

# **Suspended, Settleable, and Total Dissolved Solids in Waters and Effluents 1980**

**Methods for the Examination of Waters and Associated Materials**

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## **Methods for the Examination of Water and Associated Materials**

The methods contained in this booklet are all empirical. As these determinands are sometimes used in the estimation of charges for the discharge of trade effluents, it must be emphasized that, to avoid disputes, when negotiating the consent to a discharge, the parties concerned should agree in detail the procedures to be used.

This booklet contains three methods for Suspended Solids and one each for Settleable Solids and Total Dissolved Solids, together with notes on the more important minor variations and their effects on the analytical results.

As these methods are highly dependent on the filters used, those actually used in the development of the methods are mentioned. This in no way endorses these materials as superior to other similar materials. Equivalent materials are acceptable though it must be understood that the performance characteristics may be different and when agreement between contracting parties is essential, agreement on such details may be necessary.

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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 whether for one's self, one's colleagues in the laboratory, outsiders or subsequently for maintenance workers. 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 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.

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; '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.

Where the Committee have considered that a special unusual hazard exists, attention has been drawn to this in the text so that additional care must 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 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. 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.

## 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 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 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 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*

*31 October 1983*

# Suspended Solids

## Introduction

The separation of suspended matter from water is dependent on a number of variables some of which can be controlled only with difficulty. The chemical and physical nature of the material in suspension, the pore size of the filter, the area and thickness of the filter paper and the amount and physical state of the materials deposited on it are the principal factors involved. For these reasons, although these tests for the total content of suspended solids of various kinds are very useful, the results cannot represent the facts accurately.

## Volatile and non-volatile matter

The various types of suspended matter are defined arbitrarily by the methods used for their determination. Thus results will not include materials in the sample which are volatile under the conditions of the test procedure. Many mineral impurities contain water of crystallisation the loss of which on heating depends upon the substance, the temperature and the time. No set of conditions can be prescribed under which all the water of crystallization is either retained or lost without either retention of some water or loss by decomposition or evaporation of organic or volatile impurities. To obtain reproducible or comparable results close attention must be given to such procedural details as drying time, temperatures and filter characteristics.

## Choice of method

The amount and type of suspended matter in the sample, the purpose of the water analysis and the relative ease of making the determination, will dictate the choice of method. Three methods of determining suspended matter are described namely.

1. Filtration on a glass fibre paper
2. Filtration on a cellulose ester membrane filter
3. By use of a centrifuge

It should be noted that direct comparison of the results obtained by the three methods is not possible.

Glass fibre filtration is the most widely used procedure, and is particularly useful when information as to the volatile and ash content is required.

When the suspended solids content is expected to be low the use of membrane filtration should be considered.

The centrifugal method is used when filtration methods are not applicable, but cannot be used for samples containing floating suspended matter. The centrifugal method is much quicker than either of the filtration methods especially when large numbers of samples are being examined, but may give slightly lower results as it does not include any colloidal matter that tends to be retained by the filtration methods.

The insertion of a gauze between the filter paper and the funnel base is gaining popularity. This has advantages in improving the uniformity of filtration over the filter, and increasing filtration rates. The adoption of this procedure may lead to lower results however when compared with the method currently described, and performance characteristics may be different.

## Sampling and sample preservation

Whenever possible, the suspended solids should be determined on a separate sample as soon as it is taken, since biological, chemical and physical changes may occur which will alter the amount and nature of suspended matter. Where this is not possible, the sample should be stored at a temperature below 5° (but not frozen) in the dark for a maximum period of 24 hours prior to analysis. Cooling and storage may affect the measured concentration of solids.

The sample should be well shaken before analysis.

Samples should be collected in wide-mouth glass bottles. Plastic screw caps may be used provided they are fitted with nondegradable liners.

Sample bottles should be cleaned by standing overnight filled with 20% v/v hydrochloric acid, emptying and washing well with water. Bottles which have previously contained oily or greasy samples should be treated with a surface active cleaning agent and then well rinsed.



# SMP Determination of suspended matter (paper filtration method)

**SMP1 Performance characteristics of the method**  
(Drying at 105°C)

| 1.1                        | Substance determined       | Organic and/or inorganic matter which can be removed from water by filtration (under reduced pressure) using a GF/C glass-fibre paper.                                                                                                                                                                                                                                                                                                                                                                                        |  |           |                          |              |                           |              |                           |          |                            |          |            |          |
|----------------------------|----------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--|-----------|--------------------------|--------------|---------------------------|--------------|---------------------------|----------|----------------------------|----------|------------|----------|
| 1.2                        | Type of sample             | Natural and polluted waters.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  |  |           |                          |              |                           |              |                           |          |                            |          |            |          |
| 1.3                        | Basis of the method        | Filtration through a glass-fibre paper. The weight of recovered matter is determined by drying and weighing.                                                                                                                                                                                                                                                                                                                                                                                                                  |  |           |                          |              |                           |              |                           |          |                            |          |            |          |
| 1.4                        | Range of application       | Up to 25mg of recovered material.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             |  |           |                          |              |                           |              |                           |          |                            |          |            |          |
| 1.5                        | Standard deviation (a)(b)  | <table border="1"> <thead> <tr> <th></th> <th>Std. Dev.</th> </tr> </thead> <tbody> <tr> <td>Kaolin suspension 5 mg/l</td> <td>0.2-0.6 mg/l</td> </tr> <tr> <td>Kaolin suspension 50 mg/l</td> <td>0.5-2.2 mg/l</td> </tr> <tr> <td>Kaolin suspension 81 mg/l</td> <td>2.5 mg/l</td> </tr> </tbody> </table> <p>Within batch deviation on real samples is:</p> <table border="1"> <tbody> <tr> <td>Suspended matter 1.0 mg/l:</td> <td>0.2 mg/l</td> </tr> <tr> <td>42.8 mg/l:</td> <td>2.8 mg/l</td> </tr> </tbody> </table> |  | Std. Dev. | Kaolin suspension 5 mg/l | 0.2-0.6 mg/l | Kaolin suspension 50 mg/l | 0.5-2.2 mg/l | Kaolin suspension 81 mg/l | 2.5 mg/l | Suspended matter 1.0 mg/l: | 0.2 mg/l | 42.8 mg/l: | 2.8 mg/l |
|                            | Std. Dev.                  |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               |  |           |                          |              |                           |              |                           |          |                            |          |            |          |
| Kaolin suspension 5 mg/l   | 0.2-0.6 mg/l               |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               |  |           |                          |              |                           |              |                           |          |                            |          |            |          |
| Kaolin suspension 50 mg/l  | 0.5-2.2 mg/l               |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               |  |           |                          |              |                           |              |                           |          |                            |          |            |          |
| Kaolin suspension 81 mg/l  | 2.5 mg/l                   |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               |  |           |                          |              |                           |              |                           |          |                            |          |            |          |
| Suspended matter 1.0 mg/l: | 0.2 mg/l                   |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               |  |           |                          |              |                           |              |                           |          |                            |          |            |          |
| 42.8 mg/l:                 | 2.8 mg/l                   |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               |  |           |                          |              |                           |              |                           |          |                            |          |            |          |
| 1.6                        | Limit of detection         | 2 mg/l                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |  |           |                          |              |                           |              |                           |          |                            |          |            |          |
| 1.7                        | Sensitivity                | For a 500 ml sample volume each 1 mg/l suspended matter corresponds to a change in weight of 0.5 mg on the filter paper.                                                                                                                                                                                                                                                                                                                                                                                                      |  |           |                          |              |                           |              |                           |          |                            |          |            |          |
| 1.8                        | Bias                       | <p>Dissolved solids may bias the result by retention of soluble solids on the filter. See Section 10.2.</p> <p>If the amount of suspended matter is high, filter blinding may cause retention of particles smaller than the listed porosity of the filter paper.</p>                                                                                                                                                                                                                                                          |  |           |                          |              |                           |              |                           |          |                            |          |            |          |
| 1.9                        | Interferences              | Oil or other organic liquids. Samples with dissolved solids greater than about 1000 mg/l. (see section 10)                                                                                                                                                                                                                                                                                                                                                                                                                    |  |           |                          |              |                           |              |                           |          |                            |          |            |          |
| 1.10                       | Time required for analysis | Depends on type of sample (within one day).                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |  |           |                          |              |                           |              |                           |          |                            |          |            |          |

