The Determination of Methane and Other Hydrocarbon Gases in Water 1988

Methods for the Examination of Waters and Associated Materials

This document contains 21 pages

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Methods for the Examination of Waters and Associated Materials

Method B is capable of adaptation to air analysis.

London: Her Majesty's Stationery Office

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About this Series

This booklet is part of a series intended to provide both recommended methods for the determination of water quality, and in addition, 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, have 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 These volumes inevitably took some years to prepare, so that they were often partially out of date before they appeared in print.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 series of booklets on single or related topics; 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 and notes 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 technical staff to decide which of these methods to use for the determination in hand. Whilst the 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 a committee of the Department of the Environment set up in 1972. Currently 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 Microbiological methods
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 Sewage Works Control Methods
9.0 Radiochemical methods

The actual methods and reviews 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.

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 and minor additions to published booklets not warranting a new booklet in this series will be issued periodically as the need arises. 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 that booklet.

L R PITTWELL
Secretary

1 July 1987

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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 for one's self, one's colleagues, those outside the laboratory or work place, or subsequently for maintenance or waste disposal workers. 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. Reagents of adequate purity must be used, along with properly maintained apparatus and equipment of correct specifications. 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.

Lone working, whether in the laboratory or field, should be discouraged.

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. Hazardous reagents and solutions should always be stored in plain sight and below face level. Attention should also be given to potential vapour and fire risks. 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.

There are numerous handbooks on first aid and laboratory safety. Among such publications are: 'Guide to Safe Practices in Chemical Laboratories' and 'Hazards in the Chemical Laboratory', issued by the Royal Society of Chemistry, London: 'Safety in Biological Laboratories' (Editors Hartree and Booth), Biochemical Society Special Publication No 5, The Biochemical Society, London, which includes biological hazards; and 'The Prevention of Laboratory Acquired Infection', Public Health Laboratory Service Monograph 6, HMSO, London.

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 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. If an ambulance is called or a hospital notified of an incoming patient give information on the type of injury, especially if poisoning is suspected, as the patient may be taken directly to a specialised hospital.

The Determination of Methane and Other Hydrocarbon Gases in Water

1 Introduction

1.1 Determinands

Methane and other hydrocarbon gases can occur in water due to geochemical sources, microbial activity or leakage from installations. In theory, aqueous concentrations of methane as low as 1 mg 1^{-1} can give rise to explosion risks in poorly ventilated situations[1]. This booklet presents gas chromatographic methods and performance data for methane but the methods will also detect and can be used to measure other gases eg ethane, ethylene, acetylene, propane, propylene, and the butanes. (For information on other hydrocarbon gases see part C).

1.2 Analytical Procedures

Two methods are presented in the booklet.

Method	Determinand	Range of Concentration	Basis
A	Methane	0–20 mg 1^{-1}	Direct aqueous injection GC
B	Methane	0–20 mg 1^{-1}	Vacuum degassing, GC

2 Sample Collection and Preservation

Where possible, samples should be taken directly into gas sampling bulbs, fitted with a septum port, or the special sampling device used in Method B, making sure the sample is well flushed through and that no gas bubbles are trapped. Alternatively, samples may be sealed in crimp top septum vials. Analysis should be performed as soon as possible. If significant degassing of samples occurs Method B is recommended.

3 Sample Stability

9 replicate standards of approximtaely 3 mg 1^{-1} methane were prepared in crimp top vials. Duplicate analyses were carried out on each of 3 vials without delay and the remainder were stored in a 20°C incubator. Sets of 3 vials were analysed as before after 48 and 96 hours.

Storage Time Hours	Methane Concentration mg 1^{-1}
0	2.84±0.19
48	2.35±0.36
96	2.49±0.12

A

The Determination of Methane and Other Hydrocarbon Gases in Water by Direct Aqueous Injection Gas Chromatography

A1 Performance Characteristics Of The Method

Note: The performance characteristics are both instrument and column dependent. The typical data given here is for the main method as written. Users should evaluate their own equipment.

