ENVIRONMENTAL POLLUTANTS– SELECTED ANALYTICAL METHODS

SCOPE 6

INTERNATIONAL COUNCIL OF SCIENTIFIC UNIONS

Scientific Committee on Problems of the Environment (SCOPE)
Working Group on Methodology of Determination
of Toxic Substances in the Environment

ENVIRONMENTAL POLLUTANTS—SELECTED ANALYTICAL METHODS (SCOPE 6)

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BUTTERWORTHS
LONDON

THE BUTTERWORTH GROUP

ENGLAND
Butterworth & Co (Publishers) Ltd
88 Kingsway, London WC2B 6AB

AUSTRALIA
Butterworths Pty Ltd
Sydney: 586 Pacific Highway, NSW 2067
Melbourne: 343 Little Collins Street, 3000
Brisbane: 240 Queen Street, 4000

CANADA
Butterworth & Co (Canada) Ltd
Toronto: 2265 Midland Avenue, Scarborough M1P4S1

NEW ZEALAND
Butterworths of New Zealand Ltd
Wellington: 26-28 Waring Taylor Street, 1

SOUTH AFRICA
Butterworth & Co (South Africa) (Pty) Ltd
Durban: 152-154 Gale Street

First published 1975

ISBN 0 408 70633 3

Printed in England by Unwin Bros Ltd Woking

INTRODUCTION

This compilation of analytical methods was prepared by the Working Group set up by SCOPE on "The Methodology of Determination of Toxic Substances in the Environment".

SCOPE was formed as a committee of ICSU, following a recommendation of an ad hoc committee set up by the latter. In its report, the ad hoc committee listed substances of importance in environmental problems. Another listing was given by the SCOPE Commission on Monitoring in their report entitled "Global Environmental Monitoring". In selecting which pollutants to consider, the Working Group has given special consideration to these listings, and during its discussions decided on the addition of other substances. It is recognized by the Working Group that the list of substances to be considered is by no means complete, but it was felt advisable to publish these at this juncture, and to follow these with supplements as necessary.

The range of substances covered by the methods chosen should not itself be taken as indicative of a need for a monitoring program, nor should it be assumed that a substance for which a method has been included is necessarily a pollutant. The sensitivity or limit of detection of the analytical methods described are not necessarily indicative of the toxicity of the substance being measured.

It is well known that some elements play a beneficial role in living organisms, and this in some instances is a matter of essentiality. At higher levels, the same elements may exert toxic effects, and there is need in all cases to establish precise dose-effect relationships to evaluate these regions. In addition, the ratio of concentration of one element to another may be of importance. The work of the Ecotoxicology Commission of SCOPE will have a direct bearing on these matters.

It is apparent that many methods have been published and used in the past for these substances. The Group was aware of other compilations of methods which are of interest in the same field and of the work, in some cases still in progress, of other national and international bodies in this connection. A considerable amount of information is available on experience of the extent of validity and shortcomings of the various methods. There is in fact a growing need for a wider discussion of these activities on a common basis, especially against a background of an increasing understanding of the extent of occurrence of the various compounds in the environment and of their significance with respect to environmental quality. This is of particular importance in view of the increased sensitivity and specificity of analytical methods now available and of the prospect that even greater sensitivities may yet be achieved. The number of individual organic substances which theoretically could be present, for example, increases enormously as these aspects of analysis are improved.

The Working Group has devoted a great deal of consideration to the mass of information available. The choices of the methods presented in this compilation, based on the considered opinion of the Working Group, have necessarily been somewhat arbitrary. Unless designated as reference methods, the procedures described should be regarded as offering guidance in the choice of methods rather than as mandatory procedures. Other methods, or variations of the same method, may be available. Although specific advice relating to the choice has in some cases been given indicating why certain methods should not be used, this should not be regarded as excluding all other variations where they can be shown to give equivalent results or better results in individual circumstances. For example where a good neutron activation facility is available, this may be suitable for the estimation of trace metals.

