
**Petroleum and related products —
Determination of emulsion stability of
fire-resistant fluids —**

**Part 2:
Fluids in category HFB**

*Pétrole et produits connexes — Détermination de la stabilité d'émulsion
de fluides difficilement inflammables —*

Partie 2: Fluides de catégorie HFB



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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 20783-2 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

ISO 20783 consists of the following parts, under the general title *Petroleum and related products — Determination of emulsion stability of fire-resistant fluids*:

- *Part 1: Fluids in category HFAE*
- *Part 2: Fluids in category HFB*

Petroleum and related products — Determination of emulsion stability of fire-resistant fluids —

Part 2: Fluids in category HFB

WARNING — The use of this part of ISO 20783 may involve hazardous materials, operations and equipment. This part of ISO 20783 does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this part of ISO 20783 to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This part of ISO 20783 specifies three test methods to assess the stability of emulsions within the category HFB, as defined in ISO 6743-4^[5].

Method A describes a method for the determination of stability during storage at ambient temperature [(20 ± 2) °C] and is applicable to HFB and HFB.LT¹⁾ fluids.

Method B describes a method for the determination of stability during storage at medium temperature [(70 ± 2) °C] and is again applicable to HFB and HFB.LT¹⁾ fluids.

Method C describes a method for the determination of stability during storage at low temperature [(-10 ± 2) °C] and is applicable only to HFB.LT¹⁾ fluids.

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 648:1977, *Laboratory glassware — One-mark pipettes*

ISO 3170:—²⁾, *Petroleum liquids — Manual sampling*

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

ISO 3733:1999, *Petroleum products and bituminous materials — Determination of water — Distillation method*

1) LT is the designation for low temperature applications.

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

3 Method A — Determination of stability of HFB fluids at ambient temperature

3.1 Principle

A graduated measuring cylinder is filled with 450 ml of emulsion above a 50 ml layer of water and is stored for 1 000 h at ambient temperature. At the end of this time, 50-ml sub-samples are pipetted from two defined levels in the upper and lower regions of the emulsion column. The water contents of these test portions are compared with the measured initial water content. Measurements of the surface oil layer volume and the volume of accumulated free water are also made.

3.2 Reagents

3.2.1 Water-free petroleum spirit, (see ISO 1998-1:1998^[2], 1.20.146) with a boiling range of 90 °C to 160 °C.

3.2.2 Water, meeting the requirements of Grade 3 of ISO 3696:1987.

3.2.3 Reagents for distillation procedure, as specified in ISO 3733, for the determination of water.

3.3 Apparatus

3.3.1 Graduated measuring cylinders, of 500 ml capacity, with a scale length from the 50 ml to 500 ml graduations of (250 ± 25) mm and an overall height of approximately 390 mm.

Cylinders conforming to ISO 4788^[4] are suitable.

3.3.2 Pipettes, of 50 ml capacity, conforming to ISO 648.

3.3.3 Polyethylene film, approximately 0,05 mm thick.

3.3.4 Round-bottomed flasks, of 500 ml capacity, used as distillation vessels for the determination of water in accordance with ISO 3733.

