British Standard

BRITISH STANDARD

BS 76:1974

Incorporating
Amendment no. 1

Specification for

Tars for road purposes

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Co-operating organizations

The Road Engineering Industry Standards Committee, under whose supervision this British Standard was prepared, consists of representatives from the following Government departments and scientific and industrial organizations:

Asphalt and Coated Macadam Association*
Association of Consulting Engineers
British Quarrying and Slag Federation*
British Tar Industry Association*
Cement and Concrete Association
Concrete Society (Design and Development Divisional Committee)
Contractors' Plant Association
County Surveyors' Society*
Department of the Environment*
Department of the Environment. Transport and Road Research Laboratory*
Federation of Civil Engineering Contractors
Federation of Manufacturers of Construction Equipment and Cranes
Greater London Council*
Institute of Petroleum
Institute of Quarrying
Institution of Civil Engineers*
Institution of Highway Engineers*
Institution of Municipal Engineers*
Institution of Structural Engineers
Ministry of Defence, Army Department
Refined Bitumen Association Limited
Road Emulsion Association Limited
Road Surface Dressing Association*
Sand and Gravel Association Limited*
Society of Chemical Industry*
Individual experts

The organizations marked with an asterisk in the above list, together with the following, were directly represented on the committee entrusted with the preparation of this British Standard:

British Steel Industry
London Chamber of Commerce (Inc.)
Low Temperature Coal Distillers Association
Standardization of Tar Products Tests Committee

This British Standard having been approved by the Road Engineering Industry Standards Committee, was published under the authority of the Executive Board on 31 December 1974

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Amendments issued since publication

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1961	May 1976	Indicated by a sideline in the margin

Contents

Foreword

This British Standard was last fully revised in 1964. The principle of distinguishing between two types of road tar was then retained, but it was noted that either type might be used for many purposes.

This revision is intended to be more specific, and to give more information of direct value to the user of road tar. There are still two types but these are more clearly defined as binders for surface dressing and binders for coated macadam respectively. The two types S and C correspond approximately to the type A and type B tars respectively of the earlier editions of the standard.

Some tests, such as for naphthalene and phenol, have been omitted because their continued inclusion does not seem to be justified, whilst, in the interests of rationalization, the number of viscosity grades has been reduced. The distillation tests have been retained but the retention of these tests will be reconsidered in due course, this revision being regarded as an interim stage in passing from a composition specification to a performance specification. It should be noted that the density at 20 °C is quoted instead of specific gravity at 15.5 °C/15.5 °C. This change is in line with current practice.

Although there are no specification requirements for insoluble content in toluene, trichloroethylene or dichloromethane (methylene chloride) this information will be required by the purchaser for the analysis of coated macadams. In addition information should also be given on the density of the binder at 20 °C to enable the user to make mass/volume conversions when necessary.

Two new performance tests have been introduced. The SEGAS test for surface dressing tars allows an assessment to be made of the hardening a tar will experience on the road. This helps rule out any binder that may take too long to set, or which may embrittle prematurely. The Beckton tray test is intended to assess the degree of hardening of coated macadam tars during commercial mixing at the specified temperatures, or, in the case of the 58 °C evt coating tar, at the temperature most likely to be used to meet other special conditions.

By agreement with the Standardization of Tar Products Tests Committee, hereinafter referred to as STPTC, the methods of testing and the descriptions of apparatus contained in the appendices have been reproduced, with editorial modifications, from their publication “Standard methods for testing tar and its products”, Sixth Edition 1967[1]. Tests approved since that date are also included in the standard.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 26, 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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[1] These methods are intended to be carried out in accordance with the general principles and apparatus requirements laid down in the STPTC publication mentioned.

1 Scope

This British Standard specifies the essential properties of a number of viscosity grades of two types of road tar as given below. These types differ mainly in their setting properties as controlled by the content of flux oil and its volatility.

a) Surface dressing tars, designated as the S series in Table 1, are intended to set more rapidly than coated macadam tars.

b) Coated macadam tars, designated as the C series in Table 2, are less volatile and their behaviour at a coating plant as well as on the road is indicated by the table.

Appendix K gives a list of publications and is intended as a guide to the selection of the appropriate viscosities of road tar used for various purposes.

2 References

The titles of the British Standards referred to in this standard are listed on the inside back cover.

3 Definitions

For the purposes of this British Standard the following definitions apply:

3.1
tar

a viscous liquid, black in colour, having adhesive and waterproofing properties, obtained by the destructive distillation of coal, wood, etc. Where no specific source is stated it is implied that the tar is obtained from coal

3.2
crude tar

tar which has not been subjected to change by distillation or other treatment

3.3
road tar

tar for use in road work prepared entirely from crude tars produced wholly or substantially as a by-product in the carbonization of coal at above 600 °C in externally heated retorts or coke ovens

3.4
equiviscous temperature (evt)

the temperature in degrees Celsius at which a tar has a viscosity of 50 s as determined on the standard tar viscometer (10 mm orifice)

3.5
repeatability

a measure of the precision of the test results obtained by one operator using one set of apparatus. In this standard repeatability *r* is the difference between duplicate results on the same sample that would be equalled or exceeded, in the long run, in only one case in twenty in the normal and correct operation of the test method

3.6
reproducibility

a measure of the precision of test results obtained by different operators using different sets of the specified apparatus. In this standard reproducibility *R* is defined as the difference between a single result by one operator at one laboratory and a single result on the same sample by another operator at another laboratory that would be equalled or exceeded, in the long run, in only one case in twenty

4 Properties

The tar shall be road tar as defined in **3.3**. The type and viscosity grade shall be specified by the purchaser; surface dressing tars shall comply with the appropriate column of Table 1 and coated macadam tars with the appropriate column of Table 2.

NOTE Although there are no specification requirements for insoluble content in toluene, trichloroethylene or dichloromethane (methylene chloride) this information will be required by the purchaser for the analysis of coated macadams (see Appendix H). In addition information will also be required on the density of the binder to enable the user to make mass/volume conversions when necessary (see Appendix J).

Table 1 — Properties of surface dressing road tars

Grade		S34	S38	S42	S46
Viscosity, equiviscous temperature (°C)		34 ± 1.5	38 ± 1.5	42 ± 1.5	46 ± 1.5
Water (maximum % by mass)		0.5	0.5	0.5	0.5
Distillation yield					
a) Oils below 200 °C	maximum % by mass	1.0	0.5	0.5	0.5
b) 200 °C to 270 °C	% by mass	4 – 13	3 – 10	2 – 8	2 – 7
c) 270 °C to 300 °C	% by mass	4 – 9	4 – 9	3 – 8	2 – 7
b) + c)	maximum % by mass	18	16	14	12
SPR[a] (°C R and B)		35 – 52	35 – 53	35 – 54	35 – 55
SEGAS test (°C evt increase)		20 ± 5	16 ± 5	12 ± 5	10 ± 4
Density[a] (g/ml) at 20 °C	min.	1.110	1.115	1.120	1.125
	max.	1.250	1.255	1.260	1.265

[a] For tars produced at carbonization temperatures below 950 °C (low temperature tars), the softening point and density shall be as follows:

		S34	S38	S42	S46
SPR (°C R and B)		30 – 52	30 – 53	30 – 54	30 – 55
Density (g/ml) at 20 °C	min.	1.060	1.063	1.067	1.070
	max.	1.250	1.255	1.260	1.265

Table 2 — Properties of coated macadam road tars

Grade		C30	C34	C38	C42	C46	C50	C54	C58
Viscosity, equiviscous temperature (°C)		30 ± 1.5	34 ± 1.5	38 ± 1.5	42 ± 1.5	46 ± 1.5	50 ± 1.5	54 ± 1.5	58 ± 1.5
Water (maximum % by mass)		0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Distillation yield									
a) Oils below 200 °C	maximum % by mass	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
b) 200 °C to 270 °C	% by mass	4 – 11	3 – 10	1 – 8	1 – 6	1 – 5	0 – 4	0 – 3	0 – 3
c) 270 °C to 300 °C	% by mass	4 – 9	4 – 9	4 – 9	3 – 8	2 – 7	2 – 7	2 – 7	1 – 6
b) + c)	maximum % by mass	16	15	13					
SPR (°C R and B)		35 – 46	35 – 46	35 – 47	35 – 48	35 – 49	35 – 50	52 max.	56 max.
Beckton tray test (maximum °C evt increase)									
at 80 °C		6	6	6	6	—	—	—	—
at 100 °C		—	—	—	—	8	8	8	—
at 140 °C		—	—	—	—	—	—	—	10
Density[a] (g/ml) at 20 °C	min.	1.105	1.110	1.115	1.120	1.125	1.130	1.130	1.135
	max.	1.245	1.250	1.255	1.260	1.265	1.270	1.275	1.275

[a] For tars produced at carbonization temperatures below 950 °C (low temperature tars), the density shall be as follows:

		C30	C34	C38	C42	C46	C50	C54	C58
Density (g/ml) at 20 °C	min.	1.060	1.060	1.063	1.067	1.070	1.073	1.077	1.080
	max.	1.245	1.250	1.255	1.260	1.265	1.270	1.275	1.275

5 Sampling

5.1 General. In practically all cases, samples of road tar are only required from bulk quantities of the material; in isolated cases small quantities in multiple containers may require sampling, and in these cases the general principles described in the appropriate clauses of BS 616 should be followed.

In sampling from bulk quantities of hot road tar it can be assumed that the material is essentially homogeneous except for the extreme top and bottom. Thus any sample taken with clean implements and placed in a clean, dry metal container will be representative of the bulk from which it is taken.

When a sample is taken from a bulk-delivery tanker, the material that the sample represents will be that particular delivery; but in the case of material in a purchaser's storage tank the sample will represent the contents of the tank, which will be a thorough mixture of the residue from a previous delivery and the new material provided the mixture has been circulated for at least 15 min.

5.2 Methods of sampling

5.2.1 *Sampling from a delivery tanker or storage tank.* Several subsamples of the hot road tar shall be taken from below the surface of the tar using a clean, dry and heat-proof ladle or similar vessel. Sufficient subsamples shall be taken to provide a main sample of at least 10 litres.

5.2.2 *Sampling from a pipeline.* It is first essential to check that the road tar flowing in the pipeline is the actual material that requires sampling; this check shall be jointly carried out by both parties interested in the material being sampled. It is also essential to ensure that there is no contamination from residues in the pipeline at the point of sampling. A main sample of at least 10 litres shall then be collected by any suitable method.

NOTE Extreme care is required to ensure that the adopted procedure is as safe as possible.

5.3 Subdivision of the main sample. The main sample, while still hot, shall be subdivided into three approximately equal parts, taking care to avoid undue loss of volatile material. The three portions shall be poured into clean and dry metal containers capable of being securely closed to prevent any leakage of the contents.

Each of the three portions shall be clearly labelled with the names of the supplier and the purchaser, details of the grade of road tar, time and date of sampling, and any other relevant information needed for identification.

