

Methods of test for

Filter papers

Committees responsible for this British Standard

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- British Paper and Board Industry Federation
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- Motor Industry Research Association

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Foreword

This British Standard has been prepared under the direction of the Paper and Printing Standards Policy Committee. It revises clauses **3, 7, 8, 12, 14, 17, 20, 21, 23, 25, 29** and **32** and Appendix A and Table 1 of the 1984 edition and adds methods for the determination of acid solubility. Figure 2 and Figure 9 of the 1984 edition have also been revised and new Figure 5, Figure 13, Figure 14 and Figure 15 added. It supersedes BS 6410:1984, which is withdrawn.

The methods described in this standard do not cover all the desired fields and further methods may be added to future revisions of this standard.

Appropriate precautions are necessary to ensure the safety of personnel carrying out the tests in this standard. In particular, it is essential that suitable safety precautions are taken when curing resin-treated filter papers.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations. In particular attention is drawn to the provisions of the Health and Safety at Work etc. Act 1974.

Summary of pages

This document comprises a front cover, an inside front cover, pages i to iv, pages 1 to 32, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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Section 1. General

1 Scope

This British Standard describes methods of test for filter papers made by the papermaking process.

NOTE 1 Some of the test methods may also apply to filter media made by other processes.

NOTE 2 Appendix A gives further information on the types of filter paper to which these test methods are applicable.

NOTE 3 The titles of the publications referred to in this standard are listed on page 32.

2 Definitions

For the purposes of this British Standard, the following definitions apply.

2.1

filter paper

a paper, made by a papermaking process, of organic and/or inorganic fibrous composition intended to provide selective retention of solid particles or liquid droplets from a fluid suspension

2.2

corrugation depth

the difference between the thickness measured across the corrugations and that measured in the valleys of the corrugations

2.3

grammage

the mass of a unit area of paper (see clause 6)

2.4

total resin content

the difference, expressed in g/m^2 , between the oven dry grammages of an unimpregnated sample and an impregnated sample

2.5

extractable resin content

the quantity of resin, expressed in g/m^2 , that can be extracted from the treated paper by a solvent

2.6

equivalent pore diameter

the diameter of a capillary of constant circular cross section that requires the same air pressure to force a wetting liquid from it as from the paper pore

2.7

maximum equivalent pore diameter

the equivalent pore diameter at which air just starts to flow through the filter paper under the conditions defined in the method of test (see clause 14)

2.8

minimum equivalent pore diameter

the equivalent pore diameter at which all liquid has just been removed from the smallest pores

2.9

tensile strength

the maximum tensile force that a test piece will withstand before it breaks under the conditions defined in the method of test (see clause 18)

2.10

tearing resistance

the mean force required to continue the tearing of an initial cut in a single sheet of paper

2.11

bursting strength

the maximum uniformly distributed pressure, applied at right angles to its surface, that a test piece will withstand before it fails under the conditions defined in the method of test (see clause 20)

2.12

flexibility

the downward deflection under load, expressed in mm/g, of a test strip supported as described in the method of test (see 23.2)

2.13

brittleness

the percentage loss in tensile strength after creasing

2.14

air flow resistance

the resistance offered by a paper to the passage of air

2.15

media migration

the contamination of a filtrate by particles or fibres shed by the filter paper during use

2.16

water resistance

the hydrostatic head supported by a filter paper at which penetration of water first occurs

2.17

water filtration time

the time, in s, for 100 cm^3 of clean, prefiltered, de-aerated water to pass through the filter under a constant hydrostatic head

2.18

choking life

the time after which a filter fails to perform at an acceptable filtration rate and/or pressure differential

2.19
flammability

the ability of a filter paper to retard the spread of flame when ignited and to be self-extinguishing when the source of ignition is removed

2.20
afterflame time (flaming time)

the length of time for which the test piece continues to flame under specified test conditions after the burner flame has been removed

2.21
afterglow time (glowing time)

the length of time for which the test piece continues to glow under specified test conditions after it has ceased to flame

2.22
retentivity to particles in liquids

the particle size, in μm , at which a retention level of 98 % of the total number of particles challenging the filter paper is obtained

3 Sampling and pretreatment of test pieces

3.1 Sampling

Sampling shall be in accordance with BS 3430, except where modified by individual testing methods.

3.2 Pretreatment of test pieces

WARNING. Some resin-treated filter papers will evolve toxic or inflammable fumes during curing. It is essential that suitable safety precautions are taken.

Unless otherwise specified in a particular test method, the resin in resin-treated filter papers shall be cured before commencing the test.

NOTE Curing may be carried out by exposure to high temperature for a period, e.g. 150 °C for 10 min, or to high humidity or to ultraviolet light. The curing method used depends on the resin involved.

4 Conditioning of test pieces

Where specified in the test method, test pieces shall be conditioned at 50 ± 2 % r.h. and a temperature of 23 ± 1 °C, in accordance with BS 3431.

5 Test report

In addition to the test reports required by individual methods, the following shall be reported:

- a) precise identification of the samples;
- b) pretreatment conditions used;
- c) conditioning atmosphere used;
- d) reference to the clauses of this British Standard detailing the test methods applied;
- e) the mean results of the applied tests;
- f) if required, the range or standard deviation of results of the applied tests;
- g) details of any departure from the method of test and any circumstances or influences that may have affected the results;
- h) details of any deviations from the sampling procedure of 3.1.

Section 2. Test methods for general and structural properties

6 Grammage

Use the method of test described in BS 3432, except that for test pieces taken from narrow coils, instead of using the nominal width, measure the width to an accuracy of $\pm 1\%$.

NOTE 1 The test piece may be in the “as taken”, “after conditioning” (see clause 4) or “oven-dry” (see note 2) condition, and the condition should be reported.

NOTE 2 If the filter paper has been treated with a resin, it is recommended that the grammage (see 2.3) be measured in the oven-dry condition after curing the resin (see 3.2). Other times and temperatures may be agreed between the parties concerned.

7 Thickness

Use the method of test described in BS 3983 but, because filter papers are more compressible than other papers, reduce the applied load or increase the test area.

An applied load of 100 kPa or 50 kPa may cause excessive compression of the test piece. For low-density filter papers, especially those of types 3 and 4 (see Appendix A), it is recommended that an applied pressure of 10 ± 1 kPa over an area of 200 mm² is used. Other loads or test areas may be used, as agreed between the parties concerned.

NOTE 1 The use of grammage and thickness to calculate apparent density may not be reliable for filter papers because of their more compressible nature.

NOTE 2 The precision micrometer may be of the hand-operated mechanical type or the automatic digital readout type, but it may be difficult to obtain consistent loading conditions with the hand-operated type.

8 Corrugation depth

8.1 Principle

The thickness across the corrugations and that in the valleys of the corrugations is measured and the depth of the corrugations (see 2.2) is calculated.

8.2 Apparatus

8.2.1 *Precision micrometer*, in accordance with BS 907. The micrometer shall be fitted with a pressure foot as shown in Figure 1 or Figure 2 and shall have a maximum plunger load of 50 g.

8.3 Procedure

Cure resin-treated filter papers before commencing the test (see 3.2). Measure across the tops of the corrugations at a minimum of 10 positions wire side up and 10 positions wire side down. Repeat the measurements in the valleys of the corrugations at positions along a line parallel to the first set of measurements.

8.4 Calculation of results

Calculate the corrugation depth by taking the difference between the mean values of the two sets of measurements.

9 Moisture (volatile) content

Use the method of test described in BS 3433, but, for resin-treated filter papers, use a temperature of 105 ± 2 °C or 150 ± 5 °C.

NOTE This method does not distinguish between water and any other matter that may be volatile at the drying temperature.

10 Ash

WARNING. The combustion of some kinds of fibre and resins produces toxic fumes. It is essential that suitable safety precautions are taken.

Use the method of test described in BS 3631, but vary the ashing temperature from that given in that standard.

NOTE 1 An ashing temperature of 900 ± 25 °C is recommended, but another temperature may be used if appropriate and should be reported.

Condition each test piece in accordance with clause 4 of this standard. Cure resin-treated filter papers in accordance with 3.2 of this standard.

NOTE 2 The results obtained will include any residue from impregnants.

11 Ignition loss

WARNING. The combustion of some kinds of fibre and resins produces toxic fumes. It is essential that suitable safety precautions are taken.

Use the method of test described in BS 3631 to determine the ignition loss of filter papers containing a substantial proportion of inorganic fibres, but reduce the furnace temperature to 450 ± 25 °C. Report the ignition loss as a percentage of the mass of the test piece.

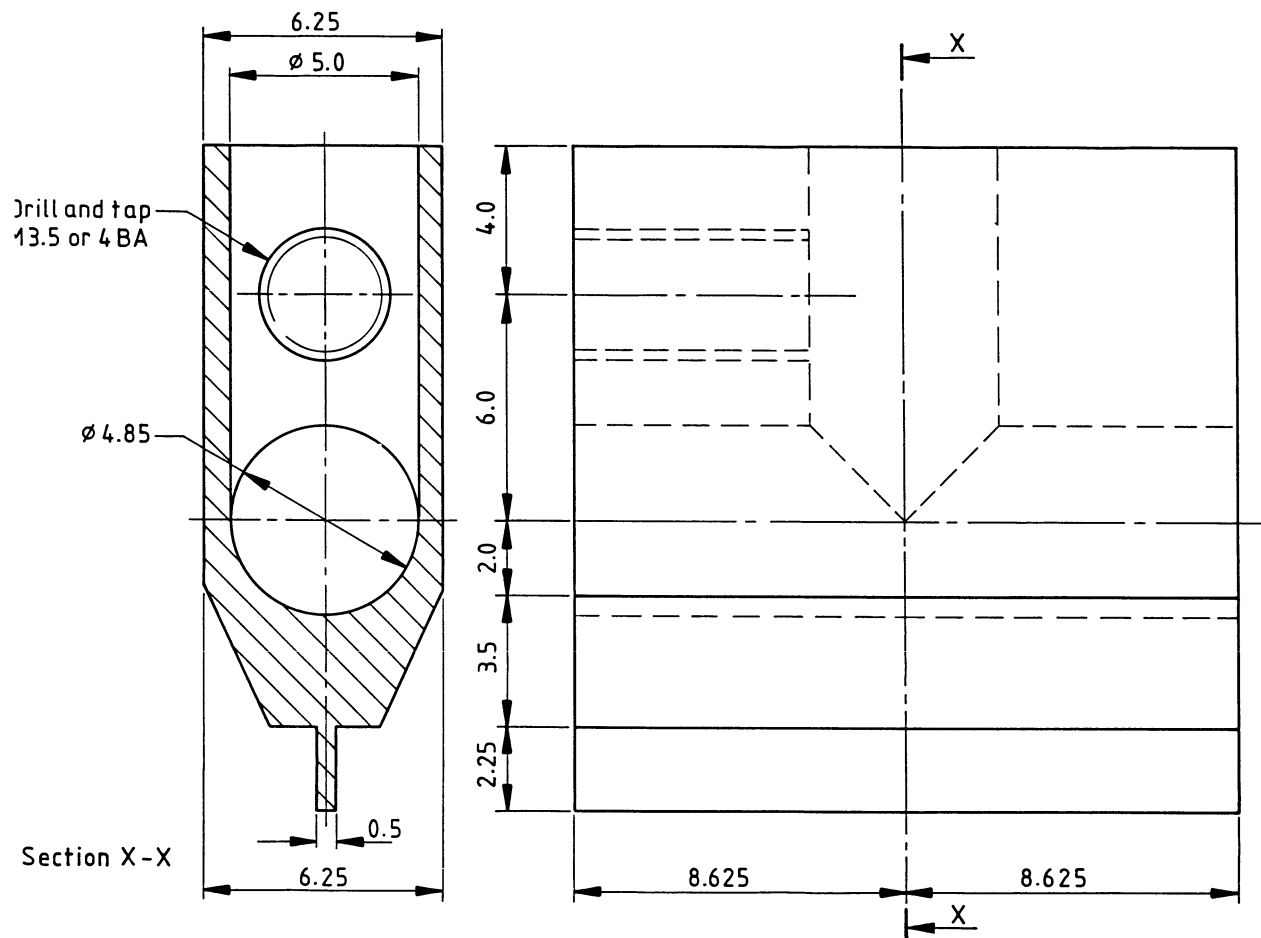
12 Resin content

WARNING. It is essential that suitable safety precautions are taken when working with solvents and resin systems. In particular, the following points should be observed:

- solvent extraction should be carried out in a fume cupboard;
- solvent trays should be covered;
- excess solvent should be blotted off test pieces before they are transferred to the oven;
- a flameproof oven should be used.

12.1 General

These methods apply to filter papers treated with substantial quantities of thermoplastic or thermosetting resins, e.g. a phenol formaldehyde resin.



All dimensions are in millimetres.

Figure 1 — Pressure foot for micrometer

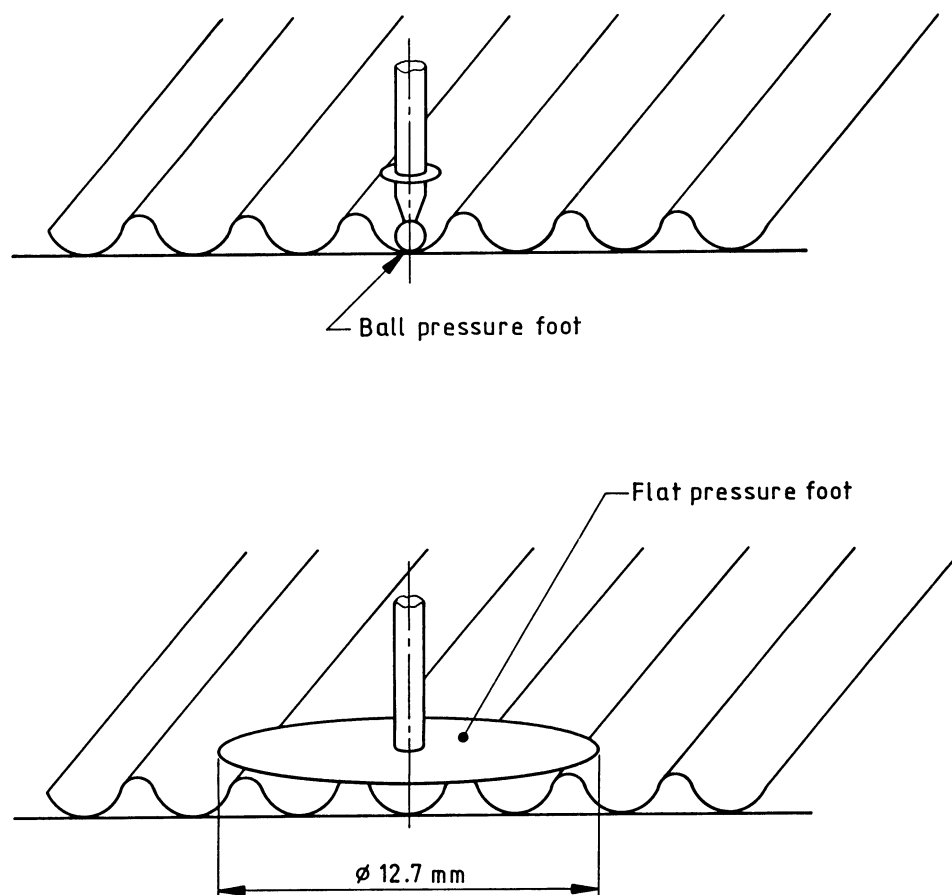


Figure 2 — Alternative pressure foot types for micrometer

NOTE Certain types of filter papers are treated during manufacture to increase their wet strength by the addition of a small quantity of a melamine formaldehyde resin, a polyamide resin or some similar material. If nitrogenous, these agents may be detected and estimated by the procedures given in BS 4497.

