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**Diesel engines — Fuel filters —  
Method for evaluating fuel/water  
separation efficiency**

*Moteurs diesel — Filtres à carburant — Méthode d'évaluation de  
l'efficacité de séparation carburant-eau*

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Published in Switzerland

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see [www.iso.org/directives](http://www.iso.org/directives)).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see [www.iso.org/patents](http://www.iso.org/patents)).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: [www.iso.org/iso/foreword.html](http://www.iso.org/iso/foreword.html)

This document was prepared by Technical Committee ISO/TC 22, *Road vehicles*, Subcommittee SC 34, *Vehicle propulsion, powertrain, and powertrain fluids*.

This document cancels and replaces the first edition ISO/TS 16332:2006 which has been technically revised. The main changes compared to the previous edition are as follows:

- test fuel definition;
- change of IFT measurement standard and interface age;
- droplet size distribution;
- test duration;
- additional preconditioning cycle; and
- validation of test procedure by conduction of two round robin tests (see [Annex G](#)).

## Introduction

Modern fuel injection systems, installed in passenger cars, as well as in heavy duty or off-road applications, require high and stable separation efficiencies for all insoluble contaminants in the fuel to ensure a prolonged life. Beside solid contamination, undissolved water, in finely or coarsely emulsified form, can also reduce the lifetime of injection systems. Suitable fuel/water separators, having a high level water separation efficiency, are an absolute necessity for system longevity.

Factors found to affect the separation efficiency of undissolved water in the field are mainly due to the fuel quality, which can differ widely in different regions of the world and which can also differ when biogenic components are added to the fuel. Additionally the separation efficiency is strongly influenced by fuel composition.

Separation efficiency tests can be applied mainly for two purposes:

- To evaluate the field performance of a fuel/water separator

To evaluate the performance of a fuel/water separator close to field conditions, the usage of commercially, untreated fuel as test fluid is necessary.

- To compare fuel/water separators under repeatable test conditions

For a fuel/water separator comparison in the laboratory, fuel conditioning is necessary to achieve constant and repeatable test conditions. Water separation efficiency results obtained with treated fuel can be significantly different from those with commercially available, untreated fuel.

Tests performed with new fuel/water separators can lead to considerably higher water separation efficiencies.

**NOTE** Ageing of the fuel/water separator due to operational conditions can strongly affect the water separation function of a fuel/water separator. To test a fuel/water separator in an “end of life” state, it can be aged in advance. It is proposed to do this by a standardized ageing procedure, to get comparable “end of life” states. However, it is not a part of this document nor any other ISO standard. This procedure may be explored in future.

# Diesel engines — Fuel filters — Method for evaluating fuel/water separation efficiency

## 1 Scope

This document specifies a fuel/water separator comparison test under defined and simplified laboratory conditions.

This test is intended for pressure side fuel/water separators as well as for suction side fuel/water separators. Pressure side fuel/water separators are tested with fine droplets and suction side filters are tested with coarse droplets using the same test rig layout.

The rated flow (in litres per hour) is intended for the range between 50 l/h and 1 500 l/h. By agreement between customer and fuel/water separator manufacturer, and with some modifications, the procedures can be used for fuel/water separators with higher or lower flow rates.

## 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 1219-1, *Fluid power systems and components — Graphical symbols and circuit diagrams — Part 1: Graphical symbols for conventional use and data-processing applications*

ISO 9101, *Surface active agents — Determination of interfacial tension — Drop volume method*

ISO 6889, *Surface active agents — Determination of interfacial tension by drawing up liquid films*

ISO 12937, *Petroleum products — Determination of water — Coulometric Karl Fischer titration method*

ISO 13320, *Particle size analysis — Laser diffraction methods*

ASTM D4176-04 (2009), *Standard Test Method for Free Water and Particulate Contamination in Distillate Fuels (Visual Inspection Procedures)*

## 3 Terms and definitions

For the purpose of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp>

### 3.1 interfacial tension IFT

work which is required to increase the interface of the liquid by one surface area unit

Note 1 to entry: In case of additivated liquids, the IFT-value has a significant time dependency. Therefore the default value for the interface age within ISO 16332-usage is defined at 10 s.

Note 2 to entry: The conditions and parameters for determination of IFT by the drop volume method according to ISO 9101 are defined in [Annex C](#).

Note 3 to entry: Interfacial tension is equivalent to the specific interfacial energy and is expressed in Millinewtons per meter (mN/m). Alternative methods to determine the IFT (at 10 s) can be used, as long as the comparability to ISO 9101 is ensured.

### 3.2 droplet size distribution

#### DSD

percentage of the droplet population in different size ranges

Note 1 to entry: For further information, see [B.3](#).

### 3.3 water concentration at the saturation level of dissolved water

#### $c_S$

concentration of water in water saturated test fuel with the IFT adjusted by Monoolein

Note 1 to entry: The determination of  $c_S$  is defined in [Annex E](#).