The three portions of the main sample shall be distributed as indicated below:

Portion 1; to the purchaser to be used for his tests.

Portion 2; to the supplier to be used for his tests if he so requests.

Portion 3; to be retained in safe custody.

6 Testing

The road tar shall be prepared and tested in accordance with Appendix A to Appendix J as required. The thermometers required by Appendix B, Appendix D, Appendix E, Appendix F and Appendix J shall be as given in Appendix L

7 Compliance with the specification

The purchaser's test result shall be compared with the appropriate limit(s) given in clause **4**, Table 1 or Table 2. If the comparison indicates non-compliance the purchaser shall repeat the tests for which non-compliance is found and compare any differences in test results with the value for the repeatability of the test method.

Appendix A Preparation of sample for testing

A.1 Procedure

The procedure shall be as follows:

1) If the sample when received is cold, warm it until it is sufficiently fluid for pouring or mixing, avoiding any direct intense source of heat. For this purpose partially immerse the container in water at not more than about 30 °C higher than the expected equiviscous temperature (evt) of the material. Alternatively, a controlled temperature oven at a temperature not more than 30 °C above the expected evt may be used, provided that the container is insulated from the base if it is directly heated. The period of warming shall be the minimum required to secure the necessary degree of fluidity.

2) If the sample when received is hot, cool it without undue exposure to the air to a temperature about 30 °C higher than the expected evt of the material.

3) Strain the warm sample through a 600 μm test sieve complying with the requirements of BS 410. Make all tests on the strained material and, immediately before any portion is withdrawn for testing, mix the sample thoroughly; it should, therefore, be contained in a vessel large enough to permit adequate agitation.

NOTE While the tar is hot it is important that the container remain covered as far as is practicable, otherwise loss of oil is likely to take place with consequent change in the character of the sample.

A.2 Reporting

Report the presence of any foreign matter remaining on the sieve.

Appendix B Method for the determination of equiviscous temperature

(Technically identical with STPTC Methods RT 2-67 and RT 3-67)

B.1 General

Determine the evt in two stages as follows:

a) Determine the viscosity of the tar in seconds by using the standard tar viscometer as described in this appendix. The range of viscosity for conversion to evt [see **B.1**b)] is limited to 33 s to 75 s and the temperature of test shall be such that the result of the test is within these limits. A convenient temperature is the multiple of 5 °C nearest to or 2.5 °C above the expected evt and consequently the multiple nearest to the grade temperature will usually give the required efflux time. If however the test is not in the range of 33 s to 75 s the test shall be repeated at the temperature which will give the required result. This temperature can be found by using Table 3.

b) Find the evt by applying the correction shown against the viscosity in Table 3, to the test temperature. For example, if the viscosity is 55 s at a test temperature of 40 °C, the correction from Table 3 is ± 0.6° and the evt is 40.6 °C.

NOTE Figure 11 shows the approximate relationship between evt and efflux times at various test temperatures and also the relationship with the penetration values. This diagram can *not* be used for the calculation of evt.

B.2 Apparatus

The following apparatus is required:

a) *Standard tar viscometer* with 10 mm cup, as shown in Figure 1 to Figure 3 and specified below.

1) *Tar cup*, as shown in Figure 3 consisting of a hard brass tube, fitted at the upper (open) end with an external brass collar serving to support the cup in the sleeve of the water bath. The bottom of the cup is made slightly conical and consists of a circular phosphor bronze plate screwed to the cylinder; it is provided centrally with a phosphor bronze (90/10 cast) extension which is drilled and polished internally to give an accurately cylindrical opening having dimensions within the following tolerances:

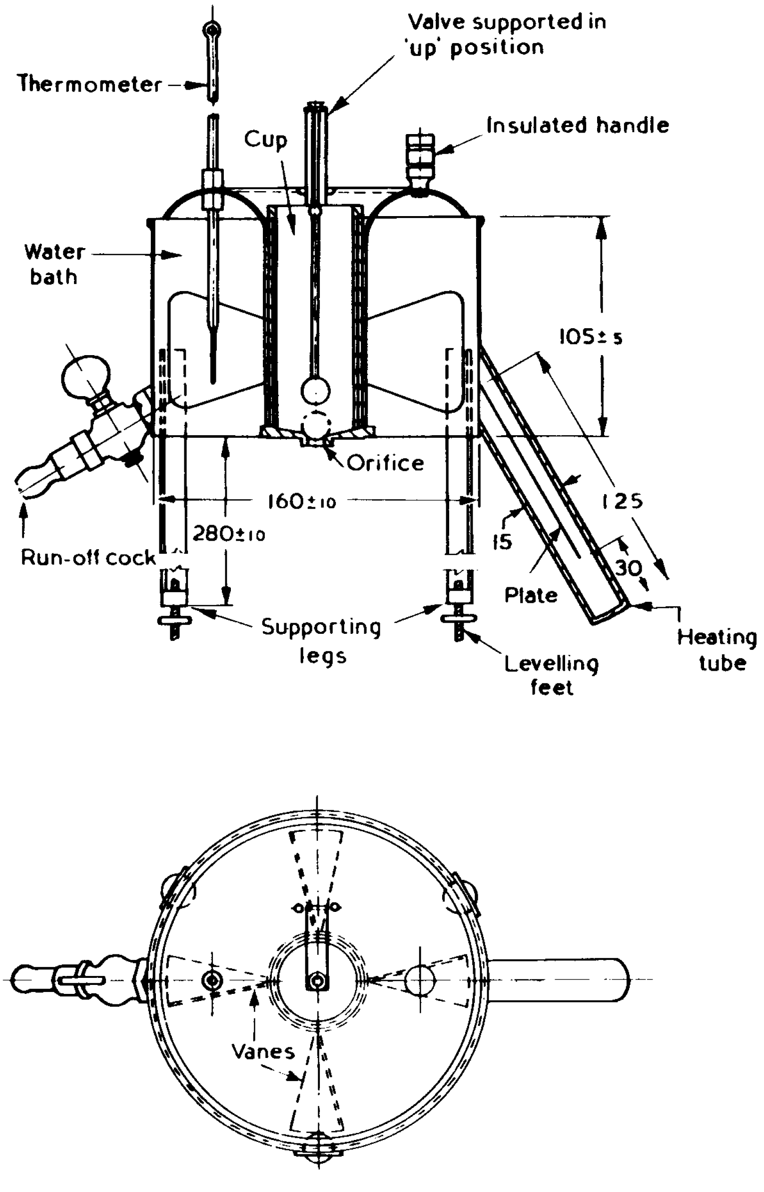
Diameter of opening	10 ± 0.025 mm
Length of jet	5 ± 0.025 mm

2) *Valve*. The jet is closed by a valve, shown in Figure 3, which consists of a phosphor bronze sphere attached to a rod of nickel-copper alloy complying with the requirements of BS 3076 grade NA 13. The rod is provided with a level-indicating peg, and at the upper end with a hemisphere by which the valve can be rested in the valve support.

3) *Water bath*, a cylindrical vessel of a suitable metal which is provided with a small brass run-off cock and a side tube for heating. This tube is partitioned for part of its length by a flat plate extending from the wall of the bath to about 30 mm from the closed lower end. Both the cock and the side tube are brazed or soldered to the bath and, to ensure normal convection, it is essential that the internal joints be smooth and free from projecting metal. A stout brass tube, 105 mm in height and of such a bore as to provide an easy sliding fit for the cup, is brazed or soldered into a central hole in the bottom of the bath to form a sleeve for the cup. It is essential that the upper rim of the sleeve be in a plane at right angles to the longitudinal axis of the sleeve. Three equidistant legs of sufficient length to allow the receiver to stand beneath the orifice of the cup are riveted to the cylindrical wall of the bath. Levelling screws are provided at the lower extremities of the legs.

NOTE It is not intended that viscometers which are already in use and are not provided with levelling screws should no longer be regarded as standard instruments.

The water bath may be adapted for electrical heating, provided that care is taken to avoid the possibility of local overheating.



All dimensions are in millimetres

Figure 1 — Standard tar viscometer, assembled

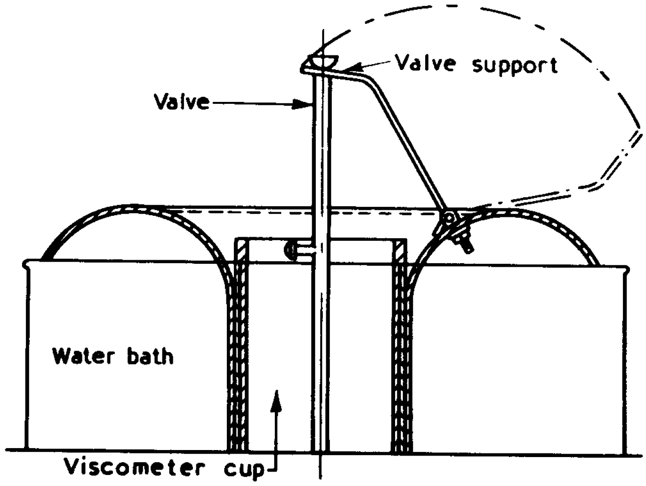


Figure 2 — Section showing arrangement of valve support

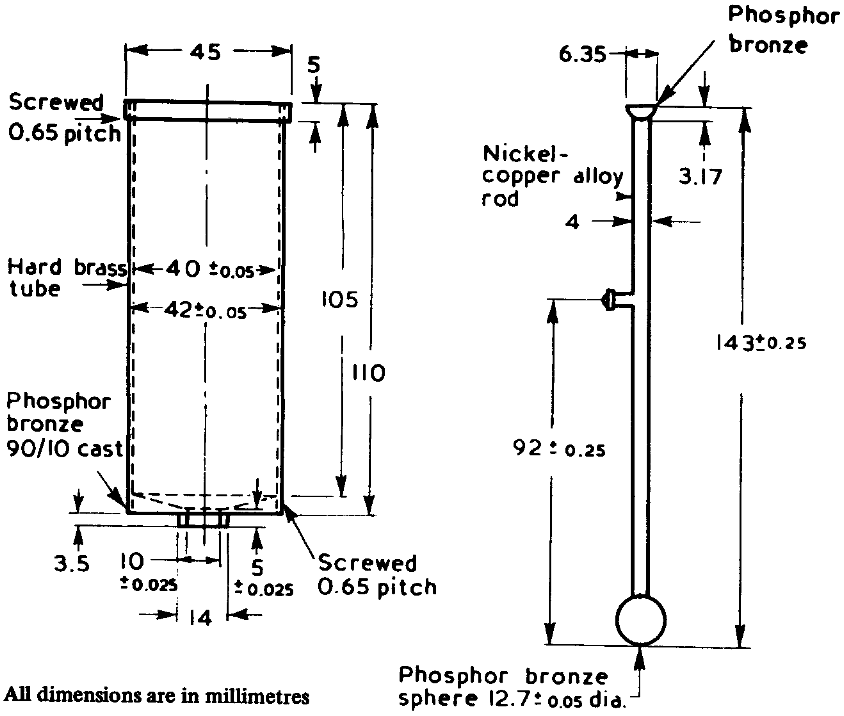


Figure 3 — Standard 10 mm tar cup and valve

4) *Stirring device,* consisting of a cylinder which surrounds the sleeve with an easy sliding fit and provided with four vanes; the upper and lower portions of the vanes are twisted in opposite directions. The cylinder is cut away between the vanes to facilitate heat transfer from the water in the bath to the contents of the tar cup. Vertical grooves are provided on the inner surface of the cylinder, to prevent water in the bath from entering the tar cup if the stirrer is raised. A curved shield is attached to the upper edge of the cylinder and extends to within about 5 mm of the rim of the water bath. This shield carries an insulated handle for rotating the stirrer, a support for the bath thermometer and a swivelled support for the valve (see Figure 2). When this valve support is in use in its inner position with the valve resting on it, the base of the valve hangs vertically over and 16 ± 1 mm above the orifice in the tar cup.