3.3.5 Sampling jig, consisting of a retort stand, clamps and set square as shown in Figure 1.

3.3.6 Stop clock.

3.3.7 Wash bottle.

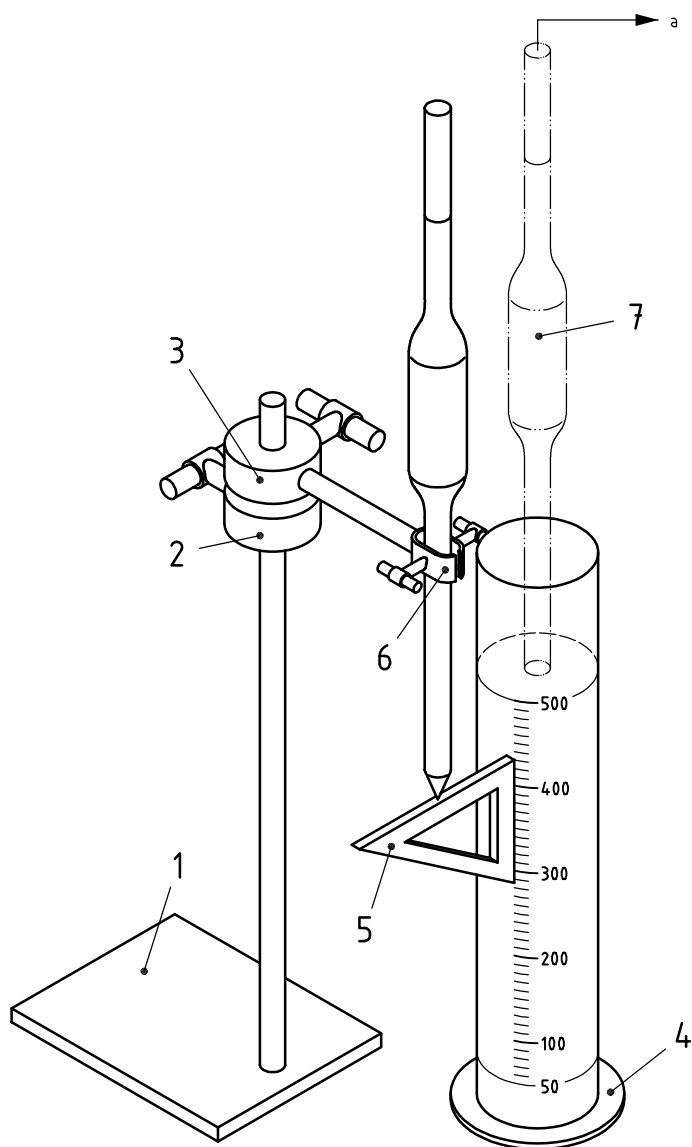
3.3.8 Apparatus for distillation procedure, as specified in ISO 3733, for the determination of water.

3.4 Samples

3.4.1 Unless otherwise specified, samples of fluid shall be obtained using the procedures specified in ISO 3170.

3.4.2 A fluid sample of no less than 1,5 l in volume shall be supplied for the test. Care shall be taken to ensure that the sample is representative of the bulk.

Dimensions in millimetres



Key

- 1 retort stand
- 2 Stop C
- 3 Clamp B
- 4 500 ml measuring cylinder
- 5 set square for external alignment of pipette
- 6 Clamp A
- 7 50 ml pipette in sampling position

a To vacuum pump

Figure 1 — Sampling apparatus

3.5 Procedure

3.5.1 General

Mix the sample thoroughly by agitation in a vessel having a capacity that is substantially greater than the sample size.

3.5.2 Initial water content of the emulsion

Take two sub-samples of 50 ml each from the sample (3.4.2) using a pipette (3.3.2). Drain each 50 ml test portion from the pipette into a clean, dry 500 ml round-bottomed flask (3.3.4). Rinse the residual fluid from the pipette into the flask using several charges of petroleum spirit (3.2.1) from a wash bottle (3.3.7). A total volume of approximately 100 ml of petroleum spirit shall be used for this operation. Drain the pipette thoroughly to ensure that all of the sub-sample is transferred to the round-bottomed flask. Use the sub-sample as the test portion specified in ISO 3733 and subject each of them to the distillation procedure given in ISO 3733 to obtain two determinations of the initial percentage water content (volume fraction), φ_1 , of the emulsion to the nearest 0,2 %.

Some emulsions contain water-soluble liquid, which distils over during the determination of the water content (volume fraction, %) by the distillation method. When such water-soluble liquid is present, the result of the water content determination by the distillation method is termed the apparent water content. Determine the apparent water content where appropriate.

