

Bitumen road emulsions (anionic and cationic) —

Part 1: Specification for bitumen road
emulsions

UDC 625.856.063:665.775.5

1/85

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Road Engineering Standards Committee (RDB/-) to Technical Committee RDB/14 upon which the following bodies were represented:

British Aggregate Construction Materials Industries
 County Surveyors' Society
 Federation of Civil Engineering Contractors
 Greater London Council
 Institute of Petroleum
 Institution of Civil Engineers
 Institution of Highway Engineers
 Institution of Municipal Engineers
 Road Emulsion Association Ltd.
 Society of Chemical Industry
 Coopted member

This British Standard, having been prepared under the direction of the Road Engineering Standards Committee, was published under the authority of the Board of BSI and comes into effect on
 29 February 1984

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First published December 1931
 First revision December 1935
 Second revision February 1952
 Third revision April 1960
 Fourth revision as BS 434-1
 October 1973
 Fifth revision February 1984

The following BSI references relate to the work on this standard:
 Committee reference RDB/14
 Draft for comment 80/13165 DC

Amendments issued since publication

Amd. No.	Date of issue	Comments
4756	December 1984	
5690	May 1988	
6376	June 1991	Indicated by a sideline in the margin

Contents

	Page
Committees responsible	Inside front cover
Foreword	ii
<hr/>	
1 Scope	1
2 Definitions	1
3 Classification of road emulsions	1
4 Composition	2
5 Particle charge	2
6 Properties of emulsions	2
7 Containers and marking	2
<hr/>	
Appendix A Sampling	5
Appendix B Preparation of specimens	5
Appendix C Method for determination of particle charge	5
Appendix D Methods for determination of residue on sieving	6
Appendix E Methods for determination of stability to mixing	7
Appendix F Method for determination of water and binder content	9
Appendix G Methods for determination of viscosity	12
Appendix H Method for determination of coagulation of emulsion at low temperature	14
Appendix J Methods for determination of storage stability	15
<hr/>	
Figure 1 — Assembled apparatus for water content test	10
Figure 2 — Engler viscometer	11
Figure 3 — Redwood No. II viscometer: section of cup	13
Figure 4 — Suitable apparatus for sedimentation test	16
<hr/>	
Table 1 — Properties of anionic road emulsions	3
Table 2 — Properties of cationic road emulsions	4
<hr/>	
Publications referred to	Inside back cover

Foreword

This revision of this Part of this British Standard, prepared under the direction of the Road Engineering Standards Committee, specifies requirements for anionic and cationic bitumen road emulsions. It supersedes the 1973 edition which is withdrawn. Recommendations for the use of these emulsions for roads and other surfaces are covered by Part 2 of this standard.

No major changes have been made to the previous edition of this standard but the opportunity has been taken to make a large number of minor alterations which have been shown to be necessary as a result of users' experience.

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.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 16, 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.

1 Scope

This Part of BS 434 specifies requirements for anionic and cationic bitumen emulsions used for the preparation and treatment of road and other surfaces carrying wheeled and foot traffic.

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

2 Definitions

For the purposes of this Part of BS 434, the following definitions apply.

2.1

bitumen

a viscous liquid or a solid, consisting essentially of hydrocarbons and their derivatives, that is soluble in carbon disulphide¹⁾; it is substantially non-volatile and softens gradually when heated. It is black or brown in colour and possesses water-proofing and adhesive properties. It is obtained by refinery processes from petroleum and is also found as a natural deposit or as a component of naturally occurring asphalt in which it is associated with mineral matter

2.2

bitumen road emulsion

a liquid product in which a substantial amount of bitumen is suspended in a finely divided condition in an aqueous medium by means of one or more suitable emulsifying agents. There are two types, anionic and cationic

2.3

anionic emulsion

an emulsion in which the anion of the emulsifier is at the interface with the bitumen particle that is negatively charged and in which the aqueous phase is normally alkaline

2.4

cationic emulsion

an emulsion in which the cation of the emulsifier is at the interface with the bitumen particle that is positively charged and in which the aqueous phase is normally acid

2.5

binder content (including emulsifier)

the difference between 100 % and the percentage water content determined in accordance with appendix F

3 Classification of road emulsions

3.1 Anionic road emulsions. Anionic road emulsions shall be classified in order of stability as follows.

a) *Class A1: labile.* An emulsion characterized by rapid breakdown on application and normally unsuitable for mixing with aggregate; used cold.

b) *Class A2: semi-stable.* An emulsion with sufficient stability to permit mixing with certain grades of aggregate before breakdown occurs; used cold.

c) *Class A3: stable.* An emulsion with sufficient mechanical and chemical stability for all purposes involving mixing with aggregates including those containing large proportions of fines or chemically active materials such as cement or hydrated lime; used cold.

d) *Class A4: slurry seal.* An emulsion formulated for the slurry seal process; used cold.

NOTE Additives may be used at the time of laying if required to adjust the setting time or viscosity of the mix.

This class shall be subdivided as follows.

1) *Class A4: slow setting.* Suitable for mixing in simple mixers for hand laying, in bulk transit concrete mixers or mobile mixing machines.

2) *Class A4: rapid setting.* Suitable for handling in special mobile mixing machines only.

NOTE 1 Classes A1 and A2 are subdivided according to their nominal binder contents (see Table 1).

NOTE 2 Due to the wide variation in chemical activity and physical structure of aggregates from different sources, not all are suitable for the slurry seal process. It is necessary, therefore, to test each aggregate in the laboratory with class A4 emulsion and to subject the mixes with class A4 emulsion to the track abrasion test in accordance with appendix A of Part 2:1984 of this standard. The loss in mass of the specimen after abrasion should not exceed 500 g/m².

3.2 Cationic road emulsions. Cationic road emulsions shall be classified in order of stability as follows.

a) *Class K1: rapid acting.* An emulsion characterized by rapid deposition of binder on contact with road surfaces and aggregates followed by early resistance to rain. Unsuitable for mixing with aggregates; class K1-70 grades used hot, other grades cold.

b) *Class K2: medium acting.* An emulsion in which the rate of deposition of binder is sufficiently delayed to permit mixing with certain clean coarse aggregates before breaking to form a continuous adhesive film without stripping; used cold.

¹⁾The internationally agreed definition of bitumen relates its solubility to carbon disulphide but in the UK trichloroethylene is used since it is safer than carbon disulphide.

c) *Class K3: slow acting.* An emulsion in which the rate of deposition of binder is sufficiently delayed to permit mixing with certain fine aggregates before breaking to form a continuous adhesive film without stripping; used cold.

Cationic slurry seal emulsions are included in class K3 and are suitable for handling in special mobile mixing machines only.

NOTE 1 Class K1 is subdivided according to its nominal binder content (see Table 2).

NOTE 2 Due to the wide variation in chemical activity and physical structure of aggregates from different sources, not all are suitable for the slurry seal process. It is necessary, therefore, to test each aggregate in the laboratory with class K3 emulsion and to subject the mixes with class K3 emulsion to the track abrasion test in accordance with appendix A of Part 2:1984 of this standard. The loss in mass of the specimen after abrasion should not exceed 500 g/m².

4 Composition

4.1 General. Road emulsions shall contain bitumen, modified if necessary as specified in 4.2, emulsifying agents and water. Small amounts of surfactants and additives shall be permitted to modify performance, as in slurry seal and other emulsions for special purposes.

4.2 Bitumen. The bitumen used for the manufacture of the emulsion shall comply with BS 3690 and shall be selected from the range grade 70 penetration to grade 300 penetration, as defined therein.

NOTE 1 The bitumen may be modified by the addition of fluxing agents ranging from light volatile distillates to heavy oils, the amount of fluxing agent used normally being not more than 5 % by mass of the bitumen for anionic emulsion and 10 % for cationic emulsion, or as agreed between the purchaser and the manufacturer of the emulsion.

NOTE 2 If required by the purchaser, the supplier should provide a typical sample of the penetration bitumen used as the basic ingredient of the emulsion and/or should disclose the nominal viscosity of the bitumen/flux mixture used.

