BS 434-1:2011

BSI Standards Publication

# Bitumen road emulsions –

## Part 1: Specification for anionic bitumen road emulsions

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# Contents



**Summary of pages**
This document comprises a front cover, an inside front cover, pages i to ii, pages 1 to 20, an inside back cover and a back cover.

# Foreword

## Publishing information

This part of BS 434 is published by BSI and came into effect on 31 January 2011. It was prepared by Technical Committee B/510, *Road materials*. A list of organizations represented on this committee can be obtained on request to its secretary.

## Supersession

Together with BS EN 13808, this part of BS 434 supersedes BS 434-1:1984, which is withdrawn.

## Relationship with other publications

The requirements for specifying cationic bitumen road emulsions are given in BS EN 13808. Recommendations for the use of these emulsions for roads and other surfaces are covered by BS 434-2.

## Information about this document

This is a full revision of the standard, and introduces the following principal changes:

- the requirements for cationic bitumen road emulsions have been removed;
- Appendix C, Appendix D and Appendix F have been replaced by references to relevant test method standards. Appendix E has been partially replaced;
- the relevant requirements of new European standards have been incorporated, where appropriate.

## Presentational conventions

The provisions of this standard are presented in roman (i.e. upright) type. Its requirements are expressed in sentences in which the principal auxiliary verb is "shall".

*Commentary, explanation and general informative material is presented in smaller italic type, and does not constitute a normative element.*

## Contractual and legal considerations

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

**Compliance with a British Standard cannot confer immunity from legal obligations.**

# 1 Scope

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

# 2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

BS 410-1, *Test sieves – Technical requirements and testing – Part 1: Test sieves of metal wire cloth*

BS 507, *Specification for ethanol for industrial use*

BS 509-1, *Acetone for industrial use – Part 1: Specification for acetone*

BS 615:1953+A2:1963, *Specification for Kohlrausch flasks*

BS 2000-0.1, *Methods of test for petroleum and its products – General introduction – Specifications – IP Standard thermometers – Section 0.1: Specifications – IP standard for thermometers (Identical with IP Annex A)*

BS 2000-0.2, *Methods of test for petroleum and its products – Part 0 – Section 0.2: Specifications for IP standard reference liquids (Identical with IP Appendix B)*

BS 2000-15, *Methods of test for petroleum and its products – Part 15: Petroleum products – Determination of pour point*

BS 6376-3, *Reagents for chemical analysis – Part 3: Specifications (second series)*

BS EN 1428, BS 2000-291, *Methods of test for petroleum and its products – BS 2000-291: Bitumen and bituminous binders – Determination of water content in bituminous emulsions – Azeotropic distillation method (Identical with IP 291-2000)*

BS EN 1429, *Bitumen and bituminous binders – Determination of residue on sieving of bituminous emulsions, and determination of storage stability of sieving*

BS EN 1430, *Bitumen and bituminous binders – Determination of particle polarity of bituminous emulsions*

BS EN 12591:2009, *Bitumen and bituminous binders – Specifications for paving grade bitumens*

BS EN 12848, *Bitumen and bituminous binders – Determination of mixing stability with cement of bituminous emulsions*

BS EN ISO 3170:2004, BS 2000-475:2004, *Methods of test for petroleum and its products – BS 2000-475: Petroleum liquids – Manual sampling (Identical with IP 475-2005)*

# 3 Terms and definitions

For the purposes of this part of BS 434, the following terms and definitions apply.

**3.1 bitumen**
virtually involatile, adhesive and waterproofing material derived from crude petroleum, or present in natural asphalt, which is completely or nearly completely soluble in toluene, and very viscous or nearly solid at ambient temperatures

[BS EN 12597]

**3.2 bitumen road emulsion**
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

*NOTE There are two types of bitumen road emulsions: anionic and cationic.*

**3.3 anionic emulsion**
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

**3.4 cationic emulsion**
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

**3.5 binder content (including emulsifier)**
difference between 100% and the percentage water content determined in accordance with BS EN 1428

# 4 Classification of 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 surfacing.* An emulsion formulated for slurry surfacing; used cold.