**SMP2 Performance characteristics of the method**  
(Ignition at 500°C)

| 2.1      | Substance determined       | Residual matter after ignition of filtered residue at 500°C.                                                                                                                                                                                                                                                                                                                |          |         |          |              |         |              |         |              |
|----------|----------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------|---------|----------|--------------|---------|--------------|---------|--------------|
| 2.2      | Type of sample             | Natural and polluted waters.                                                                                                                                                                                                                                                                                                                                                |          |         |          |              |         |              |         |              |
| 2.3      | Basis of the method        | Filtration through a glass fibre paper, weighing and recovered matter, igniting at 500°C and weighing the residue.                                                                                                                                                                                                                                                          |          |         |          |              |         |              |         |              |
| 2.4      | Range of application       | up to 25 mg residual material.                                                                                                                                                                                                                                                                                                                                              |          |         |          |              |         |              |         |              |
| 2.5      | Standard deviation (b)(c)  | Within batch standard deviation on local river waters:<br><table border="1" style="margin-left: auto; margin-right: auto;"> <thead> <tr> <th>Mean ash</th> <th>Std Dev</th> </tr> </thead> <tbody> <tr> <td>6.0 mg/l</td> <td>0.3-0.8 mg/l</td> </tr> <tr> <td>14 mg/l</td> <td>0.2-1.4 mg/l</td> </tr> <tr> <td>50 mg/l</td> <td>0.5-2.8 mg/l</td> </tr> </tbody> </table> | Mean ash | Std Dev | 6.0 mg/l | 0.3-0.8 mg/l | 14 mg/l | 0.2-1.4 mg/l | 50 mg/l | 0.5-2.8 mg/l |
| Mean ash | Std Dev                    |                                                                                                                                                                                                                                                                                                                                                                             |          |         |          |              |         |              |         |              |
| 6.0 mg/l | 0.3-0.8 mg/l               |                                                                                                                                                                                                                                                                                                                                                                             |          |         |          |              |         |              |         |              |
| 14 mg/l  | 0.2-1.4 mg/l               |                                                                                                                                                                                                                                                                                                                                                                             |          |         |          |              |         |              |         |              |
| 50 mg/l  | 0.5-2.8 mg/l               |                                                                                                                                                                                                                                                                                                                                                                             |          |         |          |              |         |              |         |              |
| 2.6      | Limit of detection         | 2 mg/l                                                                                                                                                                                                                                                                                                                                                                      |          |         |          |              |         |              |         |              |
| 2.7      | Sensitivity                | For a 500 ml sample volume each 1 mg/l residual matter corresponds to change in weight of 0.5 mg on the filter paper.                                                                                                                                                                                                                                                       |          |         |          |              |         |              |         |              |
| 2.8      | Bias                       | Not known                                                                                                                                                                                                                                                                                                                                                                   |          |         |          |              |         |              |         |              |
| 2.9      | Interferences              | Not known                                                                                                                                                                                                                                                                                                                                                                   |          |         |          |              |         |              |         |              |
| 2.10     | Time required for analysis | About 2 hours                                                                                                                                                                                                                                                                                                                                                               |          |         |          |              |         |              |         |              |

- (a) Estimates are within batch with nine degrees of freedom obtained by laboratories of the Yorkshire Water Authority, Northumbrian Water Authority and Fife Regional Council.
- (b) Estimates are within batch with 16 degrees of freedom obtained by 7 laboratories in the Harmonised Monitoring Scheme.
- (c) For additional test data see *Analyst* 1983, **108**, 1365-1373.

**SMP3 Field of application**

- 3.1 The method determines organic and/or inorganic matter which can be removed from water by filtration using a GF/C glass filter paper and is recommended for use at up to 25 mg of recovered material.
- 3.2 The method is applicable to natural and polluted waters.
- 3.3 Samples containing an excessive amount of suspended matter, colloidal material or matter in finely divided form, may often be difficult to filter, even under vacuum. When it is impracticable to use the filtration method the centrifugal method should be used, but centrifugation is not applicable if any part of the suspended matter floats.

**SMP4 Principle**

- 4.1 Suspended matter is removed from a measured volume of sample by filtration under reduced pressure through a pre-treated, pre-weighed, glass-fibre filter paper and determined gravimetrically after washing and drying at 105°C to constant weight.
- Volatile material and ash in the suspended matter may be determined by ignition at 500°C.

**SMP5 Interferences**

- 5.1 Oil; samples with more than 1000 mg/l dissolved solids. (see section 11)

**SMP6 Hazards**

- 6.1 Reduced pressure filtration requires the use of a safety screen.

**SMP7 Reagents**

- 7.1 Industrial methylated spirit 74% v/v
- 7.2 Petroleum spirit (40°/60°C) of analytical reagent grade
- 7.3 Water-deionized or freshly distilled water is suitable