3.5.3 Preparation of test portions

Clean and dry two 500 ml measuring cylinders (3.3.1). Fill each cylinder to the level of the 50 ml graduation with water (3.2.2) using a pipette (3.3.2), taking care not to splash the internal surface of the cylinder. Take portions of approximately 50 ml from the sample (3.4.2) and introduce them carefully on top of the water layer in each cylinder using a pipette (3.3.2) to minimize the mixing of the emulsion and the water layer. Fill the cylinders to the 500 ml graduations by pouring emulsion down a glass rod. Seal the mouth of each test cylinder by a piece of polyethylene film (3.3.3) bound in place.

3.5.4 Storage

Store the cylinders undisturbed for 1 000 h at a temperature of (20 ± 2) °C, remote from sources of heat. Protect them from direct sunlight and draughts.

3.5.5 Oil layer and free water measurement

After 1 000 h, take readings from each cylinder of the surface oil layer volume and the change in water layer volume, i.e. the volume of accumulated free water.

3.5.6 Water content at defined levels

3.5.6.1 Remove the polyethylene seal from the mouth of one of the test cylinders. Mount a 50 ml pipette (3.3.2) vertically in Clamp A of the sampling jig as shown in Figure 1. Seal the mouth of the pipette. Externally align the tip of the pipette with the 425 ml graduation of the measuring cylinder using a set square, and position Stop C directly beneath Clamp B. Release Clamp B, raise the pipette and align its stem with the vertical axis of the measuring cylinder. Lower the pipette carefully into the fluid until Clamp B comes to rest on Stop C, then retighten Clamp B. Remove the seal from the mouth of the pipette and fill the pipette at a steady rate in a time of no less than 60 s (3.3.6) by applying suction, until the fluid level is approximately 50 mm above the pipette graduation mark. It is advisable that the suction used for this purpose should be established in advance by trial sampling runs from a uniformly dispersed sample of the fluid under test, at a temperature of (20 ± 2) °C.

3.5.6.2 Remove the pipette from the sampling jig and wipe all excess fluid from the exterior of the pipette stem. Adjust the fluid volume in the pipette to 50 ml by dispensing excess fluid. Drain the 50 ml sub-sample from the pipette into a clean, dry 500 ml round-bottomed flask (3.3.4). Rinse the residual fluid from the pipette

into the flask using several charges of petroleum spirit (3.2.1) from a wash bottle (3.3.7). A total volume of approximately 100 ml petroleum spirit shall be used for this operation. Drain the pipette thoroughly to ensure that all of the sub-sample is transferred to the round-bottomed flask.

3.5.6.3 Use the pipetted sub-sample (3.5.6.2) as the test portion specified in ISO 3733 and subject the test portion to the distillation procedure given in ISO 3733 to determine the percentage water content (volume fraction), φ_{425} , of the 425 ml level to the nearest 0,2 % (see 3.5.2).

3.5.6.4 Repeat the procedure given in 3.5.6.2 in order to obtain a 50 ml test portion at the 125 ml level of the same measuring cylinder.

3.5.6.5 Repeat the procedure given in 3.5.6.3 on the test portion obtained in 3.5.6.4 to determine the percentage water content (volume fraction), φ_{125} , of the pipetted test portion from the 125 ml level to the nearest 0,2 % (see 3.5.2).

3.5.6.6 Repeat the procedures given in 3.5.6.1 to 3.5.6.5 in order to obtain the water contents of test portions from the 425 ml and the 125 ml levels of the second measuring cylinder.

3.6 Calculation

3.6.1 Initial water content mean

Calculate the mean, φ_{im} , of the two values of initial water content (volume fraction, %) (3.5.2).

3.6.2 Water content mean for test portions taken at the 425 ml level

For each test portion taken from the 425 ml level, calculate the changes in water content (volume fraction, %), $\varphi_{\Delta 425}$, as follows:

$$\varphi_{\Delta 425} = \varphi_{425} - \varphi_{im}$$

where

φ_{425} is the water content (volume fraction, %), of the test portion taken from the 425 ml level of the measuring cylinder as determined in 3.5.6.3;

φ_{im} is the mean of the two values of the initial water content (volume fraction, %), as determined in 3.5.2.

