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Petroleum and related products — Preparation of a test portion of high-boiling liquids for the determination of water content — Nitrogen purge method

Pétrole et produits connexes — Préparation d'une prise d'essai de liquides à haut point d'ébullition en vue du dosage de l'eau — Méthode par purge à l'azote

Reference number
ISO 20764:2003(E)

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Foreword

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ISO 20764 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

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Petroleum and related products — Preparation of a test portion of high-boiling liquids for the determination of water content — Nitrogen purge method

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

1 Scope

This International Standard specifies two procedures for the preparation of test portions from petroleum and related products boiling above 200 °C, which can then be used for the determination of total water content within the range of 3 mg/kg to 1 000 mg/kg. The method is applicable to unused and used lubricating oils, non-aqueous hydraulic fluids and high-boiling solvents. The test portions generated by these procedures are suitable for standard Karl Fischer water-content determinations, such as those described in ISO 6296 (see [1] in the Bibliography) and ISO 12937 (see [2] in the Bibliography).

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.

ISO 383:1976, *Laboratory glassware — Interchangeable conical ground joints*

ISO 3170:—1), *Petroleum liquids — Manual sampling*

3 Principle

3.1 Atmospheric pressure

The water is driven out of a weighed portion of the material under test by a nitrogen purge at 120 °C. It is then condensed in methanol (4.2) held in a titration vessel (5.4) at approximately −77 °C. The resulting water/methanol solution is immediately available for the determination of water content by Karl Fischer titration.

3.2 Reduced pressure

For used lubricating oils and high-boiling materials with high-water retaining characteristics, the water is driven out of a weighed portion by a nitrogen purge at 60 °C under an absolute pressure of 0,30 kPa to 0,65 kPa. It is collected and titrated in the same manner as the test portion in 3.1.

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1) To be published. (Revision of ISO 3170:1988)

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4 Reagents and materials

4.1 Nitrogen, of pure grade, containing less than 500 mg/kg of oxygen. The source shall be regulated to a low pressure that gives agitation without vortex at a flow rate of 3 l/h. It shall be dried through a drying agent upstream of the wash-bottle inlet.

NOTE The maximum water-content target of the nitrogen is 1 µg/l.

4.2 Methanol, of commercial grade, dried to a water content of not more than 100 mg/kg. Dried methanol shall be continuously protected by a drying tube containing a suitable drying agent.

4.3 Solid carbon dioxide

4.4 Cooling medium: commercial grade of methanol, ethanol or acetone.

5 Apparatus

5.1 General

The layout of the extraction apparatus is illustrated in Figure 1, with details of the titration vessel and inserts shown in Figures 2, 3 and 4. The major components are described in 5.2 to 5.5.

Ground-glass joints from different sources may have one of two diameter-to-length ratios. For the purposes of this International Standard, either is suitable, and for some applications, the diameter itself can be one of two. However, it is critical that the male and female parts of each joint are from the same series to avoid recession or protuberance.

NOTE In Europe, ground-glass joints normally come from a series known as “k = 6” as described in ISO 383, whereas in North America, the series generally used is “k = 8”. For joints specified in the figures in this International Standard, these result in joints with dimensions of 14,5/23 and 14,5/30, respectively.

5.2 Heating bath, consisting of a liquid bath of sufficient capacity to hold the wash bottle (5.3), and capable of being controlled at temperatures of 60 °C ± 1 °C and 120 °C ± 1 °C.

5.3 Wash bottle, made of a borosilicate glass, of inside diameter 55 mm and insert, with a graduation line at 250 ml, as illustrated in Figure 1.

5.4 Titration vessel, consisting of a borosilicate glass three-necked flask as shown in Figure 2, fitted with inserts as shown in Figures 1, 3 and 4. Figure 4 is illustrative only, as the design will depend upon the Karl Fischer detection system used for the final analysis.

5.5 Cooling vessel, consisting of a Dewar flask or similar, of sufficient capacity to hold the titration vessel (5.4) surrounded by cooling medium (4.4), and with access to allow the addition of solid carbon dioxide (4.3). A temperature-controlled bath containing cooling medium at – 77 °C ± 3 °C is suitable.

5.6 Vacuum source, a pump or other source of vacuum, capable of maintaining an absolute pressure of 0,3 kPa to 0,65 kPa for a minimum period of 90 min.