**SMP8 Apparatus**

- 8.1 Whatman Type GF/C glass-fibre filter papers, 70mm diameter, particle size retention 1.2 microns. (70 mm papers are normally used and the performance characteristics relate to this size of filter, although other sizes may be more convenient in certain circumstances).
- 8.2 Hartley pattern, 3 part filter assembly with 70mm diameter disc, or appropriate size if a smaller diameter paper is being used.
- 8.3 Buchner filtration flask
- 8.4 Drying oven controlled at  $105 \pm 5^{\circ}\text{C}$
- 8.5 500 ml polythene graduated cylinder from which the centre of the base has been cut out flush with the inner wall. This may be used in place of the upper component of the Hartley filter for slow filtering samples of volume greater than about 50 ml. But see also Step 9.3 note f.
- 8.6 Muffle furnace controlled to operate at a temperature of  $500 \pm 20^{\circ}\text{C}$
- 8.7 Desiccator (containing dry self-indicating silica gel)
- 8.8 Analytical balance capable of weighing to 0.1 mg
- 8.9 Mechanical or water suction vacuum pump to give reduced pressure (2.6 to 3.3 KPa)
- 8.10 Blunt nosed forceps
- 8.11 Muffle tongs
- 8.12 Measuring cylinder of appropriate size for sample taken (500, 250 or 100 ml).  
(1 mm Hg = 133.32 Pa, 1 KPa = 0.145 16/in<sup>2</sup> =  $10^3\text{N/m}^2$  = 0.01 bar)

| Step                                     | Procedure                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     | Notes                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                |                                          |                       |     |     |        |     |      |     |
|------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------|-----------------------|-----|-----|--------|-----|------|-----|
|                                          | <b>Analysis of samples (a)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                | (a) For accurate work read Section 10 before doing these analyses.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                                          |                       |     |     |        |     |      |     |
| 9.1                                      | <p>Carry out the following process on a number of papers which have been identified by marking near the outer edge using an HB pencil.</p> <p>Insert a glass-fibre filter paper into the funnel assembly and clip together. Using a slight suction wash with about 100 ml of water and, when free from excess water, remove the paper and place on a layer of aluminium foil (crumpled to prevent sticking) in an adjacent oven. Dry at <math>105 \pm 5^\circ\text{C}</math> for 1 hour. Using forceps transfer the paper to a dessicator and allow to cool for 30 min. Transfer the paper to the balance pan and weigh. Repeat the drying, cooling and weighing cycle until constant weight (<math>A_{\text{mg}}</math>) is attained. (notes b, c and d)</p> | <p>(b) If ignition at <math>500^\circ\text{C}</math> is required, carry out step 9.1 on a number of papers taken from the same box used in the test procedure (at least 5) and follow by heating for 30 min at <math>500 \pm 20^\circ\text{C}</math>. Use a watch glass in place of aluminium foil. Furnace tongs must be used for removing the filter papers from the muffle furnace. Cool for 60 min in a dessicator before weighing. Determine the weight loss per paper between <math>500^\circ\text{C}</math> and <math>105^\circ\text{C}</math> and from this calculate the average weight loss per paper due to ignition (<math>D_{\text{mg}}</math>). This value is to be used as a blank when suspended matter at <math>105^\circ\text{C}</math> is subsequently ignited at <math>500^\circ\text{C}</math>. Ignition at temperature above <math>400^\circ\text{C}</math> will alter the porosity of filter papers. These papers should therefore be discarded and not used.</p> <p>(c) Constant weight is attained when the weights after successive drying &amp; weighing cycles differ by 0.5 mg or less. (If it can be determined by experience that for certain types of sample constant weight is attained after two drying and weighing cycles, only one cycle is required).</p> <p>(d) A nylon gauze (<math>120 \mu\text{m}</math> mesh) may be inserted between the filter disc and the glass fibre paper in order to improve the filtration rate. This could give low results.</p> |                                          |                       |     |     |        |     |      |     |
| 9.2                                      | Place a dried, marked pre-weighed glass-fibre paper on the perforated disc of the filter unit, moisten with a few ml of water and assemble the filter unit, securing it with spring clips. Attach the assembled funnel to a Buchner flask.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |                                          |                       |     |     |        |     |      |     |
| 9.3                                      | Measure a suitable volume ( $V$ ml) of the well mixed sample using a measuring cylinder. Filter under gentle vacuum (2.6 to 3.3 KPa), ensuring that all solids are transferred to the paper. (notes e and f).                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 | <p>(e) An indication of the maximum volume which may be required is given below:—<br/>(the actual volume taken depends on the nature of the suspended matter)</p> <table border="1"> <thead> <tr> <th>Concentration of suspended matter (mg/l)</th> <th>Volume of sample (ml)</th> </tr> </thead> <tbody> <tr> <td>&lt;20</td> <td>500</td> </tr> <tr> <td>20–100</td> <td>250</td> </tr> <tr> <td>&gt;100</td> <td>100</td> </tr> </tbody> </table> <p>(f) If large numbers of large volumes of sample are being handled the sample may be measured into a suitable volumetric flask, the flask inverted and clamped over the filter funnel so that the neck of the flask is below the liquid level in the funnel top. On completion of the transfer the neck of the flask should be thoroughly washed to ensure the removal of all solids from the neck of the flask. This technique improves the transfer of solids and minimizes operator time, since heavy granular solids fall to the neck of the flask and are filtered first.</p>                                                                                                                                                                                                                                                                                                                                                                                                                                                            | Concentration of suspended matter (mg/l) | Volume of sample (ml) | <20 | 500 | 20–100 | 250 | >100 | 100 |
| Concentration of suspended matter (mg/l) | Volume of sample (ml)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |                                          |                       |     |     |        |     |      |     |
| <20                                      | 500                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |                                          |                       |     |     |        |     |      |     |
| 20–100                                   | 250                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |                                          |                       |     |     |        |     |      |     |
| >100                                     | 100                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |                                          |                       |     |     |        |     |      |     |
| 9.4                                      | Wash the walls of the measuring cylinder, the filter funnel, and the residue on the paper using three 10 ml portions of water. Suck dry.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |                                          |                       |     |     |        |     |      |     |

| Step | Procedure | Notes |
|------|-----------|-------|
|------|-----------|-------|

9.5 Release the vacuum and carefully remove the filter paper from the perforated disc. Dry on a layer of crumpled aluminium foil in an adjacent oven at  $105 \pm 5^\circ\text{C}$  to constant weight (B mg) (Step 9.1 and note c).

9.6 Total suspended matter (mg/l) = 
$$\frac{(B-A) \times 1000}{V}$$

Where A = initial weight of filter paper  
 B = weight of paper plus dried residue at  $105^\circ\text{C}$

### Volatile matter and ash

9.7 Place the dried, ignited and pre-weighted filter paper and residue from Step 9.5 in a muffle furnace at  $500 \pm 20^\circ\text{C}$  for 30 min.

9.8 Remove the paper and residue from the furnace, cool for 60 min in a dessicator and re-weigh (C mg).

### Calculation of results

9.9 Volatile suspended matter (mg/l) = 
$$\frac{(B-C+D) \times 1000}{V}$$

Ash (mg/l) = 
$$\frac{(C+D-A) \times 1000}{V}$$

Where C = weight of paper plus ash at  $500^\circ\text{C}$   
 D = mean weight loss per paper due to ignition at  $500^\circ\text{C}$

**SMP10 Improvement of accuracy** Tests have shown that papers need substantial washing to achieve constant weight. Many users compensate for this by incorporating blanks using the same volume of prefiltered distilled water as the volume of sample taken and using this blank value instead of A in the calculation.

**SMP11 Special cases** Tests have also shown that some makes of filter paper differ in appearance and also slightly in porosity depending on which side is uppermost in the filter. For accurate work, always ensure that such papers are used with the same side on top. The use of gauzes may improve speed. It does not improve precision.

