acidification on receipt in the laboratory should be sufficient to dissolve any suspended magnesium. Highly turbid samples (eg some raw waters and sewage effluents) may require pretreatment to solubilise all forms of magnesium. Experience will indicate to analysts whether pretreatment is necessary for their particular type of samples. It is recommended that this should be checked by analysts for their particular water samples by comparing the results obtained by carrying out the procedure in steps 9.1 to 9.13 with those obtained from the procedure in steps 9.4 to 9.13.

Notes

9 Analytical Procedure

Step Experimental Procedure

READ SECTION 4 ON HAZARDS BEFORE STARTING THIS PROCEDURE

| Step | Experimental Procedure | inotes |
|------|--|---|
| | If pretreatment necessary (note a) | (a) If pretreatment is not required start at step 9.4. |
| | Samples | • |
| 9.1 | Add 50.00 ± 0.05 ml of well mixed sample (note b) to a 100 ml beaker. Add 5 ± 1 ml nitric acid (d_{20} 1.42). Cover the beaker with a watch-glass and evaporate carefully to dryness on a regulated hot plate. Remove from the source of heat, cool and add 2.0 ± 0.1 ml of 5M hydrochloric acid. Replace the beaker on the hot plate and heat gently. Add approximately 5 ml of water and heat almost to boiling. Cool and transfer the contents quantitatively to a 50-ml calibrated flask. Dilute with water to the mark and mix. Allow any precipitate to settle before taking an aliquot from the supernatant liquid for step 9.6. | (b) If the sample was not collected into acid then 2.0 ± 0.1 ml of 5M hydrochloric acid per litre of sample must be added to it on receipt in the laboratory. |
| | Blank determination | |
| 9.2 | A blank must be run with each batch (eg up to 10 samples) of determinations for which pretreatment was carried out, using the same batch of reagents as for samples. Carry out step 9.1 using 50.0 ± 0.5 ml of water instead of the sample and then proceed to step 9.6 treating the solution as if it were a sample. | |
| | Calibration standards | |
| 9.3 | Duplicate calibration standards must be run with each batch (eg up to 10 samples) for which pretreatment was carried out, using the same batch of reagents as for samples. Dilute 2.50 ± 0.02 ml of standard magnesium solution A with water to 50 ml and carry out step 9.1 using this solution instead of the sample and then proceed to step 9.6 treating the solution as if it were a sample. | |

| Step | Experimental Procedure | Notes |
|------|--|--|
| | Pretreatment not required (note c) | (c) Samples start at step 9.6. |
| | Blank determination | |
| 9.4 | A blank must be run with each batch (eg up to 10 samples) of determinations for which pretreatment was not carried out, using the same batch of reagents as for the samples. Carry out step 9.6 using 5.00 ± 0.02 ml of water instead of the sample. | · |
| | Calibration standards | |
| 9.5 | Duplicate calibration standards must be run with each batch (eg up to 10 samples of determinations for which pretreatment was not carried out (see Section 13.3), using the same batch of reagents as for the samples. Carry out step 9.6 using 25.00 ± 0.03 ml of standard magnesium solution B instead of the sample. | |
| | All samples, blanks and standards (note d) | (d) See Section 12 for the concentration range of the |
| 9.6 | Add 5.00 ± 0.02 ml of the well mixed sample (note b) or pretreated sample to a 50-ml calibrated flask. Add 5.0 ± 0.1 ml of the 1% m/V lanthanum/5M hydrochloric acid solution. Dilute with water to the mark (note e) and proceed to step 9.7. | method. (e) All samples, standards and blanks must be processed to this stage before proceeding to the atomic absorption stage. |
| | Atomic absorption stage | |
| 9.7 | Set up an air/acetylene flame instrument according to the manufacturer's instructions. The wavelength required is 285.2 nm. | |
| 9.8 | Aspirate water until equilibrium conditions are established and adjust the zero. Aspirate one of the calibration standards (see step 9.3 or step 9.5 as appropriate) and adjust the instrument to give a suitable response (eg approximately 80% of full scale deflection). | |
| 9.9 | Aspirate water and re-adjust the zero. Aspirate both calibration standards with an aspiration of water after each and measure the maximum instrument responses C_1 and C_2 (eg peak height). | |
| 9.10 | Aspirate the blank, followed by an aspiration of water. Measure the maximum instrument response B_1 . | |
| 9.11 | Aspirate the samples with an aspiration of water after each. Measure the maximum instrument response for the sample, S. | |
| 9.12 | After each batch (eg 10 samples) re-aspirate the blank and the calibration standards with an aspiration of water between each. Measure the maximum instrument responses B_2 , C_3 and C_4 respectively. If B_1 and B_2 , and C_1 , C_2 , C_3 and C_4 are in satisfactory agreement calculate the means \overline{B} and \overline{C} respectively. | |