NOTE 3 Attention is drawn to the fact that bitumen originally complying with BS 3690 may not give the same test results on analysis after extraction from the emulsion.

4.3 Toxicity. Any drainings, washings or the like, passing from the work into streams, ponds, rivers, etc., shall not, after dilution by these waters, have any toxic effect upon plant, animal or fish life.

5 Particle charge

When tested in accordance with appendix C, anionic emulsion shall deposit on the anode, indicating that the particles have a negative charge and cationic emulsions shall deposit on the cathode, indicating that the particles have a positive charge.

NOTE The character and amount of deposit will depend on the class of emulsion under test.

6 Properties of emulsions

After sampling in accordance with appendix A, emulsions shall be tested in accordance with appendices B to J. The properties of anionic road emulsions shall be as given in Table 1 and the properties of cationic road emulsions shall be as given in Table 2.

7 Containers and marking

7.1 Containers. No corrosion or attack shall occur between the emulsion and the container or bung and the emulsion shall not be adversely affected by the container or bung.

NOTE The use of light metals or their alloys may lead to the evolution of hydrogen and the subsequent explosion of the container.

7.2 Marking. Containers shall be legibly labelled with the following minimum information:

- the name or trade mark of the manufacturer or supplier of emulsion;
- the date of filling;
- the number of this British Standard, i.e. BS 434/1:1984²⁾;
- the class of emulsion and nominal binder content (see Table 1 and Table 2);
- the mass of contents in kilograms or volume in litres;
- the word "Anionic" or "Cationic" on the head of the container which shall be coloured black for Anionic and green for Cationic;
- the instructions to roll the drums before use, particularly in the case of 40 % binder content emulsions;
- an instruction to protect from frost.

Documents relative to bulk deliveries shall provide similar relevant information.

²⁾ Marking BS 434/1:1984 on or in relation to a product is a claim by the manufacturer that the product has been manufactured in accordance with the requirements of the standard. The accuracy of such a claim is therefore solely the manufacturer's responsibility. Enquiries as to the availability of third party certification to support such claims should be addressed to the Director, Quality Assurance Division, British Standards Institution, Linford Wood, Milton Keynes MK14 6LE in the case of certification marks administered by BSI or to the appropriate authority for other certification marks.

Table 1 — Properties of anionic road emulsions

Property	Appendix or clause	Class of anionic road emulsions						
		A1-60	A1-55	A1-40	A2-57	A2-50	A3	A4
Particle charge	C	neg.	neg.	neg.	neg.	neg.	neg.	neg.
Residue on 710 μm BS sieve (% <i>m/m</i> , maximum)	D.1	0.05	0.05	0.05	0.05	0.05	0.05	0.05
Residue on 150 μm BS sieve (g per 100 mL, maximum)	D.2	0.15	0.15	0.15	0.15	0.15	0.15	0.15
Stability to mixing with coarse aggregate (% coagulation)	E.1	20 to 80	20 to 80	20 to 80	< 40	< 40	< 5	< 5
Stability to mixing with cement (% coagulation)	E.2	—	—	—	> 2	> 2	< 2	—
Binder content (% <i>m/m</i> , minimum)	F	58	53	38	55	48	55	56
Viscosity (degrees Engler ($^{\circ}\text{E}$) at 20 $^{\circ}\text{C}$) ^a	G.1	6 to 9	5 to 8	4 max.	8 max.	5 max.	9 max. ^b	8 max. ^b
Coagulation of emulsion at low temperature	H	nil	nil	nil	nil	nil	nil	nil
Storage stability (short period test) (inversions to clear sediment, maximum)	J.1	60	60	60	60	60	60	60
Storage stability (long period test) (% water content difference, maximum)	J.2	2	2	—	2	2	2	2

NOTE In special circumstances, class A1-60 emulsion with a higher viscosity can be used for hand application provided that the emulsion complies with all the other requirements of this standard.

^a The viscosities given in the table are suitable for use in bulk spreaders complying with BS 1707 or BS 3136 or for use in hand operated sprayers complying with BS 3136. As a general guide, a maximum viscosity of 9 $^{\circ}\text{E}$ at the temperature of the spraying is considered to be suitable.

^b These viscosity values are intended to serve as a guide only and other values may be agreed between the purchaser and the supplier.

Table 2 — Properties of cationic road emulsions

Property	Appendix or clause	Class of cationic road emulsion				
		K1-70	K1-60	K1-40	K2	K3
Particle charge	C	pos.	pos.	pos.	pos.	pos.
Residue on 710 µm BS sieve (% <i>m/m</i>), maximum)	D.1	—	0.05	0.05	0.05	0.05
Residue on 150 µm BS sieve (g per 100 mL, maximum)	D.2	—	0.15	0.15	0.15	0.15
Binder content (% <i>m/m</i>), minimum)	F	67	57	38	57	56
Viscosity (degrees Engler (°E) at 20°)	G.1	—	6 to 9	4 max.	10 max.	10 max.
Viscosity Redwood No. II (s at 85 °C)	G.2	25 to 35 ^a	—	—	—	—
Coagulation of emulsion at low temperature	H	—	nil	nil	nil	nil
Storage stability (short period test) (inversions to clear sediment, maximum)	J.1	—	60	60	60	60
Storage stability (long period test) (% water content difference, maximum)	J.2	—	2	—	2	2

NOTE In special circumstances, class K1-60 emulsion with a higher viscosity can be used for hand application provided that the emulsion complies with all the other requirements of this standard.

^a The viscosity range in the table is intended to serve as a guide only. Bitumen emulsions can change in viscosity during storage and transit and viscosities outside this range may be used by agreement between the purchaser and the supplier provided that the transverse distribution requirements of BS 1707 are met when the emulsions are sprayed in hot binder distributors complying with BS 1707.

Appendix A Sampling

A.1 General. Draw samples of emulsion as soon as possible after delivery, preferably within 24 h, and test within 7 days from the date of drawing. Do not expose the samples to temperatures below 0 °C.

A.2 Deliveries in drums. Select and sample at least three drums.

NOTE This standard does not specify the exact number of drums to be sampled and this should be agreed between the manufacturer and the purchaser.

Thoroughly mix the contents of each drum to be sampled by rolling the container to and fro for a period of 2 min to 3 min successively in opposite directions, making at least five revolutions in each direction and then up-ending each drum through two revolutions, first in one direction and then in the opposite direction. Then pour the whole contents into a clean drum of just sufficient capacity, normally 200 L, stir and immediately take a specimen of at least 4 L. If residual matter is present in the original container, submit a report with the specimen.

Use one of the three, or more, specimens for carrying out the tests given in appendices C to J inclusive and keep the other specimens in reserve.

Seal each drum so that it is airtight and label with the following information:

- a) the manufacturer's name and description of the emulsion;
- b) the class of emulsion;
- c) the date of delivery;
- d) the date of sampling;
- e) the water content of the 4 L specimen, determined in accordance with appendix F.

Use one of the drums and its contents for carrying out the test given in J.2.

A.3 Deliveries in bulk. Either sample by means of a sampling container over the whole depth of the emulsion, or take a sample at the level of half the depth of the emulsion, using a weighted container as shown in Figures 1 and 2 of BS 3195-1:1978.

Take three samples of at least 4 L each. Use one of the samples to prepare specimens for carrying out the tests described in appendices C to J inclusive and keep the other two samples in reserve.

NOTE Reference should also be made to Part 2 of this standard.

A.4 Test requirements. The test result from one of the 4 L samples tested shall be compared with the appropriate limit(s) given in Table 1 or Table 2. If the test results do not comply with the limit(s) given in Table 1 or Table 2, two further reserved samples shall be tested and the test results of two out of any of the three samples tested shall comply with the limit(s) given in Table 1 or Table 2.

Appendix B Preparation of specimens

NOTE 1 Not applicable to class K1-70 emulsion.

Prepare the 4 L specimen by passing it through a 710 µm sieve as described in D.1, and use this emulsion, after gentle stirring immediately before use, for all tests.