5) *Multiple-cup water bath*. When it is desired to carry out a number of tests concurrently, it may be advantageous to use a multiple-cup water bath accommodating a number of tar cups. Such a bath shall be fitted with a mechanical stirrer and with thermostatic control, and be provided with several cylindrical sleeves in which the tar cups can be supported. It is important that the distances between the sleeves and also between the sleeves and the sides of the bath shall be about the same as the distance between the sleeve and the side of the bath in the single-unit form of the viscometer. With larger baths more heat will be required to compensate for radiation loss. It is essential that the source shall be so located that heat is not conducted preferentially to one or more of the tar cups, resulting in local overheating.

b) *Thermometers,* for the water bath and tar cup, conforming to STPTC Schedule No. T5d or T17d, as specified in Appendix L to this standard, according to the temperature of test.

c) *Receiver*. A 100 ml graduated measuring cylinder complying with the requirements of BS 604-1, or a 100 ml Crow receiver complying with the requirements of BS 658, Type 1 or 2, Class B or a similar cylinder of not greater than 24 mm internal diameter and graduated only for 20 ml, 25 ml and 75 ml capacities.

d) *Timing device*. If the device is a stop-watch, it shall be of the jewelled-lever type, fully but not tightly wound, graduated in divisions of 0.2 s or less and accurate to within 0.07 % when tested over a period of not less than 15 min and when in use shall be held in the same position as when tested.

NOTE The National Physical Laboratory tests stop-watches mainly in the "dial-up" position.

B.3 Procedure

The procedure shall be as follows:

a) The procedure described below is applicable to the operation of a single-cup instrument. The procedure for a multiple-cup instrument is precisely the same except that the water bath thermometer is held in a clamp attached to the bath itself and, with the provision of mechanical stirring, there is no need for hand stirring. If thermostatic control is fitted it should maintain the water bath temperature automatically within the required limits.

b) Set the water bath by the aid of a spirit level and the levelling screws so that the upper rim of the sleeve is horizontal. Place the stirrer assembly, carrying the water bath thermometer, in position and fill the water bath to within approximately 10 mm of the top.

c) Place a cup, with orifice and valve appropriate to the material under examination, in the sleeve.

d) Bring the contents of the water bath to a temperature 0.05 °C higher than the temperature of test and maintain that temperature to within ± 0.05 °C throughout the test, i.e. maintain the water bath temperature between that of the test and 0.1 °C higher. Rotate the stirrer at frequent intervals or, as far as possible, continuously.

e) After preliminary treatment as described in Appendix A, warm the tar to a temperature approximately 20 °C higher than the temperature of the test and mix thoroughly. Pour not less than 250 ml of the sample into a beaker or similar vessel and allow to cool, with continuous stirring, to a temperature about 0.5 °C above the temperature of test. Close the orifice of the cup by the valve and pour tar from the beaker into the cup until, when the valve rod is vertical, the levelling peg is just immersed.

f) Place the second thermometer, as specified, in the tar in the cup and use it to stir the tar gently, to attain uniformity of temperature, until the temperature indicated is that of the test or is within 0.1 °C higher. On no account shall the temperature be lower than that of the test. Check that the temperature of the water bath is within the limits specified above. Place the receiver, containing 20 ml of mineral oil or of a 1 % *m*/*m* solution of soft soap (see note 1) under the orifice of the cup.

g) Suspend the tar thermometer coaxially within the cup with the 100 mm immersion mark on the thermometer level with the top of the hemisphere on the upper end of the valve; the bulb will then be approximately at the geometric centre of the tar. Allow the apparatus to stand until the thermometer reading falls to the temperature of the test. Withdraw the tar thermometer with minimum disturbance of the tar and quickly remove any excess of tar from the thermometer, for example with a strip of paper held tightly round the thermometer. Remove any excess tar in the cup so that the final level is on the centre line of the levelling peg when the valve is in the vertical position. Lift the valve support to the position over the tar cup and then lift the valve and hang it immediately in the valve support. Tar will flow from the cup into the receiver. Commence timing when the meniscus of the oil or of the soap solution reaches the 25 ml mark and stop when it reaches the 75 ml mark; i.e. note the time in seconds for the efflux of 50 ml of tar ignoring the first 5 ml which flows out. The flow of tar may be stopped after the completion of the timing by replacing the valve so as to close the orifice.

NOTE 1 If the receiver is emptied immediately after a test, the liquid initially present in the cylinder prevents soiling of the internal walls by the tar.

NOTE 2 The tar cup is a critical part of the viscometer and special precautions should be observed in its treatment and use. It should be cleaned after each test and any cleaning process should be of a gentle nature; non-corroding solvents such as the lighter tar oils free from phenols and bases are recommended rather than mechanical means such as brushes, which are liable to cause abrasion of the metal, especially at the orifice. Finally a soft cloth should be used to render the cup clean and dry.

The dimensions of the orifice of the cup should be tested at frequent intervals with a GO-NOT GO gauge having appropriate dimensions.

B.4 Reporting

Report the evt obtained by correcting the test temperature in accordance with **B.1**b).

B.5 Precision

Repeatability r. Duplicate results by the same operator should be considered suspect if they differ by more than 0.3 °C evt.

Reproducibility R. Single results submitted by each of two laboratories should be considered suspect if they differ by more than 0.9 °C evt.

Table 3 — Correction in degrees Celsius to be applied to temperatures of test to give evts

Viscosity in seconds	0	1	2	3	4	5	6	7	8	9
10	– 10.4	– 9.8	– 9.2	– 8.7	– 8.2	– 7.7	– 7.3	– 6.9	– 6.5	– 6.1
20	– 5.7	– 5.4	– 5.1	– 4.8	– 4.5	– 4.3	– 4.0	– 3.8	– 3.5	– 3.3
30	– 3.1	– 2.9	– 2.7	– **2.5**	– **2.3**	– **2.2**	– **2.0**	– **1.9**	– **1.7**	– **1.5**
40	– **1.4**	– **1.2**	– **1.1**	– **0.9**	– **0.8**	– **0.6**	– **0.5**	– **0.4**	– **0.3**	– **0.1**
50	**0**	**+ 0.1**	**+ 0.2**	**+ 0.3**	**+ 0.5**	**+ 0.6**	**+ 0.7**	**+ 0.8**	**+ 0.9**	**+ 1.0**
60	**+ 1.1**	**+ 1.2**	**+ 1.3**	**+ 1.4**	**+ 1.5**	**+ 1.6**	**+ 1.7**	**+ 1.7**	**+ 1.8**	**+ 1.9**
70	+ **2.0**	+ **2.1**	+ **2.2**	+ **2.2**	+ **2.3**	+ **2.4**	+ 2.5	+ 2.5	+ 2.6	+ 2.7
80	+ 2.8	+ 2.8	+ 2.9	+ 3.0	+ 3.0	+ 3.1	+ 3.1	+ 3.2	+ 3.3	+ 3.3
90	+ 3.4	+ 3.5	+ 3.5	+ 3.6	+ 3.6	+ 3.7	+ 3.7	+ 3.8	+ 3.9	+ 3.9
100	+ 4.0	+ 4.0	+ 4.1	+ 4.1	+ 4.2	+ 4.2	+ 4.3	+ 4.3	+ 4.4	+ 4.4
110	+ 4.5	+ 4.6	+ 4.6	+ 4.7	+ 4.7	+ 4.8	+ 4.8	+ 4.9	+ 4.9	+ 5.0
120	+ 5.0	+ 5.1	+ 5.1	+ 5.2	+ 5.2	+ 5.2	+ 5.3	+ 5.3	+ 5.4	+ 5.4
130	+ 5.5	+ 5.5	+ 5.5	+ 5.6	+ 5.6	+ 5.7	+ 5.7	+ 5.7	+ 5.8	+ 5.8
140	+ 5.9	+ 5.9	+ 6.0	+ 6.0	+ 6.0	+ 6.1	+ 6.1	+ 6.1	+ 6.2	+ 6.2

NOTE That part of the table giving corrections for tars having viscosities between 33 s and 75 s inclusive (indicated by bold type) may alone be used in calculating the evt to be reported. The remainder of the table will be useful in ranging tests.

Appendix C Method for the determination of water content

(Technically identical with STPTC Serial No. RT 4-67)

C.1 Apparatus

The following apparatus is required:

a) *Dean and Stark apparatus* complying with the requirements of BS 756, with a 2 ml receiver.

A glass or metal flask of about 500 ml capacity is used as distillation vessel.

b) *100 ml graduated measuring cylinder* complying with the requirements of BS 604-1.

C.2 Reagent

One of the following solvents is required:

a) *Coal-tar solvent naphtha* (90/160) complying with the requirements of BS 479/3.

b) *3° xylene or 5° xylene* complying with the requirements of BS 458/1 or BS 458/4 as appropriate.

C.3 Procedure

The procedure shall be as follows:

a) Weigh 100 ± 0.5 g of the thoroughly mixed sample into the flask and add 100 ml of solvent.

b) Attach the flask to the Dean and Stark apparatus. Heat the flask so that condensate falls from the end of the condenser at the rate of 2 to 5 drops per second.

c) Continue the distillation until condensed water is no longer visible in any part of the apparatus except the bottom of the graduated tube and until the volume of water collected remains constant.

If a persistent ring of condensed water forms in the condenser tube, remove it by increasing the rate of distillation by a few drops per second.

d) Bring the water collected to laboratory temperature, note its volume, and correct for any errors of the receiver.

C.4 Reporting

Assume the density of the water collected in the graduated tube to be 1.00 g/ml and report the volume (ml) at laboratory temperature as the percentage by mass in the sample.