A1.1	Substances determined	Methane and other gaseous hydrocarbons.
A1.2	Type of sample	Ground, surface and potable waters.
A1.3	Basis of method	Direct aqueous injection into a gas chromatograph fitted with a porous polymer packed column and a flame ionization detector.
A1.4	Range of application	0–8 mg l^{-1} is convenient, but the method can be extended up to 20 mg l^{-1}.
A1.5	Calibration curve	Linear to 20 mg l^{-1}.

A1.6 Standard deviation*

	Within Batch mg l^{-1}	Between Batch mg l^{-1}
4 mg l^{-1} standard calculated using aqueous standards	0.16(5)	0.19(4)
4 mg l^{-1} standard calculated using gas standards	0.18(5)	0.29(4)

Values in parentheses are the degree of freedom.

A1.7	Limit of detection* (5 degrees of freedom)	0.13 mg l^{-1} based on the standard deviation of a 0.2 mg l^{-1} standard and calculated using either aqueous or gas standards.
A1.8	Sensitivity	4 mg l^{-1} methane in water gives a recorder deflection of approximately 50%.
A1.9	Bias	A 4 mg l^{-1} standard showed a bias of <2% when calculated against aqueous or gas standards.
A1.10	Interferences	Any compound with the same gas chromatographic retention time as the compound of interest. None known.
A1.11	Time required for analysis	4 Hours for 10 samples using aqueous standards or 2 hours for 10 samples using gas standards.

*Information provided by North West Water.

A2 Principle

The response given by methane in water is compared to that of standards using gas chromatography with a flame ionization detector. Standards can be prepared by 2 methods, either direct injection of gas standards of known concentration or standards prepared from a saturated solution of methane at a known temperature.

A3 Interferences

Any substance capable of producing a detector response at a retention time indistinguishable from that of methane or any other gaseous hydrocarbons being determined will interfere. In practice the low molecular weight and volatility of these hydrocarbons render the analysis free from interfering compounds.

A4 Hazards

Methane is toxic and flammable and care must be taken when the standards are prepared to ensure that excess methane is well vented.

A5 Reagents

A5.1 Methane

BDH or BOC. Natural gas is 96% methane and may be used if the analytical requirement is not exacting.
0.1% v/v methane in nitrogen. Phase Separations Ltd., Altech Associates Ltd or other chromatographic equipment suppliers.

A5.2 Water

The water used for blank determinations and standard solutions should have a methane content which is negligible compared with the smallest concentration of methane to be determined in the samples.
Distilled, deionized or tap water will usually be suitable, if not methane can be removed by purging with helium or nitrogen for 30 minutes.

A5.3 Purge and Carrier Gas

Hydrocarbon free helium, nitrogen or argon. (Argon is rarely used for purging as some supplies contain methane.) See A5.2 and A6.3.

A6 Apparatus

A6.1 Septum vials

30 ml capacity sealed with a teflon faced septum and an open screw or crimp top.

A6.2 Pipettes

5 and 10 ml graduated.

A6.3 Gas chromatograph

A gas chromatograph with flame ionization detector which should be operated in accordance with the manufacturer's instructions. Various types of column have proved suitable for the analysis particularly a variety of porous polymer supports. A typical set of conditions is given below and the corresponding chromatogram is shown in Figure 1.

Column:	Glass 1.5 m × 3 mm id; 80–100 mesh Chromosorb 101
Detector temperature	250°C
Injector temperature	180°C
Column temperature	70°C
Carrier gas	Helium; 20 ml min^{-1} (alternatively oxygen-free nitrogen or hydrogen)
Injection volume	1 ul (aqueous phase)

Typical retention times (min.)		
	Methane	0.8
	Ethane	1.5
	Propane	3.8
	Butane	11.9

Alternatively a bonded phase non-polar WCOT column (1 μm or greater film thickness) can be used, operated at 40°C isothermally with split, splitless or on-column injector and a detector temperature of 200°C, and if a heated injector is used, an injector temperature of 200°C. Helium or hydrogen should be used as the carrier gas.

