Zinc in Potable Waters <u>by Atomic Absorption Spectrophotometry</u> 1980

Methods for the Examination of Waters and Associated Materials

Zinc in Potable Waters

by Atomic Absorption Spectrophotometry 1980

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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 properly equipped laboratories. 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. 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. Among such publications are: 'Code of Practice for Chemical Laboratories' and 'Hazards in the Chemical Laboratory' issued by the Royal Society of Chemistry, London; and 'Safety in Biological Laboratories (Editors Hartree and Booth), Biochemical Society Special Publication No 5, The Biochemical Society, London, which includes biological hazards.

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, decon-

tamination, 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 radio chemical 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 correct protective clothing and 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 the hazard may exist and take reasonable precautions, rather than to assume that no hazard exists until proved otherwise.

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About this series

This booklet is part of a series intended to provide recommended methods for the determination of water quality. In addition, the series contains short reviews of the more important analytical techniques of interest to the water and sewage industries. 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 nonmetallic 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.

Whilst an effort is made to prevent errors from occurring in the published text, a few errors have been found in booklets in this series. Correction notes for booklets in this series are given in the Reports of The Standing Committee of Analysts, published by the Department of the Environment but sold by the National Water Council, 1 Queen Anne's Gate, London SW1H 9ET. Should an error be found affecting the operation of a method, the true sense not being obvious, or an error in the printed text be discovered prior to sale, a separate correction note will be issued for inclusion in the booklet.

TA DICK Chairman

LR PITTWELL
Secretary

4 December 1980

Zinc in Potable Waters by Atomic Absorption Spectrophotometry 1980 version

Note: Throughout this method zinc is expressed as the element (Zn).

| the Method |
|--------------------|
| (For further |
| information on the |
| determination and |
| definition of |

Performance Characteristics of

performance characteristics see General Principles of Sampling and Accuracy of Results 1980, also published in this series.

| 1.1 | Substance determined | All forms of zinc likely to occur in potable waters (see Sections 2 and 8). | |
|------|---------------------------------------|--|--|
| 1.2 | Type of sample Potable waters. | | |
| 1.3 | Basis of the method | Concentration of the sample by evaporation followed by atomic absorption spectrophotometry. | |
| 1.4 | Range of application (a) | Up to $100 \mu g/l$ (see Section 11). | |
| 1.5 | Calibration curve (a) | Linear to $100 \mu g/l$ (see Section 10). | |
| 1.6 | Standard deviation (a) (within batch) | Zinc concentration $(\mu g/l)$ $(\mu g/l)$ $(\mu g/l)$ $(\mu g/l)$ 0.0 (b) 0.3 to 0.8 10.0 (b) 0.8 to 1.0 50.0 (b) 0.8 to 3.6 100.0 (c) 0.5 to 1.4 0.0 (c) 0.5 to 1.4 0.0 (c) 0.5 to 2.1 0.0 (c) 0.7 to 0.5 to 0.7 to 0.8 to 0.0 (c) 0.7 to 0.8 to 0.0 (c) 0.7 to 0.8 to 0.0 (c) 0.7 to 0.8 0.0 (c) 0.9 0.0 (c) 0.0 0.0 (c) 0.0 0.0 (c) 0.0 0.0 (c) (c) 0.0 (c) | |
| 1.7 | Limit of detection (a) | 1.7 to 4.6 μ g/l (each estimate has 5 degrees of freedom. | |
| 1.8 | Sensitivity (a) | $100 \mu g/l$ gives an absorbance of approximately 0.3. | |
| 1.9 | Bias (a)(b)(c) | Average bias $-1.6 \mu g/l$ on 196 analyses made in the range 10 to 100 $\mu g/l$. | |
| 1.10 | Interferences | See Section 3. | |
| 1.11 | Time required for analysis (a) | The total analytical time for a batch of 10 samples is approximately 4 hours of which the pretreatment stage occupies approximately 3 hours. | |
| | | | |

⁽a) These data were obtained from interlaboratory exercises in which either 4 or 8 laboratories participated⁽¹⁾.

⁽b) Deionized water spiked with the stated zinc concentration.

⁽c) Tap water spiked with the stated zinc concentration. The unspiked tap water contained about 10 μ g/l zinc.