NOTE 2 The residue on the 710 µm sieve may be used to decide if the specimen complies with limit(s) given in Table 1 and Table 2.

Appendix C Method for determination of particle charge

NOTE This method is technically equivalent to IP 292/73.

C.1 General. This method identifies the type of bitumen emulsion. A direct current is passed through a bitumen emulsion between two parallel plate electrodes. Deposition of the bitumen on one of the two electrodes indicates whether the emulsion is of the anionic or cationic type. Positively charged particles indicate that the emulsion is cationic, negatively charged particles that it is anionic.

C.2 Apparatus

C.2.1 Current source, direct current, approximately 12 V to 15 V.

C.2.2 Milliammeter

C.2.3 Variable resistor

C.2.4 Electrode leads

C.2.5 Electrodes, consisting of two stainless steel plates approximately 75 mm long and approximately 25 mm wide with an immersion mark at 25 ± 2 mm. An insulating device is used to hold the plates parallel 25 ± 2 mm apart in the emulsion sample during the test.

C.2.6 Beaker, of 150 mL capacity.

C.2.7 Timing device, stop-watch or other suitable device.

C.3 Procedure. Suspend the electrode assembly in the beaker.

Pour the specimen of the emulsion into the beaker until the electrodes are immersed to a depth of 25 mm.

Connect the leads from the electrodes to the current source through the milliammeter and the variable resistor.

Adjust the current to 8 mA and start the timing device.

When the current reading has dropped to 2 mA or at the end of 30 min, whichever occurs first, disconnect the current source and gently wash the electrodes in running water.

Observe the electrodes for bitumen deposit.

C.4 Reporting of results. Report the emulsion as cationic if the deposit appears on the cathode and as anionic if the deposit appears on the anode.

NOTE The cathode is the electrode connected to the negative pole of the d.c. source and the anode is the electrode connected to the positive pole.

Appendix D Methods for determination of residue on sieving

NOTE This method is based on IP 91/76.

D.1 Residue on 710 µm sieve

D.1.1 Apparatus

D.1.1.1 Sieve, of 710 µm nominal aperture, approximately 100 mm in diameter and 40 mm deep and complying with BS 410.

D.1.1.2 Small metal or glass dish, approximately 110 mm in diameter (e.g. a clock glass).

D.1.1.3 Well ventilated oven, thermostatically controlled to 105 ± 5 °C.

D.1.1.4 Balance, of 250 g capacity, readable and accurate to 0.01 g.

D.1.1.5 Balance, of 10 kg capacity, readable and accurate to 1 g.

D.1.1.6 Container, 4 L capacity, clean, weighed and dry.

D.1.2 Materials

D.1.2.1 Solutions

D.1.2.1.1 For anionic emulsion: a 2 % (m/m) solution of potassium or sodium oleate in water.

D.1.2.1.2 For cationic emulsion: a 1 % (m/m) solution of cetrinide (a mixture of alkyltrimethylammonium bromides) in 0.1 N hydrochloric acid.

D.1.2.2 Solvents

D.1.2.2.1 Xylene, complying with any grade specified in BS 458.

D.1.2.2.2 Acetone, complying with BS 509.

D.1.3 Procedure. Wash the sieve with xylene and then with acetone. Place the sieve in the dish, dry it in the oven at 105 ± 5 °C for 1 h, cool and weigh the sieve, together with the dish, to the nearest 0.01 g (W_1).

Remove the sieve from the dish and moisten with the appropriate solution.

Render uniform the 4 L specimen by gentle agitation and strain immediately through the sieve into the clean, dry, weighed container (W_2).

When all of the emulsion has passed through the sieve, remove the sieve and weigh the container containing the emulsion to the nearest 1 g (W_3).

Wash the sieve repeatedly with the appropriate solution until the washings are no longer discoloured and then wash with distilled water until free from the solution. Discard the washings. Place the sieve in the small dish to dry for 2 h in the oven at 105 ± 5 °C. Cool and reweigh the sieve and dish together to the nearest 0.01 g (W_4).

D.1.4 Calculation. Calculate the percentage retained, which is given by the expression:

$$\frac{W_4 \times W_1}{W_3 \times W_2} \times 100$$

where

W_1 is the mass of the sieve and dish (in g);

W_2 is the mass of the container (in g);

W_3 is the mass of the container and emulsion (in g);

W_4 is the mass of the sieve, dish and residue (in g).

D.1.5 Reporting of results. Report the percentage retained on the 710 µm sieve to the nearest 0.01.

D.2 Residue on 150 µm sieve

D.2.1 Apparatus

D.2.1.1 Sieve, of 150 µm nominal aperture, approximately 100 mm diameter and 40 mm deep and complying with BS 410.

D.2.1.2 Small metal or glass dish, approximately 110 mm in diameter (e.g. a clock glass).

D.2.1.3 Measuring cylinder, of 100 mL capacity.

D.2.1.4 Balance, of 250 g capacity, readable and accurate to 0.01 g.

D.2.1.5 *Well ventilated oven*, thermostatically controlled to 105 ± 5 °C.

D.2.2 *Materials*

D.2.2.1 *Solutions*

D.2.2.1.1 *For anionic emulsion*: a 2 % (m/m) solution of potassium or sodium oleate in water.

D.2.2.1.2 *For cationic emulsion*: a 1 % (m/m) solution of cetrimide (a mixture of alkyltrimethylammonium bromides) in 0.1 N hydrochloric acid.

D.2.2.2 *Solvents*

D.2.2.2.1 *Xylene*, complying with any grade specified in BS 458.

D.2.2.2.2 *Acetone*, complying with BS 509.

D.2.3 *Procedure*. Wash the sieve with xylene and then with acetone. Place the sieve in the dish, dry it in the oven at 105 ± 5 °C for 1 h, cool and weigh the sieve, together with the dish, to the nearest 0.01 g (W_1).

Measure 100 mL of the specimen into the cylinder previously moistened with the appropriate solution.

Pour the specimen rapidly on to the sieve, previously moistened with the appropriate solution, and maintain a layer of emulsion constantly upon the sieve. Gently tap the side of the sieve, if necessary, to ensure easy passage of the emulsion.

When all of the specimen has passed through the sieve, wash the sieve repeatedly with the appropriate solution until the washings are no longer discoloured and then wash with distilled water until free from the solution. Place the sieve in the dish.

Dry the sieve in the dish for 2 h in the oven, cool and reweigh together to the nearest 0.01 g (W_2).

D.2.4 *Calculation*. Calculate the residue, which is given by the expression:

$$W_2 - W_1$$

where

W_2 is the mass of the sieve, dish and residue (in g);

W_1 is the mass of the sieve and dish (in g).

D.2.5 *Reporting of results*. Report the residue on sieving as "less than 0.1 g" if this is the case, or, if not, as the mass of residue per 100 mL of emulsion to the nearest 0.01 g.

Appendix E Methods for determination of stability to mixing

NOTE For the purposes of this British Standard emulsions are classified in relation to the aggregate specified in E.1.3 and in relation to the Portland cement specified in E.2.2.

E.1 Stability to mixing with coarse aggregate

E.1.1 *General*. Although this method may be used to study coagulation of any emulsion on contact with stone, as a means of classification it is only applicable to anionic emulsions, classes A1, A2 and A3, and can be applied accurately only when related to the actual aggregate to be used and to all factors likely to modify the performance of the emulsion at the time of application.

Hence, although the limits shown in Table 1 provide a general guide to the suitability of an emulsion for the purpose for which it is employed, compliance with these values does not necessarily ensure satisfactory performance on application.

E.1.2 *Apparatus*

E.1.2.1 *Clear glass storage bottle*, approximately 180 mm high and 90 mm in diameter (e.g. a 1 kg fruit preserving jar) with a wide mouth and rubber sealing ring.

E.1.2.2 *Machine*, for rotating the bottle end over end at the rate of 60 r/min to 70 r/min.

E.1.2.3 *Two flat-bottomed nickel dishes*, weighed and approximately 90 mm diameter and 15 mm deep.