5.7 Analytical balance, of minimum capacity 500 g, capable of weighing to the nearest 0,01 g.

5.8 Flowmeter, capable of reading the nitrogen flow of 3 l/h ± 0,3 l/h.

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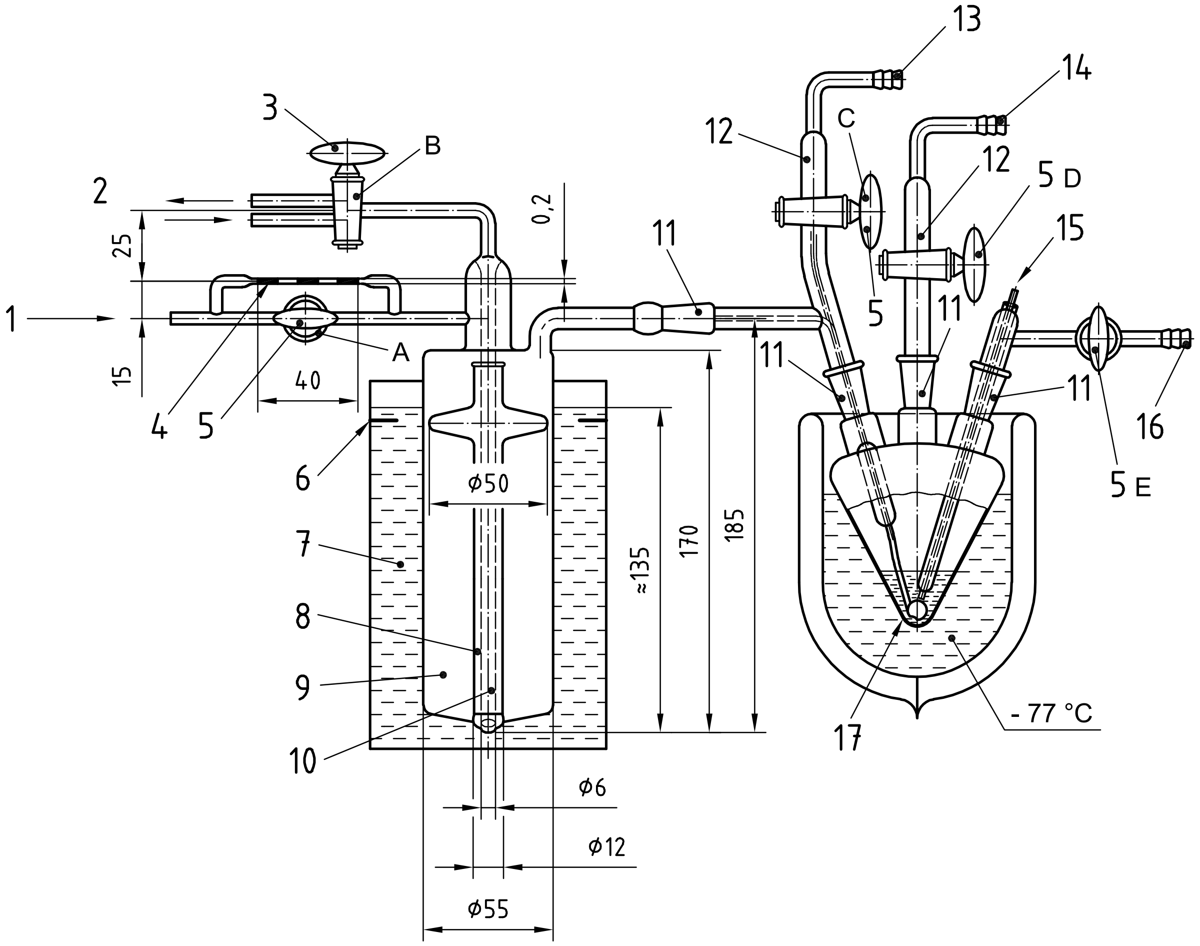
Dimensions in millimetres



Key

1	nitrogen in
2	test portion
3	two-way cock B
4	vacuum capillary
5	one-way cocks A, C, D and E
6	graduation line at ≈ 250 ml
7	heating bath
8	riser tube
9	graduated wash bottle
10	filling tube
11	ground-glass sockets/cones
12	0,1 to 0,2 bore tube
13	methanol connection
14	Karl Fischer solution connection
15	Karl Fischer detector connection
16	vacuum pump connection
17	titration vessel

Figure 1 — Layout of apparatus

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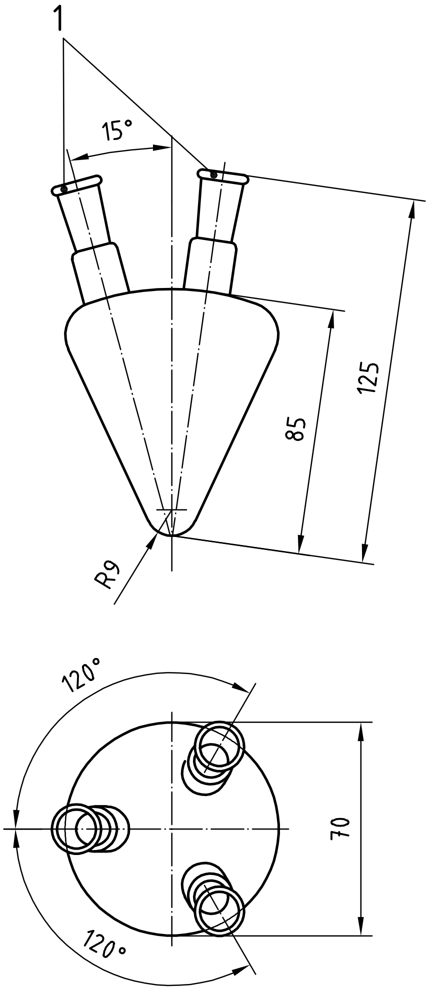
Dimensions in millimetres