The methods described assume the availability of laboratory facilities and qualified analytical chemists. Where possible, attempts have been made to avoid the use of very expensive equipment normally found only in the largest laboratories. Wherever feasible, general purpose (utility) methods have been described as alternatives. These methods have good reliability and precision, but accuracy and degree of sensitivity may be somewhat less than with a reference method. Where feasible, methods have been described for some substances in different substrates. When using the methods indicated, it is assumed that the analyst will establish the normal criteria of satisfaction with respect to control and recovery experiments.

Sampling is a most important consideration and this is thoroughly recognized by all analysts. The details and the precautionary measures to be taken have been widely discussed and described elsewhere. For air analysis, the methods may be based on high or low volume sampling techniques or on high or low flow rates, according to program requirements. This is discussed in more detail in the methods concerned. The methods based on the ring oven technique are relatively new in practice but have proved themselves over years of research. The technique has the advantage of being simple, cheap and rapid. It is well suited for field studies and for use in laboratories where sophisticated equipment and highly specialized personnel are unavailable. It appears likely that this technique will be adapted to a wider range of application in the future, including the analysis of various substances in water. The methods described for traces of lead and cadmium in water can also be simply adapted for use in the estimation of traces of other elements such as zinc, copper, nickel, manganese, cobalt and beryllium.

The format recommended by the International Organization for Standardization has been used for the presentation of methods.

FOREWORD

The Scientific Committee on Problems of the Environment (SCOPE) was established by ICSU in 1969 to (a) advance knowledge of the influence of man and his activities upon his environment, as well as the effects of these alterations upon man, his health and his welfare with particular attention to those influences and effects which are either global or shared in common by several nations; and (b) serve as a non-governmental, interdisciplinary and international council of scientists and as a non-governmental source of advice for the benefit of governments and intergovernmental agencies with respect to environmental problems.

Much of the work of SCOPE is concerned with the effects of small or trace quantities of a variety of chemical elements and compounds in various segments of the environment, in air, water, soil and biota. The determination of trace amounts of certain substances entail difficulties. It is obviously of paramount importance that results obtained in various parts of the world be strictly comparable, and the present volume presents a number of carefully chosen analytical methods for use by those seeking advice on the choice of method.

SCOPE expressed its gratitude to the Chairman and members of the working group on methods of analysis for selected pollutants for the extensive work which they have carried out. It is hoped that this task will be further extended in a succeeding volume dealing with other chemical substances including the revision of methods as required.

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Determination of Airborne Particulate Lead by Atomic Absorption Spectrophotometry

1. INTRODUCTION

Lead in air and biological materials may be measured by dithizone methods. These methods suffer from pH dependency and instability of developed colours. Polarography may be used, but unless the test sample is very carefully prepared to eliminate all traces of organic materials, the lead wave may be masked and lead thought to be absent. Methods using anodic stripping voltammetry show promise but more practical experience must be obtained. The sensitivity of methods using emission or X-ray spectrography is low and the accuracy is affected by matrix error. A limit of identification of 0.1μg of lead is reached by using the Weisz ring oven (12.1).

In the method to be described, airborne particulates are collected by drawing the air through a filter. The filter, or an areal aliquot, with collected particulates, is digested to prepare a test sample. Preparation of this sample is simple and rapid. The aqueous acidic sample is scrutinized by an atomic absorption spectrophotometer at a suitable wavelength and the lead is calculated by referring the absorbance found to the appropriate calibration curve. By the use of a graphite furnace, additional sensitivity may be achieved beyond that of the usual flame mode. The lead levels found are expressed in micrograms per cubic metre of air sampled. Some practical details of accuracy and precision obtainable will be found under the section "Expression of Results" (9).

2. FIELD OF APPLICATION

The method is applicable to the measurement of the concentrations of airborne particulate lead found in either ambient or industrial atmospheres. The method is not applicable to the measurement of lead compounds in vapor form, such as tetraethyl or tetramethyl lead. The method may be readily adapted to the measurement of tetraethyl lead in gasoline. As airborne particulate lead may make up 1 to 5% of airborne particulate matter, analytical sensitivity is ordinarily not a problem. Based upon the flame mode, washed glass filters and a nominal air sample volume of 2,000 cubic metres, lead is measurable to 0.010 micrograms per cubic metre. A slight improvement in sensitivity results from the use of cellulose filters, or down to 0.0075 micrograms per cubic metre. For the same test sample, the use of the flameless mode increases the sensitivity considerably.