Appendix D Method of distillation

(Technically identical with STPTC Method RT 5-73)

D.1 Apparatus

The following apparatus is required:

a) *Tared 750 ml distillation flask* complying with the requirements of BS 658. A further flask of suitable size may be required to distil the water from 1 000 ml of sample.

b) *Thermometer* with a range from – 2 °C to + 400 °C, so fitted in the flask that the bottom of the capillary is level with the lower edge of the side tube. The thermometer to be used is STPTC T4d, as specified in Appendix L to this standard, having a minimum scale length of 250 mm graduated in degrees, a maximum error of ± 4 °C and an immersion of 100 mm.

c) *Type 2 draught screen* complying with the requirements of BS 658. When the flask is placed in position on the shelf it is pressed down to close completely the 110 mm hole in the shelf.

d) *Air condenser* complying with the requirements of BS 658 Type 2. The side arm of the flask extends at least 25 mm beyond the cork in the upper end of the condenser.

e) *Retort stand and clamp* to hold the condenser.

f) *One 25 ml and two 100 ml Crow receivers* complying with the requirements of BS 658 Type 1, Class B, all tared.

g) *Large bunsen burner*.

D.2 Procedure

If the water content of the sample, as determined according to Appendix C, is found to be less than 0.5 % *m*/*m*, carry out the test on the sample as received after the preliminary treatment described in Appendix A.

If the water content as found above is greater than 0.5 % *m*/*m*, take about 1 000 ml of the sample and, after the preliminary treatment, distil it from a suitable flask fitted with a condenser until all the water has come over. Allow the distillate to stand and, after gentle warming if necessary, separate the water from the oil which has also distilled over. After the necessary cooling return this oil to the dehydrated tar in the flask, and thoroughly mix the contents of the flask before using for the test as described below.

a) Weigh 750 ± 1 g of the prepared tar into the weighed 750 ml distillation flask and assemble the apparatus. When the flask is placed in position on the shelf of the draught screen press it down to close completely the 110 mm hole in the shelf. Collect the first fraction in the 25 ml weighed Crow receiver. Place this and the subsequent 100 ml receivers so that the condensate flows down the side. Warm the larger receivers immediately prior to use.

b) Heat the flask so that the temperature indicated by the thermometer reaches 200 °C within 35 ± 5 min from the start. With certain types of tar this is not always possible due to frothing or difficulty associated with condensed water in the neck of the flask. In these cases difficulties can often be reduced by gently warming the upper surface of the flask with a non-luminous bunsen or similar flame. Such external heat shall not be applied after the temperature has reached 200 °C.

c) If the time taken to reach 200 °C exceeds 40 min, record the time taken in the analyst's report.

d) It is essential to maintain a steady rate of distillation throughout the whole test, and while the actual rate is important, it is more important to avoid fluctuations than to attempt to achieve a particular overall average rate. The nominal rate of distillation is 5 ml/min. To achieve uniformity, record the time taken for each 5 ml of distillate which should be 54 s to 66 s. These conditions do not apply to the first 5 ml of distillate for which the collection period may extend to 75 s (see note to **D.3**).

e) Only for work of the greatest accuracy is it necessary to correct the thermometer readings for scale errors and barometric pressure. Change the receivers at 200 °C and 270 °C (running points); 300 °C is the stop-point.

If solids tend to deposit during the distillation, warm the condenser so that they are collected in the fraction in which they come over.

Extinguish the bunsen flame immediately the highest specified temperature is reached and include in the final fraction all oil which drains from the condenser within 5 min after the flame has been extinguished.

After cooling, ascertain the mass in grams of the distillate to 200 °C, note the volume of any water collected and, assuming a density of 1.00 g/ml, record its mass in grams. The difference gives the mass of the light oil in the fraction to 200 °C. Determine the mass of the other fractions to 0.1 g.

After extinguishing the flame, leave the flask and contents with the cork and thermometer in position, to cool, but not to a temperature below which the residue is no longer fluid. Weigh the flask and contents, then mix the latter thoroughly by shaking and pour out about half. Mix the remainder again and pour the warm material directly from the flask into the pitch rings, preheated for use in the ring and ball test as described in Appendix E.

D.3 Reporting

Report the following information calculated to the first place of decimals on the original mass of prepared tar:

	% water (*m*/*m*)
Fraction (a)	% oil distilling to 200 °C (*m*/*m*)
Fraction (b)	% oil distilling between 200 °C and 270 °C (*m*/*m*)
Fraction (c)	% oil distilling between 270 °C and 300 °C (*m*/*m*)
Total (b) + (c)	% (*m*/*m*)

Report the time in seconds for each 5 ml to distil.

NOTE Each 5 ml should distil in 54 s to 66 s, but the limits may be extended to 50 s to 70 s provided that the time for any 5 ml does not differ from that for any other by more than 5 s.

The specified distillation rate corresponds approximately to 90 drops per minute or 3 drops in each 2 s. This may be taken as a guide, the receiver being moved slightly from time to time to enable the drops to be counted; but graduated receivers are specified for the collection of the distillate so that the time taken to collect each 5 ml may be kept under close observation.

D.4 Precision

The precision of this test has not been determined by the usual rigorous procedure. However, from some work done by five different laboratories, there is reason to believe that the reproducibility is as indicated below.

The mean of two results in any one laboratory should not differ from the mean of two results in another laboratory by more than the following:

Fraction (b)	1 % *m*/*m*
Fraction (c)	1 % *m*/*m*
Fractions (b) + (c)	1.5 % *m*/*m*

It is essential however to exercise close attention to detail if this reproducibility is to be achieved.

Appendix E Method for the determination of softening point of distillation residue (ring and ball test)

(Technically identical with STPTC Serial No. PT 3-67)

E.1 Apparatus

The apparatus shown in Figure 4 and specified below is required:

a) *Rings*. Tapered pitch rings [Figure 4(b)] made of brass to the following dimensions:

	mm
Depth	6.35 ± 0.10
Internal diameter at bottom	15.88 ± 0.10
Internal diameter at top	17.46 ± 0.10
External diameter above shoulder	20.60 ± 0.10
External diameter below shoulder	18.45 ± 0.45

b) *Ring support*. A convenient form of ring support is shown in Figure 4(a) and Figure 4(c). It consists essentially of two brass plates, the upper one of the form and dimensions shown in Figure 4(c) and designed to carry two rings. The support is attached to a circular brass plate or rectangular strip which rests on the top of the beaker and is provided with a hole to support the thermometer in the vertical axis of the beaker, as shown in Figure 4(a).

The depth of the rings below the shoulder is approximately the same as the thickness of the ring-supporting plate. The lower is about 25 mm in width and about 75 mm in length, and is fixed so that its upper surface is 25 ± 0.5 mm below the lower surface of the ring-supporting plate.

c) *Steel balls*, each having a diameter of 9.53 mm and a mass of 3.50 ± 0.05 g.

d) *Thermometer*, conforming to STPTC Schedule No. T3d as specified in Appendix L to this standard.

e) *Heating bath*, consisting of an 800 ml beaker, of squat form.

f) *Tripod and draught screen*. The apparatus when assembled is supported on the tripod on a square of open mesh wire gauze. The whole assembly is protected from draughts by a draught screen of sufficient height.

g) *Bunsen burner*, with a governor if the gas supply is liable to fluctuate.

h) *Oven*.

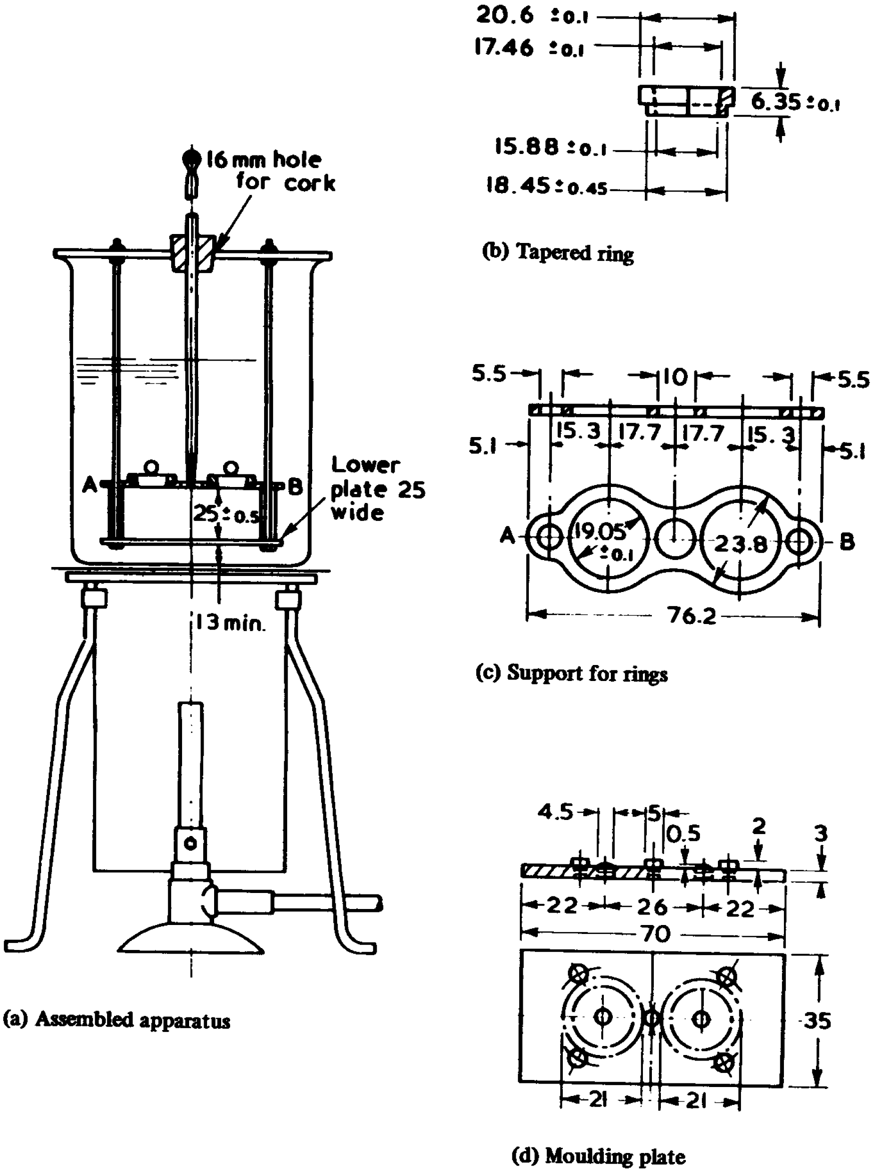
j) *Moulding plate*, of the dimensions shown in Figure 4(d).

E.2 Procedure

Place the pitch rings in an oven at about 100 °C for approximately half an hour. Invert the rings and place one within each of the sets of guide pins on the moulding plate which has been treated, e.g. by amalgamating with mercury or by lightly covering with dextrin and glycerol paste, to prevent adhesion of the pitch (i.e. the residue from the distillation test). Immediately pour the second half of the residue remaining from the distillation [see **D.2**d)] directly from the distillation flask into the pitch rings such that a slight excess of residue remains above the level of the ring. After the residue in the rings has cooled, remove the excess with a knife and without applying heat. (Retain the residue remaining in the flask in case a repeat determination is required.)