E.1.2.4 *Balance*, of 250 g capacity, readable and accurate to 0.1 g.

E.1.2.5 *Balance*, of 250 g capacity, readable and accurate to 0.001 g.

E.1.2.6 *Sieve*, of 710 μm nominal aperture, as described in D.1.1.1.

E.1.2.7 *Sieve*, of 3.35 mm nominal aperture, 200 mm in diameter and complying with BS 410.

E.1.3 Materials

E.1.3.1 Aggregate, consisting of criggon chippings³⁾ passing a 10 mm nominal aperture sieve and retained on a 6.3 mm nominal aperture sieve (both sieves complying with BS 410). Place about 1 kg in the 3.35 mm sieve (E.1.2.7) and agitate thoroughly in tap water about 100 mm deep in a sink. Repeat this procedure four times with fresh water each time. Drain the aggregate and transfer it to a glass beaker and wash three times, using 500 mL distilled water each time, while the aggregate is stirred with a glass rod. Air dry the washed aggregate to constant mass.

E.1.3.2 Xylene, complying with BS 458, with an evaporation residue not exceeding 0.02 g/L.

E.1.3.3 Soap solution, 2 % (m/m) solution of potassium or sodium oleate in water.

E.1.4 Procedure. Clean the glass bottle with xylene, dry it, wash with the soap solution and then with distilled water, and finally dry with a soft cloth or jap paper.

Bring the bottle and all the materials to a temperature between 20 °C and 25 °C before the start of the test and maintain them within this temperature range throughout the test.

Weigh 200 ± 1 g of the aggregate into the bottle, followed by 20 ± 0.2 g of emulsion which has been passed through the 710 μm sieve (E.1.2.6). Screw on the lid immediately, invert the bottle and sharply tap on the base with the hand so that the aggregate and emulsion fall freely.

Place the bottle in the rotating machine in the inverted position and rotate end over end 140 times, (i.e. 140 complete 360° revolutions). Allow a period of not more than 1 min to elapse between adding the emulsion to the aggregate and starting the rotating machine.

Remove the bottle from the rotating machine, add 200 ± 1 mL of freshly boiled, distilled water at 20 °C to 25 °C, replace the lid and rotate in the machine 60 to 70 times. Then pass the wash water and residual emulsion through the 710 μm sieve (E.1.2.6) and repeat the process until the washings remain clear.

After draining away as much as possible of the water, place the sieve in a beaker. Pour 100 mL of xylene into the beaker to dissolve the bitumen on the sieve and then pour this solution into the bottle. Wash the sieve and beaker with a further 100 mL of xylene and add this solution also to the bottle. Then close the bottle, replace it in the machine and rotate it for a period of 15 min to dissolve the coagulated bitumen.

Allow the bottle to stand for 30 min to permit dispersed water and any aggregate dust to settle. Remove two 25 mL aliquots of the xylene solution by pipette and evaporate in the two weighed nickel dishes by placing them for 1.5 h on the metal surface of a boiling water bath in a fume cupboard. Cool in a desiccator. Determine accurately the masses of bitumen residue in the dishes and if they differ by more than 0.002 g repeat the procedure.

E.1.5 Calculation. Use the mean b of the two masses to calculate the amount of binder coagulated as a percentage of the total binder originally present. The coagulation value, expressed as a percentage, is given by the expression:

$$\frac{b \times 8 \times 100}{20(1 - W/100)}$$

where

- b is the mass of the binder in 25 mL of xylene solution (in g);
 W is the water content of the original emulsion (percentage by mass) estimated in accordance with appendix F.

E.1.6 Reporting of results. Report coagulation value as a percentage to the nearest whole number.

E.2 Stability to mixing with cement (classes A2 and A3 only)

E.2.1 Apparatus

E.2.1.1 Sieve, of 1.4 mm nominal aperture, weighed and approximately 100 mm in diameter and 40 mm deep and complying with BS 410.

E.2.1.2 Sieve, of 180 μm nominal aperture, approximately 200 mm in diameter and complying with BS 410.

E.2.1.3 Round-bottomed iron dish, or similar metal utensil of approximately 500 mL capacity.

E.2.1.4 Steel rod with rounded ends, approximately 13 mm in diameter.

E.2.1.5 Balance, of 250 g capacity, readable and accurate to 0.1 g.

E.2.1.6 Graduated cylinder, of 100 mL capacity.

E.2.1.7 Shallow pan, weighed and of approximately 50 mL capacity and approximately 110 mm in diameter.

E.2.1.8 Well ventilated oven, thermostatically controlled at 105 ± 5 °C.

E.2.2 Material

E.2.2.1 Ordinary Portland cement, complying with BS 12, kept in sealed containers and not exposed to the atmosphere before use.

³⁾ Obtainable from Amey Roadstone Corporation, 160-162 Abbey Foregate, Shrewsbury, SY2 6AL.

E.2.3 Procedure. If the water content of the emulsion, determined in accordance with appendix F, is below 50 %, dilute the emulsion with freshly boiled distilled water at 20 °C to 25 °C, to a water content of 50 %.

Sift a portion of the cement through the 180 µm sieve (E.2.1.2) and weigh 50 g of the sifted cement into the iron dish.

Weigh the 1.4 mm sieve (E.2.1.1) and the shallow pan to the nearest 0.1 g (W_1).

Add 100 mL of the 50 % emulsion to the cement in the dish and stir the mixture at once with the steel rod with a circular motion, making 60 complete revolutions during 1 min. immediately at the end of the 1 min mixing period, or after a longer period if necessary to obtain a homogenous mixture, add 150 mL of freshly boiled distilled water at 20 °C to 25 °C and continue stirring for 3 min. Maintain the ingredients and apparatus at a temperature of 20 °C to 25 °C during the mixing period.

Pour the mixture through the 1.4 mm sieve (E.2.1.1) and rinse with distilled water poured from a receptacle held at a height of approximately 150 mm. Place the sieve in the pan, heat them both in the oven at 105 ± 5 °C until dry and weigh them together to the nearest 0.1 g (W_2).

E.2.4 Calculation. Calculate the coagulation value, as a percentage, which is given by the expression:

$$\frac{W_2 - W_1}{W_3} \times 100$$

where

- W_1 is the mass of the weighed sieve and pan (in g);
- W_2 is the mass of the sieve and pan, and the material retained in and on them (in g);
- W_3 is the mass of the binder in 100 mL of the diluted emulsion (in g) determined by the procedure in appendix F.

E.2.5 Reporting of results. Report the coagulation value as a percentage to the nearest whole number.

Appendix F Method for determination of water and binder content

NOTE This method is based on IP 291/73.

F.1 General. The water content as determined by this method is used to calculate the binder content of an emulsion by deducting the result from 100 (see 2.5)

F.2 Apparatus

NOTE The apparatus is illustrated in Figure 1.

F.2.1 Glass still, of 500 mL capacity, comprising a round-bottom short-necked flask with a ground socket for either a 24/29 or 34/35 joint, complying with BS 572, at the neck.

F.2.2 Condenser, consisting of a water-cooled reflux glass tube type having a jacket not less than 300 mm in length, with an inner tube of 15 ± 3 mm external diameter. The lower end of the inner tube shall be ground to an angle of approximately 60° to the axis. The drainage tip shall finish in a 19/26 cone.

F.2.3 Receiver, of annealed glass complying with the dimensions given in Figure 1.

F.2.4 Heater, such as an electric heater.

F.3 Materials

F.3.1 Carrier liquid, petroleum or coal-tar spirit, free from water and yielding not more than 5 % (V/V) of distillate at 125 °C and not less than 20 % (V/V) at 160 °C when tested as described in BS 2000-123.

NOTE A suitable carrier liquid is grade 3° or 5° xylene complying with BS 458.

F.4 Specimen. Ensure that the specimen is thoroughly representative of the material to be tested.

F.5 Procedure. Weigh 30 g to 50 g of the emulsion to the nearest 0.1 g into the distillation still. Add 100 mL of the carrier liquid and one or two glass beads.