2 Principle

- 2.1 Zinc is determined by atomic absorption spectrophotometry after concentration of the sample by evaporation in the presence of nitric acid. The pretreatment by evaporation with nitric acid ensures that any zinc present in suspended or colloidal forms is converted into a soluble form.
- 2.2 With some atomic absorption spectrophotometers it may be possible to determine zinc in the range quoted in Section 1.4 directly without the need for concentration by evaporation. Direct atomic absorption can only be applied to samples for which the acid into which the sample is collected is sufficient to convert all forms of zinc to a soluble form. If direct atomic absorption is used the performance characteristics quoted in Section 1 no longer apply.
- 2.3 Some laboratories may wish to determine the filtrable zinc content of samples. The method described (for "total" zinc) may be suitable as the basis for a method for the determination of filtrable zinc if it is combined with a filtration step, for example, using a membrane filter of suitable pore size. If such a procedure were used the performance characteristics quoted in Section 1 would no longer apply. Filtration of samples may result in contamination of the filtrable fraction or of loss of zinc by absorption processes. A recent publication⁽²⁾ gives details of the problems which have been reported and guidance concerning testing of the filtration apparatus.

3 Interferences

The effect of other substances on the determination of zinc by the method described is shown in Table 1. The data were obtained by one of the participating laboratories⁽³⁾

Table 1

| Other substance | Concentration of other substance (mg/l) | Other substance added as | Effect in μ g/l Zn of other substance at a concentration of (d) 10.0 μ g/l 50.0 μ g/l | |
|----------------------------------|---|--------------------------|---|------|
| Sulphate (as SO_4^{2-}) | 200 | acid | +0.3 | +0.4 |
| Chloride (as Cl ⁻) | 200 | acid | +0.7 | -0.4 |
| Phosphate (as PO_4^{3-}) | 20 | acid | 0.8 | -1.2 |
| Calcium (as Ca ²⁺) | 200 | nitrate | -0.4 | -0.8 |
| Sodium (as Na ⁺) | 100 . | nitrate | 0.0 | -0.7 |
| Magnesium (as Mg ²⁺) | 20 | nitrate | +0.7 | +0.8 |
| Potassium (as K ⁺) | 10 | nitrate | -1.0 | -1.4 |
| Iron III (as Fe ³⁺) | 1 | nitrate | -1.0 | -1.9 |

⁽d) If the other substances did not interfere, the effect would be expected to lie (95% confidence) within the ranges 0.0 ± 1.9 and $0.0 \pm 1.8 \mu g/l$ at 10.0 and $50.0 \mu g/l$ Zn respectively.

4 Hazards

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

Reagents

All reagents and standard solutions may be kept in glass or polyethylene bottles (see Section 6.2). Analytical reagent grade chemicals are suitable unless otherwise stated.

5.1 Water

The water used for blank determinations and for preparing reagent and standard solutions should have a zinc content that is negligible, compared with the smallest concentrations to be determined in the samples (see Section 12.2). Deionized 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 (d₂₀1.18) with water to 1 litre in a stoppered measuring cylinder and mix well.

5.3 Nitric Acid $(d_{20}1.42)$.

5.3.1. 5M Nitric Acid approximately

Dilute 320 \pm 5 ml of nitric acid (d₂₀ 1.42) with water to 1 litre in a stoppered measuring cylinder and mix well.

5.3.2 10% V/V Nitric Acid

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

5.4 Standard zinc solutions

5.4.1 Solution A 1 ml contains 1 mg Zn.

Weigh 1.000 ± 0.005 g of electrolytic zinc metal (greater than 99.9% purity), and dissolve it in a mixture of 65 ± 2 ml of nitric acid (d_{20} 1.42) and approximately 65 ml of water, carrying out the operation in a fume cupboard. Quantitatively transfer the solution to a 1-litre calibrated flask, dilute with water to the mark and mix well. The solution is stable for at least several months.

5.4.2 Solution B 1 ml contains 4 μ g Zn.

Dilute 4.00 ml of solution A with water to 1 litre in a calibrated flask and mix well. 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 zinc hollow cathode lamp. Background correction must be used, unless experience has shown that it is not essential. If the instrument is not equipped with facilities to make this correction automatically, a separate measurement for background must be made using a suitable continuum source⁽⁴⁾.

6.2 Cleanliness

Cleanliness is essential for this determination. If possible, apparatus should be reserved solely for zinc determinations: all residual zinc from previous zinc determinations must be removed. Clean all new glass and polyethylene ware, by filling with, or soaking in, 10% V/V nitric acid for several hours. 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 6.2, add 2.00 \pm 0.05 ml of 50% V/V of hydrochloric acid per litre of sample to be collected, and then collect the sample. Alternatively 1.00 \pm 0.05 ml of nitric acid d₂₀ (1.42) per litre of sample may be used. The acidification minimizes the adsorption of zinc onto the walls of the bottle.

