International Standard 6614

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Petroleum oils and synthetic fluids — Determination of demulsibility characteristics

Huiles de pétrole et fluides synthétiques — Détermination des caractéristiques de désémulsion

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 6614 was developed by Technical Committee ISO/TC 28, *Petroleum products and lubricants*, and was circulated to the member bodies in December 1981.

It has been approved by the member bodies of the following countries:

Australia	Israel	South Africa, Rep. of
Austria	Italy	Sri Lanka
Belgium	Japan	Sweden
Brazil	Korea, Rep. of	Switzerland
Canada	Netherlands	United Kingdom
Egypt, Arab Rep. of	Peru	USA
France	Poland	USSR
Germany, F.R.	Portugal	Venezuela
Hungary	Romania	
India	Spain	

No member body expressed disapproval of the document.

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Petroleum oils and synthetic fluids — Determination of demulsibility characteristics

1 Scope and field of application

This International Standard specifies a method for measuring the ability of petroleum oils or synthetic fluids to separate from water. Recommended test temperatures are 54 ± 2 °C and 82 ± 1 °C, the latter being used when the viscosity at 40 °C is more than 90 cSt*.

NOTE — Although developed specifically for steam-turbine oils having viscosities of 32 to 95 cSt at 40 °C, this method may be used to test oils of other types having various viscosities, and synthetic fluids. It is recommended that the test temperature be raised to 82 ± 1 °C when testing products more viscous than 90 cSt at 40 °C. Other test temperatures may also be used.

This method may be unsuitable for high viscosity oils where it is apparent that there is insufficient mixing of oil and water during stirring.

When testing synthetic fluids whose densities are greater than that of water, the procedure is unchanged, but it should be noted that the water will probably float on the emulsion or liquid.

2 Principle

A 40 ml sample and 40 ml of distilled water are stirred for 5 min at the test temperature in a graduated cylinder. The time required for the separation of the emulsion thus formed is recorded. If complete separation does not occur after standing for 1 h, the volumes of oil (or fluid), water, and emulsion remaining at the time are reported.

3 Materials

3.1 Water, reagent grade, demineralized by distillation and/or ion exchange. For referee testing use distilled water having a conductivity less than 10^{-4} S/m at 25 °C.

3.2 Cleaning solvents, light hydrocarbon, such as precipitation naptha or pentane for petroleum oils. Use other appropriate solvents for dissolving synthetic fluids.

3.3 Acetone.

3.4 Chromosulphuric acid.

4 Apparatus

4.1 Cylinder, graduated measuring cylinder, capacity 100 ml, conforming to ISO 4788, made of glass, preferably heat resistant. The inside diameter shall be no less than 27 mm and no more than 30 mm throughout its length, measured from the top to a point 6 mm from the bottom of the cylinder.

4.2 Heating bath, sufficiently large and deep to permit the immersion of at least two test cylinders in the bath liquid up to their 85 ml graduations. The bath shall be capable of being maintained within ± 1 °C of the test temperature, (see the note in clause 1), and shall be fitted with clamps which hold the cylinder in a position so that the longitudinal axis of the paddle corresponds to the vertical centre line of the cylinder during the stirring operation. The clamps shall hold the cylinder securely while its contents are being stirred.

NOTE — A glass housing for the bath may be preferable as it will allow volume readings of evolving layers without removal of the cylinder from the bath.

4.3 Stirring paddle, made of chromium-plated or stainless steel and conforming to the following dimensions:

Length, mm	120 ± 1,5
Width, mm	19 ± 0,5
Thickness, mm	1,5 to 1,6

It is mounted on a vertical shaft of similar metal, approximately 6 mm in diameter, connected to a drive mechanism which rotates the paddle on its longitudinal axis at 1 500 ± 15 r/min. The apparatus is of such design that, when the cylinder is clamped in position and the paddle assembly is lowered into the cylinder, a positive stop engages and holds the assembly when the lower edge of the paddle is 6 mm from the bottom of the cylinder. During the operation of the stirrer, the centre of the bottom edge of the paddle shall not deviate more than 1 mm from the axis of rotation. When not in operation, the paddle assembly can be lifted vertically to clear the top of the graduated cylinder.

* 1 cSt = 1 mm^2/s = 10^{-6} m^2/s.

5 Sampling

Take a representative sample of the product to be tested according to ISO 3170 or ISO 3171 (or other relevant method).