A7 Analytical Procedure

CAUTION
BEFORE PROCEEDING WITH ANALYSES READ SECTION A4 HAZARDS AND SECTION A8 CONTAMINATION

Step	Procedure	Notes
A7.1	Preparation of Aqueous Standards	
A7.1.1	Gently bubble methane through approximately 1 litre of distilled water for 1 hour. Record the temperature of the water which should be at ambient.	(a) Methane is flammable and toxic. Efficient fume ventilation must be provided. (b) Natural gas which contains 96% methane may be used for less exacting requirements.
A7.1.2	From the graph (Fig 2) read off the value for the concentration in the saturated solution at the appropriate temperature.	See reference 2
A7.1.3	Measure accurately the volume of vials used for the tests. (note c). Using graduated pipettes, add to a series of vials sufficient of the saturated solution from A7.1.1 to produce standards in the appropriate range. Suitable standards are 0.5, 1.0, 2.5, 5.0, and 8 mg 1^{-1}. (note d) Add water to the vial until it is completely full, slide on the seal excluding all air bubbles and firmly tighten the screw cap or crimp top. (note e).	(c) 30 ml or 60 ml vials are convenient. Calibrate the vials by weighing, empty and filled with water, to ±0.1g. (d) the saturated solution should be added with a minimum of disturbance. A convenient procedure is to add a little water to the vial, then add the saturated solution, swirl and top up. (e) The meniscus should just rise above the top of the vial to ensure that losses are less than 0.1 ml.
A7.1.4	Leave the standards at ambient temperature for 30 minutes to ensure that uniformity is achieved by diffusion.	
A7.1.5	Use of Gas Standards Gas standards (0.1% v/v methane in nitrogen) in pressurised aerosol containers fitted with a syringe adapter can be used.	
A7.2	Preparation of Samples The temperature of samples and standards should be the same, a constant temperature bath can be used if necessary.	
A7.3	Gas Chromatography	
A7.3.1	Condition the column by heating to 150°C for 10–15 minutes before allowing to equilibrate at the operating temperature. (note f)	(f) To improve baseline stability for the analysis.
A7.3.2	Inject 1 μl of distilled water as a blank. Repeat 2 or 3 times.	

A7.3.3   Inject 1 μl of each aqueous standard and record the peak heights obtained.
Alternatively inject 5 and 10 μl aliquots of a gas standard. Plot a calibration graph of peak height versus concentration. (notes g and h).

(g)   The volumes injected need to be relatively large to minimize dead volume effects of the needle.

A7.3.4   Inject the sample in the same way and compare the peak heights with those of the standards by directly reading off the concentrations of methane in the samples from the calibration graph. (note i)

(h)   If gas standards are used, measure the volume of the sample syringe needle before and after an injection in order to estimate the absolute amount injected. (Fill the syringe with water and adjust plunger to 1 μl mark. Draw plunger back and note the volume of water held in the barrel. Inject the water into the GC in the normal way and withdraw the syringe. Draw the plunger back again and note the volume of water remaining. The difference in values represents the actual volume injected.) A consistent and reproducible injection technique is required.

(i)   Repeated injections of water may cause baseline upsets with some columns, if so re-condition the column as in A7.3.1.

A8   Contamination

Contamination is not normally a problem but occasionally atmospheric methane concentrations can be sufficient to cause blank problems, for example at sewage works.

B

The Determination of Methane and Other Hydrocarbon Gases in Water by Vacuum Degassing and Gas Chromatography

B1 Performance Characteristics of the Method

Note: The performance characteristics are both instrument and column dependent. The typical data here given is for the method as written. Users should evaluate their own equipment.