## 11.1 Samples Containing oil or other organic liquids

Oil or other organic liquids may be retained on the filter and only partially volatilized on drying at  $105^\circ\text{C}$ . Where, however immiscible oil is important and is to be determined separately, the filtered, water-washed residue must be freed from oil. This may be done by washing with industrial methylated spirit and petroleum spirit before drying at  $105^\circ\text{C}$ . When this procedure is performed, it should be recorded with the results as it may have extracted material other than immiscible oil.

| Step   | Procedure                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                            | Notes                                                                                        |
|--------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------|
| 11.1.1 | <p>After step 9.4, and without applying any suction, wash the residue on the glass-fibre paper using three 30ml portions of industrial methylated spirit. Continue washing with three 30 ml portions of petroleum spirit, allowing the paper to drain free from liquid after each wash. (note g). Apply sufficient suction to remove all traces of the solvent vapour.</p> <p>If the last wash is not colourless, continue washing with 30 ml portions of petroleum spirit until the last washing is colourless.</p> | (g) Apply vacuum only after allowing sufficient time for the oil to dissolve in the solvent. |
| 11.1.2 | Apply vacuum for a few minutes, and continue as in step 9.5.                                                                                                                                                                                                                                                                                                                                                                                                                                                         |                                                                                              |

## 11.2 Samples containing more than about 1000 mg/l dissolved solids

A preliminary test for bias by dissolved solids should be made and if a correction is necessary the procedure below should be followed.

Two procedures are described, procedure 1 using two filter papers is easy to operate and is normally used. Procedure 2 uses a single filter paper but requires greater manipulative ability. For high salinity samples, however, the use of membrane filters is preferred for the determination of low suspended matter.

### 11.2.1 Procedure 1 see footnote (h)

At step 9.2 take two filter papers, each weighed separately, and assemble the Hartley funnel with the papers one on top of the other, wetting the papers before finally assembling the funnel. Continue through steps 9.2 to 9.5 and weigh each paper separately both after drying and, if required, after ignition. The lower paper, which is used as a blank, is assumed to retain the same amount of dissolved solids as the Upper paper. The uppermost paper retains the suspended matter.

Subtract the change in weight of the lower paper from the increase in weight of the upper paper.

### 11.2.2 Procedure 2

Use a single filter paper and proceed as in step 9.4 but using 3 × 50 mls water. Partially dismantle the filter funnel and wash the annular ring of the paper with the jet from a wash bottle. Then proceed as in step 9.5.

| (h) Synthetic sea water + Kaolin suspension | <u>% recovery</u> | <u>Std dev</u> |
|---------------------------------------------|-------------------|----------------|
| 50 mg/l Kaolin                              | 96.0 ± 4.0        | 3.8            |
| 100 mg/l Kaolin                             | 95.3 ± 3.0        | 2.7            |

Estimates are within batch with six degrees of freedom obtained by the Water Research Centre.

# SMM Determination of suspended matter (membrane filtration method) tentative method

## SMM1 Performance characteristics of the method

|      |                            |                                                                                                                                                                                                            |
|------|----------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 1.1  | Substance determined       | Organic and/or inorganic matter which can be removed from water by filtration using a 0.45 $\mu\text{m}$ membrane filter.                                                                                  |
| 1.2  | Type of sample             | Natural and polluted waters.                                                                                                                                                                               |
| 1.3  | Basis of the method        | Filtration through a membrane filter. The weight of recovered matter is determined by drying and weighing.                                                                                                 |
| 1.4  | Range of application       | Up to 10 mg of recovered material.                                                                                                                                                                         |
| 1.5  | Standard deviation         | Very sample dependent, but generally less than 10% relative standard deviation. There is evidence of absorption of some soluble metals by filter membranes, which should be checked for if thought likely. |
| 1.6  | Limit of detection         | See 1-5 above.                                                                                                                                                                                             |
| 1.7  | Sensitivity                | For a 2 litre sample volume each 1 mg/l suspended matter corresponds to a change in weight of 2 mg on the membrane.                                                                                        |
| 1.8  | Bias                       | Results may be high if metals, usually transition metals, are absorbed. Due to filter blinding, particles less than 0.45 $\mu\text{m}$ may be retained if there is much suspended matter.                  |
| 1.9  | Interference               | Oils or other organic liquids, samples with dissolved solids greater than 1000 mg/l. See Section 9.                                                                                                        |
| 1.10 | Time required for analysis | Depends on type of sample (within one day).                                                                                                                                                                |

## SMM2 Scope and field of application

2.1 The method determines organic and/or inorganic matter which can be removed from water by filtration through a 0.45  $\mu\text{m}$  pore size membrane filter and is recommended for use at up to 10 mg of recovered material.

2.2 The method is applicable to natural and polluted waters, having a suspended matter content of less than 10 mg/litre.

## SMM3 Principle

3.1 Suspended matter is removed from a measured volume of sample by filtration under reduced pressure through a pre-treated, pre-weighed, membrane filter and determined gravimetrically after washing and drying at 105°C to constant weight.

## SMM4 Interferences

4.1 Oil; samples with more than 1000 mg/l dissolved solids.

## SMM5 Hazards

5.1 Reduced pressure filtration requires the use of a safety screen.

**SMM6 Reagents**

6.1 Water, deionized or freshly distilled water is suitable.

6.2 Industrial methylated spirit

**SMM7 Apparatus**

7.1 Cellulose ester membrane filters (47 mm diameter), 0.45  $\mu$ m effective pore diameter.

7.2 Blunt forceps for handling membrane filters must be used at all times.

7.3 A suitable, commercial, membrane filtration apparatus consisting of a detachable filter funnel incorporating a support plate for the membrane filter.

7.4 Buchner flask (3 litres).

7.5 One litre measuring cylinder.

7.6 Drying oven controlled at 105°C  $\pm$  5°C.

7.7 Desiccator (containing self indicating silica gel).

7.8 Analytical balance capable of weighing to 0.1 mg.

7.9 Mechanical or water suction vacuum pump to give reduced pressure (2.6 –3.3 KPa).

Reduced pressure filtration must be carried out behind a safety screen

**SMM8 Analytical procedure**

| Step                | Procedure                                                                                                                                                                                                                                                                                                                                                                                                              | Notes                                                                                                                                                                                                                                                           |
|---------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Analysis of samples |                                                                                                                                                                                                                                                                                                                                                                                                                        |                                                                                                                                                                                                                                                                 |
| 8.1                 | Place the membrane filter (note a) on the support plate of the filter unit, moisten with water and assemble the top of the unit.<br><br>Filter one litre of water through the membrane under gentle vacuum (2.6–3.3 KPa).                                                                                                                                                                                              | (a) It is permissible to wash a batch of up to 10 membrane filters simultaneously. For identification purposes, cut the edges of the filters in some distinguishing way, eg with a razor blade.                                                                 |
| 8.2                 | Release the vacuum, remove the top part of the filter assembly and then remove the membrane filter and transfer it on to a suitable filter paper. Cover it with a clock glass and place in an oven at 105°C $\pm$ 5°C for 1 hour. Remove the membrane filter, allow to cool for 30 min in a dessicator and weigh.<br><br>Repeat the drying, cooling and weighing cycle until constant weight (A) is attained (note b). | (b) Constant weight is attained when the weights after successive drying and weighing cycles differ by 0.5 mg or less. If it can be determined by experience that constant weight is attained after two drying and weighing cycles, only one cycle is required. |
| 8.3                 | Place the prepared membrane filter centrally on the support plate of the filter unit. Moisten with water and assemble the top of the unit.                                                                                                                                                                                                                                                                             |                                                                                                                                                                                                                                                                 |
| 8.4                 | Measure two litres of the well mixed sample; using a measuring cylinder. Filter under gentle vacuum 2.6–3.3 KPa ensuring that all solids are transferred to the paper.                                                                                                                                                                                                                                                 |                                                                                                                                                                                                                                                                 |
| 8.5                 | Wash the walls of the measuring vessel, the filter funnel and the residue on the membrane filter using about 50 ml of water.                                                                                                                                                                                                                                                                                           |                                                                                                                                                                                                                                                                 |