6 Preparation of apparatus

6.1 Clean the graduated cylinder (4.1) by removing any film of oil (or fluid) with cleaning solvent (3.2) followed by a wash first with acetone and then with tap water. Completely immerse the cylinder in a cleaning solution of hot chromosulphuric acid. Rinse thoroughly with tap water and then with reagent water.

6.2 Clean the stirring paddle (4.3) and shaft with absorbent cotton or tissue wet with cleaning solvent (3.2) and air dry. Care shall be taken not to bend or misalign the paddle assembly during the cleaning operation.

7 Procedure

7.1 Heat the bath to the test temperature and maintain it at that temperature ± 1 °C throughout the test. Add distilled water (3.1) (see note 1) at the test temperature to the graduated cylinder (4.1) up to the 40 ml mark and then add to the same cylinder a representative sample of the oil (or fluid) under test, preheated to the test temperature, until the top level of the oil reaches the 80 ml mark on the cylinder. Place the cylinder in the bath (4.2), immerse it up to its 85 ml graduation, and allow the cylinder and contents to reach bath temperature (see note 2). Normally, 10 min is sufficient.

NOTES

1 A 1 % sodium chloride (NaCl) solution or synthetic sea water (as described in ISO 7120[1]) may be used in place of distilled water when testing certain oils or fuels used in marine applications.

2 The water level in the heating bath should be maintained at or above the 85 ml mark on the cylinder.

7.2 Clamp the cylinder (4.1) in place directly under the stirring paddle (4.3). Lower the paddle into the cylinder until the stop engages at the required depth. Start the stirrer (4.3) and a stop watch simultaneously and adjust the stirrer, as required, to a rotational frequency of 1 500 ± 15 r/min^{-1}. At the end of 5 min, stop the stirrer and raise the stirring assembly until it is just clear of the graduated cylinder.

Wipe the paddle with a rubber-covered glass rod, allowing the liquid thus removed to drop into the cylinder. Remove the cylinder from the retaining clamps and transfer it carefully to another section of the bath. At 5 min intervals, lift the cylinder out of the bath, inspect, and record the volumes of the oil (or fluid), water, and emulsion layers.

NOTE — If the heating bath housing is transparent and the volumes can be read, it is not necessary to lift the cylinder out of the bath. The rubber-covered glass rod should be made of materials resistant to the oil or fluid.

7.3 Record the time (at 5 min intervals) required until the emulsion is reduced to 3 ml or less. If the emulsion is more than 3 ml 1 h after the end of the stirring period, discontinue the test and record the amounts, in millilitres, of oil, water, and emulsion remaining.

8 Expression of results

8.1 Method of reporting

8.1.1 Report the test results in the manner shown in the following examples, showing oil, water, emulsion and time (in parentheses) in that order.

40-40-0 (20)	Complete separation occured in 20 min. More than 3 ml of emulsion had remained at 15 min.
40-37-3 (20)	Complete separation had not occured, but the emulsion reduced to 3 ml so the test was ended.
39-35-6 (60)	More than 3 ml of emulsion remained after 60 min — 39 ml of oil, 35 ml of water, and 6 ml of emulsion.

8.1.2 The appearance of each layer may be described in the following terms:

8.1.2.1 Oil (or fluid) layer

a) clear;

b) hazy;

c) cloudy (or milky).

8.1.2.2. Water layer

a) clear;

b) lacy or bubbles present, or both;

c) hazy;

d) cloudy (or milky).

8.1.2.3 Emulsion

a) loose and lacy;

b) cloudy (or milky);

c) creamy (like mayonnaise).

NOTES

1 A hazy layer is defined as being translucent and a cloudy layer opaque.

1) At present at the stage of draft.

2 The principal difference between cloudy and creamy emulsions is that the former is quite fluid and probably unstable while the latter has a thick consistency and is probably stable. A cloudy emulsion will readily flow from an inclined graduate while a creamy emulsion will not.