3. PRINCIPLE

A prepared test sample, containing the inorganic constituents in aqueous acidic solution, is reduced in the flame or graphite furnace to the atomic state. The amount of the element of interest is measured by making use of its property of absorbing light of its characteristic frequency when in the atomic state.

4. REACTIONS

The most important reactions relate to the complete dissolution of the particulate sample in acidic media to form a homogeneous test sample. As microgram quantities of material may be involved, losses due to incomplete solution, volatility, or adsorption must be avoided.

5. REAGENTS

5.1 *Air, compressed*

In pressure cylinders or online.

5.2 *Acetylene, compressed*

In pressure cylinders.

5.3 *Glass filters*

Commercially available material, in size 203 by 254mm, is exhaustively washed prior to use.

5.4 *Cellulose filters*

Ashless, acid washed, analytical grade, in size 203 by 254mm.

5.5 *Water*

Distilled at least twice from glass or quartz.

5.6 *Hydrofluoric acid*

49%. Reagent grade, in polyethylene containers.

5.7 *Nitric acid*

71%. Reagent grade.

5.8 *Standard solution of lead*

Dissolve 1.598g of lead nitrate and make up to 1 litre with 1% nitric acid. 1ml of this stock solution contains 1,000μg of lead, Pb.

6. APPARATUS

6.1 *Spectrophotometer, atomic absorption*

With meter, recorder or digital readout and monochromator with wavelength dial reading to 0.1 nanometre.

6.2 *Pipettes, glass*

Millilitre capacities, "to deliver".

6.3 *Volumetric flasks*

Borosilicate with ground glass stopper, 25ml capacity.

6.4 *Bottles, polyethylene*

Screw cap, for storage of test samples, 30ml capacity.

6.5 *Beakers, teflon*

Griffin form, 100ml capacity.

6.6 *Beakers, borosilicate*

Graduated, Griffin form, with teflon coated rim, 150ml capacity.

6.7 *Hot plate*

Electric, with temperature control, thermostat and ceramic heating surface.

6.8 *Büchner funnel*

Polypropylene, custom made, with sintered false bottom and vacuum connection, 216 by 267 by 85mm in free depth.

6.9 *Graphite furnace*

One suitable instrument is available commercially to the Massmann design.

6.10 *Strip chart recorder*

To display and record the response from the furnace.

6.11 *Pipettes, automatic*

Eppendorf design, with capacities 10 to 100 microlitres.

7. SAMPLE AND SAMPLES

7.1 *Air Sampling*

7.2 *Preparation of the laboratory sample*

Cellulose filters (5.4) may be used without further treatment. Glass fibre filters (5.3) are purified by placing a group of 100 such filters in the special Büchner funnel and extracting repetitively with distilled water. Mount the filter in a conventional high volume or other sampler head. Draw air through the filter at a flow rate between 1.13 and 1.60 cubic metres per minute, for an appropriate period, such as 24 hours. The resistance to flow offered by the cellulose filters is much greater than for glass and an appreciably smaller total volume of air will be taken with cellulose. Calculate and record the total volume of air sampled, in cubic metres, as the product of mean flow rate and time.

7.3 *Preparation of test sample*

Cut areal aliquots from the exposed surface of a filter using a circular metal punch. The cutting edge of the punch is carefully wiped with lens tissue between each use to prevent carry over of contamination from one sample to another. Place one or more such discs in a teflon beaker. Initiate the dissolution of the filter matrix by the dropwise addition of 1ml of hydrofluoric acid (5.6). Gently warm the contents of the beaker, at low heat, until the hydrofluoric acid is almost completely evaporated. At this point, add 1 to 2ml of nitric acid (5.7) and continue to heat gently until a few drops of nitric acid are left. Add about 10ml of water, bring nearly to the boil and filter through a Whatman 41 filter into a glass beaker (6.6). Transfer to a 25ml volumetric flask (6.3). Rinse down the teflon beaker with another 10ml quantity of water, warm and filter into the same beaker. Transfer to the 25ml volumetric flask and make up test sample to mark at 25°C. Mix the contents of the volumetric flask thoroughly after adjustment to volume. Transfer contents of volumetric flask to polyethylene storage bottle (6.4). The test sample is now ready for analysis. The above method applies to the digestion of glass fibre filters. Cellulose filters can be digested or extracted using nitric acid.