Key

1 ground-glass sockets

Figure 2 — Titration vessel

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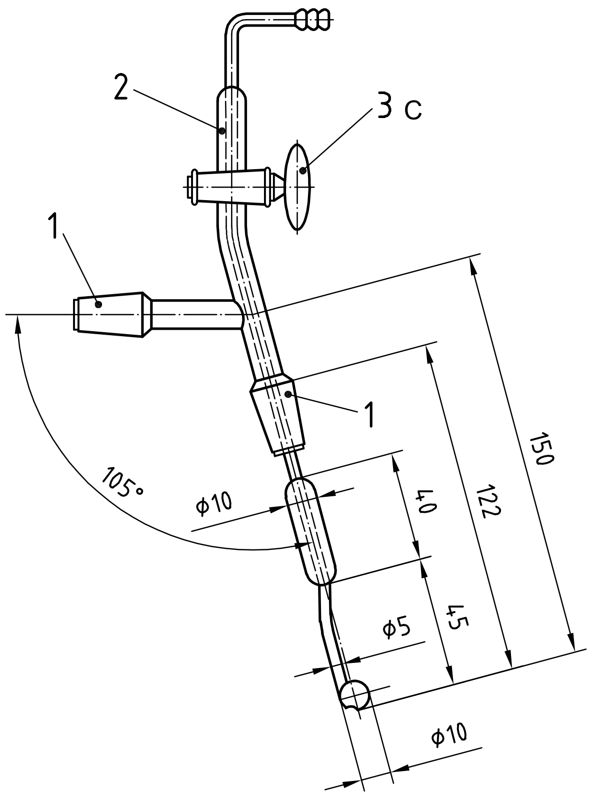
Dimensions in millimetres



Key

1 ground-glass sockets/cones

2 0,1 to 0,2 bore tube

3 one-way cock C

Figure 3 — Methanol and water vapour insert

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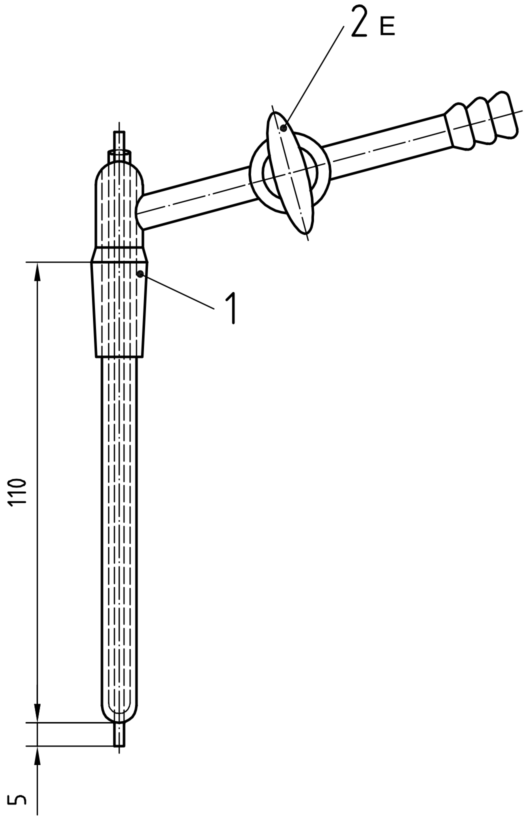
Dimensions in millimetres



Key

1 ground-glass sockets/cones

2 one-way cock E

Figure 4 — Vacuum and detector insert

6 Samples and sampling

6.1 Unless otherwise specified, samples shall be obtained by the procedures described in ISO 3170.

6.2 The laboratory samples shall be drawn into absolutely dry dark-glass containers fitted with ground-glass stoppers. The stoppers shall be made airtight by the application of a suitable water-resistant sealing medium.

6.3 Samples shall be withdrawn directly into the sample containers with the container immersed in the bulk. The stoppers shall not be removed until immersion is complete. No subsequent compositing of samples is allowed.

If alternative sample containers are all that are available, maximum precautions shall be taken to prevent ingress of moist air during and following sampling, without the risk of removing water from the sample.