B1.1	Substances determined	Methane and other gaseous hydrocarbons.
B1.2	Type of sample	Ground, surface and potable waters.
B1.3	Basis of method	Samples taken in a purpose made apparatus are degassed using a vacuum technique and analysed using a gas chromatograph fitted with a gas sampling valve, a packed column and a flame ionization detector.
B1.4	Range of application	0–20 mg 1^{-1}
B1.5	Calibration curve	Linear to 20 mg 1^{-1}
B1.6	Relative Standard deviation* (within batch) (n = 5)	1.43% for 2 ppm methane 0.56% for 20 ppm methane
	Total rsd (5 replicate batches)	7.95% for 20 mg 1^{-1} aqueous solution of methane
B1.7	Limit of detection*	0.4 ppm methane in headspace (equivalent to 0.057 μg 1^{-1} aqueous methane).
B1.8	Sensitivity	Injection of 0.5 ml of 10 ppm methane in headspace (equivalent to 1.4 mg 1^{-1} aqueous methane) gives a recorder deflection of approximately 50%.
B1.9	Bias	A recovery of 85% was obtained from de-ionized water 'spiked' with 4 mg 1^{-1} methane.
B1.10	Interferences	Any compound with the same gas chromatographic retention time as the compound of interest. None known. See B3.
B1.11	Time required for analysis	10 samples can be analysed in 5 hours.

*Information provided by Thames Water Authority

B2 Principle

Samples are vacuum degassed and the extracted gases diluted with nitrogen and compared to standards using gas chromatography with a flame ionization detector and gas sampling valve. Standards can be prepared by dilution in a gas pipette using nitrogen as dilutent, or commercially available standards in aerosol containers can be used.

B3 Interferences

Any substance capable of producing a detector response at a retention time indistinguishable from methane or other gaseous hydrocarbons will interfere. In practice the low molecular weight and volatility of these hydrocarbons render the analysis free from interfering compounds.

B4 Hazards

Methane is toxic and flammable and care must be taken when the standards are prepared to ensure that excess methane is well vented. The method requires the evacuation of glassware. Glassware must be checked for fractures each time prior to evacuation, safety glasses should be worn and safety screens and any other method of reducing the danger from flying glass should be used as appropriate.

B5 Reagents

B5.1 Methane

Methane in nitrogen (10, 100 and 1000 ppm v/v). Phase Separations Ltd., Altech Associates Ltd. or other chromatography equipment suppliers.

B5.2 Water

The water used for blank determinations and standard solutions should have a methane content which is negligible compared with the smallest concentration of methane to be determined in the samples.

Distilled, deionized or tap water will usually be suitable, if not, methane can be removed by purging with helium or nitrogen for 30 minutes.

B6 Apparatus

B6.1 Sampling and Degassing Apparatus

The apparatus is shown in fig. 3 and is an adaptation of a design by Darling[3].
Gas sampling bulbs fitted with septum port (various volumes from 100 ml to 1000 ml.) and gas syringes (both obtainable from chromatography equipment suppliers).

B6.2 Gas chromatograph

A gas chromatograph with flame ionization detector which should be operated in accordance with the manufacturer's instructions. Various types of column have proved suitable for the analysis particularly a variety of porous polymer supports, carbon adsorbents or molecular sieves.
A typical set of conditions is given below.

Column:	Glass 3 m × 2 mm id; 80–100 mesh Spherocarb
Detector temperature	250°C
Injector temperature	180°C
Column temperature	170°C
Carrier gas	Nitrogen; 25 ml min^{-1}
Injection volume	0.5 ml via gas sampling valve.

B6.3 Vacuum System

A service vacuum system or pump providing a vacuum of 1 mbar or better.