| Step                           | Procedure                                                                                                                                                                               | Notes |
|--------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------|
| 8.6                            | Release the vacuum, remove the top part of the filter assembly, carefully remove the membrane filter, then dry in an oven at $105 \pm 5^\circ\text{C}$ to constant weight (B) (note b). |       |
| <b>Calculations of Results</b> |                                                                                                                                                                                         |       |
| 8.9                            | Suspended solids (mg/l) = $\frac{(B-A) \times 1000}{2000}$                                                                                                                              |       |

**SMM9 Special Cases** 9.1 Samples containing oil or other organic liquids

Oil or other organic liquids may be retained on the membrane and only partially volatilized on drying at  $105^\circ\text{C}$ . Where, however immiscible oil is important and is to be determined separately, the filtered, water-washed residue must be freed from oil. This may be done by washing with industrial methylated spirit and petroleum spirit before drying at  $105^\circ\text{C}$ . When this procedure is performed, it should be recorded with the results as it may have extracted material other than immiscible oil.

| Step  | Procedure                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     | Notes                                                                                        |
|-------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------|
| 9.1.1 | After step 8.5 and without applying any suction, wash the residue on the glass fibre paper using three 15 ml portions of industrial methylated spirit. Continue washing with three 15 ml portions of petroleum spirit, allowing the paper to drain free from liquid after each wash. (Note c). Apply sufficient suction to remove all traces of the solvent vapour.<br><br>If the last wash is not colourless, continue washing with 15 ml portions of petroleum spirit until the last washing is colourless. | (c) Apply vacuum only after allowing sufficient time for the oil to dissolve in the solvent. |
| 9.1.2 | Apply vacuum for a few minutes, and continue as in step 8.6.                                                                                                                                                                                                                                                                                                                                                                                                                                                  |                                                                                              |

9.2 Samples containing more than about 1000 mg/l dissolved solids

A preliminary test for bias by dissolved solids should be made and if a correction is necessary the procedure below should be followed.

9.2.1 Analytical procedure

Using a single filter paper and proceed as in step 8.5 but using 3 x 50 mls water. Partially dismantle the filter funnel and wash the annular ring of the paper with the jet from a wash bottle. Then proceed as in step 8.6.

# SMC Determination of suspended matter (centrifugal method) tentative method

## SMC1 Performance characteristics of the method

|      |                            |                                                                                                    |
|------|----------------------------|----------------------------------------------------------------------------------------------------|
| 1.1  | Substance determined       | Organic and/or inorganic matter which can be removed from water by centrifuging at 1400–3000g.     |
| 1.2  | Type of sample             | Natural and polluted waters.                                                                       |
| 1.3  | Basis of the method        | Centrifuging at 1400–3000g. The weight of recovered matter is determined by drying and weighing.   |
| 1.4  | Range of application       | 200 mg/l upwards can be examined. See Section 8.4 for samples containing less than 200 mg/l.       |
| 1.5  | Standard deviation         | Very sample and equipment dependent, but generally less than a 10% relative standard deviation.    |
| 1.6  | Limit of detection         | Very sample and equipment dependent.                                                               |
| 1.7  | Sensitivity                | For a 50 ml aliquot each 10 mg/l of suspended matter will correspond to a weight change of 0.5 mg. |
| 1.8  | Bias                       | Very sample and equipment dependent.                                                               |
| 1.9  | Interference               | Oil or other organic liquids. Floating material (See Selection 3.2).                               |
| 1.10 | Time required for analysis | Depends on type of sample (within one day).                                                        |

## SMC2 Scope and field of application

2.1 The method determines organic and/or inorganic suspended matter which can be removed from water by centrifuging at 1400–3000 g and is recommended for use in the range 10 mg upwards of suspended matter recovered.

2.2 The method is applicable to natural and polluted waters where high concentrations of suspended matter are present, eg activated sludge samples.

2.3 This method is not applicable if floating solids are present in the sample.

## SMC3 Principle

3.1 Suspended matter is separated from a measured volume of sample by centrifuging, washing and drying at  $105 \pm 5^\circ\text{C}$  to constant weight.

## SMC4 Interferences

4.1 Oil or other organic liquids; floating material.

## SMC5 Hazards

5.1 Use of a safety screen is required if operating with a centrifuge which is not fully enclosed.

## SMC6 Reagents

6.1 Water, deionized or freshly distilled water is suitable.

**SMC7 Apparatus**

7.1 **Laboratory centrifuge** capable of accepting four 50 ml centrifuge tubes. The machine must be operable at a relative centrifugal force (RCF) of 1400–3000 g, measured at the bottom of the tube.

$$RCF = 1.2 \times 10^{-6} \times rN^2$$

Where r = radius (mm) from centre of rotation to bottom of tube when in operation.

N = revolutions per minute

7.2 **Hard glass, conical centrifuge tubes** capacity 50 ml.

7.3 **Drying oven** controlled at  $105^\circ \pm 5^\circ\text{C}$ .

7.4 **Dessicator** (containing dry self indicating silica gel).

7.5 **Silica or porcelain dishes**, capacity 200 ml.

7.6 **Analytical balance** capable of weighing to 0.1 mg.

**SMC8 Analytical procedure**

Unless the centrifuge is fully enclosed, it must always be operated behind a safety screen

| Step | Procedure                                                                                                                                                                                                                                                                                                             | Notes                                                                                                                                                                                                                                                         |
|------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
|      | <b>Analysis of samples containing more than 200 mg/litre suspended matter</b>                                                                                                                                                                                                                                         |                                                                                                                                                                                                                                                               |
| 8.1  | Transfer 50 ml of the well mixed sample, to a dry, pre-weighed centrifuge tube. (A) Counter balance against a second tube containing water, place the tubes on opposite sides of the centrifuge rotor and spin them at 1400–3000 g for five minutes or until the solid no longer settles from the liquid. (Note (a)). | (a) Dependent on centrifuge design a second sample may be used as a counter balance. Rotors must be balanced when rotating.                                                                                                                                   |
| 8.2  | Decant the supernatant liquor from the tube and re-suspend the deposited matter by carefully filling to the original level using water applied for a wash bottle.                                                                                                                                                     |                                                                                                                                                                                                                                                               |
| 8.3  | Centrifuge for a further five minutes and decant the supernatant liquor as completely as possible. Dry the deposit in the centrifuge tube in an oven at $105 \pm 5^\circ\text{C}$ for 1 hour. Allow to cool for 30 minutes in a desiccator and weigh.                                                                 |                                                                                                                                                                                                                                                               |
|      | Repeat the drying, cooling and weighing cycle until constant weight (B) is attained (Note (b)).                                                                                                                                                                                                                       | (b) Constant weight is attained when the weights after successive drying and weighing cycles differ by 0.5 mg or less, if it can be determined by experiment that constant weight is attained after two drying and weighing cycles, only 1 cycle is required. |
|      | <b>Analysis of samples containing less than 200 mg/litre suspended matter</b>                                                                                                                                                                                                                                         |                                                                                                                                                                                                                                                               |
| 8.4  | Transfer 200 ml of the well mixed sample to four 50 ml centrifuge tubes, and counter balance the opposite pairs of tubes.                                                                                                                                                                                             |                                                                                                                                                                                                                                                               |
|      | Centrifuge as directed in steps 8.1, 8.2 and 8.3.                                                                                                                                                                                                                                                                     |                                                                                                                                                                                                                                                               |