*NOTE 1 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 2 Classes A1 and A2 are subdivided according to their nominal binder contents (see Table 1).*

*NOTE 3 Due to the wide variation in chemical activity and physical structure of aggregates from different sources, not all are suitable for slurry surfacing. Each aggregate may be tested in the laboratory with class A4 emulsion and the mixes with class A4 emulsion subjected to the wearing (track abrasion) test in accordance with BS EN 12274-5. The loss in mass of the specimen after abrasion not exceeding 500 g/m$^2$ may be an appropriate criterion for most applications.*

*NOTE 4 Cationic road emulsions are classified in accordance with BS EN 13808. BS EN 13808 provides a framework of classes for the different properties that may be specified as supplied. Surface dressing binders (e.g. former K1-70 specified as C69B2, C69B3, C69B4, C69BF2, C69BF3 or C69BF4 according to BS EN 13808:2005) are used hot and the viscosity at 85 °C for these binders may be determined using the Redwood No. II viscosity method in **D.2**. Bitumen emulsions can change in viscosity during storage and transit and viscosity at 85 °C may be by agreement between the purchaser and supplier provided that hot binder distributors conform to BS 1707:1989, Appendix A.*

# 5 Composition

## 5.1 General

Anionic road emulsions shall contain bitumen, modified if necessary as specified in **5.2**, emulsifying agents and water. Small amounts of surfactants and additives shall be permitted to modify performance, as in slurry surfacing and other emulsions for special purposes.

## 5.2 Bitumen

The bitumen used for the manufacture of the emulsion shall conform to BS EN 12591:2009 and shall be selected from the range grade 50/70 to grade 250/300, 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, 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 paving grade 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 conforming to BS EN 12591 might not give the same test results on analysis after extraction from the emulsion.*

# 6 Particle charge

When tested in accordance with BS EN 1430, anionic emulsion shall deposit on the anode, indicating that the particles have a negative charge.

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

# 7 Properties of emulsions

## 7.1 Test procedures

Procedures for sampling and specimen preparation shall be as described in Annex A and Annex B respectively. Emulsions shall be tested in accordance with the annexes or test standards referred to in Table 1.

## 7.2 Test requirements

The properties of anionic road emulsions shall be as given in Table 1. The test result from one of the 4 L samples tested shall be compared with the appropriate limit(s) given in Table 1. If the test results do not conform to the limit(s) given in Table 1, two further reserved samples shall be tested and the test results of two out of any of the three samples tested shall conform to the limit(s) given in Table 1.

Table 1 **Properties of anionic road emulsions**

| **Property** | **Annex or test standard** | **Class of anionic road emulsions** | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | **A1-60** | **A1-55** | **A1-40** | **A2-57** | **A2-50** | **A3** | **A4** |
| Particle charge | BS EN 1430 | neg. | neg. | neg. | neg. | neg. | neg. | neg. |
| Residue on 500 µm BS sieve [% (m/m), maximum] | BS EN 1429 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 |
| Residue on 160 µm BS sieve (g per 100 mL, maximum) | BS EN 1429 | 0.15 | 0.15 | 0.15 | 0.15 | 0.15 | 0.15 | 0.15 |
| Stability to mixing with coarse aggregate (% coagulation) | C | 20 to 80 | 20 to 80 | 20 to 80 | < 40 | < 40 | < 5 | < 5 |
| Stability to mixing with cement (% coagulation) | BS EN 12848 | — | — | — | > 2 | > 2 | < 2 | — |
| Binder content [% (m/m), minimum] | BS EN 1428 | 58 | 53 | 38 | 55 | 48 | 55 | 56 |
| Viscosity [degrees Engler (°E) at 20 °C] [A)] | D.1 | 6 to 9 | 5 to 8 | ≤ 4 | ≤ 8 | ≤ 5 | ≤ 9 [B)] | ≤ 8 [B)] |
| Coagulation of emulsion at low temperature | E | nil | nil | nil | nil | nil | nil | nil |
| Storage stability (short period test) (inversions to clear sediment, maximum) | F.1 | 60 | 60 | 60 | 60 | 60 | 60 | 60 |
| Storage stability (long period test) (% water content difference, maximum) | F.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 conforms to all the other requirements of this standard.*

