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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.LT[1] 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.LT[1] 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.LT[1] 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.

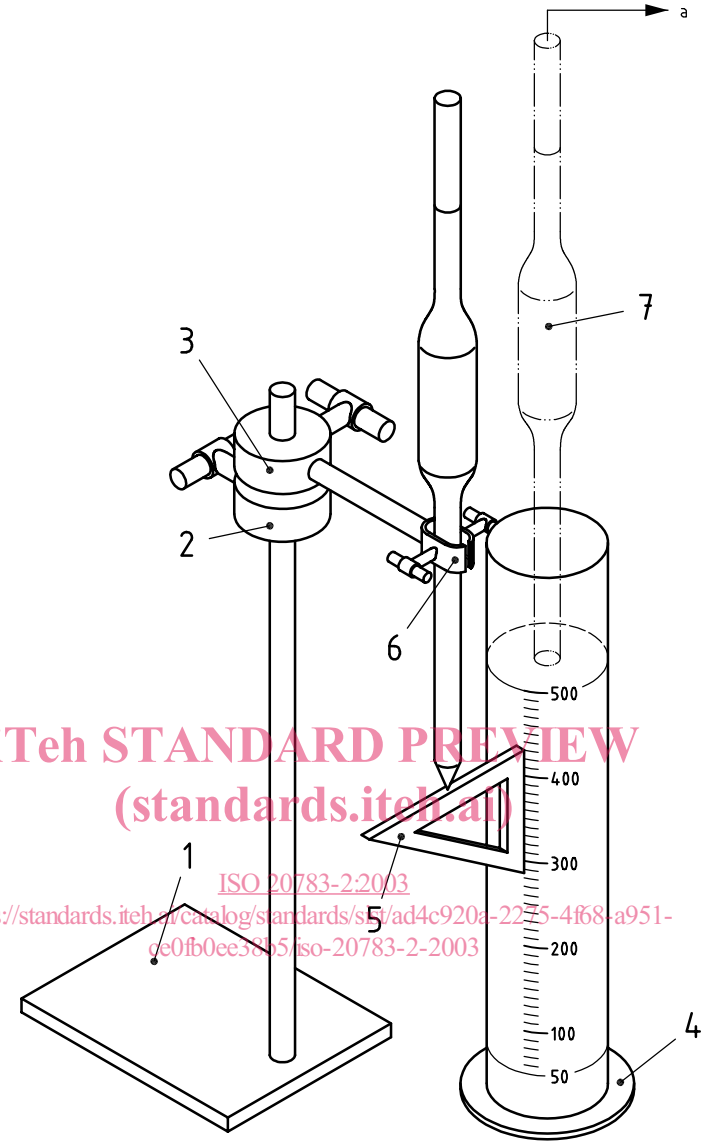
3.3.8 Apparatus for distillation procedure, as specified in ISO 3733, for the determination of water.

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 %.