Calculate the mean, $\varphi_{\Delta 425m}$, of the two determinations of $\varphi_{\Delta 425}$. Report the mean change in water content (volume fraction, %), $\varphi_{\Delta 425m}$, at the 425 ml level after 1 000 h to the nearest 0,2 %.

3.6.3 Water content mean for test portions taken at the 125 ml level

For each test portion taken from the 125 ml level, calculate the changes in water content (volume fraction, %), $\varphi_{\Delta 125}$, as follows:

$$\varphi_{\Delta 125} = \varphi_{125} - \varphi_{im}$$

where

φ_{125} is the water content (volume fraction, %), of the test portion taken from the 125 ml level of the measuring cylinder as determined in 3.5.6.5;

φ_{im} is the mean of the two values of the initial water content (volume fraction, %), as determined in 3.5.2.

Calculate the mean, $\varphi_{\Delta 125m}$, of the two determinations of $\varphi_{\Delta 125}$. Report the mean change in water content (volume fraction, %), $\varphi_{\Delta 125m}$, at the 125 ml level after 1 000 h to the nearest 0,2 %.

3.6.4 Oil layer measurements

Calculate the mean of the two oil layer measurements (3.5.5). Report the mean surface oil layer volume to the nearest millilitre after 1 000 h.

3.6.5 Accumulated free water measurements

Calculate the mean of the two measurements of accumulated free water (3.5.5). Report the mean volume of accumulated free water to the nearest millilitre after 1 000 h.

3.7 Precision

3.7.1 General

The precision of this test method was determined from the statistical analysis of interlaboratory results in accordance with ISO 4259^[3], but for tests carried out under test conditions where the maximum and minimum ambient temperatures during storage were measured and reported. Since this is different from the present test condition where the storage temperature is defined as (20 ± 2) °C, the values given in Tables 1 and 2 should be used for guidance only.

3.7.2 Repeatability (estimate)

Duplicate results by the same operator should be considered suspect if they differ by more than the values given in Table 1, which have been given for guidance only (see 3.7.1).

Table 1 — Repeatability (estimate)

Result	Range	Repeatability ^a
Surface oil layer volume	1 ml to 16 ml	$0,076(X + 4)$ ml
Accumulated free water volume	up to 5 ml	1 ml
Change in percentage water content (volume fraction) at the 425 ml level	-12 % to 0 %	$0,159(-X + 4)$ %
Change in percentage water content (volume fraction) at the 125 ml level	-3 % to +14 %	1,3 %
NOTE The stability of fluids can deteriorate with time and repeatability, in this case, refers to tests carried out concurrently in the same environment.		
^a X is the mean result.		

3.7.3 Reproducibility (estimate)

The results submitted by each of two laboratories should not be considered suspect unless the two results differ by more than the values given in Table 2, which have been given for guidance only (see 3.7.1).

Table 2 — Reproducibility (estimate)

Result	Range	Reproducibility ^a
Surface oil layer volume	1 ml to 16 ml	$0,540(X + 4)$ ml
Accumulated free water volume	up to 5 ml	3 ml
Change in percentage water content (volume fraction) at the 425 ml level	-12 % to 0 %	$0,291(-X + 4)$ %
Change in percentage water content (volume fraction) at the 125 ml level	-3 % to +14 %	$0,571(X + 4)$ %
NOTE The reproducibility for the surface oil layer volume should be regarded as tentative because this precision estimate is based on fewer degrees of freedom than are normally considered acceptable.		
^a X is the mean result.		

4 Method B — Determination of stability of HFB fluids at medium temperature

4.1 Principle

A sample of emulsion is held at (70 ± 2) °C in a 100 ml distillation cylinder (Crow receiver) for 48 h and measurements are made of the volumes of water and oil that separate out.