[A)] The viscosities given in the table are suitable for use in bulk spreaders conforming to BS 3136-2 or for use in hand operated sprayers conforming to BS 3136-2. As a general guide, a maximum viscosity of 9 °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.

# 8 Containers and marking

## 8.1 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 can lead to the evolution of hydrogen and the subsequent explosion of the container.*

## 8.2 Marking

Containers shall be legibly labelled with the following minimum information:

a) the name or trade mark of the manufacturer or supplier of emulsion;

b) the date of filling;

c) the number of this British Standard, i.e. BS 434-1:2010 [1)];

d) the class of emulsion and nominal binder content (see Table 1);

e) the mass of contents in kilograms or volume in litres;

f) the word "Anionic" on the head of the container, which shall be coloured black;

g) the instructions to roll the drums before use, particularly in the case of 40% binder content emulsions;

h) an instruction to protect from frost.

Documents relative to bulk deliveries shall provide similar relevant information.

1) Marking BS 434-1:2011 on or in relation to a product represents a manufacturer's declaration of conformity, i.e. a claim by or on behalf of the manufacturer that the product meets the requirements of the standard. The accuracy of the claim is solely the claimant's responsibility. Such a declaration is not to be confused with third-party certification of conformity.

# Annex A (normative) 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. 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. Take three or more specimens.

Use one of the specimens for carrying out the tests referred to in Table 1 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 BS EN 1428.

Use one of the drums and its contents for carrying out the test given in **F.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 BS EN ISO 3170:2004, Figure 1 and Figure 2.

Take three samples of at least 4 L each. Use one of the samples to prepare specimens for carrying out the tests described in Table 1 and keep the other two samples in reserve.

*NOTE Reference should also be made to BS 434-2 for advice on handling, storage and safety.*

## Annex B (normative) Preparation of specimens

Prepare the 4 L specimen by passing it through a 500 µm sieve as described in BS EN 1429 and use this emulsion, after gentle stirring immediately before use, for all tests.

*NOTE The residue on the 500 µm sieve may be used to decide if the specimen conforms to the limit(s) given in Table 1 .*

## Annex C (normative) Method for determination of stability to mixing with coarse aggregate

*NOTE For the purposes of this British Standard, emulsions are classified in relation to the aggregate specified in **C.3**.*

### C.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, conformity to these values does not necessarily ensure satisfactory performance on application.

### C.2 Apparatus

**C.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.

**C.2.2** *Machine*, for rotating the bottle end over end at the rate of 60 to 70 revolutions per minute (r/min).

**C.2.3** *Two flat-bottomed nickel dishes*, weighed and approximately 90 mm diameter and 15 mm deep.

**C.2.4** *Balance*, of 250 g capacity, readable and accurate to 0.1 g.

**C.2.5** *Balance*, of 250 g capacity, readable and accurate to 0.001 g.

**C.2.6** *Sieve*, of 710 µm nominal aperture, approximately 100 mm in diameter and 40 mm deep and conforming to BS 410-1.

**C.2.7** *Sieve*, of 3.35 mm nominal aperture, 200 mm in diameter and conforming to BS 410-1.

**C.2.8** *Soft cloth*.

**C.2.9** *Pipette*, of 25 mL capacity.

**C.2.10** *Water bath*.

**C.2.11** *Fume cupboard*.

**C.2.12** *Dessicator*.

**C.2.13** *10 mm nominal aperture sieve*, conforming to BS 410-1.

**C.2.14** *6 mm nominal aperture sieve*, conforming to BS 410-1.

**C.2.15** *Glass beaker*, of 1 000 mL capacity.

**C.2.16** *Glass rod*.

## C.3 Materials

**C.3.1** *Aggregate*, consisting of Criggion chippings passing a 10 mm nominal aperture sieve and retained on a 6.3 mm nominal aperture sieve. Place about 1 kg in the 3.35 mm sieve 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.