### 3.4 base water concentration

#### $c_B$

concentration of water in the test fuel, determined after the preconditioning cycle

Note 1 to entry: See [8.4.3](#) or [9.1.3](#).

Note 2 to entry: In case one of the  $c_{T,down,i}$ -values [determined in [9.1.4](#) c) and d)] is lower than  $c_B$  (determined in [9.1.3](#)) take the lowest value as  $c_B$ .

### 3.5 undissolved water concentration

#### $c_U$

concentration of free water, that is concentration above base water concentration

### 3.6 total water concentration

#### $c_T$

summation of base water concentration and undissolved water concentration

Note 1 to entry:  $c_T = c_U + c_B$ .

### 3.7 sample index

#### $i$

integer from 1 to  $n$ , where  $n$  equals the number of samples

### 3.8 instantaneous water separation efficiency

#### $\eta_i$

water separation efficiency, at test time  $t_i$

### 3.9 average water separation efficiency

#### $\eta_{av}$

average water separation efficiency, calculated based on the average downstream water concentration

Note 1 to entry: Calculation according to [9.2](#) e).

### 3.10 calibration flow rate

$Q_c$

fuel flow rate, which is adjusted for calibration purpose of the emulsifying device

Note 1 to entry: The calibration procedure is defined in [B.4](#).

### 3.11 sampling point index

< up> reference to the upstream sampling point

### 3.12 sampling point index

< down> reference to the downstream sampling point

## 4 Symbols

Graphical symbols used in this document for fluid power system components are in accordance with ISO 1219-1.

## 5 Test equipment

### 5.1 Test fluids

#### 5.1.1 Test fuels

For the validation and for each filter test one of the following three kinds of fuels can be used as test fuel.

- F1 Unmodified service station fuel
- F2 Standard test fuels: Fuels, treated according to [Annex A](#)

Test fuel F2.1: High IFT test fuel

- IFT (10 s):  $22 \pm 2$  mN/m, according to ISO 9101, parameterized according to [Annex C](#)  
Alternatively IFT (60 s):  $20 \pm 2$  mN/m, according to ISO 9101 or ISO 6889.
- Separability (ASTM D 1401): To be reported

Test fuel F2.2: Low IFT test fuel

- IFT (10 s):  $13 \pm 2$  mN/m, according to ISO 9101  
Alternatively IFT (60 s):  $11 \pm 2$  mN/m, according to ISO 9101 or ISO 6889.

To describe the test fuel used, the following parameters shall be determined:

- IFT(10 s) and IFT(60 s);
- separability (ASTM D 1401);
- water saturation level according to [Annex E](#);
- bio diesel content (optional);
- density (optional);
- kinematic viscosity (optional);
- CFPP (optional).