The two rings shall be filled with the residue from one distillation and shall be tested together.

Cool the water to be used for the heating bath to at least 45 °C below the expected result of the test, except that for residues of low softening point the temperature of the water should not be below 5 °C. Pour sufficient water into the beaker (usually about 600 ml) so that the final liquid level is at least 50 mm above the top of the rings, avoiding any aeration. Fit the filled rings into the support and place the latter concentrically in the beaker. Insert the appropriate thermometer and adjust it so that it lies along the axis of the beaker and so that the bottom of the bulb is level with the bottom of the rings and central with the central hole of the ring-supporting plate. Place the balls in the liquid in the beaker but not in position on the residue in the rings. Set up the apparatus on the tripod so that the ring-supporting plate is horizontal and allow to stand without the temperature rising above that of the liquid when the apparatus was filled. After 15 min place the balls by means of tongs in the central indentations on the upper surface of the residue in the rings.



(a) Assembled apparatus

(b) Tapered ring

(c) Support for rings

(d) Moulding plate

All dimensions are in millimetres

Figure 4 — Apparatus for ring and ball test

Place the burner under and midway between the centre and edge of the beaker and on a diameter at right angles to that which includes the rings and the thermometer. Heat so that the temperature is raised 5 °C each minute; maintain this rate within ± 0.5 °C over each 1 min period after the first 3 min; it shall not be merely the average over the period of the test. Any test in which the heating rate does not conform to these requirements shall be rejected.

Note the temperature at which the residue surrounding each steel ball first touches the lower plate of the support.

E.3 Reporting

Report the mean of the above mentioned temperatures to the nearest 0.2 °C.

If the two balls fall at temperatures differing by more than 1.0 °C discard the result and repeat the test.

If for any reason the test has to be repeated, the whole procedure shall be followed again using the residue remaining in the flask from the same distillation. This residue shall be reheated (if necessary) to about 150 °C prior to pouring directly into another pair of (preheated) rings.

E.4 Precision

Repeatability r. Duplicate results (each the mean of two figures) by the same operator should be considered suspect if they differ by more than 0.5 °C.

Reproducibility R. The reproducibility of this test has not been determined by the usual rigorous procedure. However, from some work done by five different laboratories, there is reason to believe that the reproducibility may be of the order indicated below.

The mean of two results (each of separate distillations) in any one laboratory should not differ from the mean of two results in another laboratory by more than 3 °C.

Appendix F Method for the determination of hardening by the Beckton tray test

(Technically identical with STPTC Method RT 110-73)

F.1 Introduction

The method determines the rise in evt which a film of tar undergoes during 1 h at a particular temperature and allows an assessment to be made of the hardening which will be experienced by the tar in a commercial coating plant operating at the same temperature. Thus this test only applies to the coating (C) tars.

F.2 Apparatus

The following apparatus is required:

a) *Brass tray*. A tray 100 mm × 100 mm × 10 mm in depth made of sheet brass of 0.8 ± 0.1 mm thickness.

b) *Viscometer*. A direct reading evt viscometer.

The essential component parts of the viscometer are shown in Figure 5. A specified volume of the tar contained in a rigidly held cylindrical cup is caused to exercise a viscous drag on the rotational oscillation of a cylinder which is partly immersed in the tar. The tar cup, which is made of stainless steel or heavily chromium-plated brass, has an internal diameter of 22 mm and a depth of 38 mm. The cylinder, also made of stainless steel or chromium-plated brass, is 16 mm in diameter and 34 mm in length. The cylinder is centred in the cup and the clearance between the bottom of the cylinder and the inner surface of the cup is 3 mm. The cylinder hangs from a straight beryllium-copper alloy torsion wire 200 mm in length and 0.65 mm in diameter. The wire also carries at its lower end a circular flywheel of diameter 92 mm and mass 340 g. This flywheel, which may be of any suitable metal, is positioned on the wire at a suitable distance above the level of the water in the water bath. The flywheel is graduated on its upper surface in 45° divisions and rotates beneath a fixed pointer. The upper end of the torsion wire is carried by the torsion head which can be turned through 180° between two stops. The torsion head and the tar cup are suitably attached to a supporting pillar which can be set by levelling screws (not shown in Figure 5) so that the main axes of the cup, the cylinder, the torsion wire, the flywheel and the torsion head are co-linear and vertical. The tar cup and cylinder are completely immersed in the water bath which is equipped with a manually operated paddle-stirrer.

The temperature of the water in the water bath can be raised at a controlled rate by means of a suitable heating appliance such as a gas burner with needle-valve control or an electric heating element controlled by a variable resistance.

For calibration of the instrument a gauge is used. This consists of a pointed metal rod about 40 mm long and threaded No. 4 BA, which screws through a hole in the centre of a metal cross-piece about 35 mm in length.

c) *Thermometer*. STPTC T9c or T9d. A T5d or T17d thermometer is needed for the evt determination (see Appendix L to this standard).

d) *Balance*. A balance having a direct reading scale graduated in 0.1 g divisions is suitable.

e) *Laboratory oven*. The performance of laboratory ovens is known to vary appreciably and the precision values quoted at the end of the method may not be obtainable if two laboratories use different types of oven. In determining the precision the Baird and Tatlock Stabilec Oven, Small Model No. 322/0200, Ref. 02, for 200 V to 250 V a.c. supply was used. If another oven is used this shall be calibrated against the Baird and Tatlock Oven.

The oven has an internal height, width and depth of 355 mm × 305 mm × 305 mm respectively and two shelves. It is ventilated at the top with a 19 mm diameter vent through which the thermometer, specified above, is inserted. The oven has a series of holes in its base so that air is drawn inside from beneath by convection and also a scaled dial on the outside to permit control of the working temperature.

To achieve the maximum heating rate a 200 V to 250 V a.c. model should be used with its thermostat sensing phial correctly set with respect to the heater (usually called the "long range" position of the phial[2]). The oven is lagged between its inner and outer walls with material such as glass wool or asbestos and should be placed in a draught free position in the laboratory during the test.

F.3 Use of direct reading viscometer

F.3.1 *Determination of evt.* Place the instrument, which has been recently calibrated as described in **F.3.2**, on a level, plane surface, and assemble it with the tar cup empty. Adjust the levelling screws so that, when hanging freely, the cylinder is centrally disposed within the cup. Remove the cup to a level surface and place the gauge in position. Pour the sample of tar, prepared by the standard preliminary procedure described in Appendix A, into the cup until the level of the tar just touches the tip of the screwed rod of the gauge. During this operation take care to avoid splashing tar on the upper inner surface of the cup.

Clamp the filled cup in position on the viscometer with the cylinder in place and pour water into the water bath up to the indicator line so that the cup and cylinder are immersed. Set the pointer so that it is directly over one of the graduation lines on the flywheel.

Adjust the temperature of the water in the bath to not less than 10 °C below the expected evt of the sample. Then raise the temperature at a uniform rate of 1 °C per minute. Agitate the water continuously and vigorously and periodically turn the torsion head smartly through 180°. As the tar becomes more fluid the flywheel will develop a gradually increasing overswing beyond its normal position of rest. As soon as the overswing commences, rotate the torsion head as frequently as possible, allowing the flywheel to come to rest after each rotation. Record the temperature in degrees Celsius at which an overswing of one 45° division is first attained as the evt of the tar.

F.3.2 *Calibration of viscometer.* Set the gauge so that the distance from the underside of the cross-piece to the tip of the screwed rod is about 23 mm. Carry out the procedure described above on a tar of known evt, preferably about 35 °C, as determined by the procedure described in Appendix B. If the temperature at which the flywheel overswings one 45° division is higher than the determined evt of the tar, lower the screwed rod; if the temperature is lower, raise the rod. One complete turn of the rod will alter the temperature at which the required overswing is attained, by about a quarter of a degree Celsius. Repeat the test, with a fresh quantity of tar, and continue this procedure until the temperature at which the flywheel overswings by one division is within ± 0.1 °C of the determined evt of the tar. Fix the position of the rod by soldering it to the cross-piece.

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[2] See Baird and Tatlock Leaflet No. 183.

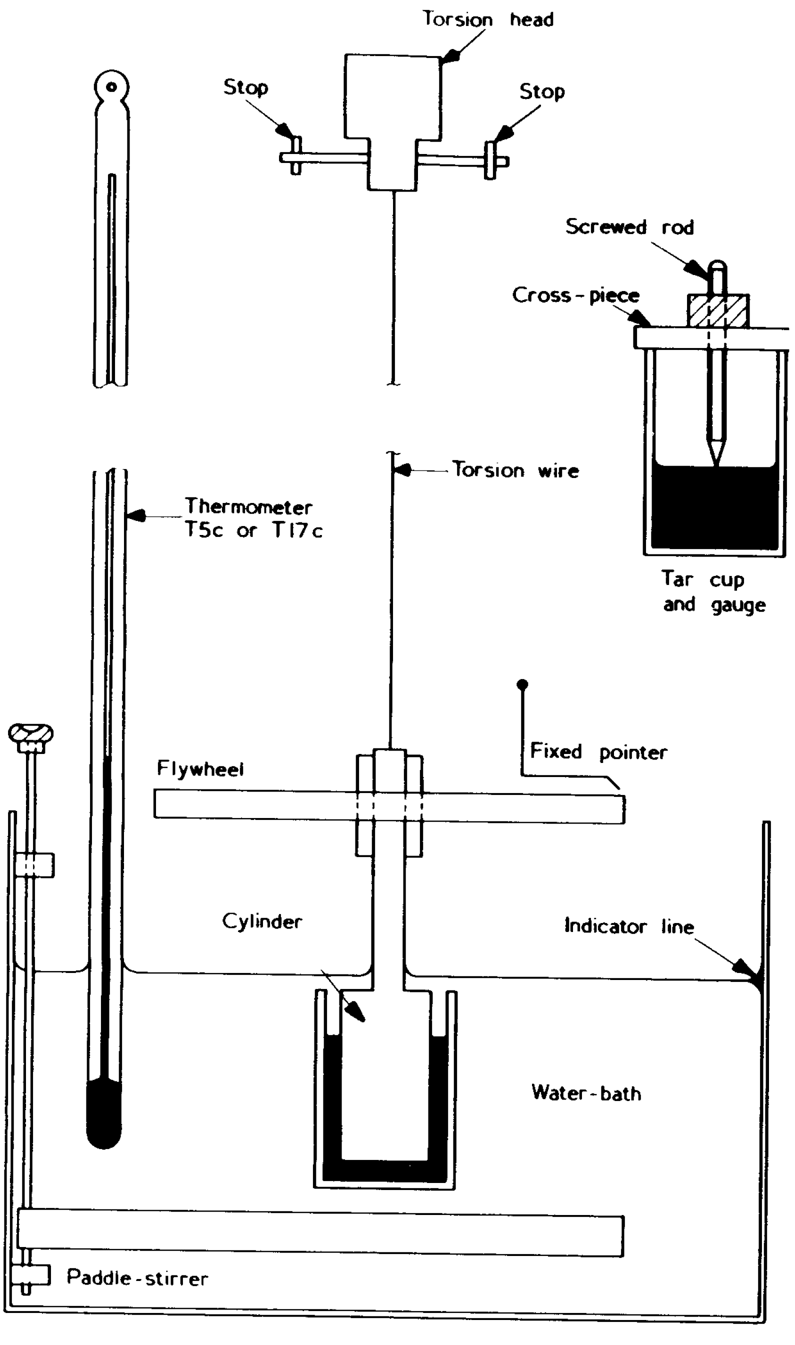

Figure 5 — Diagrammatic sketch of direct reading evt viscometer

F.4 Procedure

The procedure shall be as follows:

a) Place the tray in the oven at the centre of the upper shelf and adjust to a horizontal level by placing suitable packing underneath the oven corners and using a spirit level. Suspend the thermometer through the vent in the top of the oven by means of a piece of wire and adjust its height so that the bottom of the bulb is 10 mm above the upper edges of the tray. Remove the tray from the oven.