**C.3.2** *Xylene*, conforming to BS 6376-3, with an evaporation residue not exceeding 0.02 g/L.

**C.3.3** *Soap solution*, 2% (m/m) solution of potassium or sodium oleate in water.

**C.3.4** *Distilled water*.

**C.3.5** *Tap (drinking) water*.

## C.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.

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 $\pm$1) g of the aggregate into the bottle, followed by (20 $\pm$0.2) g of emulsion which has been passed through the 710 µm sieve. 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 $\pm$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 and repeat the process until the washings remain clear.

After draining away as much as possible of the water, place the sieve in a sufficiently large beaker. Pour 100 mL of xylene into the beaker to dissolve the bitumen on the sieve and 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. 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.

### C.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:

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

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 BS EN 1428.

### C.6 Reporting of results

Report coagulation value as a percentage to the nearest whole number.

## Annex D (normative) Methods for determination of viscosity

### D.1 Engler viscosity

*NOTE This method is based on IP 212 [1].*

#### D.1.1 General

This method describes a procedure for the determination of the viscosity of bitumen 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 could arise.*

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

#### D.1.2 Apparatus

**D.1.2.1** *Viscometer*, that is a standard Engler viscometer as shown in Figure D.1. This consists essentially of a cup with a short jet in the centre of the base which can 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.

**D.1.2.2** *Two thermometers*, conforming to BS 2000-0.1.

**D.1.2.3** *Receiving flask*, that is a standard Kohlrausch flask of 200 mL capacity at 20 °C, conforming to class A of BS 615:1953+A2:1963.

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

**D.1.2.5** *Sieve*, of 710 µm nominal aperture and conforming to BS 410-1.

**D.1.2.6** *Sieve*, of 250 µm nominal aperture and conforming to BS 410-1.

**D.1.2.7** *Pipette*.

**D.1.2.8** *Filter paper*.

**D.1.2.9** *Water bath*.

**D.1.2.10** *Glass rod.*

### D.1.3 Materials

**D.1.3.1** *Petroleum spirit*, 40/60 conforming to BS 2000-0.2.

**D.1.3.2** *Xylene*, conforming to BS 6376-3.

**D.1.3.3** *Ethanol*, conforming to BS 507.

**D.1.3.4** *Distilled water*.

### D.1.4 Calibration

Wash the cup and jet with petroleum spirit, then several times with ethanol and finally with distilled water.