4.2 Apparatus

4.2.1 Conical distillation cylinder, of 100 ml capacity, having the dimensions specified in Figure 2.

Crow receivers conforming to ISO 918^[1] are suitable.

4.2.2 Polyethylene film, approximately 0,05 mm thick.

4.2.3 Oven, with a perforated shelf and capable of maintaining a temperature of (70 ± 2) °C.

4.3 Samples

4.3.1 Unless otherwise specified, samples of fluid shall be obtained using the procedures specified in ISO 3170.

4.3.2 A fluid sample of no less than 500 ml in volume shall be supplied for the test. Care shall be taken to ensure that the sample is representative of the bulk.

4.4 Procedure

4.4.1 Preparation of the test portions

Mix the sample (4.3.2) thoroughly by agitation in a vessel having a capacity that is substantially greater than the sample size. Fill two clean dry 100 ml distillation cylinders (4.2.1) with 97 ml of the fluid (see the Note below). Seal the mouth of each test cylinder with polyethylene film (4.2.2) bound in place.

NOTE The fluid will expand to approximately 100 ml upon heating to the test temperature.

4.4.2 Storage

Make sure that the oven (4.2.3) is maintained at (70 ± 2) °C. Place the test cylinders without delay centrally in the oven onto the perforated shelf. Continue to maintain the oven temperature at (70 ± 2) °C for 48 h.

4.4.3 Oil layer and free water measurement

At the end of the 48 h storage period, take readings to the nearest 1 ml of the volume of the surface oil layer and the volume of accumulated free water for each test cylinder. Take these readings without disturbing the test cylinders and before any appreciable cooling occurs.

It is unlikely that the upper surface of the fluid will coincide exactly with the 100 ml graduation of the test cylinder. Estimate the surface oil layer as the difference in the scale readings between the upper surface of the fluid and the upper surface of the oil layer.

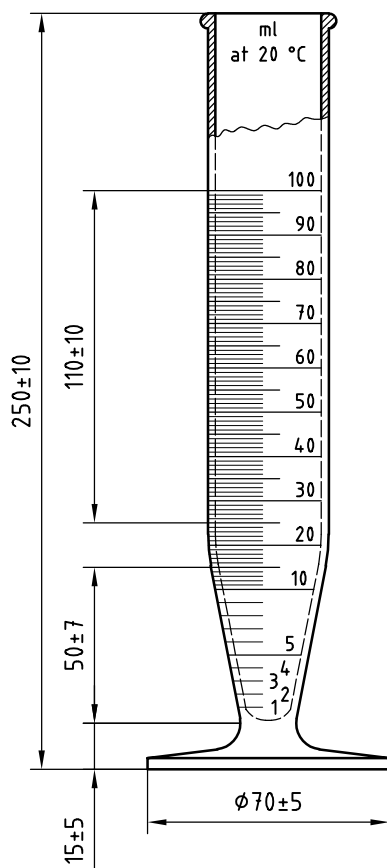


Figure 2 — Distillation cylinder

4.5 Expression of results

4.5.1 Oil layer measurements

Report the surface oil layer volume in millilitres for each test cylinder. Report the mean of the two measurements of surface oil layer volume in millilitres.

4.5.2 Accumulated free water measurements

Report the volume of accumulated free water in millilitres for each test cylinder. Report the mean of the two measurements of accumulated free water in millilitres.

4.6 Precision

The precision of this test method has not been determined. However, 3.7 of Method A gives some indication of the precision.

5 Method C — Determination of stability of HFB.LT fluids at low temperature

5.1 Principle

A sample of emulsion is held at $(-10 \pm 2) ^\circ\text{C}$ in a 100 ml distillation cylinder (Crow receiver) for 336 h. The temperature is then raised to $(+20 \pm 2) ^\circ\text{C}$ for a further 168 h and measurements are made of the volumes of water and oil that separate out. A 10 ml sample is taken from the 5 ml level in the test cylinder and the water content (volume fraction, %) of this sample is determined for comparison with the initial water content (volume fraction, %).