12.2 Total resin content

12.2.1 Principle

The oven-dry mass of test pieces of unimpregnated and impregnated filter paper is measured and the total resin content (see 2.4) is calculated.

NOTE The procedure may only be applied if representative samples of both the impregnated and unimpregnated filter paper are available.

12.2.2 Apparatus

12.2.2.1 Oven, flameproof and capable of maintaining a temperature of $150 \pm 5^\circ\text{C}$.

12.2.2.2 Weighing device, capable of weighing to an accuracy of $\pm 0.01\text{ g}$.

12.2.3 Procedure

Dry test pieces of impregnated and unimpregnated paper of known area at $150 \pm 5^\circ\text{C}$ in an oven until constant mass is achieved.

Repeat the procedure with a second set of test pieces of impregnated and unimpregnated paper.

12.2.4 Calculation of results

Take the total resin content (in g/m^2) as the difference between the grammage of the impregnated test pieces and the grammage of the unimpregnated test pieces. Calculate the mean of the results.

12.3 Extractable resin content

NOTE Two methods are given here for the determination of extractable resin content (see 2.5). Whilst the method using the Soxhlet apparatus will tend to give higher results, the rapid method may be more suitable for online quality assurance testing where results are required quickly.

12.3.1 Principle

The test piece is treated with a solvent and the quantity of resin that can be extracted is measured.

12.3.2 Apparatus and reagents

12.3.2.1 Soxhlet method

The apparatus shall comprise the following.

- Soxhlet apparatus*, in accordance with BS 2071.
- Acetone*.
- Air stream*.
- Oven*, flameproof and capable of maintaining a temperature of 150 ± 5 °C.
- Weighing device*, capable of weighing to an accuracy of ± 0.005 g.

12.3.2.2 Rapid method

The apparatus shall comprise the following.

- Weighing device*, capable of weighing to an accuracy of ± 0.005 g.
- Methanol*, or the solvent combinations used in the resin system.
- Trays*.
- Timing device*, capable of timing to an accuracy of ± 0.5 min.
- Oven*, flameproof and capable of maintaining a temperature of 150 ± 5 °C.
- Blotting paper*.

12.3.3 Procedure

12.3.3.1 Soxhlet method

Divide a number of test pieces of known area into two sets, A and B. Extract set A in a Soxhlet apparatus with acetone for 4 h at a rate of approximately 10 cycles/h. Expose the extracted test pieces to an air stream to remove the acetone left in the paper. Heat the two sets of test pieces for 10 min at 150 ± 5 °C and weigh each set to the nearest 0.01 g.

Repeat the procedure with two further sets of test pieces.

12.3.3.2 Rapid method

Divide a number of test pieces into two sets, A and B, each not less than 5 g in mass. Weigh both sets to the nearest 0.01 g. Wash set A in methanol or the solvent combinations used in the resin system, using three trays of solvent and for a total time of 10 min. Wash the test pieces in the first tray for 9 min, turning at intervals. Wash the test pieces in the second tray to remove excess solvent/resin and wash in clean solvent in the third tray. Blot the test pieces to remove excess solvent and dry at 150 ± 5 °C for 10 min. Dry set B at 150 ± 5 °C for 10 min. Weigh both sets of test pieces to the nearest 0.01 g.

Repeat the procedure with two further sets of test pieces.

12.3.4 Calculation of results

12.3.4.1 Soxhlet method

Take the extractable resin content (in g/m²) as the difference between the grammage of set A and the grammage of set B.

Calculate the mean of the results.

NOTE The extractable resin content determined by the Soxhlet method will usually be lower than the total resin content by an amount that depends upon the past history of the paper and the type of resin treatment used. However, in exceptional circumstances it may be higher.

12.3.4.2 Rapid method

Calculate the extractable resin content, *ER*, expressed as a percentage, from the following equation:

$$ER = \frac{A_1 B_2 - A_2 B_1}{A_1 B_2} \times 100 \quad (1)$$

where

- ER* is the extractable resin content;
- A*₁ is the mass of set A before extraction (in g);
- A*₂ is the mass of set A after extraction and drying (in g);
- B*₁ is the mass of set B before drying (in g);
- B*₂ is the mass of set B after drying (in g).

Calculate the mean of the results.

NOTE 1 The results may also be expressed in g/m² if the area of the test pieces is determined.

NOTE 2 The rapid method of determining extractable resin content will tend to give lower results than the Soxhlet method.

12.3.5 Test report

The test report shall include a statement giving the method used to measure the extractable resin content.

13 pH value, conductivity, chloride and sulphate contents of aqueous extracts

Use the methods of test described in BS 2924.

14 Pore size

14.1 General

The test determines the size of pores (mean, maximum and equivalent pore diameters, see 2.6, 2.7 and 2.8) that allow the passage of a fluid from one side of the filter paper to the other, designated "flow pores". Non-interconnecting pores are not determined by this method.

NOTE 1 It should be appreciated that these methods equate the normally tortuous pore structures of fibrous filter matrices with hypothetical capillaries having limiting diameters. The results from these methods allow the characterization and subsequent comparison of filter materials on the basis of pore size but should not be assumed to directly reflect particle retention levels. Other factors such as fibre type, filter depth and particle shape also affect retention efficiency, the quantification of which is best carried out by direct testing using a particle challenge technique. Another method for determination of pore size uses a mercury porosimetric technique which also determines non-interconnecting pores, but this method is not necessarily suitable for filter papers.

NOTE 2 Three procedures are given for the measurement of maximum equivalent pore diameter (see 2.7) but, due to differences between the two instruments, results may not be exactly comparable.

NOTE 3 Care should be taken when comparing the results of this test using non-aqueous liquids with the results of water-based tests. Papers with the same maximum equivalent pore diameter may swell differently in water and so have differing aqueous properties.

NOTE 4 The test piece is not normally supported on the low pressure side during the test, consequently the pressure differential tends to dome or distort the test piece. If supported, the pressure differential will cause the test piece to be compressed. Users therefore should satisfy themselves that the effects do not invalidate the results.

This problem is quite general with very porous filter paper but is particularly so when dealing with very compressible papers made of fine glass fibres or similar.

14.2 Maximum equivalent pore diameter

14.2.1 Principle

A test piece is clamped horizontally and a suitable liquid is applied to its upper surface so that the test piece is completely wetted. Increasing air pressure is applied below the test piece until an air bubble is forced through the interstices of the paper. The air pressure at this moment, together with the known surface tension of the liquid, is used to estimate the diameter of the largest equivalent capillary.

14.2.2 Apparatus and reagents

The apparatus shall be in accordance with BS 3321. The test piece shall remain flat and undistorted. Where necessary, a support (e.g. a 10 mm mesh) shall be applied to the upper side of the test piece.

A test liquid of known surface tension shall be used.

NOTE The test liquid should wet the filter paper thoroughly without interfering with its fibrous structure. The surface tension and volatility of the test liquid should be low. An example of a suitable test liquid is kerosine. Reference should be made to manufacturers' literature regarding any health and safety instructions appropriate to the test liquid.

14.2.3 Preparation of test pieces

The test pieces shall be conditioned in accordance with clause 4 of this standard.

14.2.4 Procedure

Use the procedure described in BS 3321, but record the gauge pressure when the first bubble (excluding peripheral bubbles) appears on the paper surface.

14.2.5 Calculation of results

Calculate the maximum equivalent pore diameter, d (in μm), from the following equation:

$$d = \frac{4\sigma}{P} \quad (2)$$

where

σ is the surface tension of the test liquid at the test temperature (in mN/m);

P is the air pressure (in kPa).

For any specified test liquid, used at a constant temperature,

$$d = \frac{\text{a constant}}{P} \quad (3)$$

14.3 Mean flow, maximum and minimum equivalent pore diameters

14.3.1 Manually controlled apparatus

14.3.1.1 Principle

A filter paper which has been completely saturated with a liquid of known surface tension will allow the passage of air when the air pressure exceeds the capillary attraction of the liquid contained in the largest pores. Smaller pores will exhibit the same behaviour at progressively higher pressures until all liquid is removed from the pores.

The pressure at which air just starts to flow through the filter is recorded and the maximum equivalent pore diameter is determined.

Graphs of air flow rate versus air pressure are compared for the test piece in the saturated and dry condition and the mean flow and minimum equivalent pore diameters (see 2.8) are calculated.

NOTE The principle is based on pore size determinations described in ASTM F316-80. Whilst it is possible to calculate the percentage pore size distribution from the data generated in this method, it is normally sufficient to characterize the filter in terms of mean flow, maximum and minimum equivalent pore diameters.

14.3.1.2 Apparatus and reagents

14.3.1.2.1 Pore size apparatus, shown in Figure 3.

14.3.1.2.2 Vacuum pump

14.3.1.2.3 Vacuum vessel

14.3.1.2.4 Test liquid, of known surface tension.

NOTE The test liquid should wet the test piece thoroughly without interfering with its fibrous structure. The surface tension and volatility of the test liquid should be low. An example of a suitable test liquid is kerosine. Reference should be made to manufacturers' literature regarding any health and safety instructions appropriate to the test liquid.

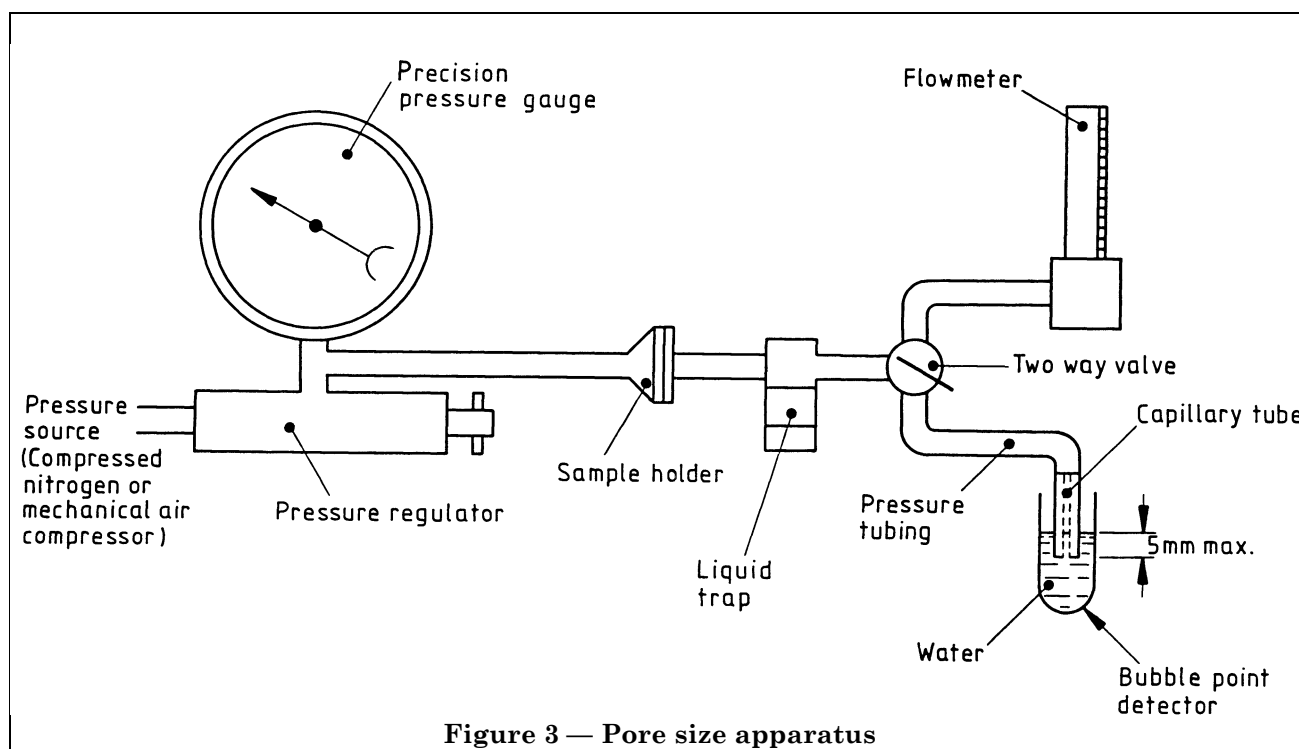


Figure 3 — Pore size apparatus

14.3.1.3 Preparation of test pieces

The test pieces shall be subjected to test either in the “as received” condition or after conditioning in accordance with clause 4 of this standard.

14.3.1.4 Procedure

Place the test piece in the “in-line” sample holder, secure the holder and slowly apply air pressure via the pressure regulator.

Plot air flow versus air pressure. Estimate the pressure range to be used by estimating the minimum and maximum equivalent pore diameters and substituting into equation (2) in 14.2.5.

Reduce the air pressure, remove the test piece and saturate it, under vacuum, in a suitable test liquid. Place the saturated test piece in the sample holder, secure the holder and slowly apply air pressure via the pressure regulator. Observe the bubble point detector and record the pressure at which initial air flow occurs, indicated by a stream of bubbles.