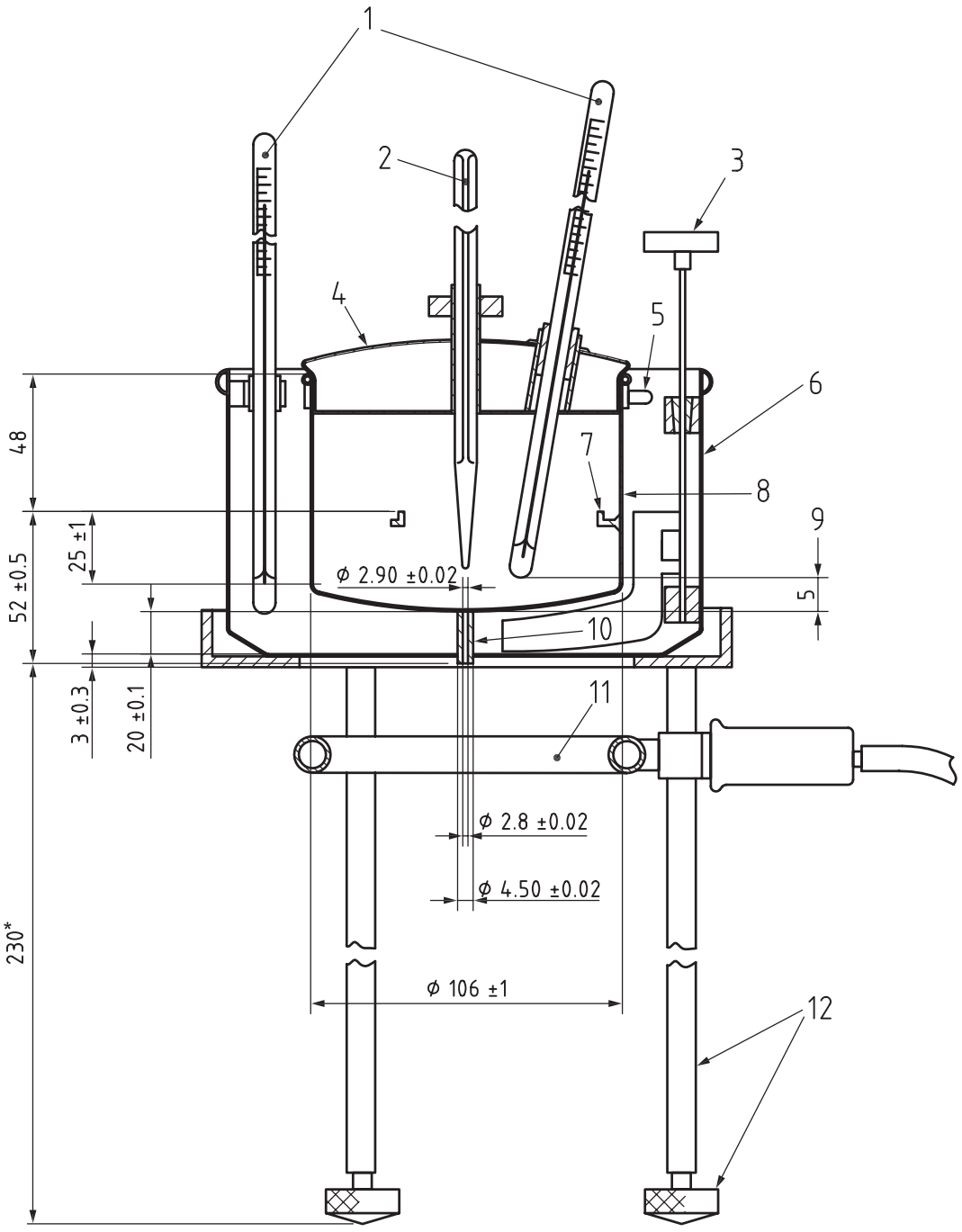
Level the viscometer approximately and insert a stopper which has been used only for the water test of the apparatus and has never been in contact with oil or emulsion. Fill the receiving flask nearly to the brim with distilled water at 20 °C and pour it into the viscometer cup, allowing the flask to 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 $\pm$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.

Figure D.1 **Engler viscometer**

Dimensions in millimetres




**Key**

| | | | |
|---|---|---|---|
| 1 | Thermometers | 7 | Measuring and levelling projections |
| 2 | Hardwood stopper | 8 | Cup |
| 3 | Stirring device | 9 | Clearance (approximate distance between thermometer and bottom of cup) |
| 4 | Lid | 10 | Outlet tube |
| 5 | Support struts for cup | 11 | Ring burner |
| 6 | Jacket | 12 | Tripod and adjusting screws |

All dimensions are requirements of the Engler viscometer apart from that marked with an asterisk (*)

### D.1.5 Preparation of specimen

Stir the emulsion gently and adjust its temperature to (20 ±0.5) °C in a water bath (see **D.1.1**, Note 2).

### D.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 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 could 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 **D.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 might result.*

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

### D.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.

### D.1.8 Reporting of results

Report the viscosity to the nearest 0.1 °E.

## D.2 Redwood No. II viscosity

### D.2.1 General

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.