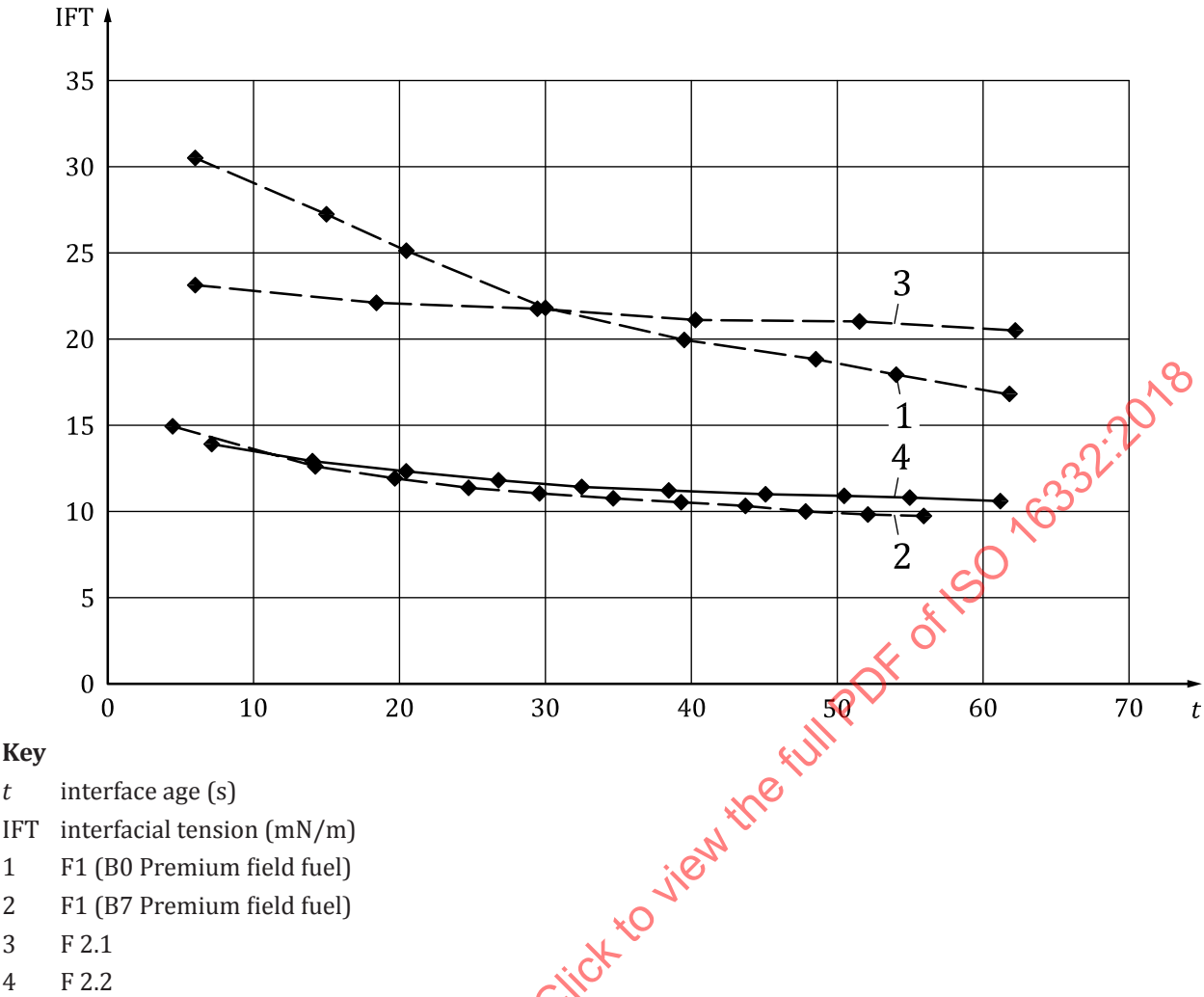


Figure 1 — Time dependency of interfacial tension [IFT(t)] for F1 fuels, F2.1 and F2.2 fuel

Figure 1 is showing two typical IFT(t)-curves for commercially available F1 fuels and for F 2.1 and F 2.2 fuel.

Depending on the F1-quality, the brand/product specific slope in IFT(t) does not allow to deduct the F1 IFT(10 s) value based on the measured F1 IFT(60 s) value.

The Monoolein specific slope in IFT(t) of F2 fuel is stable and reproducible, therefore the deduction of the F2 IFT(10 s) value - based on the measured F2 IFT(60 s) value - is valid and proven.

The test fuel shall be stored in a sealed container, protected from humidity, dust and light.

For each filter test fresh fuel shall be used. In the case of F2 fuel, fresh fuel can be achieved by retreating used fuel according to Annex A.

5.1.2 Test water

Clean, distilled or deionised water, with a surface tension of 70 mN/m -72 mN/m, measured at 20 °C ± 1,5 °C.

## 5.2 Laboratory equipment

### 5.2.1 General

All laboratory equipment and glassware, required to determine the water concentration, shall be according to ISO 12937.

### 5.2.2 Sampling bottles and glassware

100 ml sampling bottles carefully cleaned and dried, free of any residuals from the cleaning process.

### 5.2.3 Water detection system

#### 5.2.3.1 Karl Fischer (KF) titrator

As commercially available.

For biodiesel and biodiesel containing fuels the direct Karl Fischer method is recommended.

Humidity is probably the largest source of error during the titration process. Special precautions shall be taken during setup and testing. The amount of water per sample should be  $\geq 50 \mu\text{g}$  to reach a good relation between titration time and accuracy.

#### 5.2.3.2 Centrifuge

For higher water concentration as specified in [6.4.1](#), 20 000 ppm water concentration, a centrifuge according to [D.2](#) can be used. The measurement accuracy according to [Table 1](#) shall be confirmed.

### 5.2.4 Equipment for determination of IFT

The equipment for determination of the interfacial tension shall be according to ISO 9101.

## 5.3 Test stand

### 5.3.1 General

The test stand, shown diagrammatically in [Figure 2](#), shall comprise a fuel/water separator test circuit as described in [5.3.2](#).

All parts in contact with fuel, should be made of stainless steel.

### 5.3.2 Fuel/water separator test circuit

#### 5.3.2.1 Fuel sump (1)

The container with a conical bottom should be made of stainless steel. The fuel outlet shall be located at the lowest point of bottom. The container shall be able to contain the volume as specified in [6.1](#). The fuel sump shall be covered with a non-transparent cover to protect the fuel from light. The fuel sump shall contain a suitable device to maintain homogeneity of its content.

#### 5.3.2.2 Water sump (6)

The container should be made of stainless steel or corrosion resistant material with appropriate volume.

NOTE Instead of the container, a continuous water supply unit can be used.

#### 5.3.2.3 Heat exchanger (3)

The heat exchanger shall be able to maintain the test fuel temperature  $T$  within the tolerances given in [Table 1](#).

