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Methods of test for petroleum and its products

Part 19: Determination of demulsibility characteristics of lubricating oil

(Identical with IP 19/11)

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Publishing and copyright information

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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 6, an inside back cover and a back cover.

Foreword

Publishing information

This British Standard is published by BSI and came into effect on 30 June 2011. It was prepared by Technical Committee PTI/13, *Petroleum Testing and Terminology*. A list of organizations represented on this committee can be obtained on request to its secretary.

Supersession

This part of BS 2000 supersedes BS 2000-19:2003, which is withdrawn.

Information about this document

This new edition has been updated to be in line with changes implemented by the Energy Institute.

BS 2000 comprises a series of test methods for petroleum and its products that are published by the Institute of Petroleum (IP) and have been accorded the status of a British Standard. Each method should be read in conjunction with the preliminary pages of "Standard Methods for Analysis and Testing of Petroleum Products and British Standard 2000 Parts" which gives details of the BSI/IP agreement for publication of the series, provides general information on safety precautions, sampling and other matters, and lists the methods published as parts of BS 2000.

Under the terms of the agreement between BSI and the Institute of Petroleum, the revised version of BS 2000-10 will be published by BSI and by the IP (in "Standard Methods for Analysis and Testing of Petroleum Products and British Standard 2000 Parts" and as a separated publication). The numbering of the parts of BS 2000 follows that of the corresponding IP methods. BS 2000-19:2011 is thus identical with IP 19/11.

WARNING. This part of BS 2000 calls for the use of substances and/or procedures that can be injurious to health if adequate precautions are not taken. It refers only to technical suitability and does not absolve the user from legal obligations relating to health and safety at any stage.

It has been assumed in the preparation of this British Standard that the execution of its provisions will be entrusted to appropriately qualified and experienced people, for whose use it has been produced.

Presentational conventions

The provisions of this standard are presented in roman (i.e. upright) type. Its methods are expressed as a set of instructions, a description, or 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 method gives a measure of the ability of the oil to separate from an emulsion. It is commonly applied to turbine oils, but it may be used for other lubricating oils. The test is commonly applied to used turbine oils but since it is sensitive to aging and contamination of the oil, precision will be lower than that stated.

NOTE 1 Oils containing inhibitors may give much higher results than the corresponding uninhibited oils and the precision may be less satisfactory.

NOTE 2 For the purposes of this standard, the terms "% (m/m)" and "% (V/V)" are used to represent respectively the mass fraction and the volume fraction.

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 2000-0.1:1999, *Methods of test for petroleum and its products – BS 2000 Part 0: General introduction – Section 0.1: Specifications – IP standard for thermometers (Identical with IP – Annex A)*

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

BS EN ISO 3696:1995, *Water for analytical laboratory use – Specification and test methods*

3 Terms and definitions

For the purposes of this standard, the following terms and definitions apply.

3.1 demulsification number
number of seconds required for an oil to separate when it is emulsified and separated under specified conditions

4 Principle

20 mL of the oil is emulsified with steam at about 90 °C. The emulsion is then placed in a bath at about 94 °C and the time for 20 mL of oil to separate recorded.

5 Reagents and materials

5.1 Use only reagents of recognized analytical grade.

5.2 *Ammonium peroxydisulfate*, store in a cold, dry place.

5.3 *Sulfuric acid*, concentrated minimum purity 98% *(V/V)*.

5.4 *Ammonium peroxy disulfate* cleaning solution 8 g/L, dissolve 8 g of ammonium peroxydisulfate (**5.2**) in the minimum amount of water and transfer whilst stirring to a 2 L beaker containing 1 L of concentrated sulfuric acid (**5.3**).

5.5 *De-ionised water*, unless otherwise specified, meeting the requirements for grade 3 of BS EN ISO 3696:1995.

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NOTE 3 Alternative commercial cleaning solutions are available, but the user should confirm that the results obtained after cleaning the oil container tube with such cleaning solutions are within repeatability of the method. Commercial alkaline cleaning solutions will cause etching if allowed to be in contact with glassware for long periods.

6 Apparatus

6.1 Of the form and dimensions shown in Figure 1 and consisting of:

6.2 *Steam generator*, of metal or glass and, if of metal, fitted with a water-gauge.

6.3 *Separating and emulsifying baths*, of glass and provided with wooden or metal covers which hold the oil container and thermometers vertically in the bath.