### D.2.2 Apparatus

**D.2.2.1** *Standard Redwood No. II viscometer*, of the dimensions given in Figure D.2, complete with ball-valve, oil cup cover, thermometer clip, stand and screen.

**D.2.2.2** *Thermometers*, for oil cup and heating bath conforming to BS 2000-0.1.

*NOTE Alternatively, thermometers conforming to B110C/100, series B, of BS 593:1989 may be used.*

**D.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.

**D.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.

**D.2.2.5** *Loosely stoppered container*, of at least 200 mL capacity.

**D.2.2.6** *Spirit level*.

**D.2.2.7** *Soft tissue paper* or other material suitable for drying the oil cup which will not leave fluff.

**D.2.2.8** *Water/oil bath*.

### D.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.

### D.2.4 Procedure

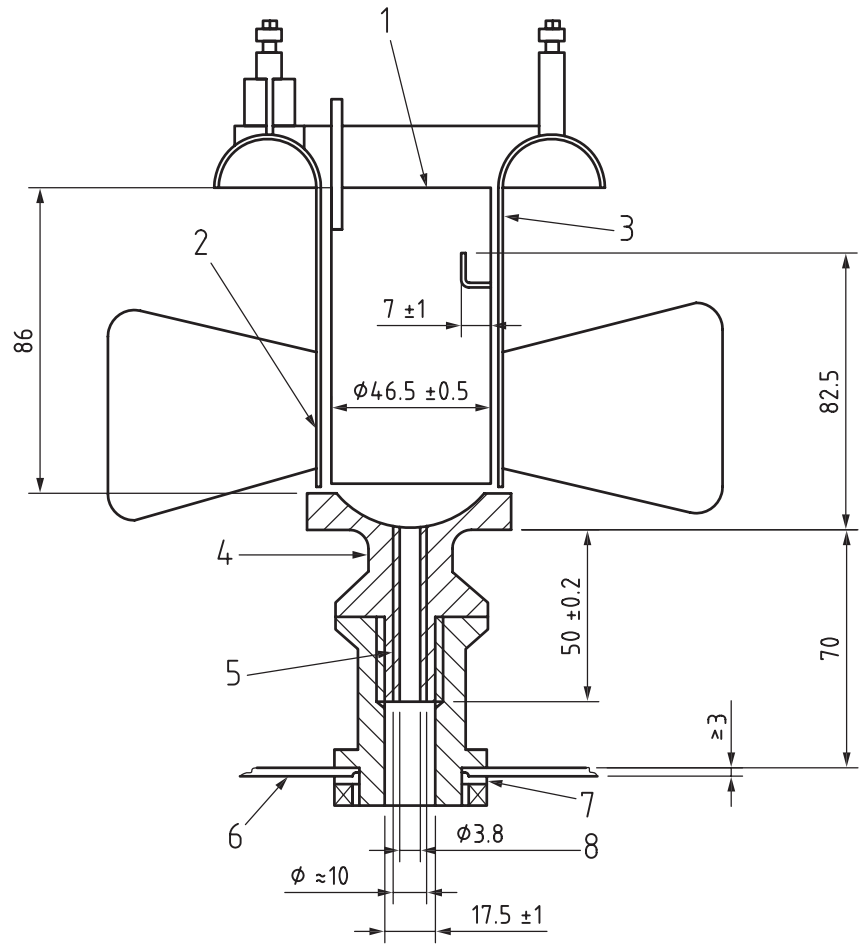
Clean the oil cup with suitable solvents and dry thoroughly with soft tissue paper or other material which will not leave fluff.

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.

Figure D.2 **Redwood No. II viscometer: section of cup**

Dimensions in millimetres




**Key**

| | | | |
|---|---|---|---|
| 1 | Oil cup | 5 | Agate jet |
| 2 | Stirrer body cut away between vanes | 6 | Bottom of heating bath |
| 3 | Stirrer | 7 | Washer |
| 4 | Brass tube | 8 | Minimum bore |

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

Slightly 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 device 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.

