INTERNATIONAL STANDARD

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Petroleum products — Determination of water separability of petroleum oils and synthetic fluids

Huiles de pétrole et fluides synthétiques — Détermination de l'aptitude des huiles de pétrole et des fluides synthétiques à se séparer de l'eau

Reference number
ISO 6614:1994(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 6614 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

This second edition cancels and replaces the first edition (ISO 6614:1983), which has been technically revised.

Annex A forms an integral part of this International Standard.

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Petroleum products — Determination of water separability of petroleum oils and synthetic fluids

WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a method for measuring the ability of petroleum oils or synthetic fluids to separate from water at a specified temperature.

NOTE 1 The normal test temperature is 54 °C ± 1 °C, but this may be increased to 82 °C ± 1 °C for products with a viscosity above 90 mm^2/s at 40 °C. Other test temperatures may also be specified.

This test method was developed specifically for steam-turbine oils in the viscosity range 32 mm^2/s to 95 mm^2/s at 40 °C, but it may be used to test the water separability of oils of different types and viscosity ranges, and also to test synthetic fluids. It may be unsuitable for high viscosity products where it is apparent that insufficient mixing of oil and water occurs.

NOTE 2 The identical procedure is used for synthetic fluids with a density greater than 1000 kg/m^3 at 15 °C, but it should be noted that the water will tend to float on the emulsion or liquid.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3170:1988, *Petroleum liquids — Manual sampling.*

ISO 3171:1988, *Petroleum liquids — Automatic pipeline sampling.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 4788:1980, *Laboratory glassware — Graduated measuring cylinders.*

ISO 7120:1987, *Petroleum products and lubricants — Petroleum oils and other fluids — Determination of rust-preventing characteristics in the presence of water.*

3 Definition

For the purposes of this International Standard, the following definition applies.

3.1 water separability: Ability of a petroleum oil or synthetic fluid to separate from water at a specified temperature.

It is expressed as a numerical code, determined in accordance with this International Standard, representing the respective volumes of oil, water and emulsion, and time (in parentheses), and a standardized description of the appearance of each layer (see annex A).

4 Principle

A 40 ml sample of petroleum oil or synthetic fluid 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 that time are reported.

5 Reagents

5.1 Water, reagent grade, conforming to grade 3 of ISO 3696.

For referee testing use distilled water having a conductivity less than 10^{-4} S/m at 25 °C.

5.2 Cleaning solvent, completely miscible with the material tested.

For synthetic fluids, use a volatile solvent appropriate to the material under test.

NOTE 3 For petroleum oils, a petroleum spirit or pentane is suitable.

5.3 Acetone, of minimum 99 % purity.

5.4 Chromosulfuric acid, or equivalent non-chromium-containing acid cleaning solution.[1)]

WARNING — Chromosulfuric acid is a health hazard. It is toxic, a recognized carcinogen as it contains Cr(VI) compounds, highly corrosive and potentially hazardous in contact with organic materials. When using chromosulfuric acid cleaning solution, eye protection and protective clothing are essential. Never pipette the cleaning solution by mouth. After use, do not pour cleaning solutions down the drain, but neutralize them with great care owing to the concentrated sulfuric acid present and dispose of them in accordance with standard procedures for toxic laboratory waste (chromium is highly dangerous to the environment).

Non-chromium-containing, strongly oxidizing acid cleaning solutions are also highly corrosive and potentially hazardous in contact with organic materials, but do not contain chromium which has special disposal problems.

5.5 Synthetic sea water, either a 1 % (*m/m*) solution of sodium chloride (NaCl) in water (5.1) or synthetic sea water as described in ISO 7120.

6 Apparatus

Ordinary laboratory apparatus and

6.1 Graduated 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.

6.2 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 note 1), and shall be fitted with clamps which hold the cylinders in a position so that the longitudinal axis of the stirring blade (6.3) corresponds to the vertical centreline of the cylinder during the stirring operation. The clamps shall hold the cylinder securely while its contents are being stirred.