Alternative to the position of the heat exchanger depicted in [Figure 2](#), the heat exchanger can as well be positioned downstream the back pressure gauge (16).

#### 5.3.2.4 Test pump (2)

A pump type shall be chosen, which does not exhibit pressure pulsation with an amplitude greater than 10 % of the average pressure at the inlet of the water emulsifying device.

#### 5.3.2.5 Water injection pump (7)

The pump type shall be adjustable to enable a water concentration in the test circuit between 1 500 ppm and 20 000 ppm over the complete flow rate of test fluid.

#### 5.3.2.6 Fuel flow meter (5a)

The equipment shall be suitable for the complete range of the flow rate of test fluid with an accuracy as specified in [Table 1](#).

#### 5.3.2.7 Water flow meter (5b)

The equipment shall be suitable for the complete range of the required injection range with an accuracy as specified in [Table 1](#).

#### 5.3.2.8 Injection device (8)

The concept shall allow a continuous water injection. The resulting DSD at the injection point shall be validated. The validation criterion is defined by:

$d_{3,50}$  shall be greater or equal to the  $d_{3,50}$  value chosen according to [6.7](#)

#### 5.3.2.9 Water emulsifying device (9)

The concept shall be able to generate a DSD as specified according to [6.7](#). Jet emulsification - as described in [Annex B](#) is recommended to be applied.

For each combination of emulsifying device, test fuel, flow rate and temperature a calibration curve is mandatory

In case the jet emulsification concept in accordance with [Annex B](#) is used, the calibration procedure is described in [B.4](#).

#### 5.3.2.10 Operating pressure gauge (10)

The operating pressure is defined at the up-stream side of the test fuel water separator (14). The required accuracy is specified in [Table 1](#).

#### 5.3.2.11 Differential pressure gauges (11)

The required accuracy is specified in [Table 1](#).

#### 5.3.2.12 Upstream sampling point (12)

The upstream sampling point shall be designed as illustrated in [Figure 4](#).

**5.3.2.13 Temperature indicator (13)**

The required accuracy is specified in [Table 1](#).

**5.3.2.14 Water drainage system (15)**

Realized as a graduated and transparent collector (e.g. laboratory measuring cylinder), located directly below the test fuel/water separator (14). The internal diameter of the connecting pipe between the test fuel/water separator (14) and the graduated water drainage system (15) shall be of at least 10 mm and allow an unconstrained removal of water. It shall be realized with pressure-tight fittings. The collector volume shall be drainable at its lowest point.

The collector volume shall be adjusted to the total amount of water injected, with a maximum of 5 % of the volume of test fuel  $V_T$  (according to [6.1](#)).

In case the collected amount of water is reaching 80 % of the collector volume, the water shall be drained out of the collector within approx. 1 min. The collector outlet valve shall be adjusted adequately. Care should be taken, not to take samples during or immediately after the water draining.

**5.3.2.15 Back pressure gauge (16)**

For determination of back pressure with an accuracy as specified in [Table 1](#).

**5.3.2.16 Back pressure control valve (17) (optional equipment)**

The backpressure control valve is to ease test fuel/water separator venting, to adjust the back pressure and allow sufficient sampling at the upstream sampling point. When adjusting the back pressure, the test fuel/water separator design pressure shall be taken into consideration.

**5.3.2.17 Downstream sampling point (18)**

For manual sampling the operating conditions at sampling point 18 shall fulfil the requirements defined in [D.1.1](#). Proper sampling can be reached by adjustment of a suitable back pressure value.

The downstream sampling point shall be designed as illustrated in [Figure 4](#).

**5.3.2.18 Clean-up system (19)**

A suitable fuel water clean-up system with the capability to separate the water – such that not more than 50 ppm by volume of undissolved water is recycled on an average basis under test conditions – shall be installed.

**5.3.2.19 Droplet size distribution measurement device (20)**

Laser diffraction measurement device according to ISO 13320.

The measurement device shall not influence the droplet size distribution. This is especially valid for an inline measurement cell, which is designed as a full flow concept.

In case no droplet size measurement device is used and the jet emulsification concept according to [Annex B](#) is applied, for each combination of orifice, batch of test fuel (independent of it being F1 or F2 fuel), flow rate and temperature, a calibration curve shall be used (further explanation is given in [B.4](#)).

**5.3.2.20 Bypass line (21)**

The total length of the bypass line shall be as short as possible.

#### 5.3.2.21 Inline water concentration measurement device (22) (Optional equipment)

Suitable inline water concentration measurement devices can be used. The measurement accuracy according [Table 1](#) shall be confirmed.

The inline water concentration measurement device shall be placed into the pipe in the full flow at the position of the up- and/or downstream sampling points (optional installation) as defined in [Figure 2](#).

#### 5.3.2.22 General requirements on the hydraulic piping system

The test stand piping shall be designed to enable the drainage of the total test fuel volume out of the test stand. This is to ensure the correct adjustment of the test fuel volume within the limits specified in [Table 1](#).