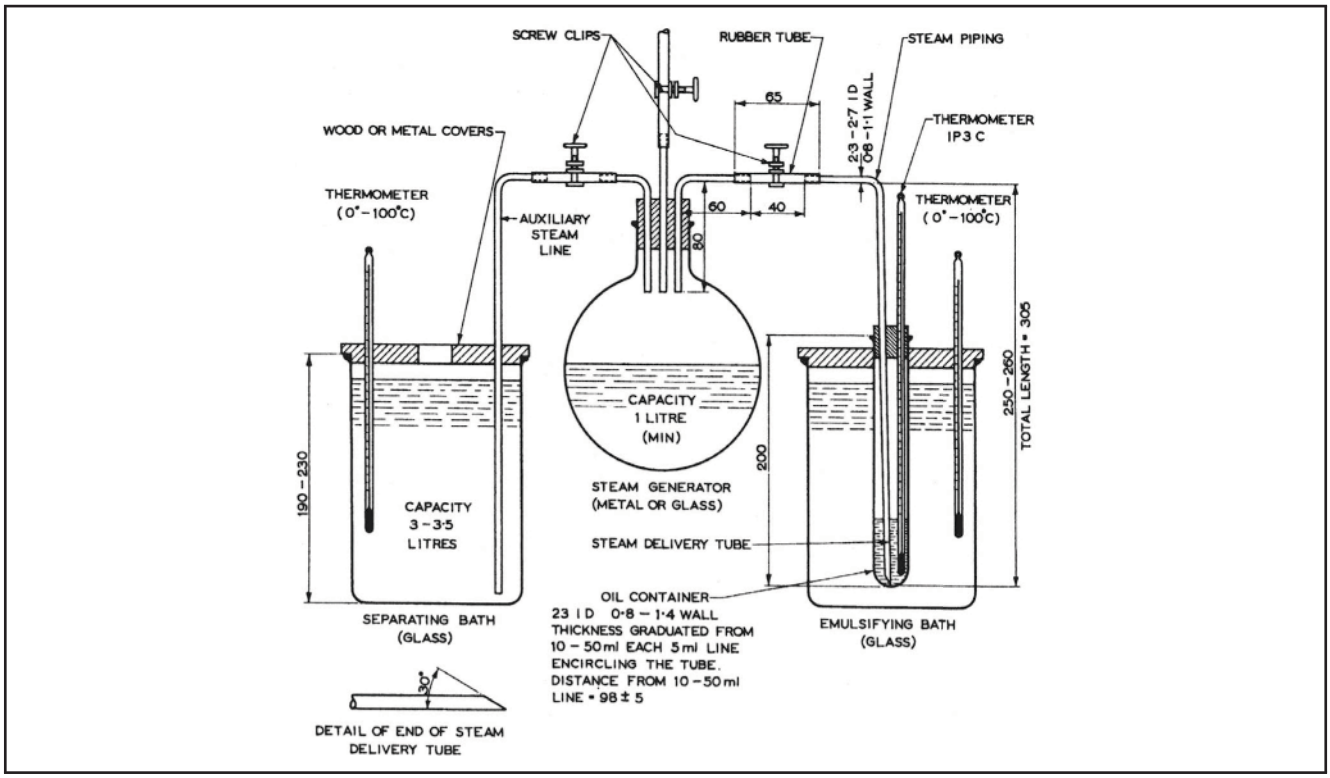
6.4 *Heat sources*, a suitable burner or electric hot-plate for the steam generator and any convenient means, such as an auxiliary steam line, for the separating bath.

6.5 *Oil-container tube*.

6.6 *Steam piping*.

6.7 *Thermometers*, for the separating and emulsifying baths, of suitable range, e.g. 0 °C to 110 °C; for the oil container, conforming to Type 3 C, as specified in BS 2000-0.1:1999.

Figure 1 **Apparatus for steam emulsion test, self-generating steam supply**



7 Sampling and sample handling

7.1 Unless otherwise specified samples shall be taken as described in BS 2000-475.

7.2 The results obtained by this method are readily affected by the presence of traces of impurities in the sample and, in consequence, it is important to avoid contamination of the sample. Take pre-cautions to exclude light from the sample until it can be tested.