8 Sample Pretreatment

The method described specifies concentration of the sample by evaporation in the presence of nitric acid. This procedure will convert any zinc present in suspended or colloidal material into a soluble form.

9 Analytical Procedure

Atomic absorption stage

9.7

Set up the instrument, according to the maufactur-

er's instructions for the determination of zinc, using an air/acetylene flame with background correction if necessary. The wavelength required is 213.9 nm.

READ SECTION 4 ON HAZARDS BEFORE STARTING THIS PROCEDURE

| Step | Experimental Procedure | Notes |
|------|--|---|
| | Analysis of samples | |
| 9.1 | Pretreatment stage (Carry out this stage in a fume cupboard) Add 200 ± 1 ml of the sample to a 400 -ml borosilicate tall form 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 (note a), until the solution volume is reduced to between 5 to 10 ml (note b), and allow to cool. | (a) The hot plate surface temperature should not exceed 160°C. This may be measured by a thermometer standing in a drilled metal block placed on the surface of the hotplate. |
| 9.2 | Cautiously wash down the watch glass and the inside of the beaker with about 10 ml of water and add 1.0 \pm 0.1 ml of nitric acid (d ₂₀ 1.42). Heat gently until the volume of the solution is again reduced to between 5 to 10 ml (note b), and allow to cool. | (b) The 5 to 10 ml volume may be judged by premarking the beaker. It is important to ensure that the liquid does not evaporate to dryness. |
| 9.3 | Transfer the contents of the beaker to a 20-ml calibrated flask. Wash the inside of the beaker with small volumes of water. Add the washings to the contents of the calibrated flask, dilute with water to the mark, stopper and mix thoroughly. Complete the atomic absorption stage during the same working day. | |
| 9.4 | Blank determination A blank must be run with each batch (eg up to 10 samples) of determinations using the same reagents as for the samples. To a 400-ml borosilicate tall form glass beaker, add 200 ± 1 ml of acidified deionized water (note c). | (c) The acidified water is prepared by adding either 2.00 ± 0.05 ml of 50% V/V hydrochloric acid or 1.00 ± 0.05 ml of nitric acid (d ₂₀ 1.42) to 1 litre of water; use the same acid as was used in Section 7. |
| 9.5 | Carry out steps 9.1 to 9.3 inclusive. | |
| 9.6 | Calibration standards Duplicate calibration standards must be run with each batch (eg up to 10 samples) of determinations (see Section 12.4). Pipette into a 200-ml calibrated flask, 5.0 ml of standard zinc solution B, dilute with acidified water (note c) to the mark and mix thoroughly. Transfer this solution to a 400-ml borosilicate tall form glass beaker, and carry out steps 9.1 to 9.3 inclusive. | 1 |

- 9.8 Aspirate acidified water (note d) until equilibrium conditions are established. Aspirate one of the calibration standards and adjust the instrument to give a suitable response.
- (d) To 100 ± 1 ml of deionized water, add 1.00 ± 0.05 ml of 5M nitric acid.
- 9.9 Aspirate acidified water and readjust the zero.
- 9.10 Aspirate the calibration standards with an aspiration of acidified water between each. Let the instrument responses of the calibration standards be C_1 and C_2 .
- 9.11 Aspirate the blank followed by acidified water. Let the instrument response of the blank be B₁.
- 9.12 Aspirate the samples with an aspiration of acidified water between each. Record the instrument response of the sample. Correct for background (note e) and let the corrected response be S.
- 9.13 After each batch of 10 samples re-aspirate the blank and both calibration standards with an aspiration of acidified water in between each. Note the instrument responses of the blank (B_2) and the calibration standards (C_3) and (C_4) .
- 9.14 Zinc concentration = $\frac{\overline{S} \overline{B}}{\overline{C} \overline{B}} \times 100 \,\mu\text{g/l}$

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

and
$$\vec{C} = \frac{C_1 + C_2 + C_3 + C_4}{4}$$

This calculation assumes a linear calibration curve. This must be checked (see Section 10).

(e) Background correction should be carried out on all samples unless the analyst has shown it to be unnecessary for particular samples.