Calculation of results (see Section 12)

9.13 Calculate the concentration, A, of magnesium in the sample from:

$$A = \frac{\overline{S} - \overline{B}}{\overline{C} - \overline{B}} \times 5 \text{ mg/l Mg (note f)}$$
Where $\overline{C} = \frac{C_1 + C_2 + C_3 + C_4}{4}$

$$\overline{B} = \frac{B_1 + B_2}{2}$$

This calculation assumes a linear calibration curve. Linearity must be checked (see Section 11).

(f) The dilution of the sample by the acid into which it is collected is negligible.

10 Measurement of Maximum Instrument Responses

The maximum instrument responses for samples, standards and blanks are measured with respect to the response of water 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 water, bracketing the peak height to be measured. Draw a parallel 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 as determined by control chart procedures (see Section 14).

To a series of 50-ml calibrated flasks add 5.0 ± 0.1 ml of 1% m/V lanthanum/5M hydrochloric acid solution. Pipette into these flasks 0.0, 5.0, 10.0, 15.0, 20.0 and 25.0 ± 0.02 ml of standard magnesium solution B and dilute with water to the mark. Mix well. These flasks contain 0, 1, 2, 3, 4 and 5 mg/l Mg respectively (based on a 5 ml effective sample). Carry out step 9.7 and plot the maximum instrument responses against mg/l Mg.

The calibration curve is normally linear to 5 mg/l Mg but this will depend to some extent on the type of instrumentation used and therefore this must be checked. If the curve departs from linearity the calibration standards (steps 9.3 and 9.5) are not appropriate, nor is the range given in Section 1.4. In such a case the calibration standard chosen 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

With appropriate instrumental settings the given procedure can be used to determine magnesium in the concentration range given in Section 1.4. When the magnesium concentration in the sample is likely to exceed 5.0 mg/l Mg, an appropriately smaller aliquot of the sample must be taken for analysis, eg an aliquot of 2.0 ml instead of 5.0 ml would increase the range to 12.5 mg/l Mg and the calculation (step 9.13) would be:

$$A = \frac{S - \overline{B}}{\overline{C} - \overline{B}} \times 12.5 \text{ mg/l Mg}$$

and for samples in the range 0 to 0.5 mg/l Mg an aliquot of 40 ml should be taken and the calculation would be:

$$A = \frac{S - \overline{B}}{\overline{C} - \overline{B}} \times 0.625 \text{ mg/l Mg}$$

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

The technique and working conditions should be critically examined and any contamination eliminated or minimized.

13.2 Interfering substances

See Section 3.

13.3 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 9.3 and 9.5 give the necessary procedure. This procedure assumes a linear calibration curve and the linearity must be checked (see Section 11).

14 Checking the Accuracy of Analytical Results

(For further infortion see another publication in this series). 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 of the accuracy 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 magnesium of suitable concentration be analysed at the same time and in exactly the same way as normal samples (see Section 5.4.2 and step 9.6). 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

- (1) Nield AH, Water Research Centre (Medmenham Laboratory), ILR No 383, September 1974.
- (2) Department of the Environment, File WS/646/53, Paper NSP/14, March 1974.
- (3) Yofé J and Finkelstein R, Anal. Chim. Acta, 1958, 19, 166.
- (4) Yofé J, Avri R and Stiller M, Anal. Chim. Acta, 1963, 28, 331.
- (5) Ramakrishna TV, West PW and Robinson JW, Anal. Chim. Acra, 1968, 40, 347.
- (6) Knutson KE, Analyst, 1957, 82, 241.
- (7) Harrison A and Ottaway JM, Proc. Soc. Analyt. Chem., September 1972, p. 205.

Appendix

Estimation of the Accuracy of Analytical Results using the Magnesium Method

1 Introduction

Quantitative investigation of the accuracy achievable when the magnesium method is used appears to be limited to work at the Water Research Centre (Medmenham and Stevenage Laboratories). Before firmly recommending the method for general use, it is desirable to know the accuracy achievable in other laboratories. It would, therefore, be of great value if any laboratory using or considering the use of this method, could estimate the accuracy of its own analytical results and report the findings to the Technical Secretary of the Metals and Metalloids Working Group of the DOE/NWC Standing Committee of Analysts*.