| Step | Procedure                                                                                                                                                                                                                                                                                                                                        | Notes                                                                                                                                                                  |
|------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 8.5  | Wash out the deposited matter from the four centrifuge tubes, using as little water as possible, and combine in a dry, pre-weighed dish (A).<br><br>Dry to constant weight (B) as directed in step 8.3.<br><br>Analysis of samples containing high concentrations of dissolved solids                                                            |                                                                                                                                                                        |
| 8.6  | Centrifuge the sample as directed in steps 8.1 and 8.2 or, if the suspended matter is less than 200 mg/l, as step 8.4.                                                                                                                                                                                                                           |                                                                                                                                                                        |
| 8.7  | Continue to wash the deposited matter, by decantation, until the supernatant liquor contains less than 50 mg/litre of dissolved solid (as determined by evaporation at an aliquot to constant weight at $105 \pm 5^\circ\text{C}$ in a tared silica dish) or the electrical conductivity falls below $70 \mu\text{S}/\text{cm}^{-1}$ (note (c)). | (c) This definition of the washing procedure is, of course, entirely empirical since extended washing may lead to dissolution of some of the original suspended solid. |
| 8.8  | Continue as in step 8.3 or, suspended matter in the sample is less than 200 mg/litre as steps 8.4 and 8.5.<br><br>Calculation of results                                                                                                                                                                                                         |                                                                                                                                                                        |
| 8.9  | Suspended matter = $\frac{(B-A) \times 1000}{V}$                                                                                                                                                                                                                                                                                                 |                                                                                                                                                                        |

# SS Determination of settleable solids, tentative method

## Introduction

The determination which is by weight of solid per unit volume, is carried out to evaluate or predict the behaviour of settlement processes. Some organisations also employ it as one method of estimating the strength of trade effluent and the procedure has been written from this viewpoint.

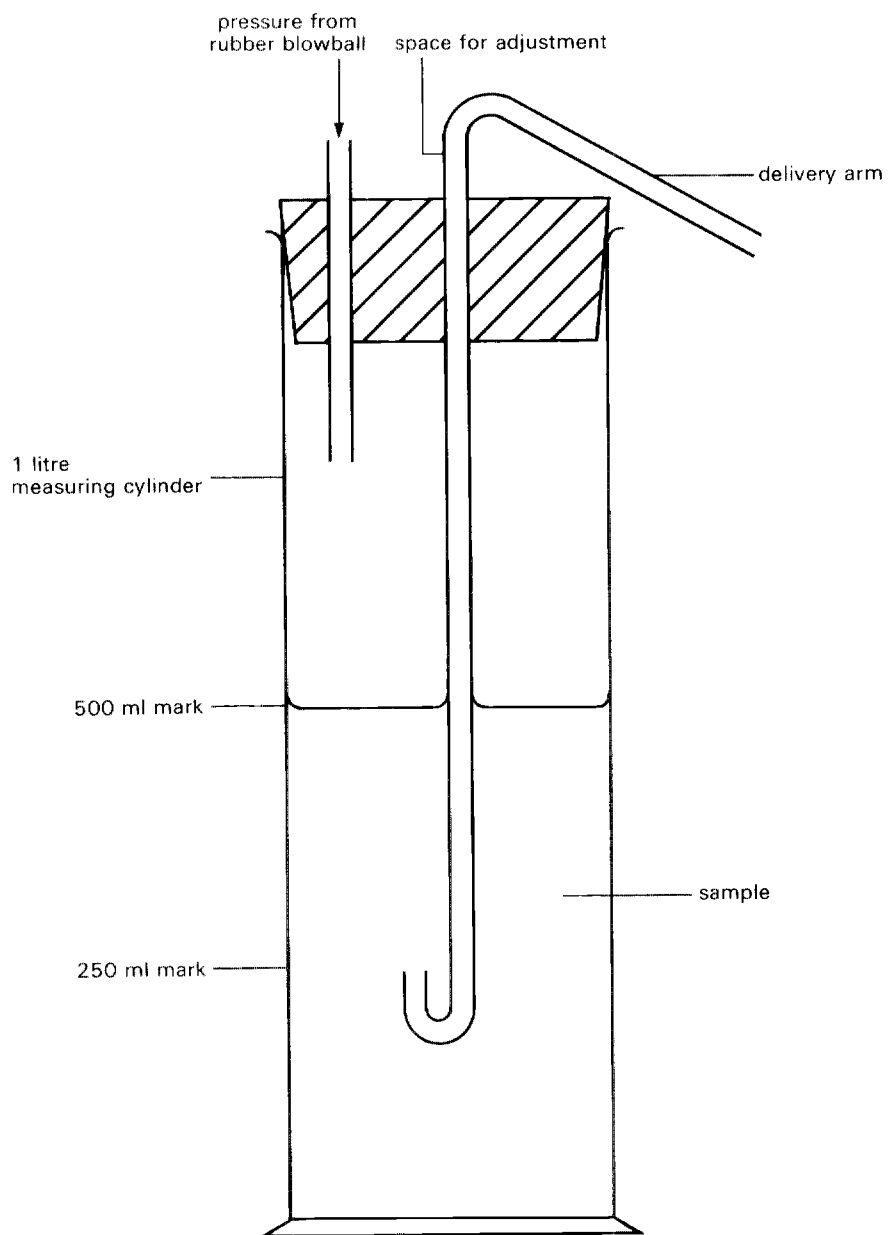
It will be appreciated that the choice of experimental conditions is essentially arbitrary and that a different pH, settling time and temperature, for example, will alter the result. The choice of conditions is, in part, a matter for negotiation between the regulatory authority and the discharger.

This method is not meant to replace existing arrangements but is recommended where such a determination is to be introduced or where there is a mutual desire to amend existing procedures.

For control purposes, some operators use the Imhoff cone, volume per unit volume method. This is appended as a note.