Assemble the apparatus. Insert a loose plug of cotton wool in the top of the condenser tube to prevent the condensation of atmospheric moisture in the condenser tube.

Heat cautiously and regulate so that the condensate falls from the end of the condenser at a rate of two drops to five drops per second. Continue until the volume of water in the receiver is constant.

Discontinue heating and cool to room temperature. If any droplets of water are adhering to the sides of the receiver or condenser, dislodge them with a fine wire spiral. Measure the volume of water.

F.6 Calculation and report. Calculate the water content, expressed as a percentage by mass, which is given by the expression:

$$\frac{A}{B} \times 100$$

where

A is the volume of water in the trap (in mL);

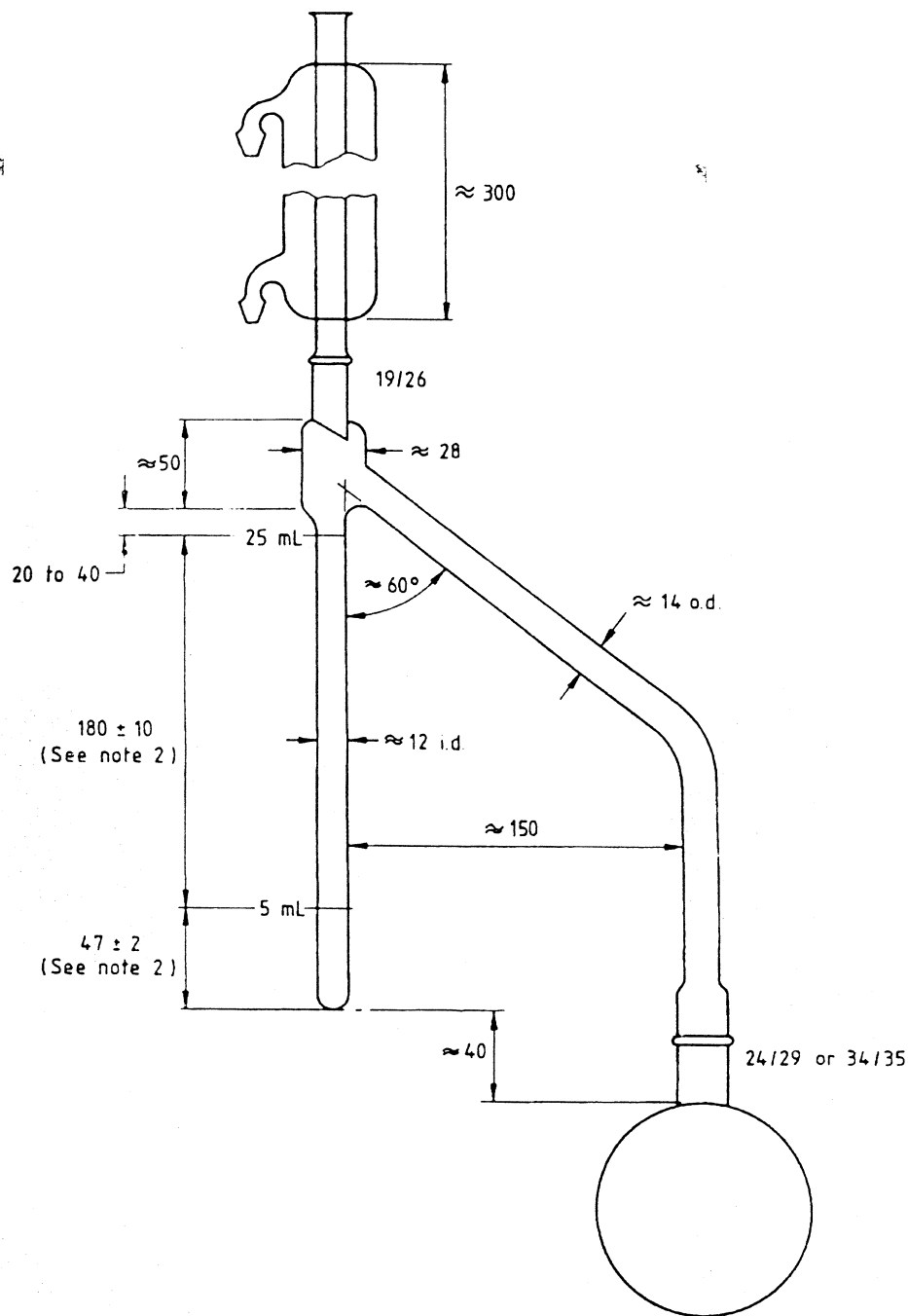
B is the mass of emulsion sample taken (in g);

Report the result to the nearest 0.1 % (m/m).

F.7 Precision. Results of duplicate tests shall not differ by more than the following amounts:

a) repeatability: 1.0 % (m/m);

b) reproducibility: 2.0 % (m/m).

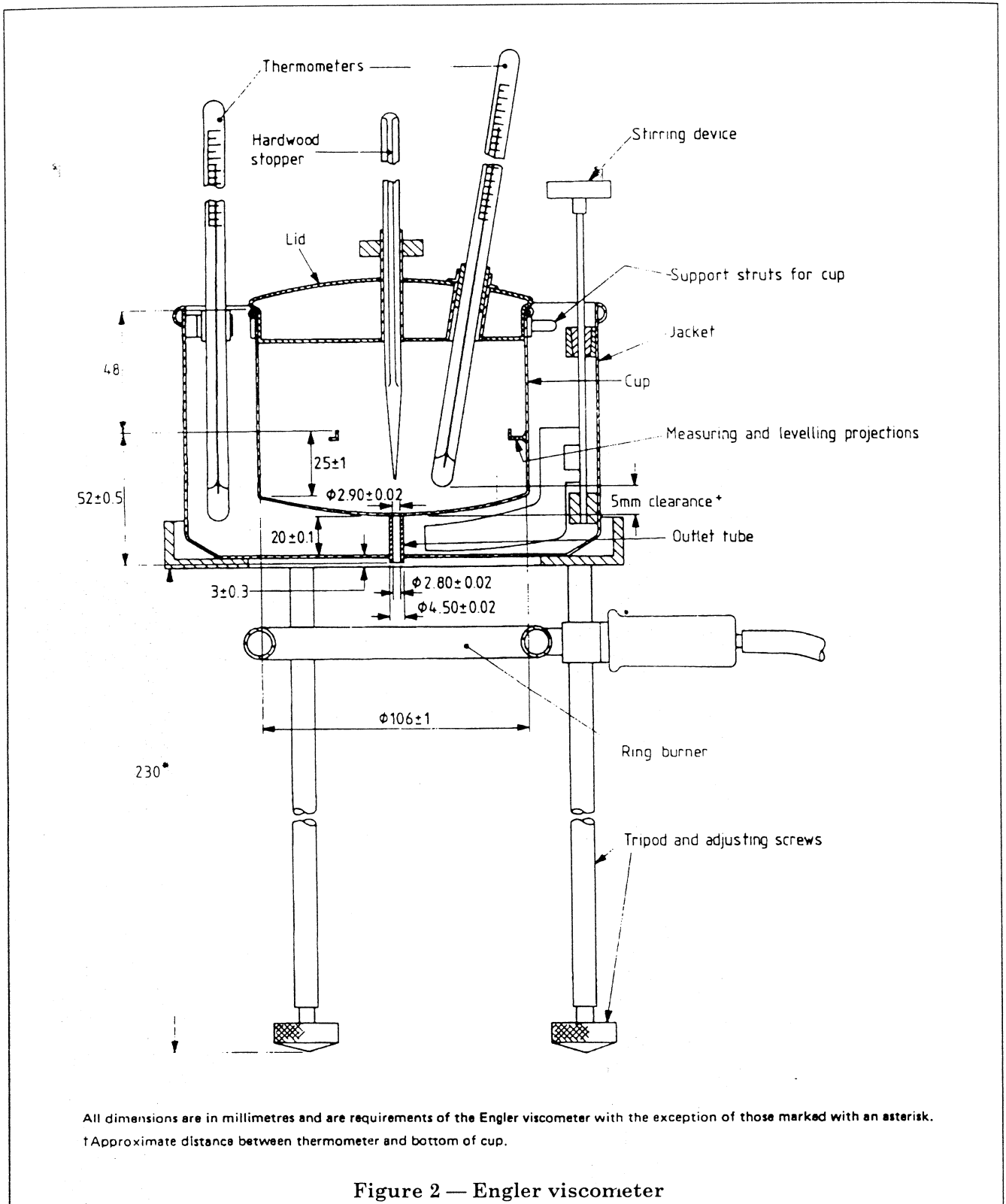


All dimensions are in millimetres.