Change to the flowmeter for air flow rate measurement and plot air flow versus air pressure on the same graph as for the dry test piece.

Continue the test until the air flow rate versus air pressure curve for the saturated test piece is coincident with the curve for the dry test piece.

14.3.1.5 Calculation of results

Calculate the mean flow, maximum and minimum equivalent pore diameter, d (in μm) from equation (2) (see 14.2.5) or equation (4) (see note). For the maximum equivalent pore diameter, use the value of pressure at which initial air flow occurred. For the mean flow equivalent pore diameter, use the value of pressure from the graph at which saturated filter flow is one-half of dry filter flow. For the minimum equivalent pore diameter, use the value of pressure from the graph at the meeting point of the plot for the saturated test piece with that for the dry test piece.

NOTE Equation (2) (see 14.2.5) assumes complete “wetting” of the filter matrix by the test liquid. If the liquid does not completely wet the filter matrix, equation (2) is modified to equation (4):

$$d = \frac{4\sigma\cos\theta}{P} \quad (4)$$

where

θ is the contact angle between the test liquid and the test piece (in degrees).

14.3.1.6 Test report

The test report shall include a statement giving the condition of the test piece (“as received” or “after conditioning”) at the time of test.

14.3.2 Automated apparatus

14.3.2.1 Principle

The test piece is saturated with the test fluid and placed in an appropriate holder. A steadily increasing pressure differential is applied to the test piece and the corresponding air flow is monitored. When all fluid has been expelled, the pressure differential is automatically reduced to zero and the cycle repeated. (This is regarded as the dry cycle). The pore size distribution and other parameters are computed.

14.3.2.2 Apparatus and reagents

14.3.2.2.1 Automated liquid porosimeter¹⁾, of suitable range with operating manual.

NOTE Commercially available equipment can measure pore diameters within the range 0.05 μm to 300 μm .

14.3.2.2.2 Test liquid¹⁾, of low, known surface tension.

NOTE The test liquid should wet the test piece thoroughly without interfering with its structure. The volatility should be low. Some fluorocarbons are examples of suitable test liquids.

14.3.2.2.3 Clean dry compressed air, at pressures up to 1 000 kPa and flows of up to 100 L/min.

14.3.2.2.4 Metal punch, or other means to cut circular test pieces.

14.3.2.2.5 Test piece holders

14.3.2.3 Procedure

Switch on the equipment and allow it to stabilize for about 20 min. Set up the equipment as detailed in the operating manual so as to provide the type of results required.

NOTE The equipment can compute the maximum, minimum and mean flow pore sizes as well as pore size distributions by flow, number or volume.

Cut circular test pieces of the required size and saturate them with the test liquid in accordance with the instructions in the operating manual. Carry out the test according to the instructions in the manual.

14.3.2.4 Calculation of results

The equipment will calculate the requested parameters and distributions before displaying the results. Any or all of the raw data or computed values or distributions can be printed out if required.

15 Alkali solubility

Use the method of test described in BS 4499.

NOTE This method does not differentiate between other soluble fractions, such as beta cellulose and gamma cellulose.

16 Acid solubility

16.1 The filter paper is exposed to sulphuric acid of specified specific gravity for a specified time under specified conditions and the loss in mass calculated.

16.2 Reflux method

16.2.1 Apparatus and reagent

16.2.1.1 Balance, with a sensitivity of 0.001 g.

16.2.1.2 Oven, capable of maintaining a temperature of 115 ± 10 °C.

16.2.1.3 Erlenmeyer flasks, (borosilicate glass), of 250 mL capacity and fitted with 24/40 ground glass joints.

16.2.1.4 Reflux condensers, fitted with 24/40 ground glass joints.

16.2.1.5 Hot-plate magnetic stirrers, with teflon-coated stirring bars.

16.2.1.6 Sintered glass crucibles, (about 5 μm opening), and 75 mm pyrex watch glasses.

16.2.1.7 Filter flask, (Buchner flask), fitted to receive the sintered glass crucible.

16.2.1.8 Desiccator, and desiccant.

16.2.1.9 Spectrograde sulphuric acid, $c(\text{H}_2\text{SO}_4) = 4.5 \text{ mol/L}$.²⁾

16.2.2 Procedure

Dry three samples, each of about 1.0 g, in tared containers for 30 min in the oven at 115 ± 10 °C. Place the containers plus samples in a desiccator until cool and weigh accurately to the nearest 0.001 g (mass A).

Remove the samples and shred into separate Erlenmeyer flasks. Add a stirring bar to each flask and to each add 100 mL of 1.26 specific gravity spectrograde sulphuric acid. Place on the hot plates and connect the reflux condensers. Adjust the temperature and stirring speed to obtain a reflux rate of 1 drop/s. Reflux for 3 h.

Allow to cool and vacuum filter the suspension through tared sintered glass crucibles.

NOTE The filtrate may be used for the analysis of the acid extractable impurities, anions or cations, if required.

Rinse the contents of each crucible with 1 L of distilled water and dry in the oven for 1 h at 115 ± 10 °C. Place the crucibles in the desiccator until cool and weigh to the nearest 0.001 g. Repeat the washing and drying procedure until a constant mass is obtained. Deduct the mass of the crucible to obtain the mass of the residue (mass B).

¹⁾ Details of suitable sources of supply may be obtained from BSI Enquiry Section, Linford Wood, Milton Keynes, MK14 6LE.

²⁾ Of specific gravity 1.26.

16.2.3 Calculation and report

Calculate the acid solubility, S , as follows:

$$S = \frac{100(A - B)}{A} \quad (5)$$

Report the acid solubility as the mean percentage mass loss to the first decimal place. Report also the range and standard deviation of the results.

16.3 Immersion method**16.3.1 General**

This method is intended primarily for papers manufactured from glass fibres.

16.3.2 Apparatus and reagent

16.3.2.1 Balance, with a sensitivity of 0.001 g.

16.3.2.2 Oven, capable of maintaining a temperature of $115 \pm 10^\circ\text{C}$.

16.3.2.3 Oven, or water bath maintained at 59°C to 60°C .

16.3.2.4 Reagent bottles, (borosilicate glass), of 250 mL capacity and fitted with polypropylene caps.

16.3.2.5 Filter funnel, and filter papers (Whatman 42, 542 or equivalent).

16.3.2.6 Pair of rollers, hand operated and specially coated.

16.3.2.7 Sulphuric acid, $c(\text{H}_2\text{SO}_4) = 4.7 \text{ mol/L}$ to 5.0 mol/L .³⁾

16.3.2.8 Muffle furnace, at a temperature of 500°C .

16.3.2.9 Desiccator, and desiccant.

16.3.3 Procedure

Take a sample of about 15 g to 20 g of paper. Ensuring that the rollers are clean and set sufficiently close together, feed the paper through the rollers so that the structure of the paper is destroyed.

NOTE If the paper does not become extremely weak the rollers need to be set closer together.

Dry the sample at $115 \pm 10^\circ\text{C}$ for 30 min and then weigh accurately to 0.001 g two test portions each of about 5 g (mass A).

With appropriate precautions put 150 mL portions of the sulphuric acid into each of two 250 mL bottles. Cap the bottles loosely and place in the oven or water bath at 59°C to 60°C until the bottle and contents achieve equilibrium.

Remove the bottles and to each add the previously weighed test portions of 5 g, taking care not to lose any of the test portion in the process. Recap the bottles tightly and shake well. Loosen the cap and replace the bottles in the oven or water bath for a period of 72 h, shaking periodically.

After the immersion period has been completed remove the bottles from the oven or water bath, loosen the caps and allow to cool.

Assemble a filter funnel, filter paper and a clean glass bottle. Tighten the cap on one of the bottles containing a test portion, shake vigorously and immediately transfer the contents onto the filter paper. Rinse the original bottle several times with distilled water so as to transfer all the fibres to the filter paper. Allow to drain.

NOTE The filtrate may be used for the analysis of the acid extractable impurities, if required.

Wash the filter paper and its contents with distilled water to remove all traces of acid. Up to 1 L of water may be required to accomplish this. Allow to drain.

NOTE Clean tap water may be used for this washing process in place of distilled water.

Transfer the filter paper and its contents to a tared silica crucible. Dry off in the oven at $115 \pm 10^\circ\text{C}$ and then ignite in a muffle furnace at 500°C for 30 min. Cool in the desiccator and weigh to the nearest 0.001 g. Deduct the mass of the crucible to obtain the mass of the residue (mass B).

Repeat the filtration, washing and incineration process with the other test portion.

16.3.4 Calculation and report

Calculate the acid solubility as in **16.2.3**.

Report the acid solubility as the mean percentage mass loss to the first decimal place. Report also the range of the results.

17 Calcium, copper, iron and manganese content

Use the methods of test described in BS 4897-1 to BS 4897-4.

NOTE 1 In addition to estimating impurities in the raw materials used to manufacture the filter paper, this method also estimates impurities introduced into the filter paper before, during and after manufacture.

NOTE 2 For cellulose fibre-based filter paper, the level of impurities may also be assessed from the following:

- the difference between total fibre content and the fraction determined by the alkali solubility of the paper (see clause 15);
- analysis for trace quantities of cations in an acid solution of the residue from ignition at $900 \pm 25^\circ\text{C}$ (see clause 10).

³⁾ Of specific gravity 1.27 to 1.29.

Section 3. Test methods for mechanical properties

18 Tensile strength

Use the method of test described in BS 4415-2.

NOTE In the case of coils of width less than 45 mm it is not possible to avoid the 15 mm within the edge of the sample. In this case report both the test piece width and the coil width.

19 Tearing resistance

Use the method of test described in BS 4468.

20 Bursting strength

Use the method of test described in BS 3137.

21 Wet tensile strength

Use the method of test described in BS 2922-2.

22 Wet bursting strength

22.1 General

The water contact method is applicable to most filter papers, but particularly to those with very low wet strength. The diaphragm method is applicable to heavier and stronger filter papers and is imprecise at low readings because of the imperfect flexibility of the diaphragm.

22.2 Diaphragm method

Use the method of test described in BS 2922-1, using distilled or deionized water for immersion of the test pieces. Carry out preliminary tests to determine the immersion time, which is the time after which no further reduction in bursting strength occurs.

22.3 Water contact method

22.3.1 Principle

The dry test piece is clamped horizontally and water is applied directly to its lower surface at a controlled pressure. The pressure is increased until failure occurs.

22.3.2 Apparatus

The apparatus shall be as given in Figure 4. It shall be constructed so that a circular area of the test piece of diameter 30.50 ± 0.05 mm is exposed to the pressure.

22.3.3 Procedure

If the test piece cannot be readily saturated, presoak it in distilled or deionized water until no further gain in mass is achieved, indicating that the test piece is saturated. Use distilled or deionized water at the conditioning temperature (see clause 4).

Clamp the test piece securely and apply clean tap water at the conditioning temperature (see clause 4) at a controlled pressure to the lower surface.

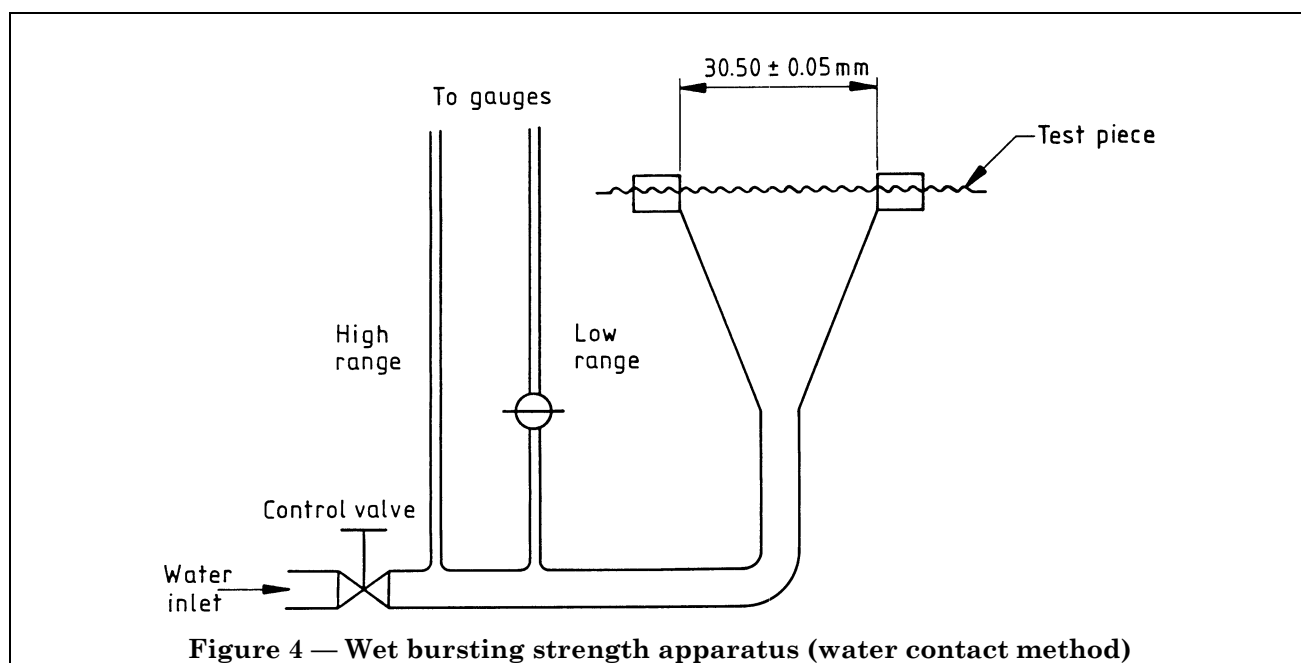
Increase the water pressure at such a rate that the test piece is ruptured in 20 ± 5 s. Repeat with nine other test pieces.

22.3.4 Calculation of results

Take the bursting strength as the mean limiting water pressure applied (in kPa).

22.3.5 Test report

The test report shall include a statement indicating whether the test piece was presoaked or not and, if presoaked, the presoaking time.



23 Stiffness and flexibility

23.1 General

Use the method of test described in BS 3748 or the method described in 23.2 to 23.7.

NOTE Various other static and dynamic methods for measuring the stiffness of paper are under consideration. See also ISO 5628, Tappi T535 and Tappi T543.