NOTE 4 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.

6.3 Stirring blade, 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

The blade shall be mounted on a vertical shaft of similar metal, approximately 6 mm in diameter, connected to a drive mechanism which rotates the blade on its longitudinal axis at 1 500 ± 15 r/min. The apparatus shall be designed such that, when the cylinder is clamped in position and the assembly is lowered into the cylinder, a positive stop engages and holds the assembly when the lower edge of the blade is 6 mm from the bottom of the cylinder. During the

1) Hot NOCHROMIX® solution or a 24-h soak in MICRO® solution gave results statistically equivalent to hot chromosulfuric acid.

NOCHROMIX and MICRO are examples of suitable non-chromium-containing products available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products. Equivalent products may be used if they can be shown to lead to the same results.

operation of the stirrer, the centre of the bottom edge of the blade shall not deviate more than 1 mm from the axis of rotation.

When not in operation, the blade assembly can be lifted vertically to clear the top of the graduated cylinder.

6.4 Glass rod, covered with a material such as rubber, resistant to the oil or fluid under test.

7 Sampling

The samples shall be obtained by the procedure described in ISO 3170, ISO 3171 or an equivalent national standard. The test portions shall be taken after thorough mixing of the sample, using mechanical aid if necessary.

8 Preparation of apparatus

8.1 Clean the graduated cylinder (6.1) by removing any film of oil (or fluid) with cleaning solvent (5.2) followed by a wash first with acetone (5.3) and then with tap water. Completely immerse the cylinder in a cleaning solution of chromosulfuric acid (5.4). Heat if necessary, but not above 50 °C. Rinse thoroughly with tap water and then with water (5.1).

8.2 Clean the stirring blade (6.3) and shaft with absorbent cotton or tissue wet with cleaning solvent (5.2) and air dry. Take care not to bend or misalign the blade assembly during the cleaning operation.

9 Procedure

9.1 Heat water (5.1), or synthetic sea water (5.5) if the product is for marine applications, to the test temperature and add it to the graduated cylinder (6.1) up to the 40 ml mark. Heat the test portion of the oil or fluid to the same temperature, and add this to the cylinder until the top level reaches the 80 ml mark. Place the cylinder in the heating bath (6.2), immerse it at least up to its 85 ml graduation mark, and allow it to reach bath temperature.

NOTE 5 Normally 20 min is sufficient for the test portion to reach test temperature.

9.2 Clamp the cylinder (6.1) in place directly under the stirring blade (6.3). Lower the blade into the cylinder until the stop engages at the required depth. Start the stirrer (6.3) and a stop watch simultaneously and adjust the stirrer, as required, to a rotational frequency of 1 500 r/min ± 15 r/min. 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 blade with a glass rod (6.4), 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.

9.3 At 5 min intervals, record the volumes of the oil (or fluid), water and emulsion layers, either by inspection through the heating bath housing if it is transparent, or by lifting the cylinder out of the bath to read the graduations.

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

9.5 The appearance of each layer and each interface shall be described in accordance with annex A.

10 Expression of results

Report the test results numerically (see 9.3 to 9.5) in the manner shown in the following examples, giving the volume of oil, water, emulsion and time (in parentheses) in that order. The maximum volume that shall be reported as the oil layer is 43 ml. Add a descriptive code in accordance with annex A, for example b) a) b) b).

Water separability	Interpretation of numerical code
40-40-0 (20)	Complete separation occurred in 20 min. More than 3 ml of emulsion had remained at 15 min.
39-38-3 (20)	Complete separation had not occurred, 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, i.e. 39 ml of oil, 35 ml of water and 6 ml of emulsion.
43-37-0 (30)	The emulsion layer reduced to 3 ml or less after 30 min. The emulsion layer at 25 min exceeded 3 ml, for example, 0-36-44 or 43-33-4.

11 Precision

11.1 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 figure 1 only in one case in 20.