8. PROCEDURE

8.1 *Safety precautions*

Follow normal precautions for the handling of compressed gases. Observe manufacturer's instructions on lighting and extinguishing flame. TEST GAS SUPPLY SYSTEM FOR LEAKS BEFORE USE AND EACH TIME A CYLINDER OF GAS IS REPLACED.

8.2 *Test Portions*

8.2.1 Test portions, flame

Introduce the test portion into the flame by *continuous* aspiration, through polyethylene tubing, of an *unmeasured* portion of the test sample. Aspirate distilled water into flame, between the introduction of each test portion, to prevent cross contamination.

8.2.2 Test portions, furnace

Measure and insert in the furnace, test portions of 10 to 100 microlitres of the test sample using an automatic pipette (6.11).

8.3 *Calibration Curves*

8.3.1 Calibration curves, flame

Using "to deliver" pipettes, prepare known concentrations of lead by dilution of standard solution 5.8 to cover the range of 1.0 to 40.0 micrograms of lead per ml. Install in the monochromator and align, if necessary, the hollow cathode source for lead. Set the wavelength of the monochromator at 283.3nm. Using an air-acetylene flame, aspirate an unmeasured portion of each dilute standard lead solution (8.2.1). At the same time, aspirate into the flame a blank of distilled water. From the instrumental responses obtained, prepare a calibration curve of absorbance against concentration of lead in micrograms per ml. Such a representative curve is given in Figure 1. Using the same dilute standard solutions of lead, and a distilled water blank, prepare a similar calibration curve with the wavelength of the monochromator set at 217.0nm. Representative curves, covering wavelengths 217.0 and 283.3nm, are given in Figure 2 (Page 11).

8.3.2 Calibration curves, furnace

By means of an automatic pipette with polyethylene tip, place identical microlitre portions of the dilute standard lead solutions and a distilled water blank in the furnace. Measure and record the response for each test portion following the predetermined measuring cycle. Cover the range 1 to 5 nano-

grams lead in steps of 1 nanogram (0.1 to 0.5 micrograms per ml for a 10 microlitre volume). Prepare a calibration curve of response, in arbitrary units, against concentration of lead in micrograms per ml. Such a representative curve is given in Figure 3 (Page 12).

8.4 *Determination*

8.4.1 Determination, flame

Set the wavelength of the monochromator at 283.3nm. Observing conditions of 8.2.1 and 8.3.1, aspirate an unmeasured portion of each test sample into the flame. Record the response for each unknown. Measure the lead in all the unknown test samples which can be accommodated to the calibration curve for 283.3nm. If greater sensitivity is required, this may be obtained by measuring at 217.0nm, at the cost of more electronic noise. If the lead content of a given test portion should be too low for both calibration curves, it will be necessary to use the flameless mode. If the lead content should prove to be too high for the 217.0nm curve it will then be necessary to dilute the test sample.

8.4.2 Determination, furnace

If insufficient sensitivity is available in the flame mode, measure into the furnace, by automatic pipette, a similar test portion of each unknown test sample. Follow the drying and heating programmes previously established. Record the response for each test portion in arbitrary units. If a test portion of 10 microlitres should provide insufficient response, repeat the measurement using larger test portions. Derive the amount of lead present, by referring to the appropriate calibration curve, Figure 3 (Page 12).

8.5 *Blank Tests*

8.5.1 Reagents

In parallel with preparation of calibration curves, prepare a reagent blank of the acids and distilled water used in the preparation of calibration curves and test samples. Change or purify reagents, as necessary, if blanks are unsatisfactory.

8.5.2 Reagents plus filters

In parallel with the preparation of calibration curves and test samples, prepare areal test portions from unexposed filter media as described in 7.3. Measure lead and express blanks in micrograms of lead per ml.