23.2 Principle

A test piece, supported at each end, is deflected by means of a small weight placed at the centre point. The deflection from the horizontal is measured.

23.3 Apparatus

23.3.1 Wires, two in number, of approximately 3 mm diameter, of smooth finish and straight.

23.3.2 Small weight, of known mass, for example 1 g.

23.3.3 Rule, graduated in millimetres.

23.4 Preparation of test pieces

Cut five test pieces of dimensions 25 mm by 150 mm, with the longer dimension in the machine direction.

23.5 Procedure

Position two wires so that they are horizontal, parallel to each other and 100 mm apart. Lay the test piece on top of the wires. Record the height of the centre point of the test piece. Place a small weight at the centre of the test piece, such that the centre is lowered by not less than 3 mm and not more than 20 mm. Record the change in height. Repeat with the test piece inverted. Repeat with the other four test pieces.

23.6 Calculation of results

Take the flexibility of the test piece as the mean of the results obtained, expressed in mm/g.

23.7 Test report

The test report shall include the mass of the weight (in g).

24 Brittleness

24.1 General

This test applies to filter papers used in the manufacture of pleated filter elements. Excessive brittleness (see 2.13) may cause failure of the filter element at the pleat.

24.2 Principle

The tensile strength of test pieces, creased under controlled conditions, is compared with the tensile strength of uncreased test pieces.

NOTE 3 Other test widths may be used provided the loading is as specified in 24.3.3 and the template is of a suitable size with

24.3 Apparatus

24.3.1 Tensile testing apparatus, as described in BS 4415-2.

24.3.2 Weight, consisting of a flat-bottomed rectangular section of metal approximately 40 mm × 40 mm and of mass 495 ± 10 g.

24.3.3 Acrylic template

(see Figure 5), 50 mm × 50 mm × 1 mm, with marked guidelines for positioning the folded strip and the weight, so as to ensure a constant loading of 1.5 g/mm^2 (see note 2 to 24.5).

24.4 Preparation of test pieces

Condition the sample in accordance with clause 4. Identify the two surfaces of the sample in some convenient way, e.g. side one and side two and cut 30 test pieces from the sample of dimensions 15 ± 0.1 mm by 250 mm minimum, with the longer dimension parallel to the machine direction. Ensure that the edges of each test piece are clean and straight.

24.5 Procedure

Carry out the tests in the conditioning atmosphere in accordance with clause 4. Form a loop in a test piece by bringing the two ends together between finger and thumb with side one on the inside of the loop. Holding the two ends together, lay the nearly closed loop on a flat rigid surface and place the template on the loop so that the strip lies between the parallel guidelines. Gently flatten the loop by exerting minimum pressure on the template to form a crease in the cross direction. Adjust the template so that the creased test piece is correctly positioned and place the weight on the template in the marked position for 30 ± 2 s. Remove the weight, unfold the test piece, and, using the procedure in BS 4415-2, determine the tensile strength of the creased strip. Repeat this procedure with another test strip, reversing the direction of fold, so that side one is on the outside of the loop. Repeat this process until 20 strips have been tested with an equal number tested with side one on the inside and on the outside of the loop. Use the remaining 10 strips to determine the tensile strength of the uncreased material using the procedure in BS 4415-2.

The same jaw speed should be used for testing both creased and uncreased test pieces.

NOTE 1 If it can be shown that there is no significant difference in results between tests with side one on the inside of the loop and those with side one on the outside of the loop only 10 tests need be carried out.

NOTE 2 For certain papers, e.g. resin-impregnated glass fibre, the brittleness value is sensitive to loading. Hence it is important to maintain a consistent loading. If a different loading to that specified is used this should be reported.

appropriate guidelines and the centreline of the mass is maintained 2 mm back from the fold.

24.6 Calculation of results

Use the procedure described in BS 4415-2 to calculate the tensile strength of both the creased and uncreased test pieces.

Calculate the brittleness B , as the percentage loss in strength, from the following equation:

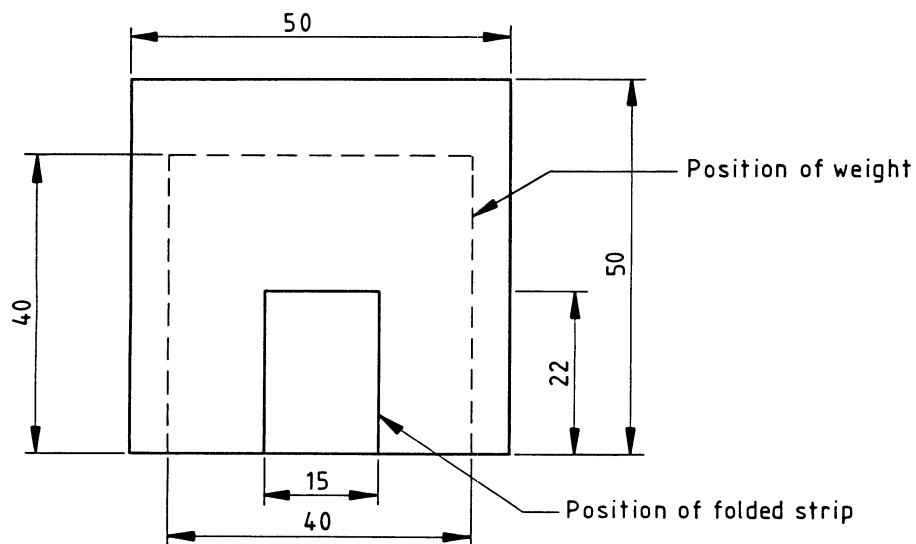
$$B = \frac{100 (T_b - T_a)}{T_b}$$

where

T_a is the mean tensile strength of the creased test pieces (in kN/m);

T_b is the mean tensile strength of the uncreased test pieces (in kN/m).

(6) If the mean tensile strength of the creased test pieces for the two directions of fold differs by more than 20 %, calculate and report the brittleness separately for each direction of fold, otherwise report the average.



All dimensions are in millimetres.

Figure 5 — Template for positioning the weight on the folded strip

Section 4. Test methods for functional properties

25 Retentivity to particles in air

Use the method described in BS 4400. The test piece shall be arranged as shown in Figure 6. The test area shall be either circular or rectangular and of a minimum area of 0.01 m^2 . Report the face velocity used.

NOTE 1 It is recommended that a value for the air flow at the filter surface (face velocity) of 1.5 m/min (0.025 m/s) is used.

NOTE 2 Various alternative methods are available for measuring the retentivity to particles in air for filter paper e.g. ASTM D2986. These methods generally use the same principle of generating an aerosol (either mono or polydispersed) from a known solution, such as DOP (Di-iso-octyl phthalate). A metered portion of this aerosol can then be characterized upstream and downstream of the filter paper by using a variety of different equipment. The most commonly used are condensation nuclei counters (CNC), laser-based equipment or light-scattering photometry. From the data generated by this equipment the efficiency of the filter paper can be calculated over various particle size ranges.

26 Retentivity to particles in liquids

26.1 Principle

The loading of monosized particulates in the test liquid is measured with a particle sizing/counting instrument. The test piece is used to filter the test liquid and the particulate loading of the filtered test liquid is measured. This process is repeated with other particulates and a graph of percentage retention against particle size is plotted. The retentivity (see 2.22) is read from the graph.

NOTE 1 If initial retentivity levels were quoted at 100 % retention of challenging particles, the result would not reflect the performance of the filter normally found under practical conditions. Because of this aspect, the practical performance levels of depth filters are rated according to their ability to retain 98 % of particles at the stated size.

NOTE 2 More elaborate test methods exist as ASTM F795 and ASTM F796, but are considered to be more suitable for research purposes than for routine tests.

26.2 Apparatus and reagents

26.2.1 Filter holder, e.g. a three-piece glass or porcelain "Hartley" pattern holder, 70 mm diameter.

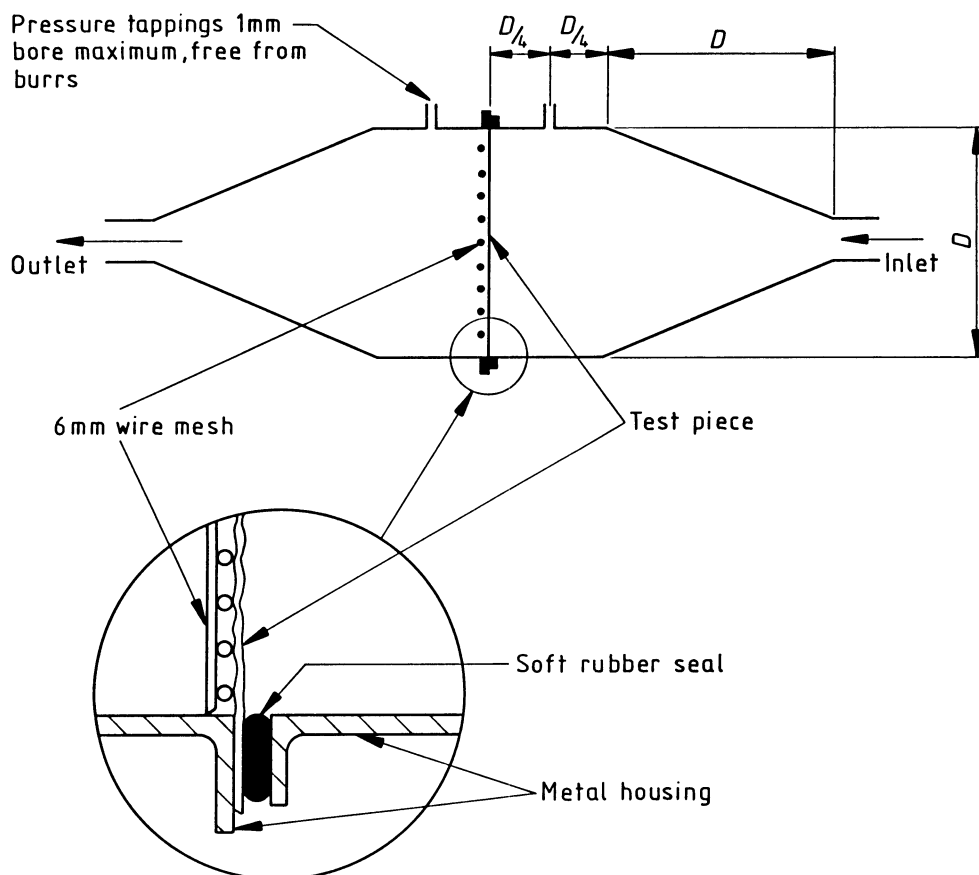


Figure 6 — Retentivity to particles in air apparatus

26.2.2 Filter support, of fine mesh.

NOTE The support is fitted beneath the filter to eliminate pinpoint filtration over the support plate holes.

26.2.3 Buchner flask, with bung to accept filter holder.

26.2.4 Vacuum pump, capable of sustaining a vacuum of at least 13 kPa.

26.2.5 Mercury manometer

26.2.6 Monosized particulates⁴⁾, of size 1 µm to 25 µm minimum.

26.2.7 Particle sizing/counting instrument⁴⁾

26.2.8 Test liquid, water-based and appropriate to the particle sizing/counting instrument used.

26.3 Preparation of test pieces

Test pieces shall be in the “as received” condition or shall be conditioned in accordance with clause 4.

NOTE Some impregnated paper, e.g. paper tested prior to conversion, may require curing. Such papers should be cured in accordance with 3.2.

26.4 Procedure

For filter papers requiring prewash, carry out the appropriate prewash procedure.

Carry out a “blank” particle count with the particle sizing/counting instrument on the test liquid to ensure that the background count level on the liquid to be used for particulate dispersion is sufficiently low that interference with the monosized particulate count does not occur.

Choose a monosized particulate that is expected to span the 2 % penetration range for the test piece. Carefully mix sufficient of the particulate into the test liquid to produce a total particle count near the optimum upper counting limit of the particle sizing/counting instrument.

NOTE 1 This level of particulate loading should be low enough to reduce secondary filtration effects.

Measure the total particle count with the instrument and adjust, if necessary, with either test liquid or particulate. Record the total count.

Pour the whole of the test dispersion into the filter reservoir and filter at 13 kPa (100 mmHg) or other desired vacuum level. Collect the filtrate in a clean, prerinsed container and measure the total particle count. Record this count.

NOTE 2 A vacuum level of 13 kPa is recommended but other levels may be used, as agreed between the parties concerned.

Repeat the procedure with at least two other particulates of different sizes.

26.5 Calculation of results

Calculate the penetration of particulate, P , through the test piece as a percentage, from the following equation:

$$P = \frac{N_1 \times 100}{N_0} \quad (7)$$

then

$$R = 100 - P$$

where

P is the percentage penetration;

N_1 is the particle count of the filtrate;

N_0 is the original particle count of the test liquid;

R is the percentage retention.

Plot a graph of percentage penetration against particulate size, with particle size (in µm) on the x-axis and percentage penetration (as a logarithmic scale) on the y-axis.

Draw a line parallel to the x-axis at 2 % penetration (98 % retention) to cut the curve. At the point of intersection, read off from the x-axis the value of retentivity (in µm) for the test piece.

26.6 Test report

The test report shall include the following particulars:

- the retentivity (in µm);
- a statement that the retentivity was determined at 98 % particulate retention;
- where test pieces were prewashed, the prewash conditions;
- nature of the monosized particulate;
- test liquid;
- test piece working area;
- original particulate loading in the test liquid;
- vacuum level used during filtration.

27 Air flow resistance

27.1 General

This method is applicable to high permeability papers.

NOTE 1 BS 6538-3 describes a method that is suitable for medium permeability papers.

NOTE 2 When measuring the retentivity to particles in air (see clause 25), the air flow resistance at 1.5 m/min (0.025 m/s) may be taken as the measured pressure drop across the test piece.

⁴⁾ For information on the availability of monosized particulates and particle sizing/counting instruments, apply to Enquiry Section, Linford Wood, Milton Keynes, MK14 6LE.

27.2 Principle

Air is allowed to flow through the test piece and the pressure differential across the test piece is recorded.

27.3 Apparatus

27.3.1 *Test apparatus*, as shown in Figure 7. The apparatus shall have a clamp capable of holding the test piece flat without lateral tension. The internal diameter of the apparatus D (see Figure 7) shall be 35.69 ± 0.05 mm.