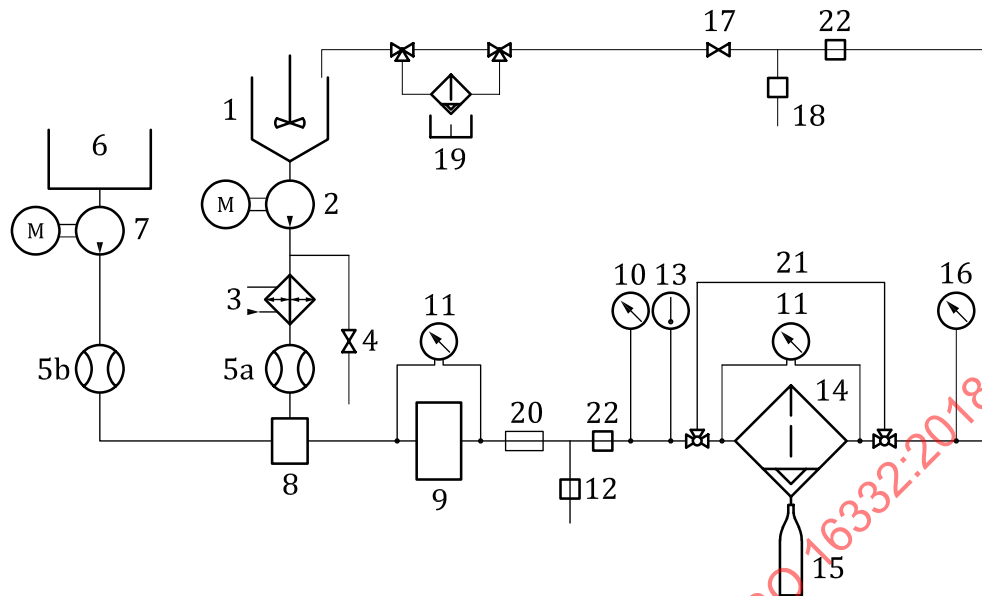
The test stand pipes shall be made of stainless steel; painted or coated pipes are not allowed.

For the adaptation of the test fuel/water separator (14) to the test stand piping, flexible lines are allowed.

The piping shall be designed with a minimum number of flanges or fittings and grounded upstream near the test fuel/water separator (potential difference <10 V between each point).

The test stand section line inner diameter  $d_i$  between water injection device (8) and downstream sampling point (18) shall allow a flow velocity  $\geq 0,75$  m/s. The overall pipe length between the water emulsifying device (9) and test fuel/water separator (14) shall not to exceed 1 m. ([Figure 3](#)).

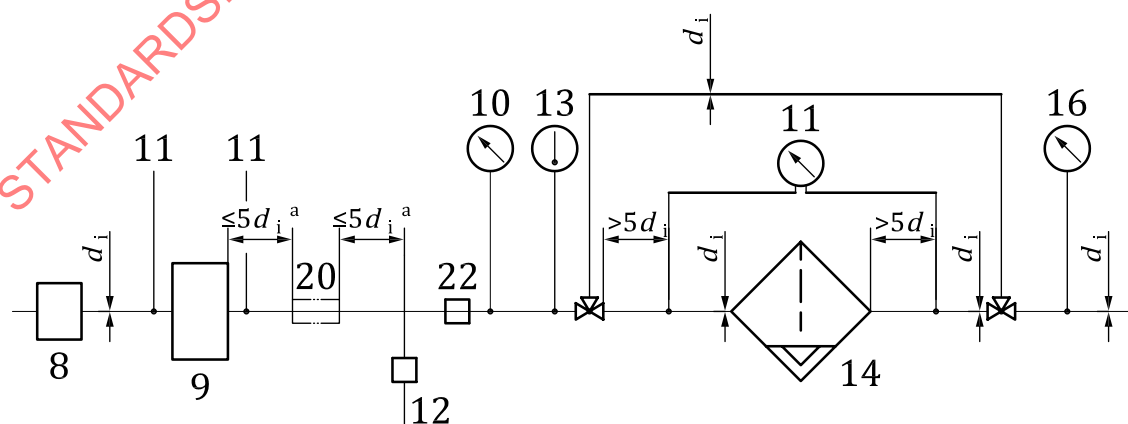
The pipes, outside of [Figure 3](#), shall be as short as possible.