5.2 Reagent

5.2.1 Water-free petroleum spirit, with a boiling range of $90 ^\circ\text{C}$ to $160 ^\circ\text{C}$.

5.2.2 Reagents for distillation procedure, as specified in ISO 3733, for the determination of water.

5.3 Apparatus

5.3.1 Conical distillation cylinder, of 100 ml capacity, having the dimensions specified in Figure 2.

Crow receivers conforming to ISO 918^[1] are suitable.

5.3.2 Pipettes, of 10 ml and 50 ml capacity, conforming to ISO 648.

5.3.3 Polyethylene film, approximately 0,05 mm thick.

5.3.4 Refrigeration chamber, capable of maintaining a temperature of $(-10 \pm 2) ^\circ\text{C}$.

5.3.5 Round-bottomed flasks, of 500 ml capacity, used as distillation vessels for the determination of water in accordance with ISO 3733.

5.3.6 Stop clock.

5.3.7 Wash bottle.

5.3.8 Apparatus for distillation procedure, as specified in ISO 3733, for the determination of water.

5.4 Samples

5.4.1 Unless otherwise specified, samples of fluid shall be obtained using the procedures specified in ISO 3170.

5.4.2 A fluid sample of no less than 1 l in volume shall be supplied for the test. Care shall be taken to ensure that the sample is representative of the bulk.

5.5 Procedure

5.5.1 General

Mix the sample thoroughly by agitation in a vessel having a capacity that is substantially greater than the sample size.

5.5.2 Initial water content of the emulsion

Take two sub-samples of 50 ml each from the sample (5.4.2) using a pipette (5.3.2). Drain each 50 ml sub-sample from the pipette into a clean, dry 500 ml round-bottomed flask (5.3.5). Rinse the residual fluid from the pipette into the flask using several charges of petroleum spirit (5.2.1) from a wash bottle (5.3.7). A total volume

of approximately 100 ml of petroleum spirit shall be used for this operation. Drain the pipette thoroughly to ensure that all of the sub-sample is transferred to the round-bottomed flask. Use the pipetted sub-samples as the test portion specified in ISO 3733 and subject each of them to the procedure given in ISO 3733 to obtain two determinations of the initial percentage water content (volume fraction) φ_1 , of the emulsion to the nearest 0,2 %.

Determine the apparent water content (volume fraction, %) where appropriate.

5.5.3 Preparation of the test portions

Fill six clean, dry 100 ml conical distillation cylinders (5.3.1) with 100 ml of the fluid (5.4.2). Seal the mouth of each test cylinder with polyethylene film (5.3.3) bound in place.

5.5.4 Storage

5.5.4.1 Make sure that the refrigeration chamber (5.3.4) is maintained at (-10 ± 2) °C. Place the test cylinders without delay centrally in the refrigeration chamber. Continue to maintain the refrigeration chamber temperature at (-10 ± 2) °C for 336 h.

5.5.4.2 After 336 h, transfer the test cylinders carefully to an ambient temperature of (20 ± 2) °C. Maintain the cylinders at this temperature for a further 168 h.

5.5.5 Oil layer and free water measurement

At the end of the further 168 h storage period, take readings to the nearest 1 ml of the volume of the surface oil layer and the volume of accumulated free water for each test cylinder. Take these readings without disturbing the test cylinders.

5.5.6 Water content at defined levels

5.5.6.1 Remove the polyethylene film from the mouth of one of the test cylinders. Lower a 10 ml capacity pipette (5.3.2) carefully into the test cylinder until the tip of the pipette is aligned with the vertical axis of the cylinder at the depth of the 5 ml graduation. Maintain the pipette in this position and fill the pipette at a steady rate in a time of no less than 60 s (5.3.6) by applying suction, until the fluid level is approximately 20 mm above the pipette graduation mark. It is advisable that the suction used for this purpose should be established in advance by trial sampling runs from a uniformly dispersed sample of the fluid under test, at a temperature of (20 ± 2) °C.