NOTE 1. Graduations to 0.2 mL are required on the receiver, though for clarity they are omitted from this drawing.

NOTE 2. If apparatus with a glass bulb is used, then a dimension 30 mm to 60 mm may be used in place of 47 ± 2 mm and 170 mm to 210 mm in place of 180 ± 10 mm, provided the graduations in the upper tube allow for the precision tolerances in F.7 to be achieved.

Figure 1 — Assembled apparatus for water content test



Appendix G Methods for determination of viscosity

G.1 Engler viscosity

NOTE This method is based on IP 212/75.

G.1.1 *General.* This method describes a procedure for the determination of the viscosity of bitumen and road emulsions in arbitrary units at 20 °C using the Engler viscometer.

NOTE 1 The flow of bitumen and road emulsions is non-Newtonian in that the relationship between shearing stress and rate of shear is not linear, but under the conditions of the test, the divergence from Newtonian behaviour does not normally lead to any difficulty in obtaining reproducible results. Nevertheless, if measurements are attempted at low temperature, or if the emulsion has an abnormally high viscosity, exceeding 25 °E (degrees Engler) for example, such difficulties may arise.

NOTE 2 The viscosity of road emulsions may be changed permanently by violent agitation, by heating or chilling, or by long storage at ambient temperature.

G.1.2 Apparatus

G.1.2.1 *Viscometer*, that is a standard Engler viscometer as shown in Figure 2. This consists essentially of a cup with a short jet in the centre of the base which may be closed by means of a tapered hard wood stopper. The cup is surrounded by a water bath fitted with a stirring device. Thermometers (10 °C to 55 °C) are carried in both the cup and the water bath.

G.1.2.2 *Two thermometers*, complying with BS 2000-0: Addendum 1.

G.1.2.3 *Receiving flask*, that is a standard Kohlrausch flask of 200 mL capacity at 20 °C, complying with class A of BS 615.

G.1.2.4 *Timing device*, that can be read to 0.2 s or less and it is accurate to 0.1 % over a period of not less than 15 min. A stop-watch of the jewelled lever type, complying with these requirements when fully put not tightly wound is suitable; when in use it shall be held in the same position as when tested.

G.1.2.5 *Sieve*, of 710 µm nominal aperture and complying with BS 410.

G.1.2.6 *Sieve*, of 250 µm nominal aperture and complying with BS 410.

G.1.3 Materials

G.1.3.1 *Petroleum spirit*, 40/60 complying with BS 2000-0: Addendum 1.

G.1.3.2 *Xylene*, complying with BS 458.

G.1.3.3 *Ethanol*, complying with BS 507.

G.1.4 *Calibration.* Wash the cup and jet with petroleum spirit, then several times with alcohol and finally with distilled water.

Level the viscometer approximately and insert a stopper which has been used only for the water test of the apparatus and has never been in contact with oil or emulsion. Fill the receiving flask nearly to the brim with distilled water at 20 °C and pour it into the viscometer cup, allowing the flask to drain in an inverted position for 3 min. thus filling the cup to a little above the pointers. Place the lid and the thermometers in position and maintain the temperature of the water in the cup and the water bath at 20 ± 0.5 °C. By raising the stopper a few times, fill the jet completely with water and wet the surface of the outlet end so that the drop completely covering the surface remains pendant. Set the water surface exactly at the pointers by sucking out excess water with a pipette or, if necessary, by adding a small quantity of water at 20 °C. When the water is completely at rest, place the drained receiving flask underneath the orifice.

Lift the stopper right out vertically so that the surface of the water suffers the minimum disturbance and at the same time start the timer. Note the time taken for the receiving flask to fill to the 200 mL mark.

Repeat the determination until three consecutive results have been obtained differing by not more than 0.5 s and showing no progressive decrease. Clean the apparatus and make another series of determinations in the same way. If agreement is obtained with the results of the first series, no further tests need be made; if not, carry out further series of determinations until constant times of efflux are found. The mean of the six values of the last two series shall be taken as the time of efflux of water and shall lie between the limits of 47 s and 53 s.

G.1.5 *Preparation of specimen.* Stir the emulsion gently and adjust its temperature to 20 ± 0.5 °C in a water bath (see note 2 to G.1.1).

G.1.6 *Procedure.* Adjust the temperature of the water in the jacket of the viscometer to 20 ± 0.5 °C and insert the stopper in the cleaned cup.

Pour the specimen of the emulsion through the 710 µm sieve (G.1.2.5) into the cup until the level makes contact with one or more of the pointers. If necessary, adjust the levelling screws and add more emulsion until the surface of the emulsion just touches all three pointers simultaneously. Remove any excess emulsion with a pipette; do not draw any off by raising the stopper because coagulation of bitumen may occur in the jet when the stopper is re-inserted.

When the temperature of the jacket and of the specimen is steady at 20 ± 0.5 °C, measure the time of outflow of 200 mL of the emulsion into the receiving flask, allowing the emulsion to flow down the side of the flask to avoid frothing.

Make the measurement at a sufficiently short interval after introducing the emulsion into the viscometer so as to minimize sedimentation or creaming and maintain the specimen at 20 ± 0.5 °C during the test. Carry out three determinations on three separate portions of the sample and take the mean value. If the three values differ by more than 5 % of the mean value, carry out three further determinations.

After each determination wash out the cup with distilled water and remove superfluous water with filter paper. Clean the jet thoroughly by means of a spill of soft filter paper moistened with xylene and dry the bottom of the instrument round the jet with filter paper. If bitumen has been deposited in the viscometer, clean by the procedure given in G.1.4.

NOTE When gas heating is used, moisture is liable to be deposited and if the bottom of the jet is damp, irregular readings may result.

In cases where difficulty is encountered through blockage of the jet of the viscometer, strain the emulsion first through the 710 μm sieve (G.1.2.5) and then through the 250 μm sieve (G.1.2.6).

G.1.7 Calculation. Calculate the viscosity, in Engler degrees (°E), which is given by the expression:

$$\frac{T_E}{T_W}$$

where

T_E is the mean time of efflux of 200 mL of emulsion;

T_W is the mean time of efflux of 200 mL of water.

G.1.8 Reporting of results. Report the viscosity to the nearest 0.1 °E.

G.2 Redwood No. II viscosity

G.2.1 Introduction. This method describes the procedure for the determination of the viscosity in seconds at the test temperature (85 °C) using the Redwood No. II viscometer.

G.2.2 Apparatus

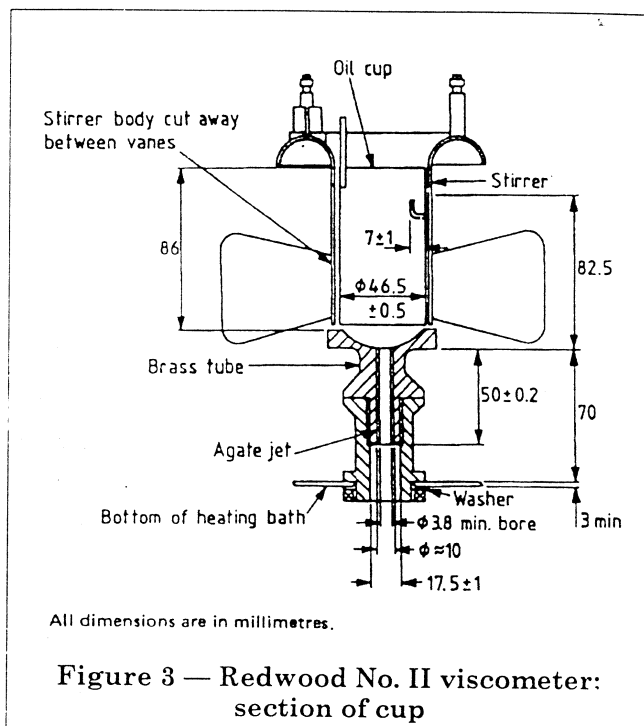
G.2.2.1 Standard Redwood No. II viscometer, of the dimensions given in Figure 3, complete with ball-valve, oil cup cover, thermometer clip, stand and screen.