### D.2.5 Reporting of results

Report the viscosity in Redwood No. II seconds to the nearest 0.5 s.

## Annex E (normative) Method for determination of coagulation of emulsion at low temperature

### E.1 Apparatus

**E.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.

**E.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 **E.1.1** and has a minimum distance from the bottom of the bulb to the –5 °C mark of 150 mm; a thermometer conforming to BS 2000-0.1 is suitable.

**E.1.3** *Sieve*, of 710 µm nominal aperture, conforming to BS 410-1.

**E.1.4** *Two beakers*, of 600 mL capacity, one provided with wire gauze to be fitted over crushed ice in the bottom.

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

### E.2 Materials

#### E.2.1 Solutions

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

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

#### E.2.2 Solvents

**E.2.2.1** *Xylene*, conforming to any grade specified in BS 6376-3.

**E.2.2.2** *Acetone*, conforming to BS 509-1.

#### E.2.3 Other materials

**E.2.3.1** *Distilled water*.

**E.2.3.2** *Ice water*.

**E.2.3.3** *Finely crushed ice*.

**E.2.3.4** *Salt*.

### E.3 Procedure

Wash the 710 µm sieve successively with xylene, acetone and distilled water. Moisten the clean sieve with the solution (**E.2.1.1**).

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 (salt added to ice water to induce melting of the ice and reduction of temperature down to below 0 °C) 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.

### E.4 Reporting of results

Report if any coagulated bitumen is retained on the sieve.

## Annex F (normative) Methods for determination of storage stability

### F.1 Storage stability (short period test)

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

#### F.1.1 Apparatus

**F.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, provided with a bung to close it.

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

**F.1.1.3** *Sieve*, of 710 µm nominal aperture, conforming to BS 410-1.

**F.1.1.4** *Centrifuge*, with swing-out head capable of accepting **F.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) can be calculated from the formula:*

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

*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.

**F.1.1.5** *Inverting machine*, capable of rotation through 360°. (A suitable apparatus is shown in Figure F.1.)

**F.1.1.6** *Clean, soft cloth or absorbent paper*, suitable for drying the glass cylinder.

## F.1.2 Materials

### F.1.2.1 Solutions

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

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

### F.1.2.2 Solvents

**F.1.2.2.1** *Xylene*, conforming to any grade specified in BS 6376-3.

**F.1.2.2.2** *Acetone*, conforming to BS 509-1.

## F.1.3 Procedure

Pass some of the specimen of emulsion through the sieve previously cleaned as described in **E.3**, and moistened with the solution (**F.1.2.1.1**). 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 washings to drain. Observe the presence or absence of