CAUTION
BEFORE PROCEEDING WITH ANALYSES READ SECTION B4 HAZARDS AND SECTION B8
CONTAMINATION

Step	Procedure	Notes
B7.1	Preparation of Standards	
	Purchased standards can be used directly. Intermediate standards can be prepared by dilution as follows. Fill a 100 ml gas sampling bulb with an appropriate standard and using a gas syringe dilute into calibrated gas sampling bulbs. Suitable gas standards are 1.0, 2.5, 5.0, 7.5 and 10.0 ppm.	(a) Atmospheric methane should be removed from the gas sampling bulbs by flushing with nitrogen.
B7.2	Degassing of Samples	
	Hold the apparatus with bulb A uppermost and open taps 1 and 2 fully. Using tap 3 drain water from the apparatus slowly until bulb A is empty and close taps 2 and 3. Evacuate bulb A using a vacuum line closing tap 1 on completion.	(b) For higher concentrations the device can be used with bulb B uppermost.
	Open tap 2 fully and immerse the whole of bulb B in an ultrasonic bath for 1 hour, remove from the bath and close tap 2.	
	Restore the pressure in bulb A by connecting to a low pressure supply of nitrogen and opening tap 1. Use a T-piece vent in the gas line to ensure the pressure in bulb A does not exceed atmospheric pressure.	
B7.3	Gas Chromatography	
B7.3.1	Flush the gas sampling valve loop with nitrogen and inject for a blank determination.	
B7.3.2	Displace each standard into the sample valve loop and inject into the gas chromatograph. Record the peak heights obtained and plot a calibration graph of peak height versus concentration.	
B7.3.3	Connect tap 1 of the apparatus to the gas sampling valve on the gas chromatograph by means of a short length of silicone rubber tubing and displace gas from bulb A into the loop by introducing water from a reservoir connected to bulb B by a short length of tubing and opening taps 2 and 3.	(c) It is usually possible to obtain 3 replicate injections in this way.
	Inject the sample and compare the peak height with those of the standards by directly reading off the concentration of methane in the gas from the calibration graph.	

B7.3.4   Calculation of Results

The concentration of methane in ppm in the
headspace is related to the original aqueous
concentration (μg 1^{-1}) at NTP by the following:

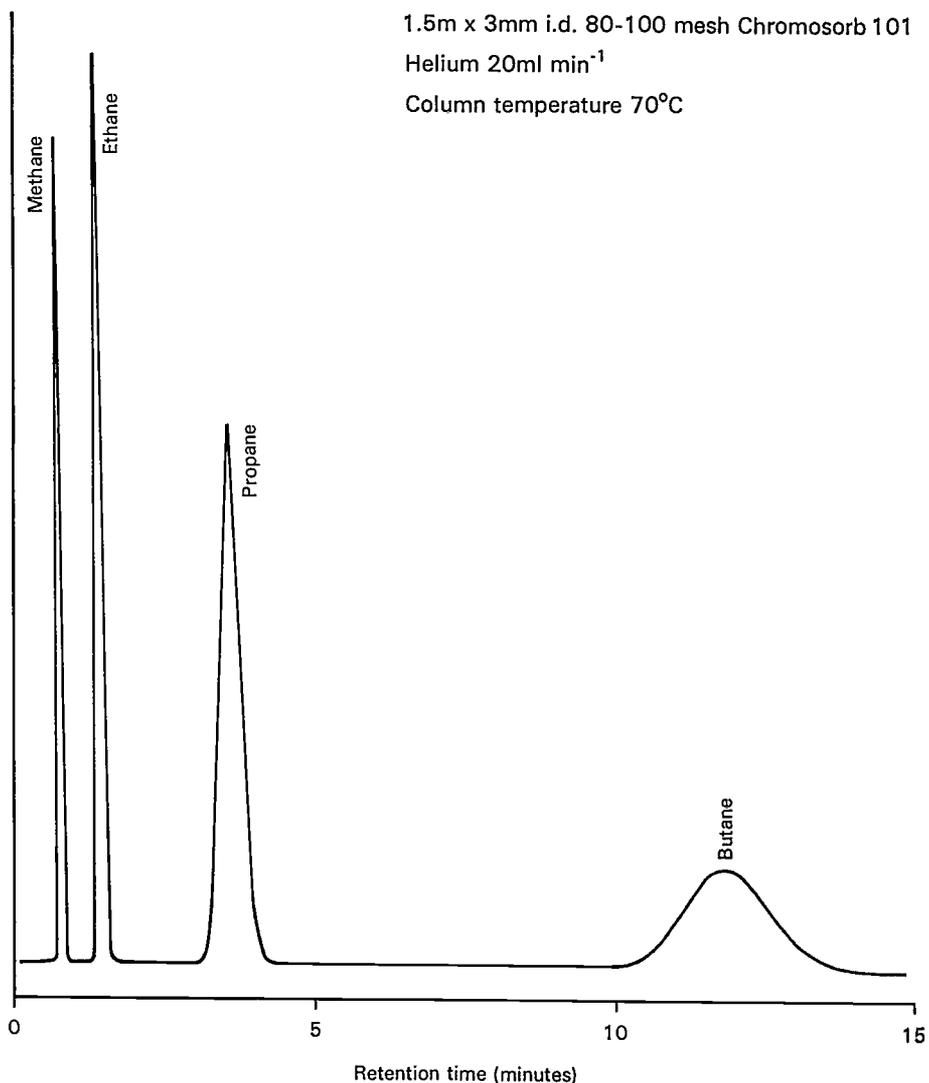$$\mu g\ 1^{-1} = H_{ppm} \cdot \frac{Vh}{1000} \cdot \frac{16}{22.41} \cdot \frac{1000}{V_1} \cdot \frac{P}{760} \cdot \frac{273}{T}$$