NOTE The area of filter paper under test should be approximately 10 cm^2 .

27.3.2 *Means of applying air to the test piece*

27.3.3 *Flowmeter*, measuring air flow in litres per minute and calibration checked to $\pm 1\%$.

27.3.4 *Means of measuring the pressure differential across the test piece*, in kPa.

27.4 Procedure

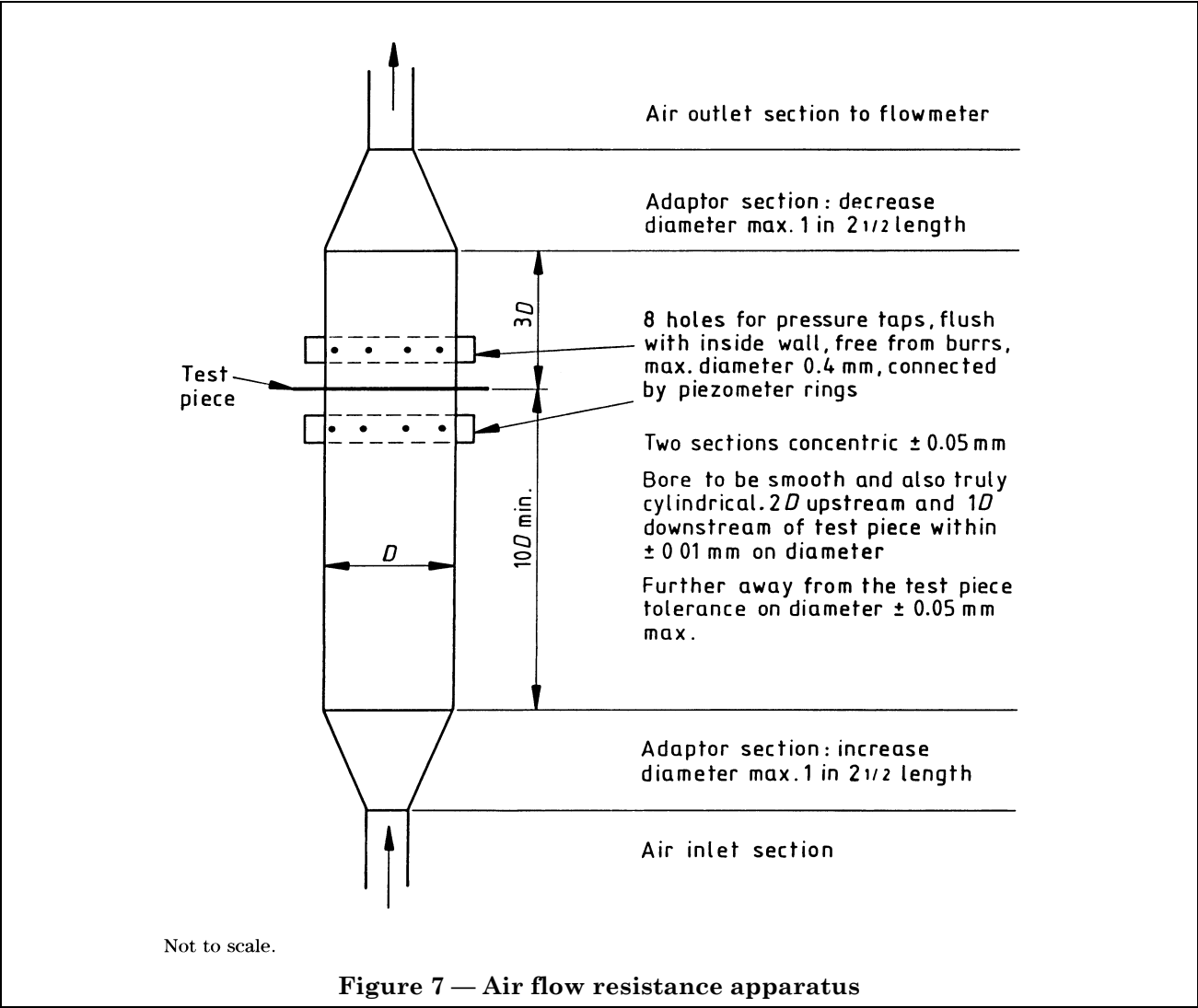
Clamp a test piece in the apparatus and turn on the air supply to the test piece. Use an air flow of 10 L/min or, for more permeable papers of air flow resistance less than 0.25 kPa at 10 L/min, an air flow of 20 L/min.

NOTE An air flow of 10 L/min with a test area of 10 cm^2 is equivalent to an apparent linear flow of 10 m/min (0.167 m/s). An air flow of 20 L/min is equivalent to an apparent linear air flow of 20 m/min (0.333 m/s).

Record the pressure differential across the test piece. Check the equipment daily with a standard orifice plate or other check piece.

27.5 Calculation of results

For an air flow of 10 L/min, take the air flow resistance (in kPa) as the recorded pressure difference. For an air flow of 20 L/min, take the air flow resistance (in kPa) as half the value of the recorded pressure differential.



27.6 Test report

Report the air flow resistance and the air flow used.

28 Media migration

28.1 Principle

Kerosine test liquid is passed through a test piece of filter paper held in a test piece holder and a sample of the filtrate is collected in a clean, glass sample bottle. The contaminant content of this sample is extracted by passing the sample through a fine membrane type filter and the membrane deposit is examined microscopically.

28.2 Apparatus and reagents

28.2.1 Kerosine test liquid, additive-free and particle-free, with a kinematic viscosity of between $1.8 \mu\text{m}^2/\text{s}$ and $2.4 \mu\text{m}^2/\text{s}$ at 37.8°C and a surface tension between 24 mN/m and 27 mN/m at 23°C , prepared by filtration through a membrane filter (see **28.2.5**).

28.2.2 Test piece holder

NOTE An example of a suitable test piece holder is shown in Figure 8. Other materials of construction may be used, but those shown are readily available and easily worked. It is recommended that the dimensions of the test piece holder are such that an area of 0.01 m^2 of the test piece is subjected to test.

28.2.3 Small-scale test rig, as shown in Figure 9.

NOTE 1 The components used in the test rig should not shed contaminants into the system.

NOTE 2 The pump rating should be chosen in conjunction with the size of the test piece holder to give a flow rate per unit area of the test piece appropriate to the type of filter paper under test. A small, electrically driven centrifugal pump is most suitable.

28.2.4 Sample bottles, made of glass and of no more than 2 L capacity, with smooth internal contours.

NOTE The bottles should preferably be treated internally with a clear silicone film that will facilitate complete removal of the solids. A satisfactory coating can be obtained by an application of 2 % dichlorodimethylsilane in a suitable solvent. Bottles are then allowed to drain and dry normally.

28.2.5 Membrane filters, cellulose ester, diameter 47 mm, pore size $0.8 \mu\text{m}$.

28.2.6 Filtration apparatus, for use with the membrane filter (**28.2.5**), comprising a support for the membrane made from sintered bronze or an acceptable alternative, a clamping ring for the membrane and a glass or stainless steel funnel, together with a large receiving vessel fitted with vacuum suction equipment.

28.2.7 Wash bottles

28.2.8 Flat-bladed forceps

28.2.9 Petri dishes

28.2.10 Binocular microscope, approximately $\times 20$ magnification.

28.2.11 Monocular microscope, capable of resolving $2 \mu\text{m}$ particles and having a gridded or calibrated eyepiece suitable for particle size estimation.

28.2.12 Balance, accurate to 0.1 g.

28.3 Preparation of test pieces

Cut a test piece of a suitable size for the test piece holder.

Carry out the test on the test piece in the “as received” condition.

28.4 Procedure

28.4.1 Paper test

Assemble the small-scale test rig shown in Figure 9 and flush thoroughly with kerosine. Insert the test piece in the holder. With the control valve closed pour 5 L of kerosine into the reservoir. Open the control valve and start the pump, allowing kerosine to pass through the test piece.

Rinse the sample bottle with kerosine. Insert the sample bottle into the flow of filtrate and collect approximately 1 L. Stopper the sample bottle with a well-fitting glass stopper or cover with a clean glass plate, a watch-glass or a piece of clean plastics film material.

NOTE Samples may be taken to include the initial flow or at any selected times during the test.

28.4.2 Extraction of contaminant from kerosine samples

Clean the filtration apparatus with kerosine, using clean wash bottles.

Select a membrane filter and mount it in the filtration apparatus, using flat-bladed forceps to avoid damaging the membrane.

Weigh the sample bottle and its contents to the nearest gram. Pour the contents of the sample bottle into the funnel of the filtration apparatus and draw the liquid through the membrane by suction. Rinse the sample bottle at least three times with kerosine and pour the rinsings into the funnel. Continue until there are no visible signs of contaminant remaining in the sample bottle. Wash down the sides of the funnel with kerosine. Remove the funnel and wet the membrane carefully with two or three drops of kerosine to ensure that any electrostatic charges are dissipated. Remove the membrane carefully, using flat-bladed forceps and place in a petri dish. Weigh the empty sample bottle to the nearest gram.

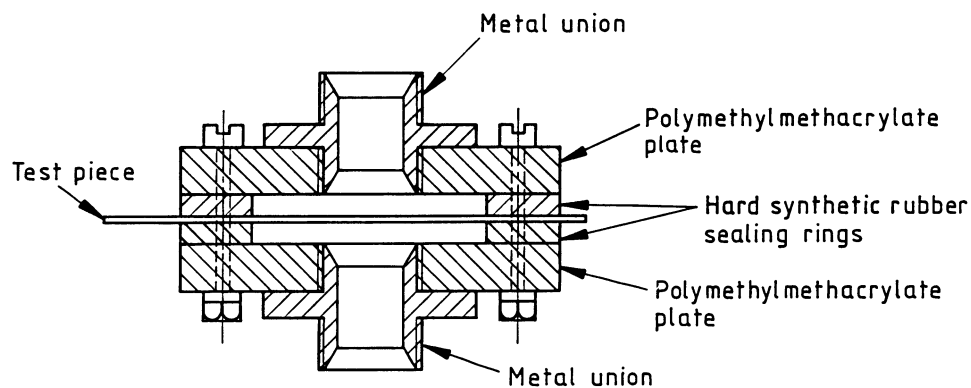


Figure 8 — Example of a media migration test piece holder

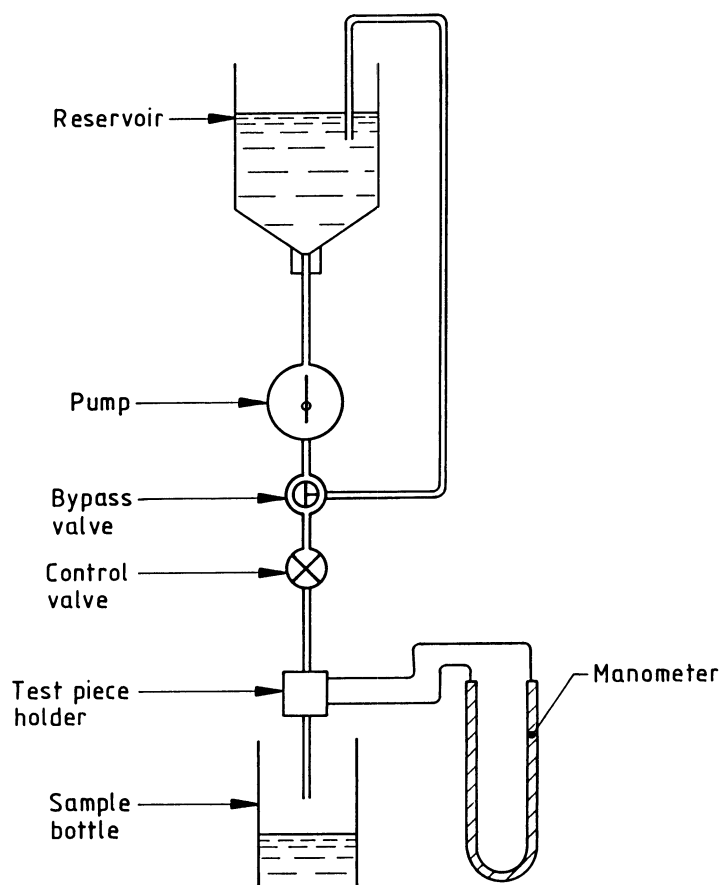


Figure 9 — Media migration test rig

28.4.3 Examination of membrane debris

Inspect the membrane visually for the presence of large particles, fibres or other obvious contaminant.

NOTE A fibre may be defined as any material having a length equal to or greater than 10 times its width.

Examine the membrane with a binocular microscope for small materials and attempt to classify these materials, e.g. metallic, fibrous, coloured, crystalline, etc.

Finally, make an approximate particle size count with the aid of a monocular microscope, grouping particles into wide size bands of less than 10 μm to 50 μm to 100 μm and over 100 μm . Record the sizes of individual large particles.

28.5 Calculation of results

Determine the sample size by taking the difference in mass of the empty and filled sample bottle. Determine the volume of solvent accurately using the density of the solvent.

28.6 Test report

The test report shall include the following particulars:

- an approximate count of the number and size of any fibres present per litre of solvent;
- details of any large particles or particular materials observed;
- the size distribution of any particles observed;
- the types of any small particles (metallic, fibrous, crystalline, etc.) observed and their number;
- any other contaminant observed.

29 Water resistance (hydrostatic head test)

29.1 Principle

The hydrostatic head supported by a paper is a measure of the opposition to the passage of water through the paper. A test piece is subjected to a steadily increasing pressure of water on one face, under standard conditions, until penetration occurs at one place.

NOTE This method is based on, but is not identical with, BS 2823.

29.2 Apparatus and reagents

29.2.1 Water resistance apparatus, shown in Figure 10. The apparatus shall be constructed from non-corrodible materials and shall enable the test piece to be clamped in such a manner that:

- it is horizontal and is not bulging;
- no leakage of water occurs;
- no slippage of the test piece occurs.

NOTE A circular test area of diameter 50 mm is recommended but other test areas may be used, as agreed between the parties concerned.

29.2.2 Water, distilled or deionized

29.3 Procedure

Ensure that the clamping surfaces are dry. Clamp the test piece in the test head so that the wire side of the test piece is in contact with the water. Ensure that water is not being forced through the test piece. Subject the test piece immediately to increasing water pressure.