b) Raise the oven to the temperature at which the test is to be carried out (to within an accuracy of ± 1 °C) by setting the control dial on the outside of the oven at the FULL ON position (i.e. to give the maximum heating rate) and then turning the dial back a few divisions when the oven is about 10 °C below the desired temperature. Make final adjustments to the dial as the oven temperature approaches the required value and once the dial has been set to give the correct temperature do not alter it during the subsequent stages of the test. The oven shall remain at the required temperature for about 1 h before commencement of the test to ensure equilibrium conditions have been attained.

c) Whilst the oven is being heated to the temperature required, measure the initial evt of the tar to the nearest 0.1 °C using the direct reading viscometer. Calibrate the instrument before use by the procedure given in **F.3.2**.

d) Weigh into the clean, dry tray an amount of tar, in grams, equal to 12.5 times its density at 20 °C, to an accuracy of 0.1 g. Heat the tar to 80 °C to 100 °C prior to the weighing operation but exercise care to avoid overheating or prolonged heating.

e) When the oven has been at the desired temperature for 1 h, quickly place the tray of tar at the centre of the upper shelf and close the door immediately; do this operation very quickly to ensure that the fall in oven temperature is at a minimum.

The oven shall contain only one tray of tar at any one time. (Tests have shown that if the initial temperature of the oven is 140 °C it will fall to about 131 °C after this operation; recovery is fairly rapid however and 137 °C to 138 °C is re-attained in about 15 min. No readjustments should be made, therefore, to the original setting of the control dial on the front of the oven.)

f) Remove the tray after 1 h, mix the tar carefully but thoroughly with a small spatula, and pour directly into the preheated viscometer cup. Pour into the viscometer cup only sufficient hot tar to carry out the normal evt test. Determine the final evt to the nearest 0.1 °C.

F.5 Reporting

Report the following:

a) temperature of test (°C);

b) initial evt (°C evt);

c) final evt (°C evt);

d) rise in evt during test (°C evt).

F.6 Precision

F.6.1 *At 140 °C*

Repeatability r. Duplicate results by the same operator should be considered suspect if they differ by more than 2.4 °C evt.

Reproducibility R. Single results submitted by each of two laboratories should be considered suspect if they differ by more than 4.0 °C evt.

F.6.2 *At 80 °C*

Repeatability r. 1.1 °C evt.

Reproducibility R. 1.6 °C evt.

F.6.3 *At temperatures between 80 °C and 140 °C.* The precision of the test at temperatures between 80 °C and 140 °C has not been determined but approximate values of the repeatability and reproducibility may be obtained by calculation. For example:

$$Repeatability\ at\ 100\ ^\circ C = 1.1 + \left(\frac{2.4 - 1.1}{3}\right) = 1.6\ ^\circ\text{C evt}$$

$$Reproducibility\ at\ 100\ ^\circ C = 1.6 + \left(\frac{4.0 - 1.6}{3}\right) = 2.4\ ^\circ\text{C evt}$$

Appendix G Method for the determination of hardening by the SEGAS test

(Technically identical with STPTC Method RT 100–73)

G.1 Introduction

The method determines the rise in evt which a film of tar undergoes during 43 h at 45 °C whilst a stream of air passes over its surface. The test allows an assessment to be made of the hardening the tar will experience during service on the road as a surface dressing binder. Thus this test only applies to the surface dressing (S) tars.

G.2 Apparatus

The following apparatus is required and shall be in accordance with Figure 6:

a)*Brass plate and cover*. A brass plate, 267 mm × 267 mm × 6.3 mm thick is machined to a plane surface and 3 mm square brass rods are riveted to the plate to form four squares of 114 mm sides. The edges between the brass strips and the plate, on the inside of each square, are lightly soldered in order to prevent tar getting between the brass strips and the plate. The plate is fitted with three levelling screws attached to pillars on its rim and it is suitably supported, for example on two bars, in a water bath so that the water level is between the upper and lower surfaces of the plate.

The type of levelling screws used and their positioning on the sides of the flat plate should not restrict the flow of air through the 8 mm diameter holes cut in the sides of the cover (see below).

A cover, constructed of tin plate or other suitable material, 235 mm square and 76 mm deep is divided by soldered partitions into four compartments corresponding to the four squares on the brass plate, so that when placed over the plate the edges of the cover and the edges of the four compartments rest on the 3 mm brass rods. Four holes, 11 mm in diameter are cut in the top of the cover on the diagonals, with their centre 57 mm from the point at which the diagonals intersect. Six 8 mm diameter holes are cut in each of the external sides of the cover, their centres 12.5 mm from the bottom, so that each compartment has six of these outlets. A piece of rubber sheeting placed over the cover, with four holes as in the cover top, serves to provide thermal insulation.

b) *Jets*. Four jets connected to a manifold rest on suitable washers in the holes in the cover. Fan spray jets are used[3]. The jets are arranged so that the flat fan of air discharges at right angles to diagonal of the cover and the four fans form the sides of a square.

c) *Pressure control devices*. A simple U-tube manometer containing water to observe pressure of the air flow. It may be used in conjunction with a rotameter and a pressure limiting device which can take the form of a T-piece with the air passing along the top arm and the vertical arm inserted in a 500 ml cylinder containing about 400 ml of water.

d) *Balance*. A balance having two scale pans and a direct reading scale graduated from 0 g to 10 g in 0.1 g divisions is suitable.

e) *EVT viscometer*, as described in Appendix F.

f) *Pump*, or other suitable equipment to provide compressed air supply.

g) *Water bath*.

h) *Oven*.

j) *Hot plate*.

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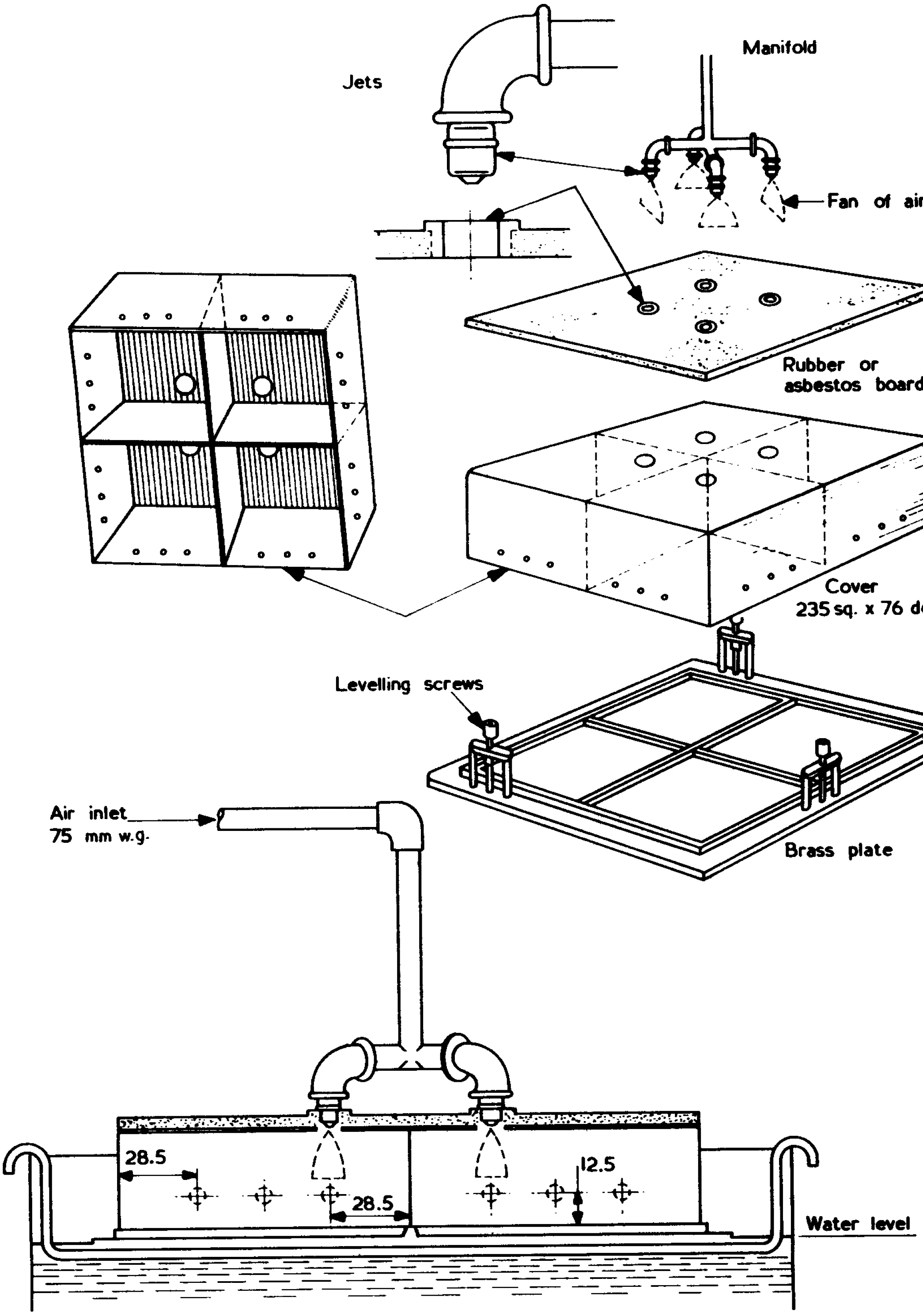
[3] This equipment is available commercially from George Bray and Co. Ltd., Leicester Place, Blackman Lane, Leeds, as fan spray jet No. 4274. size 14, spray angle 80°.