10 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. Pipette respectively to a series of 200-ml calibrated flasks 0.0, 1.0, 2.0, 3.0, 4.0 and 5.0 ml of standard zinc solution B, dilute with acidified water (see step 9.4 note c) to the mark, and mix thoroughly. These flasks contain respectively 0, 20, 40, 60, 80 and 100 $\mu g/l$ zinc. Carry out the procedure given in Section 9 treating these solutions as if they were samples. Plot the instrument response of each solution against $\mu g/l$ zinc. The calibration curve is normally linear; however, the linearity should be checked. If the calibration curve departs from linearity, the calibration standard in step 9.6 may not be appropriate, nor the range given in Section 1.4. In such a case, the calibration standard chosen for step 9.6 should be the highest concentration on the linear portion of the calibration curve, and the concentration range of the method should be amended accordingly.

11 Change of Concentration Range of Method

If the zinc concentration of the sample is likely to exceed $100 \mu g/l$, an appropriately smaller aliquot of the sample must be taken for analysis, diluted with acidified water (see step 9.4, note c) to 200 ml, and treated as described in the procedure from step 9.1 onwards.

The calculation of the result, step 9.14, must then be altered to

Zinc concentration =
$$\frac{S - \bar{B}}{\bar{C} - \bar{B}} \times \frac{200}{V} \times 100 \ \mu g/l$$

where V ml is the volume of sample taken.

Alternatively if the calibration curve is linear beyond 100 μ g/l for a particular spectrophotometer the range of the method may be extended up to the extent of linearity of the calibration curve.

12 Sources of Error

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

12.1 Contamination

It is desirable to carry out the analysis in a laboratory in which no appreciable amounts of zinc, or its compounds, are handled. Analysts should note that many cosmetics and some common laboratory equipment, for example rubber tubing, contain zinc. 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 zinc determinations solely for this purpose, and to carry out a preliminary series of blank determinations, to ensure low blank values before analysing any samples.

12.2 Zinc content of the water used for blank determinations

If the water used for the blank determinations contains zinc, the results will be falsely low. The importance of this error depends on the zinc concentration, in both the blank water and the samples. Ideally, the zinc content of the water used for each blank determination should be measured and an appropriate correction made. An upper limit for the zinc content of the water can be calculated by converting the instrument response 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 zinc content of the water:

- (a) To each of two 500-ml borosilicate glass beakers, add 200 ± 1 ml of water and 0.40 ± 0.05 ml of 50% V/V hydrochloric acid or 0.20 ± 0.02 ml of nitric acid (d₂₀ 1.42); use the same acid as was used in Section 7
- (b) To each of two 500-ml borosilicate glass beakers, add 400 ± 1 ml of water and 0.40 ± 0.05 ml of 50% V/V hydrochloric acid or 0.20 ± 0.02 ml of nitric acid (d₂₀ 1.42); use the same acid as was used in Section 7
- (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 ± 1 ml of water to each beaker from (b) and continue heating until the volumes are reduced to 200 ± 1 ml.
- (d) Analyse the contents of all four beakers as described in Section 9, and let the instrument responses be W¹₁ and W¹₂ for the two unheated beakers, and W¹¹₁ and W¹¹₂ for the two heated beakers.
- (e) The zinc content of the blank water is equivalent to an instrument response of

$$W = \frac{(W_1^{11} + W_2^{11}) - (W_1^1 + W_2^1)}{4}$$

(f) The concentration of zinc, $A_{\rm w}$ in the blank water is then given by

$$A_{\rm w} = \frac{W}{\overline{C} - \overline{B}} \times 100 \ \mu \text{g/l zinc}$$

(See step 9.14)

12.3 Interfering substances

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

12.4 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. Therefore, a calibration standard must be run for each batch of analyses, and steps 9.6 onwards give the necessary procedure. This procedure assumes a linear calibration curve and linearity must be checked (see Section 10).

13 Checking the Accuracy of Analytical Results

Once the method has 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, (5) and they should be used as appropriate. As a minimum, however, it is suggested that a standard solution of zinc of suitable concentration be analysed at the same time, and in exactly the same way as normal samples (see 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.

14 References

- (1) Department of the Environment, File WS/646/56, Paper SCA/4.4/1
- (2) Hunt D T E, Filtration of Water Samples for Trace Metal Determinations, Water Research Centre *Technical Report*, TR 104, 1979
- (3) Department of the Environment, File WS/646/56, Paper SCA/4.4/2
- (4) Standing Committee of Analysts, Atomic Absorption Spectrophotometry an Essay Review, HMSO, London, 1980
- (5) Wilson A L and Cheeseman R V, Water Research Centre *Technical Report* TR 66, 1978

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 booklet are requested to write to:

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Department of the Environment/National Water Council

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