NOTE The rate of increase of water pressure is very important. Too rapid an increase gives inaccurate and high results. A rate of increase of 0.9 kPa/min to 1.1 kPa/min is recommended but other rates such as 5 kPa/min may be used, subject to agreement between the parties concerned.

Watch continuously for evidence of penetration by water and record the pressure at which the first droplet forms and continues to grow. Ignore penetration immediately at the periphery of the test area.

29.4 Calculation of results

Take the water resistance of the test piece (in kPa) as the recorded water pressure. For water pressure up to 2.5 kPa, express the result to the nearest 0.05 kPa. For higher pressures, express the result to the nearest 0.1 kPa.

29.5 Test report

The test report shall include the following particulars:

- the rate of increase of water pressure (in kPa/min);
- the size of the test area.

30 Liquid absorbency (Klemm method)

Use the method of test described in BS 6504.

NOTE The test liquid used is normally distilled or deionized water but other liquids may be used and that fact is noted in the test report.

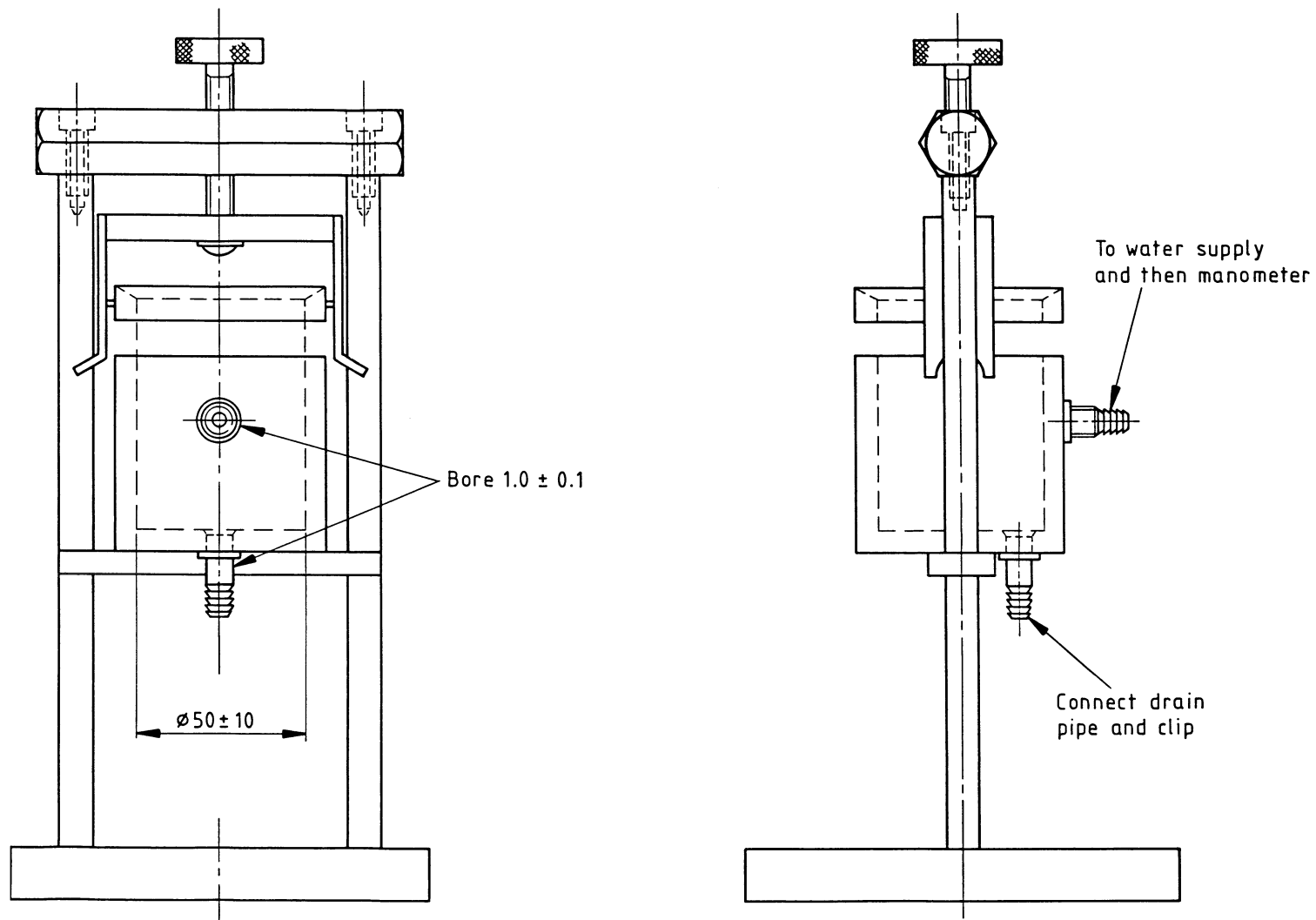
31 Water filtration time

31.1 General

Test methods intended to reproduce practical water flow rate conditions whereby a filter paper is quadrant folded and "freely suspended" using a wire loop are known to be subject to numerous variations and potential errors. In addition, this approach to the assessment of water filtration time does not lend itself to the testing of filter papers which are not compatible with the quadrant folding technique, e.g. unbound glass microfibre papers.

Other test methods have been proposed that utilize flat test filters but difficulties can be encountered in ensuring that a constant hydrostatic head is maintained on the filter during the test.

This method overcomes many of the difficulties previously encountered.



All dimensions are in millimetres.

Figure 10 — Water resistance apparatus

31.2 Principle

The test piece is clamped horizontally and freely exposed, from beneath, to a continuous supply of clean water at a constant hydrostatic head. The upper surface is open to the atmosphere and surrounded by a shallow-lipped wall to allow collection of the water passing through the test piece. The time for the collection of 100 mL of water is recorded.

31.3 Apparatus and reagents

31.3.1 *Constant hydrostatic head water filtration device*, (Herzberg apparatus)⁵⁾, shown in Figure 11. The apparatus shall include a sliding tube arrangement so that the magnitude of the hydrostatic head can be adjusted to the required value.

31.3.2 *Vacuum flask*, for de-aeration of water.

31.3.3 *Vacuum pump*, for de-aeration of water.

31.3.4 *Membrane filter*, of 0.8 μm pore size.

31.3.5 *Membrane filter holder*

31.3.6 *Vacuum system*, for use with membrane filter and holder.

31.3.7 *Measuring cylinder*, of at least 100 mL capacity.

31.3.8 *Timer*

31.3.9 *Water*

31.4 Procedure

Using the membrane filter, filter 1 L to 2 L of water, with vacuum suction. De-aerate the water using the vacuum pump and flask. Fill the reservoir of the apparatus.

Using the three-way tap, allow the water level to rise in the open cavity of the test piece holder until a distinct meniscus has formed at the test piece clamp level. Close the three-way tap.

Carefully “roll” the test piece on to the test piece holder, wire side down, so that the test piece is flat and all air is excluded from the contact area between water and paper. Clamp the test piece in place.

Set the constant hydrostatic head device at the mark on the graduated scale that will give the required hydrostatic head.

NOTE A hydrostatic head of 1 kPa (10 cm mark on the graduated scale) is recommended, but other pressures may be used. Filtration times should not be assumed to be directly proportional to the hydrostatic head.

Open the three-way tap to allow flow of water from the reservoir to the test piece.

Time the collection of 100 mL of water passing through the test piece into a 100 mL measuring cylinder. Record the time.

31.5 Calculation of results

Take the water filtration time for the test piece (in s) as the time recorded for the collection of 100 mL of water.

31.6 Test report

The test report shall include the following particulars:

- the statement that the water filtration time was the time taken to collect 100 mL of water;
- the hydrostatic head used.

32 Flammability

NOTE Some resin-treated papers may give off harmful fumes. Care should therefore be taken to avoid breathing any fumes given off during this test.

32.1 Principle

A test piece is ignited for a specified time in a draught-proof enclosure and, after removal of the ignition source, the flaming and glowing times (see 2.19, 2.20, 2.21) are recorded. The length of the charred portion is measured.

32.2 Apparatus

32.2.1 *Draught-proof enclosure*, a fume cupboard or metal cabinet, as shown in Figure 12.

NOTE The cabinet is similar to that described in Tappi method T461 om -84.

32.2.2 *Test piece holder*, capable of holding a test piece of 210 mm in length and 70 mm in width by gripping 10 mm of one or more edges. The test piece holder shall be as shown in Figure 12 or shall comprise two large clips of the “bulldog” type, mounted vertically in retort stands. The clamping arrangement shall leave an area of 50 mm $\times$ 210 mm exposed.

32.2.3 *Bunsen burner*, of 10 mm internal diameter, connected to a natural or propane gas supply.

32.2.4 *Stopclock*, or stopwatch

32.3 Preparation of test pieces

Condition the sample in accordance with clause 4. Cure a resin-treated sample in accordance with 3.2.

Cut five test pieces from the sample, each of dimensions 210 mm $\times$ 70 mm, ensuring that the longer dimension is in the machine direction.

⁵⁾ For information on the availability of the Herzberg apparatus, apply to Enquiry Section, BSI, Linford Wood, Milton Keynes, MK14 6LE.

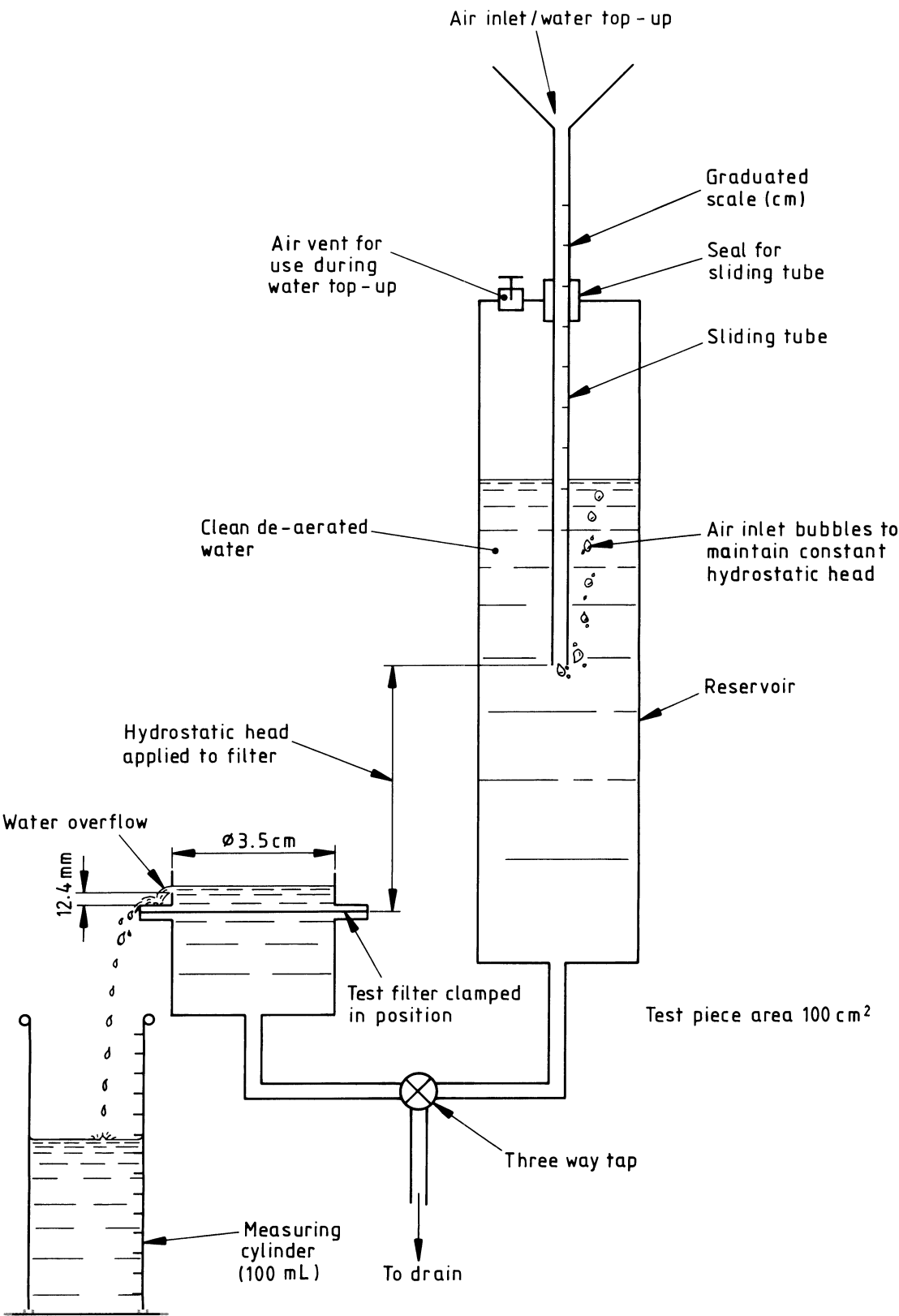
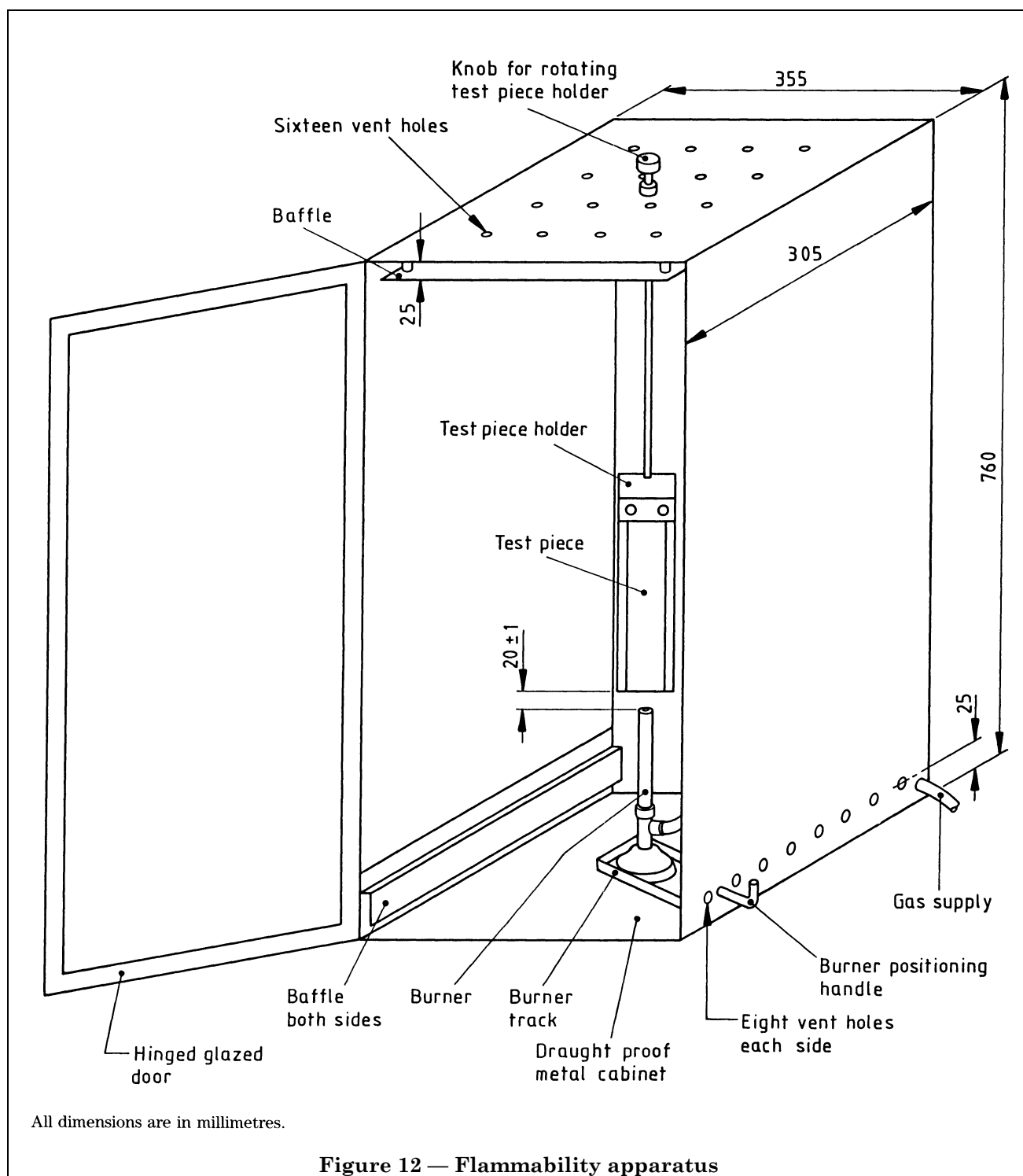