All dimensions are in millimetres

Figure 6 — Apparatus for SEGAS test

G.3 Procedure

The procedure shall be as follows:

NOTE Two tars may be tested at the same time.

a) Clean the surface of the brass plate thoroughly with acetone and dry for 30 min in an oven at 70 °C to 80 °C.

b) Remove the plate from the oven, screw down the levelling screws to the full extent and place on the supporting bars in a thermostatically controlled water bath of which the temperature has previously been set at 45 ± 0.5 °C. Adjust each of the levelling screws so that the water level is just below the upper level of the plate on all four sides.

c) Prepare the sample as specified in Appendix A.

d) Measure the initial evt of the first tar to the nearest 0.1 °C using the direct reading evt viscometer. The instrument shall be calibrated beforehand in accordance with **F.3.2**.

e) Weigh 25 g to 30 g of the warm first tar into a clean, dry 50 ml beaker which contains a short square-ended spatula. This amount is sufficient for covering two squares on the brass plate. Ascertain the gross mass of the beaker + tar + spatula[4)].

f) Transfer sufficient tar from the beaker to one of the squares on the brass plate such that the mass of tar, in grams, is 7.85 times the density of the tar at 20 °C, to an accuracy of 0.1 g (this is equivalent to a film thickness of about 0.6 mm). The recommended technique is to cover the surface of the square with a slight excess of tar and then remove the excess with the spatula until the mass is correct. Spread the film carefully and evenly over the square ensuring that the tar reaches fully into the corners. Avoid scratching the surface of the plate.

g) Then transfer sufficient of the first tar from the same beaker into the diagonally opposite square such that the mass of tar in this square is also 7.85 times the density of the tar at 20 °C.

h) If a second tar is to be tested at the same time, measure its initial evt using the direct reading evt viscometer. Weigh 25 g to 30 g of the second tar into a separate clean, dry 50 ml beaker and cover the other two diagonally opposite squares of the plate in the same way as is described in (f) and (g).

j) Place the metal cover over the plate, put the rubber sheet and washers in position and rest the jets (with manifold) in the holes. The jets shall be cleaned by washing with acetone and dried before use. Do not attempt to scrape or clean the jets with pieces of wire or other sharp objects.

k) Connect the manifold to the air supply and the manometer. Pass air which has been filtered through cotton wool into the jets at 75 mm water gauge[5)] and over the individual tar films continuously for 43 h. Maintain the temperature of the water bath at 45 ± 0.5 °C throughout the test.

During the early stages of the test, sublimed naphthalene or other solid material may block one or more of the jets and this is indicated by a change in the water levels of the manometer. If a blockage is suspected, quickly clean out and dry the jets and continue the test.

m) At the end of 43 h shut off the air and remove the cover. Whilst the tar films are still warm, transfer the film from one square as completely as possible into a clean, dry 50 ml beaker. Take particular care that any hard film is completely incorporated. A paint scraper with a thin, flexible blade about 50 mm wide is suitable for this operation. Remove the tar film from the diagonally opposite square and transfer to the same beaker.

n) Warm the contents of the beaker carefully, until fluid, and mix by means of a spatula until homogeneous. It is essential to avoid overheating or prolonged heating at this stage. Pour the mixed tar into a preheated viscometer cup and determine the evt to the nearest 0.1 °C.

p) If a second tar has been tested at the same time, transfer the two films remaining on the other two squares of the plate into another clean, dry 50 ml beaker and mix until homogeneous. Pour the mixed tar into the cleaned, preheated viscometer cup and obtain an evt result for this second tar.

[4)] A small strip of PTFE or thin steel, about 10 mm to 15 mm wide, is a suitable alternative to the spatula.

[5)] 1 mm water gauge ≈ 9.8 Pa.

G.4 Reporting

Report the following:

- a) *First tar:*
  - 1) the intial evt of the tar to the nearest 0.1 °C,
  - 2) the evt after test of the combined tars from the first two diagonally opposite squares, to the nearest 0.1 °C, and
  - 3) the rise in evt.
- b) *Second tar* (if tested):
  - 1) the initial evt of the tar, to the nearest 0.1 °C,
  - 2) the evt after test of the combined tars from the second two diagonally opposite squares, to the nearest 0.1 °C, and
  - 3) the rise in evt.

G.5 Precision

The precision obtained by using one pair of diagonally opposite squares is the same as that obtained by using the other pair of diagonally opposite squares. For one tar, the precision values for the rise in evt are as follows:

Repeatability r. Duplicate results by the same operator should be considered suspect if they differ by more than 2.0 °C evt.

Reproducibility R. Single results submitted by each of two laboratories should be considered suspect if they differ by more than 3.4 °C evt.

Appendix H Method for the determination of matter insoluble in organic solvents

(Technically identical with Appendix C of BS 598-2:1974, and for toluene and dichloromethane with STPTC Serial No. RT 8-73)

H.1 Outline of method

Matter soluble in a solvent is extracted from the sample. The solution is filtered and the insoluble part of the tar is washed, dried and weighed.

H.2 Discussion of principles

The solvent used for the determination of the insolubles content shall be the same as that used for the extraction of the binder in the analysis of tar-bound material. The determination of the insolubles content shall be carried out at a temperature similar to the temperature at which the extraction of binder takes place. For example when the extraction bottle method or the sieving extractor method is used for analysis then dichloromethane at room temperature shall be used; when the hot extractor method is used for analysis then trichloroethylene at a temperature close to its boiling point shall be used.

The method described below is generally applicable to all solvents used in the analysis of tar-bound materials. The only variation in the method is in the temperature of the solvent used for the determination of the insolubles.

H.3 Precautions

Toluene is highly flammable and it should only be heated with an electric hot plate (or steam bath) and never with a bunsen burner. All flames in the vicinity should be extinguished during its use.

Trichloroethylene and dichloromethane are highly toxic and, although the latter is used in the test at room temperature, special precautions should always be taken with either to ensure the vapour is not inhaled.

Whatever solvent is used, it is advisable to carry out the test in a fume cupboard or well ventilated laboratory.

H.4 Apparatus

The following apparatus is required:

- a) *Beaker*, 200 ml.
- b) *Clock glass* to cover the beaker.
- c) *Filtering glass crucible*, capacity 30 ml, porosity grade 4 or equivalent.

d) *Measuring cylinder,* 100 ml.

e) *Water bath or electric hot plate* when use of hot solvent is required.

f) *Glass stirring rod*.

g) *Laboratory oven*.

H.5 Reagents

One of the following solvents (as appropriate; see **H.2**) is required:

a) *Toluene*, pure, complying with the requirements of BS 805/1.

b) *Trichloroethylene*, pure, complying with the requirements of BS 580.

c) *Dichloromethane*, pure, boiling point 40 °C to 42 °C, complying with the requirements of BS 1994.

H.6 Procedure

The crucible shall be dried at a temperature of 110 ± 5 °C, cooled in a desiccator and weighed to an accuracy of 0.0002 g.

Approximately 2 g of a thoroughly mixed sample, or 1 g for samples containing approximately 12 % or more of insolubles, shall be weighed into the beaker to an accuracy of 0.002 g, spreading it over the base of the beaker as evenly as possible. 100 ml of solvent, at the appropriate temperature, shall be added and stirred occasionally by means of the glass rod until the tar has completely disintegrated. The beaker shall be covered with the clock glass and left for about 10 min to allow the insoluble matter to settle in the beaker. If hot solvent is used, the beaker shall be left for about 10 min in hot water.

The supernatant solution shall be decanted into the crucible using very gentle suction to assist filtration. With dichloromethane as the solvent, if excessive vacuum is used ice may form and interfere with the filtration. The transfer of the sediment into the crucible shall be avoided. The sediment shall be washed three or four times in the beaker, using hot solvent if appropriate, before finally being transferred into the crucible where the washing shall be continued until a total of approximately 500 ml of solvent has been used for washing.

It is imperative that the sediment in the crucible is not allowed to dry out during the filtration. If it does, cracks will develop in the layer of insoluble matter collected on the filter and the solvent used for washing will pass through the cracks without washing the insoluble material.

The crucible shall be dried at 110 °C, cooled and reweighed.

The percentage by mass of insoluble matter in the sample shall be calculated.

H.7 Precision

Repeatability r. Duplicate results by the same operator should be considered suspect if they differ by more than 0.5 % *m*/*m*.

Reproducibility R. Single results submitted by each of two laboratories should be considered suspect if they differ by more than 1.1 % *m*/*m*.

Appendix J Method for the determination of density

(Technically identical with STPTC Method GP 1-67)

J.1 Apparatus

The following apparatus is required:

a) *50 ml density bottle* complying with the requirements of BS 733, or

b) *Hubbard bottle*, as shown in Figure 7, with dimensions 40 ± 2 mm in diameter at the base, 22 ± 2 mm in diameter at the open end and 70 ± 2 mm in height. The open end is closed by a solid ground glass stopper with a central capillary 1.6 ± 0.2 mm in diameter. The lower part of the stopper is concave to a depth of 4 mm to 6 mm. The bottle and stopper are constructed of borosilicate glass.

c) *Water bath*, thermostatically controlled to within ± 0.1 °C at any convenient temperature between 10 °C and 30 °C.

d) *Thermometers*, Schedule No. T1d (– 10 °C to + 20 °C) and T5d (15 °C to 45 °C) are suitable water bath thermometers (see Appendix L to this standard).

e) *Microscope cover glass*, for use with the Hubbard bottle.

J.2 Procedure by the partial filling method

J.2.1 *Cleaning.* Before calibrating the bottle, or when any liquid fails to drain cleanly from the walls of the bottle, clean it as follows. Fill the bottle with chromic acid solution or alternatively use a suitable detergent. Allow to stand overnight, empty and rinse well with distilled water followed by anhydrous acetone. Dry the bottle with a slow stream of dry filtered air passing into it. Between determinations rinse the bottle with toluene or other suitable solvent followed by anhydrous acetone. Dry the bottle as before.

J.2.2 *Calibration.* Remove any static charge from the clean bottle, which may conveniently be done by breathing on it or alternatively by wiping it with a clean lint-free cloth slightly damped with water. Place the bottle on the balance for 15 min then weigh it to an accuracy of 0.1 mg. Include a microscope glass in the weighing of the Hubbard bottle. Fill the bottle with freshly boiled distilled water to the lower part of the ground glass neck. Carefully insert the stopper so that no bubbles are trapped in the neck and the excess water is expelled through the capillary in the stopper. Place the bottle in the water bath so that it is immersed to not more than 5 mm from the top. Maintain the water bath at *above ambient temperature* but below 30 °C.

After 30 min brush the top of the stopper with a slip of filter paper held at an angle to ensure a flat surface at the capillary opening. When a Hubbard bottle is used, place the microscope cover glass on top of the stopper to prevent evaporation. Remove the bottle from the water bath, carefully dry and allow it to cool to room temperature. Remove any static charge and weigh as described above. Calculate the mass of water in the bottle and record and temperature of the water bath to the nearest 0.1 °C.