6.4 In general, the start of test-portion preparation shall immediately follow sampling, and the maximum time difference shall be 1 h. If a longer period has elapsed, note and report the time difference.

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7 Procedure

7.1 General

7.1.1 Assemble the apparatus and add 3 ml to 5 ml of dried methanol (4.2) to the titration vessel. Adjust the nitrogen flow to 3 l/h ± 0,3 l/h and obtain a reading of the blank mass of water for the reagents, using the technique described in 7.2.4, over a period of 15 min ± 2 min, with the temperature of the dried methanol at 0 °C to 20 °C.

7.1.2 Weigh the wash bottle (5.3) without the top and riser tube, to the nearest 0,01 g. Add a quantity of the laboratory sample by nitrogen displacement of 180 ml to 220 ml, and reweigh to the nearest 0,01 g.

NOTE The volume chosen is to ensure that, at no time during the procedure, the liquid level rises above the graduated mark on the wash bottle. For samples expected to have water contents in excess of 50 mg/kg, the volume may be reduced to 100 ml.

7.1.3 Immediately assemble the apparatus and ensure that all joints are sealed.

7.1.4 Adjust the temperature of the cooling medium (4.4) to – 77 °C ± 3 °C (approximately the saturated liquid temperature of solid carbon dioxide) and start the nitrogen flow (see 7.3.2).

7.1.5 Switch on the heating bath (5.2) and adjust to the temperature specified in 7.2 or 7.3.

7.2 Atmospheric pressure

7.2.1 Raise the temperature in the heating bath to 120 °C ± 1 °C.

7.2.2 Maintain this temperature, with the nitrogen flow at 3,0 l/h ± 0,3 l/h for 60 min ± 5 min.

7.2.3 At the end of this period, remove the cooling vessel (5.5) from the titration vessel (5.4) and allow the methanol/water mixture to rise to a temperature within the range of 0 °C to 20 °C.

7.2.4 Determine the mass of water in the methanol/water mixture by potentiometric or coulometric titration, with the nitrogen still flowing to stir the mixture.

7.3 Reduced pressure

7.3.1 Raise the temperature in the heating bath to 60 °C ± 1 °C.

7.3.2 Adjust the vacuum to 0,30 kPa to 0,65 kPa absolute pressure, at the same time reducing the nitrogen flow rate. As soon as vacuum is applied, ensure that Cock C and Cock D in Figure 1 are closed.

The nitrogen flow rate should be very low, but positive, as the vacuum is established to minimize the risk of the material in the wash bottle foaming as it is degassed. A flow rate of 2 to 3 gas bubbles (50 µl) per second is recommended, and is achieved either by the use of the vacuum-capillary bypass shown in Figure 1, or by a suitable control valve.

7.3.3 Maintain the temperature of the heating bath at 60 °C ± 1 °C, with the liquid in the cooling medium at – 77 °C ± 3 °C, and the vacuum at 0,30 kPa to 0,65 kPa for 90 min ± 5 min.

7.3.4 At the end of this period, adjust the pressure back to atmospheric, increase the nitrogen flow, and follow the procedures described in 7.2.3 and 7.2.4.

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8 Calculation

Calculate the water content of the sample, w, in milligrams per kilogram, using the following equation:

$$w = \frac{m_1 - (m_0 x)}{m_3 - m_2} \times 1\,000$$

where

m_0 is the mass of water in the blank, expressed in milligrams;

m_1 is the mass of water in the test portion, expressed in milligrams;

m_2 is the mass of the empty wash bottle, expressed in grams;

m_3 is the mass of the wash bottle plus the laboratory subsample (7.1.2), expressed in grams;

x is the time multiplier (4 for atmospheric pressure, 6 for reduced pressure).

9 Expression of results

Report the method of preparation of the test portion, after reference to this International Standard, as “(A)” or “(V)” for atmospheric or reduced-pressure preparation, respectively.

Report an excess of time between sampling and the commencement of test-portion preparation as “delayed – X h”, as described in 6.4.

NOTE For reporting purposes, where this or other special test-portion preparation procedures have been used, two test method references will be required, the second one relating to the water-content determination technique used. Users of this International Standard are encouraged to ensure that it is used appropriately in conjunction with other references.

10 Precision

This procedure does not have a precision of a conventional type, but data show that the precision and accuracy of the test method chosen for the determination of water content was not affected by the application of the techniques described in this International Standard.

11 Test report

The test report shall contain at least the following information:

a) a reference to this International Standard, in addition to the standard used for determining the water content;

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

c) the procedure used (see Clause 9);

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

e) the date of the test.

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Bibliography

[1] ISO 6296:2000, *Petroleum products — Determination of water — Potentiometric Karl Fischer titration method*

[2] ISO 12937:2000, *Petroleum products — Determination of water — Coulometric Karl Fischer titration method*

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