Figure 11 — Water filtration time apparatus (Herzberg)



32.4 Procedure

Set up the apparatus so that the top of the burner tube will lie 20 ± 1 mm below the lower edge of the test piece. Light the burner and with the air supply closed adjust the gas supply to the burner to give a yellow flame 40 mm high. Clamp a test piece in the test piece holder, adjust the burner flame, if necessary, and position the burner so that the yellow flame is directly under the bottom edge of the test piece. Close the fume cupboard or cabinet. Withdraw the flame after 12 s. Record the flaming time and glowing time.

Remove the test piece from the holder. Holding the test piece horizontally, remove the loose, charred area by tapping gently with a metal or glass rod of 6 mm diameter or by rubbing between finger and thumb. Measure (to the nearest 2 mm) the maximum length of the void area from the original bottom edge of the test piece. Record the measured values.

Repeat for each test piece.

32.5 Calculation of results

For flaming and glowing times greater than 2 s, calculate the mean of the results (in s).

32.6 Test report

The test report shall include the following particulars:

- a) the statement "These results were obtained under the specified test conditions and do not necessarily represent the behaviour of the test material under other conditions of test or use";
- b) for flaming and glowing times of more than 2 s, the mean and maximum values (in s);
- c) for flaming and glowing times of less than 2 s, a statement that the filter paper is self-extinguishing;
- d) the length of the void area (in mm) for each test piece.

33 Choking life

NOTE It should be noted that choking life determinations are highly dependent on a number of factors including particle size, shape and concentration, nature and viscosity of the carrier fluid and the face velocity at which the carrier fluid approaches the filter. In view of this, it may be appropriate for the choice of particulate, fluid, test face velocity and other controlling conditions, to be agreed between the parties concerned.

33.1 Terminology

Choking life relates to the ability of a filter to load a contaminant or a particulate material into the fibrous matrix whilst maintaining a practical flow rate through and pressure differential across the filter. The choking life is exceeded when the filter fails to perform at an acceptable flow rate and/or pressure differential. It is expressed as the mass of particles fed to the filter before the end point is reached.

The predecided acceptable limits are dependent on the ultimate use for the filter. Consideration should be given to the application before deciding the limits, which shall be reported. For example with air filters, the limit may be when the differential is double the permitted maximum initial air pressure differential at the test velocity, or, in the absence of that information, double the starting pressure differential. With liquid filters, the limit may be, in some cases, as high as 300 kPa whilst in others it may be lower. For the laboratory filtration of contaminated liquids, the acceptable limit may be when the filter fails to perform at a flow rate 95 % lower than the initial rate of flow. The concept of "loading capacity" is also used to determine a filter's working limit.

The test results will change with change of test particles or of test fluid. Once a test has commenced, it should be continuous.

33.2 Principles

33.2.1 Liquid systems

In general, test methods for liquid systems are based on one of two approaches.

33.2.1.1 A liquid containing a particulate is fed to a filter in a recirculating system whereby the rate at which particulate is removed by the filter is compensated by constant addition of fresh particulate, or a single pass test may be undertaken without the need for addition of fresh particulate, provided that the supply vessel volume is sufficiently large. The pressure differential across the filter is monitored so as to assess the choking life.

33.2.1.2 Aliquots of a liquid containing a particulate are fed to a filter under constant vacuum and the filtrate rate of flow determined for each aliquot. A plot of "number of aliquots" or "cumulative volume" versus "filtrate rate of flow" will produce a curve which characterizes the particular filter/liquid/particulate system under test. Choking life may be assessed by defining the lowest acceptable "filtrate rate of flow".

33.2.2 Gaseous systems

Test methods for choking life of filters used in removal of particulates from a gas are based on one of the following.

33.2.2.1 Utilization of the standard retentivity test for particulates from air (e.g. BS 4400) with the test extended to increase particulate loading on the filter. Choking life is assessed by defining the highest acceptable pressure differential for a given loading of particulates.

33.2.2.2 Purpose-built choking life rigs in which continuously generated dust clouds are fed to the filter with choking life quantified by filter differential pressure measurements.

33.3 Apparatus

33.3.1 Liquid systems

33.1.1.1 Apparatus suitable for the test method described in **33.2.1.1** is shown diagrammatically in Figure 13 or Figure 14. For simplicity, only the apparatus based on single pass assessment of choking life for a filter is shown.

In addition, a suitable particulate (either monosized or covering a reproducible and “definable” size range) dispersed in liquid is required. Numerous liquids can be used in this method, dependent upon the applicational requirements expected for the filter.

However, the chosen liquid for preparation of the particulate dispersion should fulfil several requirements:

- a) it should be representative of the viscosity range anticipated for the application;
- b) it should exhibit suitable “polar” or “non-polar” properties to reflect the anticipated application;
- c) it should be sufficiently consistent from batch to batch that adequately defined materials will exhibit reproducible properties;
- d) it should be easily obtainable and relatively low in cost.

Suggested liquids fulfilling those requirements are:

Water, usually buffered to a known pH;

Kerosine, additive-free;

A calibrating fluid, i.e. a straight mineral oil⁶⁾ with a viscosity of approximately $4.5 \text{ mm}^2/\text{s}$ at a temperature of 20°C and a surface tension of $23 \pm 1 \text{ mN/min}$.

Fully detergent mineral oil.

The chosen particulate for the choking test should represent the type of particulate to be encountered in the application. Anomalous results may be obtained if, for example, a spherical particulate is used in the test whereas an angular or fragmented particulate is anticipated in the application. “Definable” particulates may be:

- 1) Monosized spherical particulates prepared from, for example, polystyrene divinyl benzene latex, polyvinyl toluene latex, polystyrene latex.

NOTE These materials can be expensive and therefore should be limited to small-scale choking life tests.

- 2) “Defined” size range particulates such as silica, glass beads, precipitated calcium carbonate, aluminium oxide dust.

In all cases the particulate should be purchased as a size (in microns) and material defined product. Those materials not available in size classified form should be quantified before the test by using a particulate sizing instrument.

Possible particulates include the following:

- i) fused aluminium oxide dust⁷⁾, 60 % to 80 % of the particles, by mass, being within the range $3.5 \mu\text{m}$ to $7 \mu\text{m}$;
- ii) a similar dust⁷⁾, 60 % to 80 % of the particles, by mass, being within the range $15 \mu\text{m}$ to $25 \mu\text{m}$;
- iii) any one of the range of 5 MIRA dusts⁷⁾ which have mean sizes from $5 \mu\text{m}$ to $55 \mu\text{m}$.

33.3.1.2 Apparatus suitable for the test method described in **33.2.1.2** is as follows:

- a) *filter holder* (three-piece “Hartley” pattern 7 cm diameter glass or porcelain funnel is suitable). A fine mesh support beneath the filter is recommended to eliminate pinpoint filtration over the support plate holes;
- b) *Buchner-type flask*, and bung to accept filter holder;
- c) *vacuum pump*;
- d) *mercury manometer*;
- e) *size and material defined particulate*, as defined in **33.1.1.1**.

33.3.2 Gaseous systems

Apparatus suitable for the test method described in **33.2.2.1** is given in BS 4400, although the exact design of filter holder may need to be changed so as to clamp the test piece.

⁶⁾ For information regarding suitable oils, apply to Enquiry Section, Linford Wood, Milton Keynes, MK14 6LE.

⁷⁾ For information on the source of particulate sizing equipment and of BSI, Linford Wood, Milton Keynes, MK14 6LE.

33.4 Procedure

33.4.1 Procedure for single pass choking life test as described in 33.2.1.1

33.4.1.1 Prepare the dispersion of size defined particulate in the chosen test liquid at the required concentration.

NOTE The test dispersion concentration should reflect the particulate concentration which is expected in the envisaged application. In the absence of further information a concentration of 0.1 g/L may be used.

33.4.1.2 Transfer the particulate dispersion to the holding vessel (Figure 13, item A) and keep gently agitated.

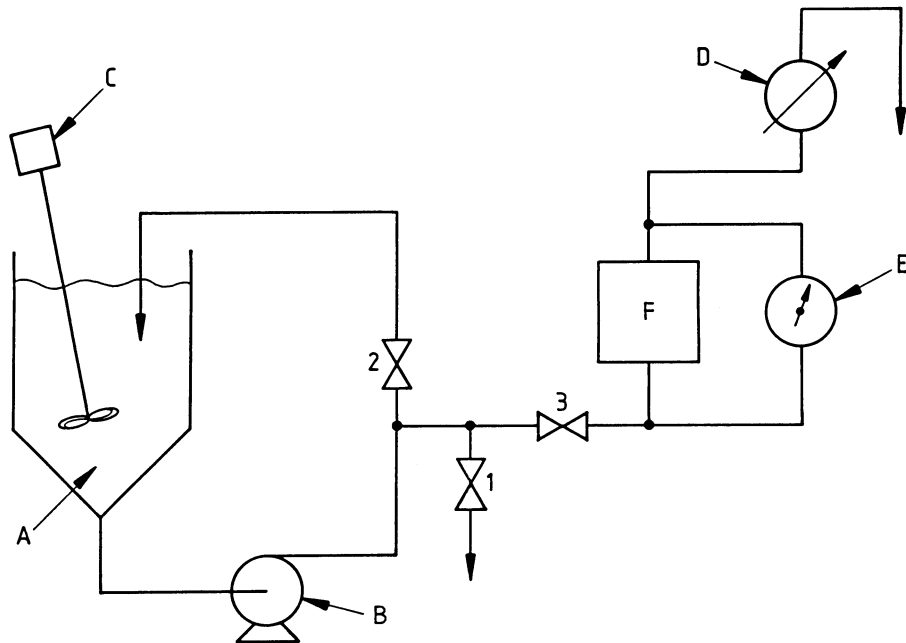
33.4.1.3 Install the filter medium in the holder (Figure 13, item F), or Figure 14.

NOTE The holder shown in Figure 14 does not permit the use of an inline flowmeter after the holder. The flow should be checked by some other suitable means.

34.4.1.4 Open valve 2, close valves 1 and 3 and commence recirculation of the particulate dispersion via pump B. Sample the dispersion in the reservoir several times to ensure that the particulate is well dispersed in the test liquid.