5.5.6.2 Remove the pipette from the test cylinder and wipe all excess fluid from the outside of the pipette stem. Adjust the volume in the pipette to 10 ml by dispensing excess fluid. Drain the 10 ml test portion from the pipette into a clean, dry 500 ml round-bottomed flask (5.3.5). Rinse the residual fluid from the pipette into the flask using several charges of petroleum spirit (5.2.1) from a wash bottle (5.3.7). Drain the pipette thoroughly to ensure that all of the test portion is transferred to the round-bottomed flask.

5.5.6.3 Use the pipetted test portion (5.5.6.2) as the test portion specified in ISO 3733 and subject the test portion to the distillation procedure given in ISO 3733 to determine the percentage water content by volume, φ_5 , of the 5 ml level to the nearest 0,2 % (see 3.5.2).

5.5.6.4 Repeat the procedures given in 5.5.6.2 and 5.5.6.3 for the other five test cylinders to provide a total of six water content determinations (see 3.5.2).

5.6 Calculation

5.6.1 Initial water content mean

Calculate the mean, φ_{1m} , of the two determinations of initial water content (volume fraction, %) (5.5.2).

5.6.2 Water content mean for test portions taken at the 5 ml level

Calculate the mean, φ_{5m} , of the determinations of water content (volume fraction, %) of the test portions taken from the six test cylinders (5.5.6.4).

For each test portion taken from the 5 ml level, calculate the change in water content (volume fraction, %), $\varphi_{\Delta 5}$, as follows:

$$\varphi_{\Delta 5} = \varphi_5 - \varphi_{im}$$

where

φ_5 is the water content (volume fraction, %) of the test portion taken from the 5 ml level of the measuring cylinder as determined in 5.5.6.3;

φ_{im} is the mean of the two values of the initial water content (volume fraction, %) as determined in 5.5.2.

Report the change in water content (volume fraction, %), $\varphi_{\Delta 5}$, at the 5 ml level for each test cylinder.

Calculate the mean change in water content (volume fraction, %) at the 5 ml level, $\varphi_{\Delta 5m}$, for the six test cylinders. Report the mean, $\varphi_{\Delta 5m}$, of the six changes in water content at the 5 ml level.

5.6.3 Oil layer measurement

Calculate the mean of the six measurements of surface oil layer volume in millilitres (5.5.5). Report the mean surface oil layer volume to the nearest millilitre.

5.6.4 Accumulated free water measurement

Calculate the mean of the six measurements of the volume of accumulated free water in millilitres (5.5.5). Report the mean volume of accumulated free water to the nearest millilitre.

5.7 Precision

The precision of this test method has not been determined. However, 3.7 of Method A gives some indication of the precision.

6 Test report

The test report shall contain at least the following information:

- a) a reference to this part of ISO 20783;
- b) the type and complete identification of the product tested;
- c) the test method used, i.e. Method A, B, or C;
- d) the results of the test (see 3.6, 4.5 or 5.6 as appropriate);
- e) any deviation, by agreement or otherwise, from the procedure specified;
- f) the date of the test.

Bibliography

- [1] ISO 918:1983, *Volatile organic liquids for industrial use — Determination of distillation characteristics*
- [2] ISO 1998-1:1998, *Petroleum industry — Terminology — Part 1: Raw materials and products*
- [3] ISO 4259:1992, *Petroleum products — Determination and application of precision data in relation to methods of test*
- [4] ISO 4788:1980, *Laboratory glassware — Graduated measuring cylinders*
- [5] ISO 6743-4:1999, *Lubricants, industrial oils and related products (class L) — Classification — Part 4: Family H (Hydraulic systems)*