### Key

- |                                           |                                                              |
|-------------------------------------------|--------------------------------------------------------------|
| 1 fuel sump including homogenizing device | 12 upstream sampling point                                   |
| 2 test pump                               | 13 temperature indicator                                     |
| 3 heat exchanger                          | 14 test fuel/water separator                                 |
| 4 sampling point                          | 15 graduated water drainage system                           |
| 5a fuel flow meter                        | 16 back pressure gauge                                       |
| 5b water flow meter                       | 17 back pressure control valve (optional)                    |
| 6 water sump                              | 18 downstream sampling point                                 |
| 7 adjustable water injection pump         | 19 clean-up system                                           |
| 8 injection device                        | 20 DSD measurement device                                    |
| 9 water emulsifying device (orifice)      | 21 bypass line                                               |
| 10 operating pressure gauge               | 22 inline water concentration measurement devices (optional) |
| 11 differential pressure gauges (2)       |                                                              |

**Figure 2 — Fuel/water separator test stand (diagrammatically)**

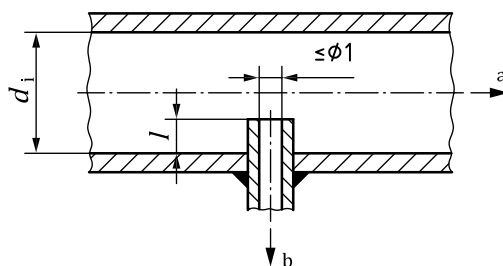


### Key

- $d_i$  inner pipe diameter  
a for  $d_i < 10$  mm:  $\leq 10$  mm

**Figure 3 — Distances of components and inner diameter  $d_i$  of test stand pipes**

Dimension in millimetres

**Key**

- $l$   $d_i / 4 < l < d_i / 3$   
 $d_i$  inner pipe diameter  
 a Flow of test fluid.  
 b Sampling flow.

**Figure 4 — Upstream and downstream sampling point****6 Test conditions****6.1 Volume of test fuel  $V_T$** 

The total volume of the fuel in litres filled into the empty test stand shall be 20 % of the test flow rate  $Q_T$  (l/h), with a minimum of 45 l and a maximum of 200 l.

Therefore for flow rates lower than 225 L/h, the volume will be 45 L and for flow rates higher than 1000 L/h, the volume will be 200 L

The total circuit volume – including the fuel sump, piping, fuel/water separator and the clean-up system shall not exceed a critical value. Thus, a suitable filling level in the fuel sump is given, when the test procedure is started.

**6.2 Test fuel temperature  $T$** 

The test shall be conducted at a test fuel temperature  $T = 23 \pm 2$  °C. The test fuel temperature is measured at the test fuel/water separator inlet (13).

**6.3 Test flow rate  $Q_T$** 

The test flow rate  $Q_T$  (l/h) is specified by the customer and is defined as the flow rate of fuel through the test fuel/water separator.

**6.4 Upstream undissolved water concentration  $c_{U,up}$** **6.4.1 General**

One of the following two conditions for the concentration  $c_{U,up}$  shall be used for the test:

- standard condition: 1 500 ppm volume fraction  $\pm$  100 ppm volume fraction;
- optional condition: 20 000 ppm volume fraction  $\pm$  1 000 ppm volume fraction.

#### 6.4.2 Water injection flow rate $Q_W$

The water injection flow rate  $Q_W$  [ml/min] for the efficiency testing is calculated by the following equation:

$$Q_W = \frac{\left( Q_T * 1000 \frac{ml}{l} \right)}{\left( 60 \frac{min}{h} \right)} * c_{u,up}$$

#### 6.5 Back pressure

The back pressure is adjusted by the back pressure control valve (17) to  $\geq 0,5$  bar.

#### 6.6 Sampling

The upstream sampling shall be taken at sampling point (12).

The downstream sampling shall be taken at sampling point (18).

In case of manual sampling according to [D.1](#), the sampling volume shall be approximately 50 ml, and the sampling time shall be between 5 s and 10 s.

#### 6.7 Droplet size distribution DSD

For standard test conditions, two droplets size distributions, fine and coarse, are defined. As following:

Fine DSD, to test pressure side fuel/water separators:

- $D_{3,50}$ :  $10 \pm 1,5 \mu m$ ;
- $D_{3,90}$ :  $\leq 30 \mu m$ .

Coarse DSD, to test suction side fuel/water separators:

- $D_{3,50}$ :  $150 \pm 10 \mu m$ ;
- $D_{3,90}$ :  $\leq 350 \mu m$ .

The choice of whether to use the fine, coarse or a customer specified DSD, depends on the application and shall be specified by the customer.

#### 6.8 Test duration $t_{test}$

The test duration  $t_{test}$  shall be 90 min, the test duration  $t_{test}$  does not include the pre-test preparation according to [9.1.2](#) and the Preconditioning cycle according to [9.1.3](#)

The test may be performed at test parameters as agreed between customer and fuel/water separator manufacturer. This shall be recorded in the test report.

In any case the test configuration shall be checked regarding the relevancy to the application.

### 7 Accuracy of measuring instruments and test conditions

The measuring instruments shall be capable of measuring to the levels of accuracy given in [Table 1](#). In addition, [Table 1](#) specifies the limits, within which the test conditions shall be maintained.

