

Material Extractable by Light Petroleum from Sewage Sludge (Tentative Method) 1985

Methods for the Examination of Waters and Associated Materials

Material Extractable by Light Petroleum (Boiling Range 40°C–60°C) from Sewage Sludge Tentative Method 1985

Methods for the Examination of Waters and Associated Materials

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First published 1987

ISBN 0 11 751942 1

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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 These volumes inevitably took some years to prepare, so that they were often partially out of date before they appeared in print.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 users 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 seven 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
- 3.0 Empirical and physical methods
- 4.0 Metals and metalloids
- 5.0 General nonmetallic substances
- 6.0 Organic impurities
- 7.0 Biological 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 1986

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 waste, 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 given information on the type of injury, especially if poisoning is suspected, as the patient may be taken directly to a specialized hospital.

Introduction

This method, like the majority of methods for the quantification of hydrocarbons and greases, is empirical. The materials determined are decided by the procedure used.

Because of the wide range of sources and properties of materials defined as oil and grease, no one analytical method can be expected to *determine all the substances* so described. The analytical method in this document is for material extractable by light petroleum (40°C to 60°C), under defined conditions. The substances determined will include fats, all types of hydrocarbons and other materials that are retained in the extract under the conditions used for the evaporation of the solvent.

The method is intended for use for the operational determination of extractable material in sewage sludge wherein the performance targets of the analytical data are less exacting. It is complementary to another method in this series for the determination of material extractable by carbon tetrachloride and of certain hydrocarbon oil and grease components (1).

It is not intended to issue a method for Material Extractable by Chloroform either from Water or Sludge, as due to the solubilities of water in chloroform and of chloroform in water, tests have shown that the effective limit of detection for such a method would only be 1.3 mg/l (Anglian WA data with 42 degrees of freedom, other laboratories have similar findings).

The method forms part of a continuing series, which when complete can be used as part of a general co-ordinated scheme of analysis.

1 Performance Characteristics of the Method

1.1	Substance determined	Those substances extracted from sewage sludge by light petroleum (40°–60°C) and remaining after evaporation of the solvent at 105°C. If the presence of fatty acid soaps is suspected, prior treatment with hydrochloric acid may liberate the free acid which may also be determined. However sludges are known where such acidification results in a slight reduction in the percentage of extractable material.
1.2	Type of sample	Sewage sludge
1.3	Basis of method	The sludge, acidified if required, is extracted with light petroleum (40°–60°C). The solvent is evaporated at 105°C and the residue weighed.
1.4	Range of Application	Up to 1 gram of previously dried sludge may be extracted.

1.5 Standard Deviation*
(as % extractable material)

Sample	Without Acid Treatment				With Acid Treatment			
	Mean Concentration Extracted %	S _w	S _T	Degrees of Freedom	Mean Concen. Extracted %	S _w	S _T	Degrees of Freedom
Blank	0.1	0.88						
Dry								
Sewage 1	14.88	0.43	0.52	7.5	13.33	0.42	0.97	4.8
2	6.17	0.19	0.45	4.8	6.04	0.12	1.10	4.0
3	32.06	2.42	4.23	5.5	19.72	0.66	1.81	4.6
4	31.28	0.296		5				
5	30.02	0.350		5	29.96	0.191		5
6	29.06	0.386		5	30.26	0.365		5
Wet								
Sewage 7	20.24	0.543		5	23.04	0.621		5
8	19.10	0.538		5	22.73	0.125		5
9					21.39	0.452		5

1.6 Limit of detection*
(as% extractable material) 0.46 (from blank)

1.7 Sources of error See section 3.

1.8 Time required for analysis Assuming a minimum of a 4 hour extraction period, total time is 6 hours. Operator time for one determination is 2 man hours. With multiple apparatus, one operator can perform 12 analyses per day.

(*) Results obtained by M.R. Wright, G. Hindle, R.C. Leech and D.A. Partridge of Cambridgeshire College of Arts and Technology.

2 Principle

Dried sewage sludge is continuously extracted for at least 4 hours using light petroleum (40° to 60°C). The solvent is then removed from the extract, and the extract dried at 105°C for 15 minutes.

3 Sources of Error

Provided the procedure is followed correctly, there are no serious sources of error, within the definition of the materials determined, for the quantification of oils and grease.

4 Hazards

The procedure uses concentrated hydrochloric acid, which is corrosive, and light petroleum 40° to 60°C, which is narcotic and highly flammable. Avoid inhalation, contact with skin and eyes, and ingestion.

5 Reagents

Analytical reagent grade chemicals must be used.

5.1 Hydrochloric acid d₂₀1.18

5.2 Light petroleum spirit (boiling range 40° to 60°C)

6 Apparatus

Greased joints and seals must not be used.

6.1 Soxhlet extraction apparatus

(of an appropriate size to fit the flat bottomed flask to take the thimble and solvent volume) and thimbles e.g. 30 mm × 100 mm.

6.2 Water bath

(boiling).

6.3 Flat bottomed flasks

(150 ml or smaller as required) and reflux condensers.

6.4 Drying oven

at $105 \pm 2^\circ\text{C}$.