G.2.2.2 Thermometers, for oil cup and heating bath complying with BS 2000-0: Addendum 1.

NOTE Alternatively, thermometers complying with B110C/100, series B, of BS 593 may be used.

G.2.2.3 Receiver, that is a Kohlrausch type flask, of 50 ± 0.5 mL capacity at 20 °C or a 100 mL cylinder with graduations at 25 mL and 75 mL.

G.2.2.4 Timing device, that can read to 0.2 s or less and is accurate to 0.1 % over a period of not less than 15 min.



G.2.3 Preparation of sample. Place approximately 200 mL of the specimen in a suitable clean, loosely stoppered container and bring to a temperature a little over the test temperature (85 °C) by immersing in a hot water or oil bath; do not heat the specimen over a flame or by the immersion of hot bodies in it.

G.2.4 Procedure. Clean the oil cup with suitable solvents and dry thoroughly with soft tissue paper or other material which will not leave fluff.

Set up the viscometer, level by using a spirit level and fill the bath with water or oil to not less than 10 mm below the rim of the oil cup at test temperature.

Heat the viscometer bath to a few degrees above the test temperature. Homogenize the specimen by stirring and pour the prepared specimen into the oil cup. Adjust the temperature of the bath until the specimen in the cup is maintained at the test temperature of 85 °C, stirring the contents of the bath and cup during the process. Stir the specimen with the thermometer during the preliminary period. Do not stir the specimen during the actual determination.

When the temperature of the specimen has become quite steady at the test temperature, adjust the liquid level, if necessary, by removing any excess until the surface of the specimen touches the filling joint. This should be achieved within 15 min of pouring the specimen into the oil cup in order to minimize loss by evaporation.

Lightly warm the oil cup cover and place it in position on the oil cup and swing the oil cup thermometer towards the closed end of the curved slot in the cover. Place the clean, dry standard 50 mL flask or 100 mL cylinder containing 25 mL of water below the jet with the neck a few millimetres from the bottom of the jet. Do not insulate the flask in any way.

Lift the ball-valve and simultaneously start the time recorder. Suspend the valve from the clip supporting the oil cup thermometer by means of the hook in the wire stem. Stop the timing recorder at the instant the specimen reaches the 50 mL graduation mark on the flask or the 75 mL graduation on the cylinder and note the final reading of the oil cup thermometer.

Reject any determination if the temperature of the specimen in the oil cup varies during the run by more than 0.3 °C.

H.2.5 Reporting of results. Report the viscosity in Redwood No. II seconds to the nearest 0.5 s.

Appendix H Method for determination of coagulation of emulsion at low temperature

H.1 Apparatus

H.1.1 Glass boiling tube, 150 mm long and 25 mm in internal diameter. The tube is provided with a cork with a central hole 13 mm in diameter through which a thermometer passes loosely. The thermometer is fitted with a cork 13 mm in diameter which is capable of being pushed down the stem into the hole in the larger cork when required in order to hold the thermometer rigidly in the centre of the tube.

H.1.2 Three thermometers, graduated at least from -5 °C to +35 °C at intervals of 0.2 °C. One thermometer is for use with H.1.1 and has a minimum distance from the bottom of the bulb to the -5 °C mark of 150 mm, a thermometer complying with BS 2000-0: Addendum 1 is suitable.

H.1.3 Sieve, of 710 µm nominal aperture, complying with BS 410.

H.1.4 Two beakers, of 600 mL capacity.

H.1.5 Water bath, or other means of heating water to 30 °C.

H.2 Materials

H.2.1 Solutions

H.2.1.1 For anionic emulsions: a 2 % (m/m) solution of potassium or sodium oleate in water.

H.2.1.2 For cationic emulsions: a 1 % (m/m) solution of cetrimide (a mixture of alkyltrimethylammonium bromides) in 0.1N hydrochloric acid.

H.2.2 Solvents

H.2.2.1 Xylene, complying with any grade specified in BS 458.

H.2.2.2 Acetone, complying with BS 509.

H.3 Procedure. Wash the 710 µm sieve successively with xylene, acetone and distilled water. Moisten the clean sieve with the appropriate solution.

Pass some of the emulsion through the moistened sieve and introduce 20 mL of sieved emulsion into the boiling tube.

Heat the specimen of emulsion by plunging the tube into the water at 30 °C and stir gently with the thermometer until the temperature of the emulsion is constant. When stirring, take care not to allow the emulsion to coat the tube above the reading level.

Remove the tube from the warm water and plunge it into a 600 mL beaker containing ice water, at the bottom of which is a mass of finely crushed ice retained by a piece of wire gauze fitted into the beaker. Stir slowly during the cooling process. As soon as the temperature of the emulsion no longer falls, add salt to the ice water until the temperature of the freezing mixture reaches -1 °C to -1.5 °C so that the temperature of the emulsion, which should still be slowly stirred, is reduced to 0 °C.

When the emulsion reaches 0 °C, discontinue the stirring, transfer the tube to a second 600 mL beaker of freezing mixture at a temperature of -3 °C to -4 °C and allow the emulsion to remain quiescent for 30 min. While this part of the test is being carried out, hold the thermometer rigidly in the centre of the tube by sliding the smaller cork down to fit firmly within the larger cork.

Remove the tube from the freezing mixture without disturbance and allow the temperature of the contents to rise spontaneously to room temperature, i.e. a temperature not less than 15.5 °C.

Wash the 710 µm sieve successively with xylene, acetone and distilled water. Moisten the clean sieve with the appropriate solution. Pass the emulsion through the sieve, wash the tube free from emulsion and other residue with the appropriate solution and pass the washings through the sieve. The coagulated bitumen, if any, will be retained on the sieve.

H.4 Reporting of results. Report if any coagulated bitumen is retained on the sieve.

Appendix J Methods for determination of storage stability

J.1 Storage stability (short period test)

NOTE The temperature of the laboratory during this test should be between 15.5 °C and 25 °C.

J.1.1 Apparatus

J.1.1.1 Glass cylinder, as specified in BS 2000-15, with an internal diameter between 30 mm and 33 mm and graduation mark at 75 mL.

J.1.1.2 Balance, of 250 g capacity, readable and accurate to 0.1 g.

J.1.1.3 Sieve, of 710 µm nominal aperture, complying with BS 410.

J.1.1.4 Centrifuge, with swing-out head capable of accepting **J.1.1.1**, the speed of which can be controlled to give a relative centrifugal acceleration measured at the outer tips of the rotating tubes of between 500 and 600 times gravitational acceleration. The gravitational acceleration is expressed in m/s^2 .

NOTE 1 The correct speed (in r/min) may be calculated from the formula:

$$\text{speed} = 1337 \sqrt{\frac{a}{d}}$$

where

a is the relative centrifugal acceleration;

d is the tip to tip diameter of the rotating tubes (in mm).

NOTE 2 In measuring d it should be remembered that this refers to the tubes and not to the buckets containing nor the cushions supporting them.

When d equals 380 mm, the speed shall be between 1 530 r/min and 1 675 r/min and when d equals 430 mm, the speed shall be between 1 435 r/min and 1 573 r/min.

J.1.1.5 Inverting machine, capable of rotation through 360°. (A suitable apparatus is shown in Figure 4.)