Table 1 — Instrument accuracy and test condition variation

| Test condition                                                             | Units                | Measurement accuracy                  | Allowed test condition variation                                                                              |
|----------------------------------------------------------------------------|----------------------|---------------------------------------|---------------------------------------------------------------------------------------------------------------|
| Fuel volume                                                                | l                    | ±2 %                                  | ±5 %                                                                                                          |
| Fuel flow rate ( $Q_C$ , $Q_T$ )<br>(fuel flow meter)                      | l/h                  | ±1 %                                  | ±2 %                                                                                                          |
| Water flow rate<br>(water flow meter)                                      | ml/min               | ±1 %                                  | ±2 %                                                                                                          |
| Pressure                                                                   | hPa                  | ±2 %                                  | —                                                                                                             |
| Differential pressure $\Delta p_0$<br>(gauge at orifice plate)             | hPa                  | ±0,5 %                                | —                                                                                                             |
| Differential pressure $\Delta p_F$<br>(gauge at test fuel/water separator) | hPa                  | ±2 %                                  | —                                                                                                             |
| Interfacial tension                                                        | mN/m                 | ±0,5 mN/m                             | ±1 mN/m                                                                                                       |
| Temperature<br>(temperature indicator)                                     | °C                   | ±1 °C                                 | ±2 °C                                                                                                         |
| DSD<br>$D_{3,50}$                                                          | µm                   | According to<br>ISO 13320             | ±20 %, or<br>a minimum of ± 2 µm,<br>whichever is larger, and<br>a maximum of ±10 µm,<br>whichever is smaller |
| Water concentration                                                        | % Volume<br>fraction | Precision accord-<br>ing to ISO 12937 | —                                                                                                             |

NOTE The overall tolerance range, resulting out of the allowed test condition variation does already include the measurement accuracy, the tolerances are not to be considered in a cumulative sense. Allowed test condition variation is valid for the whole test duration.

## 8 Validation procedures

### 8.1 General

These validation procedures reveal the effectiveness of the test circuit and the water injection system in maintaining the required DSD and water concentration. The frequency of revalidation shall be defined by internal quality specifications of the user.

### 8.2 Water detection system

#### 8.2.1 Inline water concentration measurement device (optional)

The inline water concentration measurement device shall be calibrated. In case the test fuel is changed, the validity of the calibration shall be confirmed.

NOTE Depending on the detection principle, either the total water concentration  $c_T$  or only the free water concentration  $c_U$  will be detected.

#### 8.2.2 Karl Fischer titration sytem

The Karl Fischer Titrator shall be calibrated.

In case of manual sampling, the KF titration system including the primary and secondary sampling procedure shall be validated according to [Annex D](#).

In case of automated sampling, the KF system shall be validated by the sample concentrations obtained according to [8.4](#)

The validation procedures reveal the effectiveness of a correct and representative sampling and transfer procedure out of the test circuit to the KF titration.

### 8.3 Emulsifying device

The validation of the DSD upstream the test fuel/water separator shall be done with a droplet size distribution measurement device (20) according to [5.3.2.19](#) for each combination of:

- specified droplet size distribution;
- test fuel;
- flow rate; and
- temperature.

In case the emulsifying device calibrated according to [B.4](#) is used, the validation is confirmed by the corresponding calibration data. In case a different emulsifying device is used, the droplet size distribution shall be measured *in situ* with the online DSD measurement device according to [5.3.2.19](#) and adjusted to the specified DSD.

### 8.4 Filter test circuit and water injection system

#### 8.4.1 General

The validation of the test stand shall be performed at the minimum and maximum design flow rates for every test section line size.

For each of these two flow rates, perform the following procedure in the order given.

#### 8.4.2 Validation preparation

The following validation preparation procedure shall be followed:

- a) Use fresh fuel.
- b) Determine all relevant test fuel characteristics prior to usage, as specified in [Annex F](#).
- c) Ensure that the hydraulic system is free from any residuals (e.g. remaining fuel from previous test, additives and impurities). The complete hydraulic system shall be flushed with the specified test fuel. The verification of the cleanliness level is based on the IFT value. The flushing process shall be repeated until the IFT of the flushing fuel is equal to the IFT of the fuel as out of the drum within the tolerances according to [Clause 7](#).
- d) Drain the flushing fuel completely.
- e) Replace the clean-up filters if another test fuel is used (e.g. for F1: change from B7 to B20, etc.). In case identical test fuel is used, they can be reused.
- f) Fill the specified volume of test fuel (see [6.1](#)) into the fuel sump (1).
- g) Install a straight section of pipe in place of a fuel/water separator during the validation procedure.
- h) Choose an orifice plate with a suitable orifice for the required DSD and fuel flow rate  $Q_T$  according to [Annex B](#).
- i) Install the orifice plate into the water emulsifying device (9).