6.5 Pestle and mortar

6.6 Airline

supplying filtered air.

7 Sample Collection and Preservation

The sample of sludge should be as representative as possible. Reference should be made to the general booklet on sampling and other booklets published in this series (2)(3). Light petroleum ether-extractable materials may degrade on storage giving lower results, but no quantitative information is available; samples should be analysed as quickly as possible.

8 Analytical Procedure

READ SECTION 4 ON HAZARDS BEFORE STARTING THIS PROCEDURE

Step	Procedure	Notes
Preparation:		
8.1	Place approximately 100 ml of sludge in a dish (note a), and evaporate the contents to near dryness on a boiling water bath (notes b and c). The dish is then transferred to an oven, set at $105^\circ\text{C} \pm 2^\circ\text{C}$, for at least an hour. Grind the dried material to a powder using a pestle and mortar (note d). Dry a 150 ml flat-bottomed flask to constant weight at 105°C . (W_{1g}). (note e).	(a) The volume of sample may be varied if particularly high or low grease contents are expected. (b) If measurement is to include soaps etc. the sample is acidified with 2.5 ml hydrochloric acid, $d_{20} 1.18$; it will be noted that the acidified samples tend to yield less extract than unacidified samples (see section on Performance Characteristics)

Step	Procedure	Notes
Extraction		
8.2	<p>Weigh accurately approximately 1g (W_2g) of the dried material into a grease free extraction thimble and place into the Soxhlet extraction apparatus. Add light petroleum (boiling range 40°C to 60°C), to the flask, ensuring that there is sufficient solvent for recycling to occur (note f). Connect the flask to the Soxhlet extraction apparatus and reflux extract for a minimum of 4 hours or until extraction is complete, using a water bath.</p> <p>(note g)</p> <p>Remove the Soxhlet extraction apparatus containing excess solvent and then evaporate off the remainder of the solvent from the flask using the water bath, completing with a stream of filtered air preferably in a fume cupboard.</p>	<p>(c) If a forced ventilation oven is available, this step may be carried out in the oven.</p> <p>(d) Some difficulty may be experienced grinding sludges with a high fibrous or oil and grease content. This may be improved if acidification is used.</p> <p>(e) Smaller flasks have been used satisfactorily when the oil and grease content is very low so that the final weighing of flask and residue gives a more sensitive measure of the weight of the residue.</p>
Drying:		
8.3	Dry the light petroleum (40° to 60°C), extract for 15 minutes in the flask at $105 \pm 2^\circ\text{C}$. Allow to cool and weigh (W_3 g). (note h).	(f) 75 ml have been found to be satisfactory.
Calculations		
8.4	<p>Percentage of grease in dried sample</p> $= \frac{(W_3 - W_1) \times 100}{W_2}$ <p>Percentage of grease in sample</p> $= \frac{(W_3 - W_1)}{W_2} \times \frac{X}{100}$ <p>where X is the concentration of total solids in the sludge, expressed as percent (note i).</p>	<p>(g) The extraction is complete when the solvent in the Soxhlet extraction apparatus is colourless.</p> <p>(h) Prolonged drying may result in loss of grease by volatilisation.</p>

Step	Procedure	Notes
Blank:		
8.5	Repeat steps 8.2 and 8.3 without dried sludge i.e. with solvent alone. The weight of residue is $W_5 - W_4$ where W_4 is the weight of the flask and W_5 is the weight of flask plus residue. This may then be used to calculate the blank corrected percentage of grease in dried sample (note i).	(i) This method is used for the operational measurement of extractable material in sewage sludge and because the weight of solvent residue is very small compared to the extract, any inaccuracies caused by the omission of the blank correction is usually acceptable. The performance characteristics in Section 1 are blank corrected.

9 Checking the Validity of Analytical Results

Once the method has been put into normal routine operation many factors may subsequently adversely affect the accuracy of the analytical result. It is recommended that experimental tests to check certain sources of inaccuracy should be made regularly. Many types of tests are possible, and they should be used as appropriate. As a minimum however, it is suggested that at least one sample of suitable concentration, in each batch of analyses, be analysed in duplicate. The results obtained should then be plotted on a quality control chart which will facilitate detection of inadequate precision and allow the standard deviation of routine analytical results to be estimated.

10 References

10.1 *The determination of Material Extractable by Carbon Tetrachloride and of certain Hydrocarbon Oil and Grease Components in Sewage Sludges 1978.* Methods for the Examination of Waters and Associated Materials, HMSO, London.

10.2 *The Sampling and Initial Preparation of Sewage and Waterworks Sludges, Solids, Sediments, Plants and Contaminated Wild Life 1986.* Methods for the Examination of Waters and Associated Materials, HMSO, London.

10.3 *Sampling of Oils, Fats Waxes in Aqueous and Solid Systems 1983.* Methods for the Examination of Waters and Associated Materials. HMSO, London.

Address for Correspondence

However thoroughly a method may be tested, there is always the possibility of a user discovering a hitherto unknown problem. At the present time, though based on work in several laboratories, thorough test data is not available, hence the tentative status of the method. Additional test data would be welcomed. Users with information on this method are requested to write to:

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