The precision achieved and the effects of any interfering substances that may be present in samples are of particular interest. Any information on these aspects would be useful, but the value of such information would be greatly enhanced if it were obtained to a common plan so that the information can be compared and valid conclusions drawn. Accordingly, suggestions for a suitable experimental design and analysis of results are given in the following sections and it is strongly urged that laboratories follow this design whenever possible. The design has been chosen to be as simple as possible; more complex designs are possible and would give more information.

2 Basis of suggested Tests

The limit of detection is governed by the within-batch variability of blank determinations. The precision of analytical results may depend on the concentration of magnesium in the sample analysed and on the type of sample, eg, worse precision may be obtained with samples than with standard solutions. For these reasons the basic design recommended is the analysis of one portion of each of the following solutions on each of n days, where n is at least 5 and preferably up to 10.

| Solution No | Description | | |
|-------------|-------------------------------------|--|--|
| 1 | Blank | | |
| 2 | Another blank | | |
| 3 | Standard solution 0.5 mg/l Mg | | |
| 4 | Standard solution 5.0 mg/l Mg | | |
| 5 | Typical sample† | | |
| 6 | Same sample spiked with 5.0 mg/l Mg | | |

†diluted with water if necessary so that the magnesium concentration is within the range of the method.

It is essential that these solutions be treated exactly as if they were samples and the procedure specified in Section 9 of the method be rigidly followed. These solutions should be analysed in random order in with each batch of analyses. Solutions 1 to 4 should be prepared each day exactly as described in the method and should contain the same amount of hydrochloric acid as is present in the samples. On any one day the same batch of water should be used to prepare these four solutions. For solutions 5 and 6 a total of 2 litres of typical sample are required. Prepare solution 6 each day when required by spiking solution 5 as follows: add with a pipette 5.00 ml of standard magnesium solution A to 100 ml of solution 5. When analysing solution 6 it will be necessary to take into account Section 12 and to take an appropriately smaller aliquot. The total period of the tests may be any convenient time so long as the magnesium concentration in solution 5 does not change appreciably (up to 2 weeks). The results of the analyses of solutions 5 and 6 will provide a check on the effect of sample type on precision. Any deviation of the recovery of spiked magnesium from 100% may give an indication of the presence of interfering substances.

Evaluation of Results

The raw experimental results should be sent direct to the Department of the Environment* for evaluation together with the results obtained from the standards used to establish the calibration curve in each batch of analysis. However, for those laboratories wishing to make the calculations themselves the details are given below.

- 3.1 Convert all results to concentrations as described in the method. Deduct the first of the two blank values (solution 1) from each of the other solution values.
- 3.2 Calculate the mean concentration of the n results for each solution.
- 3.3 Calculate the standard deviation, s, of the n results for each solution from:

$$s = \sqrt{\frac{\sum (x_i - \overline{x})^2}{n - 1}}$$

where x_i = the result from the ith batch

 \overline{x} = the mean value of x_i

3.4 Calculate the within-batch standard deviation, s_w, of the blank from:

$$s_w = \sqrt{\frac{\sum (x_{1_i} - x_{2_i})^2}{2n}}$$

where x_{1i} = the 1st blank result (solution 1) from the ith batch x_{2i} = the 2nd blank result (solution 2) from the ith batch

3.5 Calculate the mean percentage recovery, R, of the spiked magnesium in solution 6 from:

$$R = \frac{(1.05 \ \overline{x}_6 - \overline{x}_5)}{5}$$

where \bar{x}_s = the mean value of the results for solution 5 \bar{x}_6 = the mean value of the results for solution 6

3.6 Summarize the results as in the following table:

| Solution | No of results n | Mean Magnesium Concentration mg/l | Standard Deviation mg/l | Mean Recovery % |
|------------------------------------|-----------------------|---|-------------------------------|-----------------------|
| 2 Blank 3 Standard, 0.5 mg/l Mg | | | | |
| 4 Standard, 5.0 mg/l Mg 5 Sample | | | | _ |
| 6 Solution 5+5.0 mg/l Mg | | | | |

The appropriate sample description should be entered in the space for solution 5. The standard deviation from step 3.4 is entered for the blank solution 2 and the standard deviations from step 3.3 are entered for solutions 3 to 6. If the pretreatment procedure (steps 9.1 to 9.3) was carried out this should also be stated.