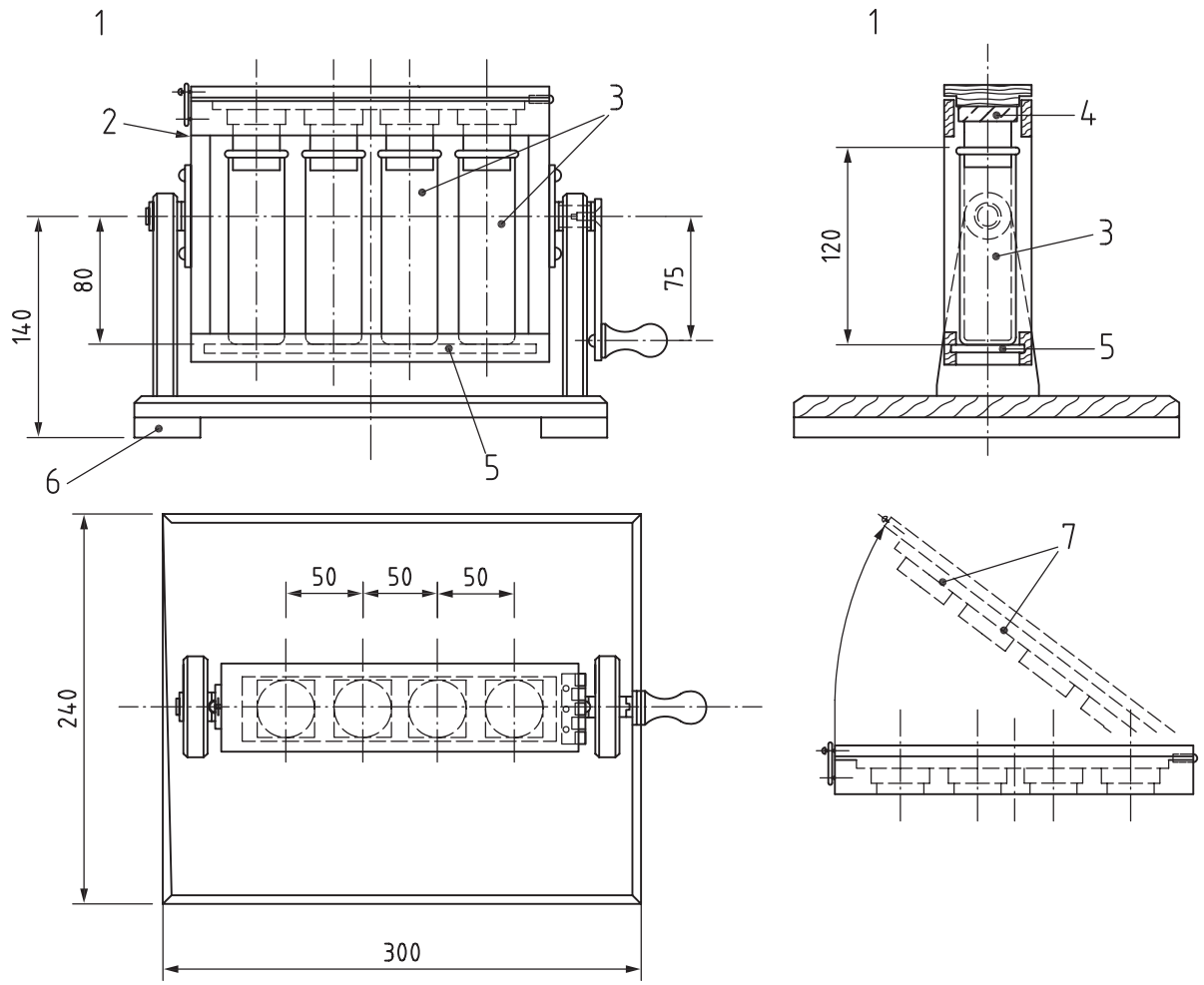
sediment. From this position rotate for a further five revolutions, allow to drain and observe sediment as before.

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.*

Figure F.1 **Suitable apparatus for sedimentation test**

Dimensions in millimetres




**Key**

1 Hinged top
2 Revolving frame
3 Glass tubes
4 Sponge rubber
5 Glass bottom
6 Stand
7 Sponge rubber blocks attached to top

### F.1.4 Reporting of results

Report the number of complete inversions required to effect complete removal of the sediment.

## F.2 Storage stability (long period test)

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

### F.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 BS EN 1429 and determine the water content in accordance with BS EN 1428.

### F.2.2 Reporting of results

Report the percentage difference between the final and initial water contents to the nearest 0.1% (m/m).

# Bibliography

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For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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BS 593:1989, *Specification for laboratory thermometers*

BS 1707:1989, *Specification for hot binder distributors for road surface dressing*

BS 3136-2, *Specification for cold emulsion spraying machines for roads – Part 2: Metric units*

BS EN 12274-5, *Slurry surfacing – Test Method – Part 5: Determination of wearing*

BS EN 13808:2005, *Bitumen and bituminous binders – Framework for specifying cationic bituminous emulsions*

## Other publications

[1] IP 212, *Determination of viscosity of bitumen emulsions – Engler method* [2)].

[2)] Available from www.energyinst.org.uk.

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