where H_{ppm} = ppm methane observed in headspace
V_h = volume of headspace (bulb A) ml
V_1 = volume of liquid degassed (bulb B) ml
16   = molecular weight of methane
P   = atmospheric pressure mm Hg
T   = temperature degrees Kelvin

B8   Contamination   Atmospheric levels of methane can be significant (and can be determined by injecting
a loop full of air) and it is important the dimensions of connecting tubes are kept to
a minimum.

Figure 1 Typical chromatogram

1.5m x 3mm i.d. 80-100 mesh Chromosorb 101

Helium 20ml min^{-1}

Column temperature 70°C

Retention time (minutes)

# C.   Extension to Other Hydrocarbon Gases

Both methods are capable, if calibrated by standard samples of the various gases, of detecting and determining hydrocarbon gases up to the pentanes. These may be prepared as in sections A7.1 or B7.1. Note that acetylenic compounds must not come into contact with copper or silver. See also ref. 4.

Figure 2 Methane solubility

(Henry's Law)
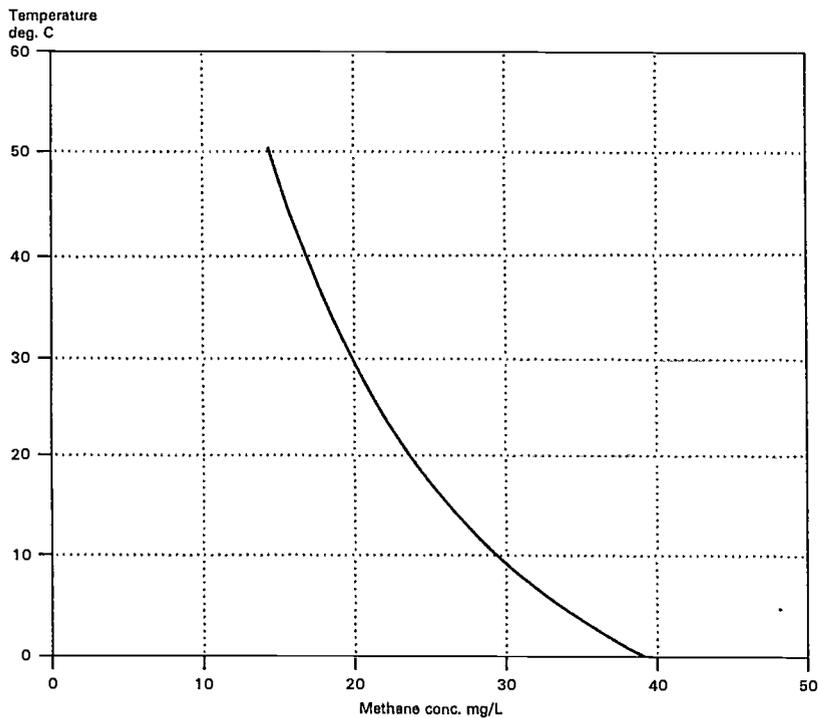
Data taken from reference 2