8.1.3 The appearance of the oil/emulsion and water/emulsion interfaces may be described in the following terms:

a) well-defined, sharp;

b) ill-defined, bubbles;

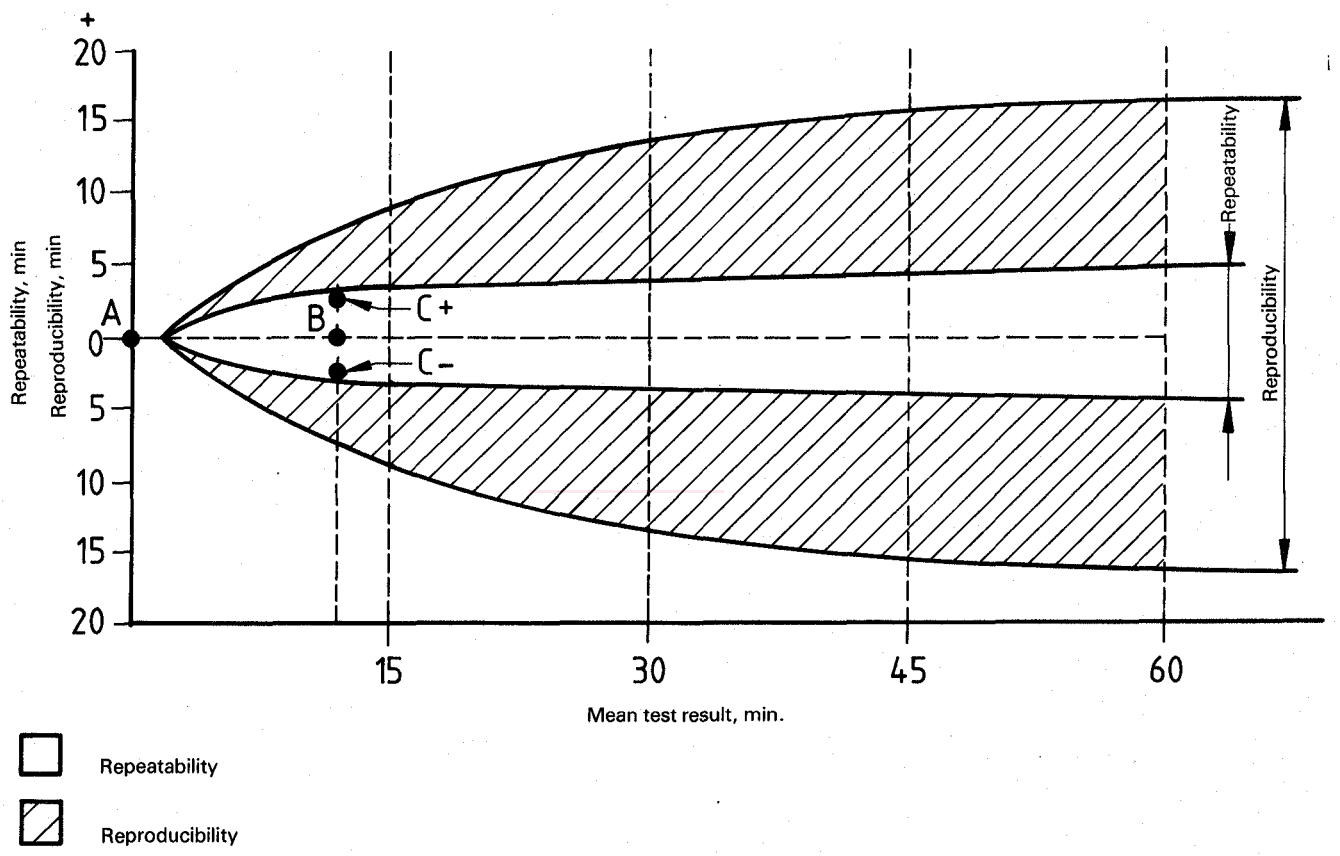
c) ill-defined, lace.

8.2 Precision

The precision of the method, as obtained by statistical examination of interlaboratory test results on steam-turbine oils having viscosities of 32 to 95 cSt at 40 °C, is as follows:

8.2.1 Repeatability

The difference between successive 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 values shown in the figure only in one case in twenty.



Use of chart

Calculate the mean test result in minutes. Enter chart at the zero point, A, on the ordinate and move to the right on the abscissa to point B. Compute and locate the deviation points C+ and C−, from the mean test result. If the deviation points fall within the repeatability area, then the results are within the precision of the test.

Example: An oil has emulsion values of 40-40-0 (10 min) and 40-40-0 (15 min). The mean test result is 12,5 min (B) and the deviation from the mean is +2,5 (C+) and −2,5 (C−).

These points fall within the repeatability.

Use this graph similarly for the reproducibility of means of different laboratories.

Figure — Chart for determining test precision