## SS1 Performance characteristics of the method

|     |                            |                                                                                                                                                      |
|-----|----------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------|
| 1.1 | Substance determined       | The amount of substance settling from a suspension in 60 minutes (or other agreed time). Floating matter is also includable if present.              |
| 1.2 | Type of Sample             | River or sea waters, sewages and effluents.                                                                                                          |
| 1.3 | Basis of the method        | Removal of a sample of supernatant liquid, without inclusion of floating solids and determination of suspended solids before and after settling.     |
| 1.4 | Standard deviation         | This is highly sample dependent, but being dependent on two determinations of suspended solids cannot be as accurate or as precise as that analysis. |
| 1.5 | Limit of detection         | This is highly sample dependent but is often 2-3 mg/l.                                                                                               |
| 1.6 | Bias                       | Not determined but from a consideration of the method is likely to be positive and variable.                                                         |
| 1.7 | Interferences              | Floating matter.                                                                                                                                     |
| 1.8 | Time required for analyses | Sample dependent, but under one day.                                                                                                                 |



EGGING DEVICE FOR REMOVING SETTLED SAMPLE

**SS2 Field of apparatus** The method determines organic and/or inorganic matter that settles from a sample under the prescribed conditions of test.

**SS3 Principle** The settleable solids are determined by subtracting the solids left in suspension after one hour's settlement, from the total suspended solids content of the sample.

**SS4 Reagents**

**4.1 5M nitric acid**

To 500 ml of distilled water, add carefully with stirring  $320 \pm 5$  ml nitric acid ( $d_{20} 1.42$ ). Transfer to a 1000 ml measuring cylinder and dilute to 1000 ml with distilled water. Mix well, and store in a glass bottle.

(Note, that for certain waters, dependent on whether solution or precipitation is likely to cause error in the measurement, some laboratories prefer to substitute, 5M sulphuric, hydrochloric or acetic acids. Nitric acid is preferred unless local conditions prevail. Hydrochloric acid is the most prone to dissolving sediments).

**4.2 5M Sodium hydroxide**

Dissolve  $200 \pm 5$  gm of sodium hydroxide pellets in approximately 500 ml distilled water. Transfer to a 1000 ml measuring cylinder and dilute to 1000 ml distilled water. Mix well, and store in a polythene bottle.

**SS5 Apparatus**

**5.1 1000-ml measuring cylinder.** This should be the open top kind and total length of the graduations must be  $33 \pm 1$  cm.

**5.2 The egging apparatus** illustrated in the Figure is required to remove the sample from the measuring cylinder.

**SS6 Analytical procedure**

| Step | Procedure                                                                                                                                                                                                                               | Notes                                                                                                                                                                                                                                                                                                                                                                                                                                |
|------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 6.1  | Adjust the temperature of the sample to $20 \pm 5^\circ\text{C}$ .                                                                                                                                                                      |                                                                                                                                                                                                                                                                                                                                                                                                                                      |
| 6.2  | Take a known volume $V_1$ ml (where $V_1$ is not less than 500). Measure the pH and adjust to 6.0 to 8.0 by the addition of 5M nitric acid or 5M sodium hydroxide as appropriate. Note the volume of reagent taken, $V_2$ ml. (Note a). | (a) A second portion of sample will be required later, see Step 6.7 and note g.                                                                                                                                                                                                                                                                                                                                                      |
| 6.3  | Shake the neutralized sample vigorously and immediately pour $500 \pm 10$ ml into the 1000-ml measuring cylinder. (note b)                                                                                                              | (b) Carefully adjust the arm so that the required volume of sample can be removed without entraining either floating matter or settled sediment.                                                                                                                                                                                                                                                                                     |
| 6.4  | Allow the sample to settle for $60 \pm 5$ minutes, insert the delivery arm of the device illustrated in the Figure taking care to avoid the introduction of floating matter into the arm during insertion (notes b, c and d).           | (c) If previously agreed other settling times may be used but should be stated with the report.<br>(d) If there is much floating matter it may help to close the delivery end of the arm thus trapping air inside it while lowering the inner end well clear of the floating matter.                                                                                                                                                 |
| 6.5  | Use this apparatus to remove a maximum of 250 ml of the liquor from midway between the settled and any floating matter, without disturbing the settled or floating material. (notes e and f)                                            | (e) The depth to which the delivery arm is inserted may need some variation from that given in Section 5.2.<br>(f) In most sewage operations, floating matter is included in with settleable solids, but if floating matter is likely to cause considerable error, it might be skimmed off with filter paper, washed off the filter paper with distilled water and estimated approximately using the suspended solids procedure SMP. |

| Step | Procedure                                                                                                                                                                                                                                                                                                       | Notes                                                                                                                                                                                                                                          |
|------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 6.6  | Remove the egging apparatus (Section 5.1) and read the volume of sample remaining in the 1000-ml measure to the nearest 10 ml. Subtract this volume from 500 ml to obtain the volume egged off.                                                                                                                 |                                                                                                                                                                                                                                                |
| 6.7  | Determine the suspended solids content of both the removed supernatant from the settled sample and the fully mixed neutralized sample by one of the methods described for suspended matter. Let $S_s$ = settled supernatant solids content in mg/l.<br>$S_o$ = original sample solids content in mg/l. (note g) | (g) The fully mixed neutralized sample is a second portion $V_1$ ml of the sample to which $V_2$ ml of acid or alkali is added as was added to the settled sample. Different volumes may be used as long as the ratio $V_1/V_2$ is maintained. |

**SS7 Calculation of result**

Settleable solids =  $(S_o - S_s) \frac{(V_1 + V_2)}{V_1}$  mg/l

Where  $S_o$ ,  $S_s$ ,  $V_1$ ,  $V_2$  are defined above.



# SSV Settleable solids by volume

## Apparatus

**Imhoff cone** (a graduated glass cone (point downwards) of at least 1 litre capacity with a narrow angle at the apex, a graduated volume scale engraved at the narrow bottom end with zero at the apex and an engraved 1 litre calibration ring at the widest top end.)

## Procedure

Fill the Imhoff cone to the 1-litre mark with well-mixed sample and allow to settle. After 45 minutes gently dislodge any deposit on the side of the vessel either with a glass rod or by spinning the cone so that any adhering material settles to the apex. After one hour, or other time specified, to relate to works conditions, read off the volume of sludge (ml) which has settled in the apex.

## Accuracy

This apparatus, although capable of indicating small quantities of settled material in the apex, is subject to considerable inaccuracy if the solids contain large fragments.

## Results

Express the result in ml settleable solids per litre of sample.