J.1.2 Materials

J.1.2.1 Solutions

J.1.2.1.1 For anionic emulsions: a 2 % (m/m) solution of potassium or sodium oleate in water.

J.1.2.1.2 For cationic emulsions: a 1 % (m/m) solution of cetrimide (a mixture of alkyltrimethylammonium bromides) in 0.1 N hydrochloric acid.

J.1.2.2 Solvents

J.1.2.2.1 Xylene, complying with any grade specified in BS 458.

J.1.2.2.2 Acetone, complying with BS 509.

J.1.3 Procedure. Pass some of the specimen of emulsion through the sieve previously cleaned as described in **H.3**, and moistened with the appropriate solution. Weigh 10 g, to the nearest 0.1 g, into the glass cylinder which has been cleaned with xylene and acetone, dried, rinsed with the solution and again dried with a clean soft cloth or with absorbent paper. Place the cylinder in the centrifuge and rotate for 5 min. Timing begins when the operating speed appropriate to the machine has been reached.

Add 30 mL of the appropriate solution and close the cylinder by pushing a bung down to the 75 mL graduation mark on the tube. Place the cylinder in the inverting apparatus and from the upright position rotate steadily through 360° five times at 1 r/s. Turn the cylinder through 150° and leave in this inclined position for 10 s to allow the washing to drain. Observe the presence or absence of sediment. From this position rotate for a further five revolutions, allow to drain and observe sediment again before.

Repeat this procedure until the sediment has been completely removed from the base of the cylinder.

NOTE In some cases a few isolated specks of about 1 mm diameter remain at the bottom of the tube after the sediment has been otherwise completely removed. These specks may be disregarded.

J.1.4 Reporting of results. Report the number of complete inversions required to effect complete removal of the sediment.

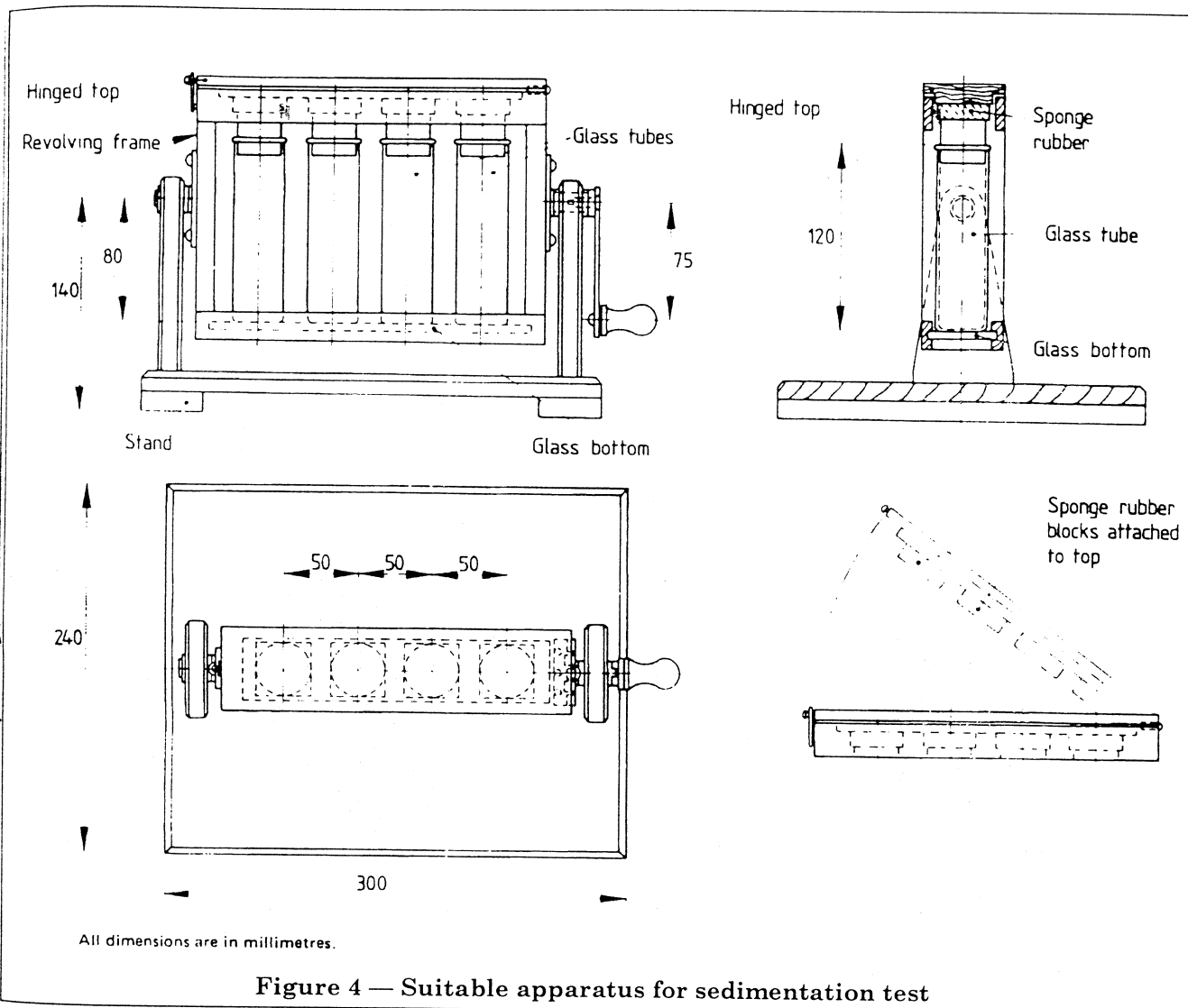
J.2 Storage stability (long period test)

NOTE By reason of their low binder content and low viscosity classes A1-40 and K1-40 emulsions are more prone to sedimentation than the others. The sedimentation resulting from a few weeks storage should easily be dispersed by agitation but longer period of undisturbed storage makes redispersal difficult and, in extreme cases, impossible. These emulsions, therefore, should not be kept in stock for long periods and are consequently not required to meet this test.

J.2.1 Procedure. Obtain 200 L of emulsion as described in **A.2** and stand the 200 L drum on end for 3 months, ensuring that it is not at any time subjected to frost or extremes of temperature.

At the end of this period, take a 4 L specimen of the drum's contents after rehomogenizing the contents as described in **A.2**. Strain this sample through a 710 µm sieve as described in appendix D and determine the water content in accordance with appendix F.

J.2.2 *Reporting of results*, Report the percentage difference between the final and initial water contents to the nearest 0.1 % (m/m).



Publications referred to

- BS 12, *Specification for ordinary and rapid-hardening Portland cement.*
BS 135, 458, 805, *Specifications for benzene, xylenes and toluenes.*
BS 410, *Specification for test sieves.*
BS 434, *Bitumen road emulsions (anionic and cationic).*
BS 434-2, *Code of practice for use of bitumen road emulsions.*
BS 507, *Ethanol.*
BS 509, *Acetone.*
BS 572, *Interchangeable conical ground glass joints.*
BS 593, *Laboratory thermometers.*
BS 615, *Kohlrausch flasks.*
BS 1707, *Hot binder distributors for road surface dressing.*
BS 2000, *Methods of test for petroleum and its products.*
BS 2000-0, *Addendum 1 Standard reagents and thermometers.*
BS 2000-123, *Distillation of petroleum products.*
BS 3136, *Cold emulsion spraying machines for roads.*
BS 3136-2, *Metric units.*
BS 3195, *Methods for sampling petroleum products.*
BS 3195-1, *Liquid hydrocarbons: manual sampling.*
BS 3690, *Bitumens for building and civil engineering.*
IP 91/76, *Bitumen emulsion residue on sieving⁴⁾.*
IP 212/75, *Viscosity — bitumen road emulsion⁴⁾.*
IP 291/73, *Water content of bitumen emulsions⁴⁾.*
IP 292/73, *Particle charge of bitumen emulsions⁴⁾.*

⁴⁾ Published by the Institute of Petroleum, 61 New Cavendish Street, London WIM 8AR.

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