11.2 Reproducibility

The difference between two single and independent 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 values shown in figure 1 only in one case in 20.

NOTE 6 The precision of the method was obtained by statistical examination of interlaboratory test results on steam-turbine oils having viscosities of 32 mm^2/s to 95 mm^2/s at 40 °C.

12 Test report

The test report shall contain at least the following information:

a) sufficient details for complete identification of the product tested;

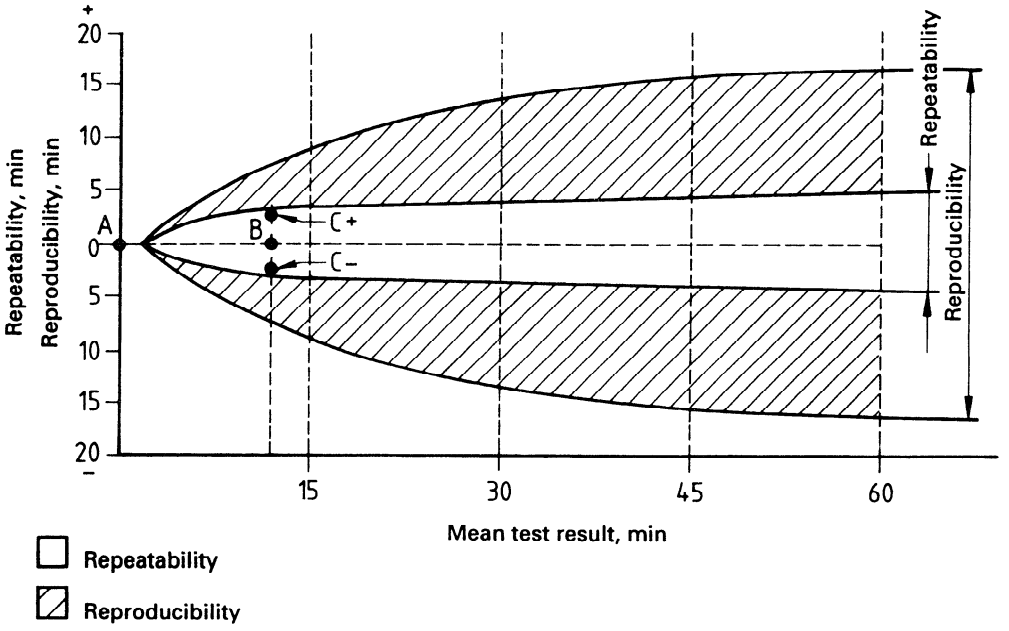
b) a reference to this International Standard;

c) the test temperature, and the aqueous solution if other than water as specified in (5.1);

d) the results of the test, expressed in accordance with clause 10;

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

f) the date of the test.



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 1 — Chart for determining test precision

Annex A
(normative)

Standardized descriptions of the emulsion, oil (or fluid) and water layers and their interfaces

A.1 Description of layers

Describe the appearance of each layer in the following terms:

A.1.1 Oil (or fluid) layer

a) clear;

b) hazy;

c) cloudy (or milky).

A.1.2 Water layer

a) clear;

b) lacy or bubbles present, or both;

c) hazy;

d) cloudy (or milky).

A.1.3 Emulsion

a) loose and lacy;

b) cloudy (or milky);

c) creamy.

A.2 Description of interfaces

Describe the appearance of the oil/emulsion and water/emulsion interfaces in the following terms:

a) well-defined, sharp;

b) ill-defined, bubbles;

c) ill-defined, lace.

A.3 Explanation of descriptive terms

A hazy layer is one that is translucent and a cloudy layer is opaque.

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 quite stable. A cloudy emulsion will readily flow from an inclined cylinder, while a creamy emulsion will not.

A lacy layer or emulsion is one where a small amount of oil is loosely held in a phase which is substantially water, possibly giving a braided or gauze-like effect.

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Descriptors: petroleum products, fluids, mineral oils, synthetical oils, emulsions, tests, determination, dewatering.

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