# TDS Total dissolved solids dried at 180°C

## TDS1 Performance characteristics of the method

|      |                            |                                                                                                                                                                                                                                                                                     |                                           |                    |
|------|----------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------------|--------------------|
| 1.1  | Substance determined       | Non volatile organic and/or inorganic matter which is not removed from water by filtration using a 0.45 $\mu$ m membrane filter.                                                                                                                                                    |                                           |                    |
| 1.2  | Type of sample             | Natural and polluted waters and some effluents, but not sea water or waters high in both magnesium and chloride, but see Section 10.                                                                                                                                                |                                           |                    |
| 1.3  | Basis of the method        | Filtration through a membrane filter. The weight of dissolved matter in the filtrate is determined by evaporation, drying at 180°C and weighing. (See also Section 10).                                                                                                             |                                           |                    |
| 1.4  | Range of application       | The lower limit is dependent on the sensitivity of the balance available. There is no real upper limit.                                                                                                                                                                             |                                           |                    |
| 1.5  | Standard deviation (a)     | Total dissolved solids 180°C<br>mg/l                                                                                                                                                                                                                                                | Standard deviation (within batch)<br>mg/l | Degrees of freedom |
|      |                            | 569                                                                                                                                                                                                                                                                                 | 5.32                                      | 9                  |
|      |                            | 343                                                                                                                                                                                                                                                                                 | 5.54                                      | 9                  |
|      |                            | 202                                                                                                                                                                                                                                                                                 | 2.89                                      | 9                  |
| 1.6  | Limit of detection         | As written, about 10 mg/l, but is balance dependent.                                                                                                                                                                                                                                |                                           |                    |
| 1.7  | Sensitivity                | For a 100 ml aliquot each 10 mg/l of total dissolved solids will correspond to a weight change of 1 mg.                                                                                                                                                                             |                                           |                    |
| 1.8  | Bias                       | Not determined, but from a consideration of the method is likely to be slightly negative and sample dependent, but may be slightly positive if the sample contains salts which are hygroscopic. Some Organic Compounds may char, some compounds (eg ammonium salts) may volatilize. |                                           |                    |
| 1.9  | Interference               | No specific tests have been made, but waters containing bicarbonate content may require prolonged drying at 180°C to ensure the complete conversion of bicarbonate to carbonate.                                                                                                    |                                           |                    |
| 1.10 | Time required for analysis | Depends on type of sample, typically within one day.                                                                                                                                                                                                                                |                                           |                    |

(a) Data from Mid Kent Water Co

## TDS2 Scope and field of application

- 2.1 The method determines non volatile organic and/or inorganic matter which is not removed from water by filtration through a 0.45  $\mu$ m pore size membrane filter.
- 2.2 The method is applicable to natural and polluted waters and some effluents.

2.3 The filtrate from the determination of suspended matter (membrane filtration method) may be used for the determination of total dissolved solids. (See Section 8 note b).

**TDS3 Principle** Suspended matter is removed from a measured volume of sample by filtration, under reduced pressure.

The filtrate is then evaporated to dryness on a steam bath, dried at 180°C and then total dissolved solids determined gravimetrically.

**TDS4 Interferences** Samples containing bicarbonates and/or hygroscopic salts.

**TDS5 Hazards** 5.1 Reduced pressure filtration requires the use of a safety screen.

**TDS6 Reagents** 6.1 Water, deionized or freshly distilled water is suitable.

**TDS7 Apparatus**

7.1 Cellulose ester membrane filters 47mm diameter, 0.45 μm effective pore size.

7.2 Blunt forceps for handling membrane filters.

7.3 A Hartley pattern filtration apparatus consisting of a detachable filter funnel incorporating a support plate for the membrane filter.

7.4 Buchner flask 500-ml capacity.

7.5 250-ml measuring cylinder.

7.6 Drying oven controlled at 180 ± 2°C.

7.7 Desiccator containing self indicating silica gel.

7.8 Analytical balance capable of weighing to 0.1 mg.

7.9 Mechanical or water suction pump to give reduced pressure (2.6-3.3 kPa).

7.10 Steam bath with suitable supports for evaporating dishes.

7.11 Platinum, glass or porcelain evaporating dishes of 150-ml capacity. Platinum dishes should not be used if phosphates are likely to be present in the sample. (For notes on the care of platinum ware, see *Silicon in Waters and Effluents 1980 p24*, in this series).

**TDS8 Analytical procedure** Safety precaution — reduced pressure filtration must be carried out behind a safety screen

| Step | Procedure                                                                                                                                                                                                                                                                             | Notes                                                                                                                                                                                                                                                                                                                                                                |
|------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 8.1  | Place the membrane filter on the support plate of the filter unit, moisten with distilled water and assemble the top of the unit. Wash the membrane with 3 × 50 ml distilled water applying gentle vacuum between each wash to remove the water. Discard the filtrate (note a and b). | <p>(a) Measurement of specific conductivity may be used as a guide to the approximate value of the total dissolved solids. However the correct conversion factor for the type of water under test must be found by experiment.</p> <p>(b) If the filtrate from the determination of suspended matter (membrane filtration method) is used proceed from step 8.4.</p> |

| Step | Procedure                                                                                                                                                                                                               | Notes                                                                                                                                                                                                                                                                                                                                                                                                                                                |
|------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 8.2  | Filter a suitable volume of well mixed sample such that the filtrate does not contain more than 200 mg dissolved solids (notes a and c).<br><br>Record the volume of sample filtered. Let this be A ml.                 | (c) Excessive residue in the evaporating dish may form a water entrapping crust.                                                                                                                                                                                                                                                                                                                                                                     |
| 8.3  | After filtration of the sample wash the membrane filter with 3 × 10 ml water to remove dissolved solids which may be entrained on the filter. Add the washings to the sample filtrate.                                  |                                                                                                                                                                                                                                                                                                                                                                                                                                                      |
| 8.4  | Dry a clean evaporating dish at 180 ± 2°C for one hour, cool in a desiccator and weigh to nearest 0.1 mg. Let this weight be B gm.                                                                                      |                                                                                                                                                                                                                                                                                                                                                                                                                                                      |
| 8.5  | Transfer quantitatively the combined filtrate and washings to the evaporating dish and evaporate to dryness on a steam bath or on a sand bath with overhead infra red heating.                                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                      |
| 8.6  | When all the water has evaporated from sample filtrate transfer the evaporating dish to the drying oven and dry to constant weight (notes d and e) at 180 ± 2°C. Cool in a desiccator and weigh. Let this mass be C gm. | (d) Constant weight is attained when the weights after successive drying and weighing cycles differ by 0.5 mg or less.<br>(If it can be determined by experience that, for certain types of sample, constant weight is attained after two drying and weighing cycles, only one cycle is required).<br>(e) Samples containing bicarbonate or hygroscopic salts will require prolonged drying and rapid weighing after cooling to ambient temperature. |

**TDS9 Calculation of results** Total dissolved solids dried at 180°C =  $\frac{C-B}{A} \times 1,000,000$  mg/l

where C = Weight of dried residue and dish in gm  
 B = Weight of dish in gm  
 A = Volume of filtered sample in ml.

**TDS10 Variants on the method**

10.1 Variations in Ignition or Drying Temperature

For some purposes samples are dried at 105°C, or ignited at 450°C or 500°C to constant weight instead of at 180°C. If one of these variants is used, this should be stated along with the result.

10.2 Sea and brackish water samples containing high concentrations of both magnesium and chloride

These hydrolyse with loss of hydrogen chloride on ignition forming magnesium hydroxychloride. For a suitable procedure see Morris A W and Riley J P, *Deep Sea Research* 1964. 11. 899.

# Address for Correspondence

No matter how thoroughly a method may have been tested there is always the possibility of users discovering a hitherto unreported problem. The Accuracy and Precision data on several of the methods in this booklet are not up to the usual standards desired by the committee. Users with problems or test data are asked to write to:

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