The Technical Secretary
The Metals and Metalloids Working Group
The Standing Committee of Analysts
The Department of the Environment
2 Marsham Street
LONDON SW1P 3EB
England

^{*}Results to be sent to the following:

Correspondence

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:

| | Main Committee | | Working Group | | Panel | |
|---------------------------|----------------|------------|---------------|-----------------|-----------|--|
| | from | to | from | to | from to | |
| Mr MJ Beckett | | | Sept 1973 | Sept 1974 | | |
| Mr DE Bond | May 1973 | Jan 1974 | - | • | | |
| Mr J Borland | June 1975 | | | | | |
| Dr GW Bryan | | | Nov 1975* | | | |
| Mr W Carr | | | Nov 1975* | | Nov 1975 | |
| Dr JM Carter | June 1975 | | Sept 1974 | June 1975 | | |
| Dr GW Clayfield | May 1973 | | | | | |
| Dr V Collins | June 1975 | Jan 1977 | | | | |
| Mr EC Conchie | | | Nov 1975 | | | |
| Dr RL Cooper | June 1975 | | | | | |
| Dr BT Croli | June 1975 | | | | | |
| Dr JAW Dalziel | | | Sept 1974 | June 1975 | | |
| Mr KG Darrall | | | Nov 1974 | June 1975 | | |
| Mr JA Davis | | | Sept 1973 | vano 1775 | Nov 1975 | |
| Dr W Davison | | | Nov 1975* | | 1107 1575 | |
| Mr TA Dick | Feb 1975 | | 1104 13/3 | | | |
| Mr JWR Dutton | 1 (0 15/3 | | Nov 1975 | | | |
| Mr GE Eden | May 1973 | June 1975 | 1707 13/3 | | | |
| Mr GE Eden Mr MG Firth | Iviay 19/3 | Julie 19/3 | Nov 1975 | | | |
| | 141072 | I 1075 | Nov 1973 | | | |
| Dr N Harkness | May 1973 | June 1975 | G 1073 | 1 - 1075 | | |
| Mr J Heron | | | Sept 1973 | June 1975 | | |
| Mr PJ Hewitt | 1 1075 | | Nov 1975 | Dec 1976 | | |
| Mr E Hodges | June 1975 | | | | | |
| Mr GJ Holland | June 1975 | D 1056 | | | | |
| Dr AJ Howard | June 1975 | Dec 1976 | N. 1055 | | N. 10554 | |
| Mr OD Hydes | 3.4 1072 | | Nov 1975 | | Nov 1975* | |
| Mr WM Lewis | May 1973 | | Nov 1975 | | Nov 1975 | |
| Mr PJ Long | June 1975 | | | | | |
| Mr GF Lowden | | | Feb 1974 | | Nov 1975 | |
| Mr JC McCullins | Jan 1976 | | | | | |
| Mr D Mercer | May 1973 | June 1974 | | | | |
| Dr HAC Montgomery | | | Sept 1973 | Feb 1974 | | |
| Mr P Morries | June 1975 | | | | | |
| Mr D Myles | June 1975 | • | Sept 1974 | June 1975 | | |
| Dr EJ Newman | | | Nov 1975 | | | |
| Mr AH Nield | Jan 1976 | | Sept 1973 | June 1975 | | |
| Dr HA Painter | June 1975 | | | | | |
| Mr JF Palframan | | | Nov 1975 | | | |
| Dr AT Palin | May 1973 | June 1975 | | | | |
| Dr SJ Patterson | May 1973 | | Sept 1973 | June 1975 | | |
| Mr LR Pittwell | Dec 1973 | | | | | |
| Dr JE Portmann | June 1975 | | | | | |
| Mr LD Purdie | June 1975 | | | | | |
| Dr L Ranson | | | Nov 1975 | | | |
| Mr BD Ravenscroft | June 1975 | | | | | |
| Prof JP Riley | June 1975 | | Nov 1975 | | | |
| Mr IR Scholes | | | Nov 1975 | | | |
| Mr DT Schofield | | | | | Nov 1975 | |
| Dr EA Simpson | | | Sept 1974 | June 1975 | | |
| Mr R Sinar | May 1973 | | - | | | |
| Mr PAH Sperring | May 1973 | Jan 1976 | | | | |
| Dr G Topping | • | | Nov 1975 | | | |
| Dr KC Wheatstone | | | Nov 1974 | June 1975 | | |
| Mr BT Whitham | June 1975 | | | - | | |
| Mr AL Wilson | May 1973 | | | | | |
| Dr R Wood | June 1975 | | | | | |

^{*}Occasional co-opted member

HER MAJESTY'S STATIONERY OFFICE

Government Bookshops

49 High Holborn, London WC1V 6HB
13a Castle Street, Edinburgh EH2 3AR
41 The Hayes, Cardiff CF1 1JW
Brazennose Street, Manchester M60 8AS
Southey House, Wine Street, Bristol BS1 2BQ
258 Broad Street, Birmingham B1 2HE
80 Chichester Street, Belfast BT1 4JY

Government publications are also available through booksellers