From these calculate the volume of the bottle as follows:

$${}_{m}V_{20} = {}_{m}M_{1} \times m_{1};$$

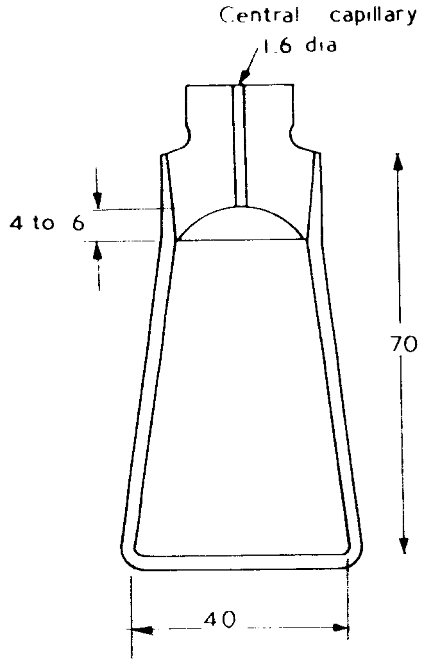
where

${}_{m}V_{20}$ is the calibration volume of the bottle at 20 °C;

${}_{m}M_{t}$ is the mass of water in the bottle at t (g);

m_1 is the multiplication factor for conversion of mass of water at t to volume of vessel at 20 °C[6];

t is the water bath temperature (°C).



All dimensions are in millimetres

Figure 7 — Hubbard density bottle

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[6] Obtainable from STPTC Measurement Conversion Tables (Table 7).

J.2.3 *Road tar density*. Warm the tar to a temperature just sufficient for it to be poured[7]. Remove any static charge from the clean bottle, which may conveniently be done by breathing on it or alternatively by wiping it with a clean, lint-free cloth slightly damped with water. Place the bottle on the balance for 15 min, then weigh it to the nearest 0.1 mg. Include a microscope cover glass in the weighing of the Hubbard bottle.

Half fill the bottle with the tar, using a small funnel if necessary to prevent contamination of the neck of the bottle. Allow the bottle to stand for half an hour at the pouring temperature, to permit the escape of trapped air bubbles and then allow the bottle to cool to ambient temperature. If necessary remove any static charge as above and reweigh the bottle. Calculate the mass of sample in the bottle.

Fill the bottle with freshly boiled distilled water to the lower part of the ground glass neck. Carefully insert the stopper so that no bubbles are trapped in the neck and the excess water is expelled through the capillary in the stopper.

Place the bottle in the water bath so that it is immersed to not more than 5 mm from the top. Maintain the water bath at *above ambient temperature* but below 30 °C.

After 30 min brush the top of the stopper with a slip of filter paper held at an angle to ensure a flat surface at the capillary opening. When a Hubbard bottle is used, place the microscope cover glass on top of the stopper to prevent evaporation. Remove the bottle from the water bath, carefully dry and allow it to cool to room temperature. Remove any static charge and weigh as described above. Calculate the mass of water in the bottle and record the temperature of the water bath to the nearest 0.1 °C.

Calculate the density (D_{20}) as follows:

$$D_{20} = \frac{{}_{s}M_t \times m_5}{{}_{m}V_{20} - {}_{m}M_t \times m_1} + a_5$$

where

${}_{s}M_1$ is the mass of tar at t (g);

${}_{m}V_{20}$ is the calibration volume of the bottle at 20 °C (ml);

${}_{m}M_1$ is the mass of water at t required to fill the bottle containing the tar (g);

m_1 is the multiplication factor for conversion of mass of water at t to volume of vessel at 20 °C[8];

m_5 and a_5 are the multiplication and addition factors for conversions of mass/volume ratio at t to density at 20 °C[8];

t is the water bath temperature (°C).

Appendix K Bibliography

As a guide to the choice of the most suitable type and viscosity of road tar for the particular maintenance or construction work concerned, the following may be useful:

Subject	**Publication**	**Publisher**
Surface dressing	Road Note No. 39	HM Stationery Office
Flexible pavements for new roads	Road Note No. 29	HM Stationery Office
Coated macadam for roads and other paved areas	BS 4987	British Standards Institution
Dense tar surfacing	Dense tar surfacing (DTS)	British Tar Industry Association

It should be Noted that the recommendations for tars contained in the above publications have particular reference to conditions in Great Britain, Northern Ireland and the Republic of Ireland.

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[7] A temperature between 20 °C and 30 °C higher than the evt is required for road tar.

[8] Obtainable from STPTC Measurement Conversion Tables (Table 7 and Table 19).

Appendix L STPTC thermometer specifications

STPTC schedule mark	Range	Graduation	Longer lines at each[a]	Fully figured at each[b]	Maximum overall length	Minimum length of main scale	Maximum error	Limit of accuracy, NPL test;[c]	Maximum use before first stability check
	°C	degree	degree	degree	cm	cm	degree	degree	h
T1d	– 10 to + 20	0.1	0.5 and 1	2	41	23	± 0.2	± 0.05	5 000
T3d	0 to 120	0.5	1 and 5	10	43	24	± 0.6	± 0.1	2 000
T4d	– 2 to + 400	1	5	10	43	25	± 4.0	± 0.5	25
T5d	15 to 45	0.1	0.5 and 1	2	43	26	± 0.4	± 0.05	5 000
T9d	50 to 210	0.5	1 and 5	10	43	24	± 0.8	± 0.1	500
T17d	39.5 to 70.5	0.1	0.5 and 1	2	40	21	± 0.4	± 0.05	2 000

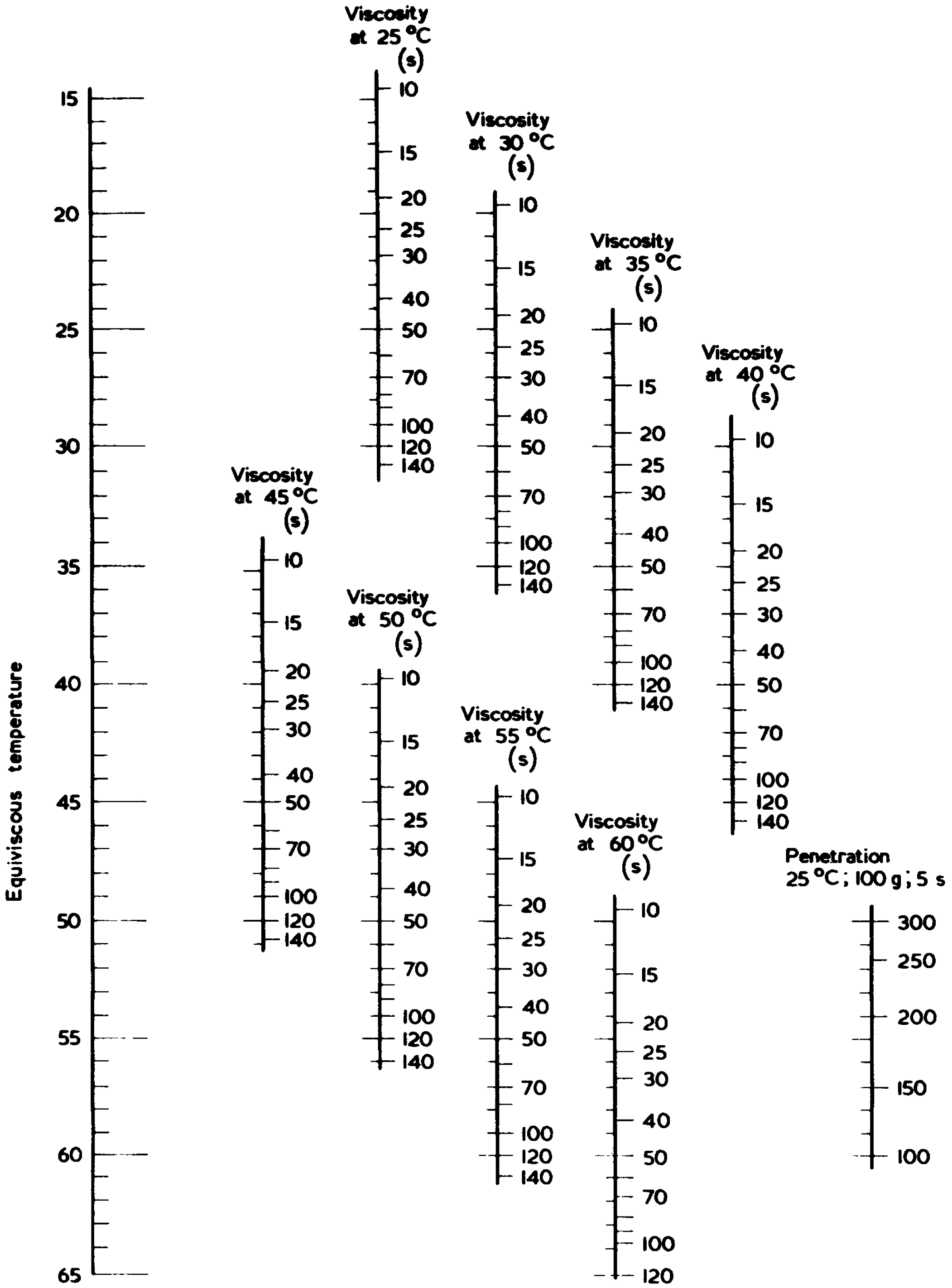
NOTE 1 British Standard thermometers A20C/100, A40C/100 and A70C/100 of BS 593 may be used, where the range is appropriate, in place of thermometers T1d, T5d and T17d.

NOTE 2 In 1967 the specified maximum distance from the bottom of the bulb to the top of the contraction chamber was increased from 30 mm to 35 mm. The schedule numbers of such thermometers carry the suffix d. Thermometers with schedule numbers carrying the earlier suffix c may be used in place of the latest version.

[a] In this column, the lines corresponding to the second number shall be slightly longer than those corresponding to the first number.

[b] The figuring of thermometers which are to be figured at each 2 degrees shall apply to the even number degrees.

[c] The corrections to be applied shall not change between successive test points by more than five times the figure in this column, which shows the limit of accuracy of the test which the National Physical Laboratory will apply to each thermometer submitted for test.



NOTE The graduations on the left of each colume are evt units and correspond to the main evt scale. Viscosities are by the 10 mm cup (see Appendix B).

Figure 8 — Chart showing approximate relationships between evt, viscosity and penetration

Publications referred to

This standard makes reference to the following British Standards:

BS 410, *Test sieves.*
BS 458, *Xylenes.*
BS 479, *Coal-tar naphthas.*
BS 580, *Trichloroethylene.*
BS 593, *Laboratory thermometers.*
BS 598, *Sampling and examination of bituminous mixtures for roads and other paved areas.*
BS 598-2, *Testing.*
BS 604, *Graduated measuring cylinders.*
BS 616, *Sampling of coal tar and its products.*
BS 658, *Apparatus for the determination of distillation range (including flasks and receivers).*
BS 733, *Density bottles.*
BS 756, *Dean and Stark apparatus.*
BS 805, *Toluenes.*
BS 1994, *Dichloromethane (methylene chloride).*
BS 3076, *Nickel and nickel alloys. Rods.*
BS 4987, *Coated macadam for roads and other paved areas.*

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