8 Apparatus preparation

8.1 Fill the oil-container tube (**6.5**) with the ammonium peroxydisulfate solution (**5.4**) (see Note 3), and allow to stand overnight in a fume cupboard. Remove the cleaning solution and rinse several times with water (**5.5**).

8.2 Fill the steam generator half-full of water, adding some anti-bumping granules for the promotion of even ebullition, and assemble the apparatus as shown in Figure 1. Take care to exclude from the steam generator any foreign materials which would cause contamination of the steam, and make sure that the steam-piping is clean. Fill each bath with (3 000 ±60) mL of water.

8.3 Boil the water in the steam generator, raise the temperature of the water in the emulsifying bath to 19 °C to 26 °C, and in the separating bath to 93 °C to 95 °C.

9 Procedure

9.1 Make duplicate determinations.

9.2 Ensure that all parts of the apparatus which come into contact with the sample are chemically clean, since this test is very susceptible to contaminants. Measure 20 mL of the sample into the oil-container and place the latter in the holder of the emulsifying bath. Place a cork with two openings, with the thermometer in one, in the mouth of the oil-container, adjusting the thermometer so that the bottom of the bulb is 20 mm to 25 mm from the bottom of the tube.

9.3 Pass steam through the steam-delivery tube until the condensate disappears, and then disconnect it from the rubber tubing and insert it through the second opening in the cork until the end of the tube touches the centre of the bottom of the oil-container. The fit between the steam-delivery tube and the cork should be loose. Re-connect the rubber tubing to the steam-delivery tube and admit steam at such a rate that the temperature of the oil, as indicated by the thermometer in the oil-container, rises to 88 °C to 91 °C. The usual time for the temperature of the oil to come to this point is 45 s to 75 s, depending on its nature. Control the temperature by regulating the screw clips on the steam pipe and the steam exhaust making sure that the steam supply is sufficient at all times to cause a generous discharge from the exhaust. Continue the steaming until the volume of the condensed water and oil in the oil-container is (40 ±3) mL, making appropriate allowance for the volume of the steam-delivery tube and the thermometer; this volume is approximately 4 mL. In order to observe the volume of condensed water and oil momentarily prevent the entry of steam into the oil container. The time for this operation should be 4 min to 6.5 min. If the time is less than 4 min, reject the test as the steam is probably wet, or the steam-delivery tube was incompletely steamed out.

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9.4 Disconnect the steam-delivery tube from the rubber tubing, and immediately start a stop-watch. Without delay, transfer the oil-container to the separating bath; it is extremely important that the separating bath be maintained at 93 °C to 95 °C. Remove the thermometer and steam-delivery tube from the oil-container (see Note 3). Every 30 s record the volume of the separated oil layer, making no differentiation between the clear and turbid oil. Where the interface between more or less clear oil and the emulsion is not a clear, straight, horizontal line, estimate the position of such a line to the nearest 0.5 mL, mid-way through the more or less clear oil-bubbles. Consider as part of the water layer any lacy emulsion. Record the time in s at the end of the 30 s interval during which 20 mL of oil separates. If 20 mL of oil has not separated in 20 min, discontinue the test and record the volume of oil separated.

NOTE 4 The oil-container tube shall not be withdrawn from the separating bath during the period of examination.

10 Calculation

Calculate the mean of the results of the duplicate determinations of the demulsification number. If these results differ from the mean by more than 15 s, or by more than 10% of the mean if this is greater, carry out a third test.

11 Expression of results

Report the mean of the Demulsification Number, in s, rounded to the nearest 15 s.

If 20 mL of oil has not separated in 20 min, report the Demulsification Number as 1 200 + (number of mL separated).

12 Precision

12.1 Repeatability, *r*

The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material would in the long run, in the normal and correct operation of the test method, exceed the value given in Table 1 in absolute value in only one case in twenty.

12.2 Reproducibility, *R*

The difference between two single and independent test results, obtained by different operators working in different laboratories on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the value given in Table 1 in only one case in twenty.

Table 1 **Precision**

	Repeatability	**Reproducibility**
Unused oil	30 s or 20% of mean, whichever is greater	60 s or 30% of mean, whichever is greater

13 Test report

The test report shall contain at least the following information:

a) a reference to this standard;

b) the type and complete identification of the product tested;

c) the result of the test (see Clause **11**);

d) any deviation, by agreement or otherwise, from the procedure specified;

e) the date of the test.

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