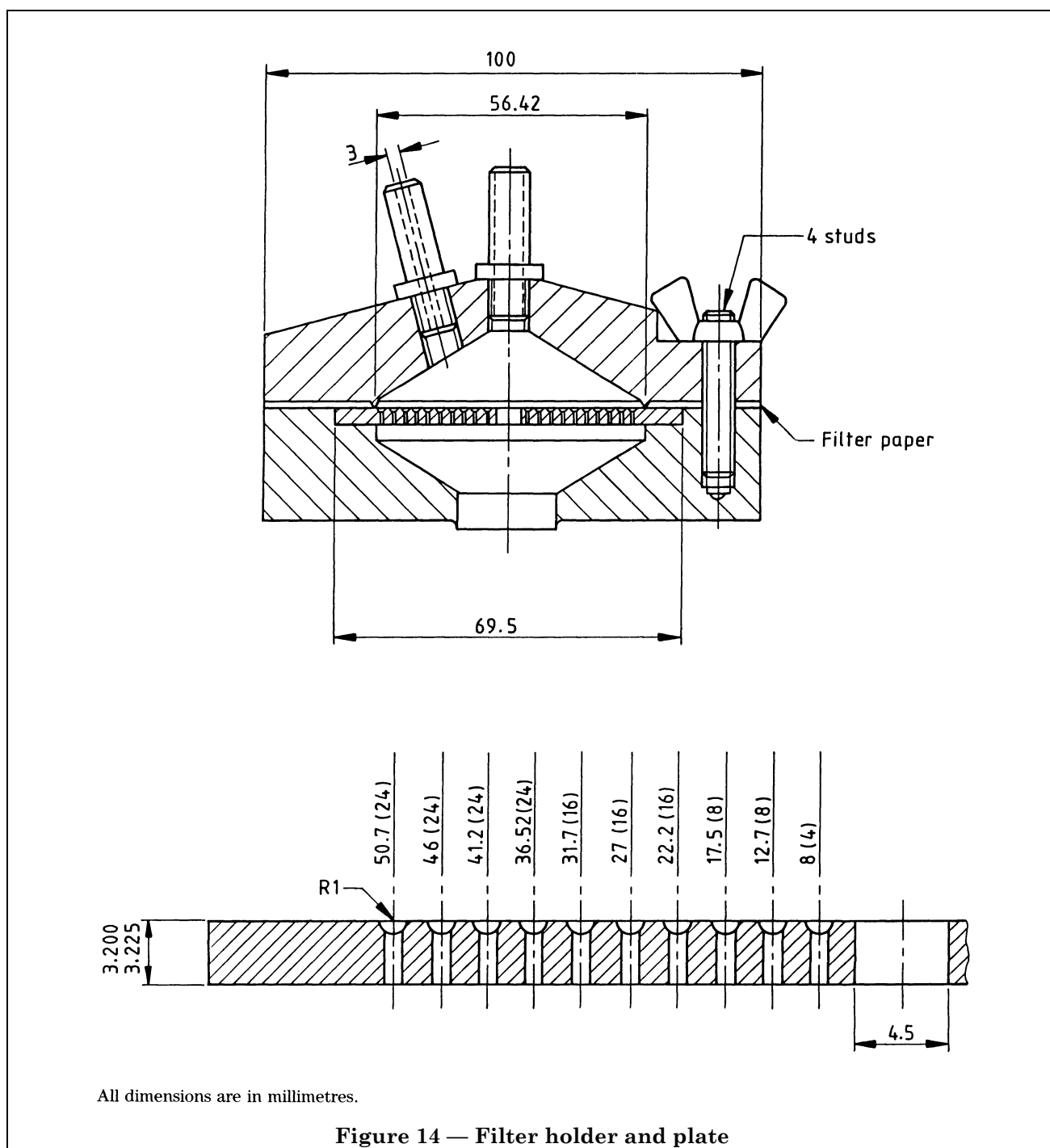
34.4.1.5 Open valve 3 and close valve 2. Adjust valve 3 to give the desired flow rate measured by flowmeter D. Record time and differential pressure across the filter.

34.4.1.6 Continue to adjust valve 3 to achieve constant flow rate and record differential pressure and time at suitable intervals.



- Key**
- 1, 2, valves
 - 3
 - A storage tank for liquid/particulate mixture
 - B pump
 - C agitator
 - D flowmeter
 - E differential pressure gauge
 - F suitable housing capable of securing test filter and tapped for differential pressure gauge

Figure 13 — Schematic layout of single pass apparatus



33.4.1.7 Stop the test when the differential pressure reaches the upper limit recommended by the manufacturer or other predetermined limit. An end point of five times the original differential pressure across the filter may be taken as a practical useful limit to filter performance.

33.4.1.8 Plot the results on a graph as differential pressure vs time and record the “choking life” as the time taken to reach the predetermined differential pressure limit. Record particulate type and size, carrier liquid, particulate concentration and dispersion temperature at the start and finish of the test.

33.4.2 Procedure for testing laboratory type filters using aliquots of particulate dispersion (see 33.2.1.2)

33.4.2.1 The apparatus should be set up as shown diagrammatically in Figure 15.

33.4.2.2 Prepare the dispersion of size-defined particulate in the chosen test liquid at the required concentration. Keep the dispersion gently agitated using a paddle or magnetic stirrer.

33.4.2.3 Install the filter medium on the filter holder ensuring that the circumference and main area of the filter is flat and in contact with the support mesh. The diameter of the latter should be 1 cm less than the filter diameter to ensure adequate sealing of the filter to the holder during the filtration process.

33.4.2.4 Wet the filter with a small quantity of the test liquid and switch on the vacuum pump.

33.4.2.5 Open valve D and adjust the air bleed B to give the desired vacuum level as monitored on the differential pressure gauge E.

33.4.2.6 Measure out the first aliquot of particulate dispersion ensuring that it is representative of the main volume of material. 50 ml aliquots may be used or other volumes dependent on filter characteristics and dispersion concentration.

33.4.2.7 Pour the first aliquot into the filter holder reservoir and record the time taken for completion of filtration.

33.4.2.8 Adjust the air bleed valve to ensure a constant vacuum level and pour in the second aliquot of particulate dispersion. Record the time taken for completion of filtration.

33.4.2.9 Repeat the procedure described in **33.4.2.8** until the time taken to complete the filtration of an individual aliquot is in excess of the predetermined practical filtration limit.

33.4.2.10 Plot the results on a graph as “filtration speed” in mL/min vs “number of aliquots”. The “choking life” is recorded as the number of aliquots (or cumulative volume) of particulate dispersion filtered before the filtration time exceeded the predetermined practical limit.

Record particulate type and size, carrier liquid, particulate concentration and dispersion temperature during the test.

33.5 Procedure for testing papers using particulates in an air stream as described in 33.2.2.1

33.5.1 Assemble the apparatus as described in BS 4400. For the purposes of this procedure the detector is not needed, although it may be used if an assessment of the initial retentivity is to be made. A suitable clamp will be needed to secure the test pieces.

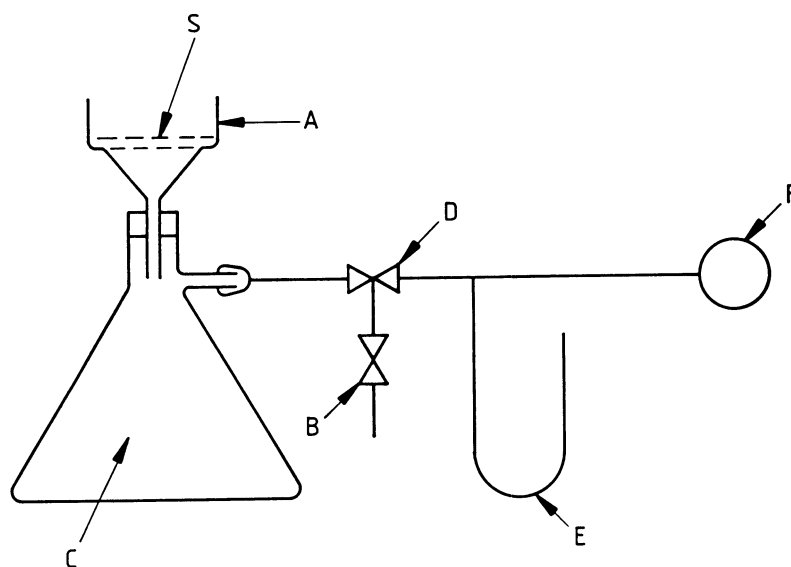
33.5.2 Ensure that the salt solution, atomizer and air flows have been prepared or adjusted as in BS 4400. The rate of particulate production is about 150 mg/min.

33.5.3 Install the test piece in the clamp. The face velocity of the air stream may be varied by altering the site of the clamp or by adjusting, within limits, the air flow. The test area should be at least 100 cm², and unless otherwise indicated, the face velocity should be 25 mm/s, corresponding to a flow of 30 L/min through a test area of 200 cm².

33.5.4 Measure the initial pressure differential across the test piece and note the time. Allow the test to continue, recording the elapsed time and corresponding pressure differential.

33.5.5 Stop the test when the pressure differential reaches some predetermined limit. In the absence of other information an end-point of twice the original pressure differential may be taken as the limit.

33.5.6 Record the face velocity and elapsed time. Calculate the mass of particulates fed to the test piece.



Key

- A three-piece filter funnel
- B air bleed valve
- C Buchner flask
- D valve
- E mercury manometer
- F vacuum pump
- S support mesh for filter

Figure 15 — Test apparatus for laboratory type filter papers

Appendix A Types of filter paper and applicability of test methods

The test methods in this standard apply to filter papers made by the papermaking process. This category of filters may be subdivided as follows.

- a) *Type 1: Laboratory filter papers.* Laboratory filter papers may be made from a variety of fibres. They are used primarily for the purpose of analysis or measurement of suspended particles in a fluid and secondly for clarification of relatively small volumes of fluid where subsequent analysis or measurement of the particles and/or their suspension vehicle is not necessarily required. Owing to the wide range of properties which may be required of this type it is only possible to include a selected group of test methods.
- b) *Type 2: Filter papers used for diagnostic applications.* These papers can be made from a variety of fibres and are primarily used for the purpose of providing a supporting matrix for diagnostic chemistries. Solutions under investigation can be added to the impregnated paper where a reaction takes place within a fibrous matrix. Detection can be carried out using various methods including colour change, colorimetry, fluorescence and radio assay techniques.

- c) *Type 3: Papers for high-efficiency air filters and battery separator papers.* These include papers made from glass fibres, from mixtures of glass fibres with other fibres or entirely from other fibres.
- d) *Type 4: Papers for filter elements.* These include papers for the manufacture of automotive and other filter elements (other than those included in type 3).
- e) *Type 5: Industrial and general filter papers (other than those included in types 3 and 4).* These include papers used in filter presses. These are supplied as cut sheets (including circles) or from a roll, but they are not shaped into elements before use. This category also includes papers used to filter beverages such as coffee, wine, beer, etc. and papers used for the manufacture of vacuum cleaner dust bags.

Table 1 lists the test methods described in this standard and the types of filter paper for which they are generally applicable. The final column of the table gives the clause in which the method is described.

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Table 1 — Applicability of test methods

Properties under test	Test method	Applicable to filter paper type					Test method described in clause
		1	2	3	4	5	
General and structural	Grammage	•	•	•	•	•	6
	Thickness	•	•	•	•	•	7
	Corrugation depth				•		8
	Moisture (volatile) content	•	•	•	•	•	9
	Ash	•	•	•	•	•	10
	Ignition loss	•		•			11
	Resin content	•	•		•	•	12
	pH value, conductivity, chloride and sulphate contents of aqueous extracts	•	•	•	•	•	13
	Maximum equivalent pore diameter	•	•	•	•	•	14.2
	Mean flow, maximum and minimum equivalent pore diameters	•	•		•	•	14.3
	Alkali solubility	•	•				15
	Acid solubility		•	•			16
	Calcium, copper, iron and manganese content	•	•	•			17
Mechanical	Tensile strength	•	•	•	•	•	18
	Tearing resistance		•	•	•	•	19
	Bursting strength	•	•	•	•	•	20
	Wet tensile strength	•	•		•	•	21
	Wet bursting strength	•	•		•	•	22
	Stiffness and flexibility		•	•	•	•	23
	Brittleness		•	•	•		24
Functional	Retentivity to particles in air	•		•			25
	Retentivity to particles in liquids	•	•		•	•	26
	Air flow resistance	•		•	•	•	27
	Media migration	•	•	•	•	•	28
	Water resistance		•	•	•		29
	Liquid absorbency	•	•			•	30
	Water filtration time	•	•			•	31
	Flammability			•	•		32
	Choking life	•			•		33

Publications referred to

- BS 907, *Specification for dial gauges for linear measurement.*
- BS 2071, *Specification for soxhlet extractors.*
- BS 2823, *Methods of test for resistance of fabrics to penetration by water (hydrostatic head test).*
- BS 2922, *Strength of wet paper and board.*
- BS 2922-1, *Method for determination of the bursting strength of paper and board after immersion in water.*
- BS 2922-2, *Method for determination of the tensile strength of paper and board after immersion in water.*
- BS 2924, *Aqueous extracts of paper, board and pulp.*
- BS 2924-1, *Method for determination of pH.*
- BS 2924-2, *Method for determination of conductivity.*
- BS 2924-3, *Titrimetric method for determination of water-soluble sulphates.*
- BS 2924-4, *General method for determination of water-soluble chlorides.*
- BS 3137, *Methods for determining the bursting strength of paper and board.*
- BS 3321, *Method for measurement of the equivalent pore size of fabrics (bubble pressure test).*
- BS 3430, *Method for sampling to determine the average quality of paper and board.*
- BS 3431, *Method for the conditioning of paper and board for testing.*
- BS 3432, *Method for determination of grammage of paper and board.*
- BS 3433, *Method for determination of moisture content of paper and board by the oven-drying method.*
- BS 3631, *Method for the determination of ash of paper and board.*
- BS 3748, *Method for the determination of stiffness of board.*
- BS 3983, *Method for determination of thickness and apparent bulk density or apparent sheet density of paper and board.*
- BS 4400, *Method for sodium chloride particulate test for respirator filters.*
- BS 4415, *Determination of the tensile properties of paper and board.*
- BS 4415-2, *Constant rate of elongation method.*
- BS 4468, *Method for the determination of the internal tearing resistance of paper.*
- BS 4497, *Recommendations for the detection and estimation of nitrogenous treating agents in paper.*
- BS 4499, *Method for determination of alkali solubility of pulp.*
- BS 4897, *Trace metal content of pulps.*
- BS 4897-1, *Method for determination of calcium content by edta titrimetric and flame atomic absorption spectrometric methods.*
- BS 4897-2, *Method for determination of copper content by extraction-photometric and flame atomic absorption spectrometric methods.*
- BS 4897-3, *Method for determination of iron content by 1,10-phenanthroline photometric and flame atomic absorption spectrometric methods.*
- BS 4897-4, *Method for determination of manganese content by sodium periodate photometric and flame atomic absorption spectrometric methods.*
- BS 6504, *Method for determination of water absorption of paper and board (Klemm method).*
- BS 6538, *Air permeance of paper and board.*
- BS 6538-3, *Method for determination of air permeance using the Gurley apparatus.*
- ISO 5628, *Paper and board — Determination of bending stiffness by static methods — General principles.*
- ASTM D2986, *Method for evaluation of air assay media by the monodisperse DOP (Di-iso-octyl phthalate) smoke test.*
- ASTM F316, *Pore size characteristics of membrane filters for use with aerospace fluids.*
- ASTM F795, *Standard practice for determining the performance of a filter medium employing a single-pass, constant-rate liquid test.*
- ASTM F796, *Standard practice for determining the performance of a filter medium employing a single-pass, constant-pressure, liquid test.*
- Tappi Method T461 om-84, *Flame resistance of treated paper and paperboard.*

Tappi Method T535 cm-85, *Stiffness of paperboard (resonance length method)*.

Tappi Method T543 pm-84, *Stiffness of paper (Gurley type stiffness tester)*.

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