Figure 3 Combined sampling and degassing apparatus
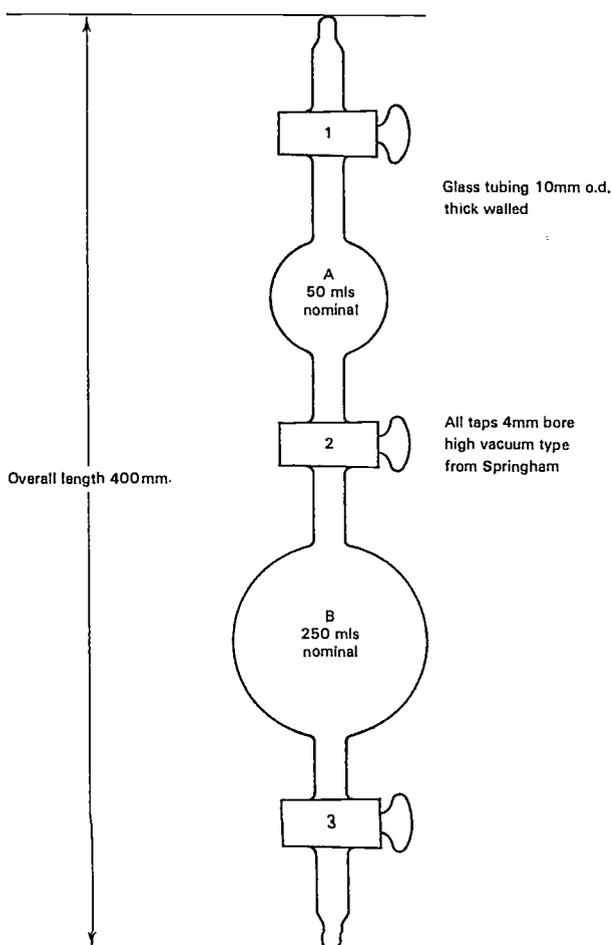
Analytical Quality Control

As can be seen from the data given in the Sample Stability Section, provision of a control sample is difficult. Furthermore, methane diffuses about 25% more rapidly than air through quite fine apertures, and great care would be needed for long leak proof storage of such samples. The solubility of these gases in water is dependent on temperature, total dissolved solids and partial pressure in the ullage above the liquid. It is suggested that analytical quality control charts should be run; but that samples be prepared afresh at least once a week and that allowances be made for slight variations in the standards. (See also ref. 4). Note also that some hydrocarbon gases such as propylene form hydrates.

Address for Correspondence

However thoroughly a method may be tested there is always the possibility of a user encountering a hitherto unreported problem.

Correspondence about these methods should be addressed to:—

    The Secretary
    The Standing Committee of Analysts
    Department of the Environment
    Romney House
    43 Marsham Street
    LONDON
    SW1P 3PY

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2. Mosey, F The solubility of anaerobic digester gases in water calculated from Henry's Law. *Laboratory Record 12S*, WRC Stevenage, November 1980.

3. Darling, W G British Geological Survey Hydrogeology Group. *Report ref. WD/St/85/3* July 1985.

4. British Standard *BS 3156.11.1.1* (1986).

Department of the Environment

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These methods were developed at the request of the Health & Safety Executive. Two panels shared the work—Volatile Substances, and Oils, Fats, Waxes and Tars.	
Main Committee Member	1
Working Group Member	2
Panel Member	3