- j) Utilized the clean-up system (19).
- k) Start circulation at the specified test flow rate  $Q_T$  (see 6.3) and test fuel temperature  $T$  (see 6.2). Bleed air from the system including water drainage system (15). Record an initial pressure loss reading at the orifice (9) immediately after the start.

#### 8.4.3 Preconditioning cycle

The preconditioning cycle is to saturate the test system between the point of water injection (8) and the clean-up system (19) including pipes, clean-up system, test fuel etc. with water.

Perform the preconditioning cycle in the following order:

- a) Apply the nominal fuel flow rate  $Q_T$ . Switch on the clean-up system (19).
- b) Start the water injection for a duration of 20 min, with a water injection flow rate  $Q_W$  [ml/min] calculated by the following equation:

$$Q_W = \frac{\left( Q_T * 1000 \frac{ml}{l} \right)}{\left( 60 \frac{min}{h} \right)} * 1500 \text{ ppm}$$

- c) Continue the circulation of the test fuel through the clean-up system until the test fuel is showing a clarity and brightness-level which is higher or equal as the fresh fuel appearance (e.g. out of the drum). Terminate the circulation when the clarity and brightness-level is comparable to the fresh fuel (according to ASTM D4176 or suitable method). Report the clean-up time.
- d) Take a sample at sampling point (12) to determine the base water concentration  $c_B$ :
  - if the base water concentration  $c_B$  is below the saturation level  $c_s + 50$  ppm, the circulation can be stopped and the preconditioning cycle is completed;
  - if the water concentration  $c_B$  is above  $c_s + 50$  ppm, the circulation through the clean-up system shall be continued for further 20 min. Repeat Step d).

#### 8.4.4 Validation cycle

The validation cycle shall be started subsequently to the preconditioning cycle. Perform the following procedure in the order given:

- a) Start the water injection to achieve the required concentration  $c_{U,up}$  according to 6.4.1.
- b) Allow the system to stabilize for at least 10 min.
- c) Take samples every 10 min for a period of 90 min:
  - 1) at the sampling point (4), for the validation of the clean-up system (19);
  - 2) at the upstream sampling point (12) and downstream sampling point (18) for the validation of the injection system and sampling devices;
  - 3) measure the water content of each sample.

NOTE Optionally, the validation can be performed in subsequent steps for the clean-up and water injection and sampling devices, when the sampling system does not allow to take 3 samples at the same point of time.

The validation shall be accepted only if:

- at the sampling point (4), the variation between the total water concentration of each sample is less than 50 ppm by volume and the average total water concentration is below the saturation level  $c_s + 50$  ppm. For reference, the saturation level  $c_s$  – determined in 8.4.2 b) – shall be used;

- at the sampling points (12) and (18), the actual concentration of water of each sample, reduced by the base water concentration, is equal to the specified water concentration and within the limits according to [6.4.1](#).

NOTE Many water in oil analytical devices will determine the water concentration in terms of micrograms. To convert micrograms to parts per million (ppm) by volume, the following equation can be used: Water concentration (ppmVol.) = titration reading (ppmMass) × fuel-water densities correlation factor. The fuel-water densities correlation factor is [fuel density, (g/l)]/[water density (g/l)].

## 9 Simplified laboratory test

### 9.1 Test procedure

#### 9.1.1 General

The test procedure applies to both testing of filters for pressure side applications and testing of filters for suction side applications. That means, suction side filters are tested also in the same pressure side setup, but with coarse droplets.

#### 9.1.2 Pre-test preparation

Perform the pre-test preparation procedure in the following order:

- a) Use fresh fuel for each filter test. In the case of the usage of F2 test fuel, fresh test fuel can be achieved by retreating used fuel according to [Annex A](#).
- b) Determine all relevant test fuel characteristics prior of the usage, as they are defined in [Annex E](#).
- c) Ensure that the hydraulic system is free from any residuals (e.g. remaining fuel from previous test, additives and impurities). The complete hydraulic system shall be flushed with the specified test fuel (F1) or (F2). The verification of the cleanliness level is based on the IFT value. The flushing process shall be repeated until the IFT of the flushing fuel is equal to the IFT of the fuel as out of the drum within the tolerances according [Clause 7](#).
- d) Drain the flushing fuel completely.
- e) Replace the clean-up filters if another test fuel is used (e.g. for F1: change from B7 to B20, etc.). In case identical test fuel is used, they can be reused.
- f) Fill the specified volume of test fuel  $V_T$  according [6.1](#) into the fuel sump (1).
- g) Install the test fuel/water separator (14) and water drainage system (15) for testing.
- h) Choose an orifice plate with a suitable orifice for the required DSD and fuel flow rate  $Q_T$  according to [Annex B](#).
- i) Install the orifice plate into the water emulsifying device (9).
- j) Utilize the clean-up system (19).
- k) Start circulation at the specified test flow rate  $Q_T$  (see [6.3](#)) and test fuel temperature  $T$  (see [6.2](#)). Bleed air from the system including water drainage system (15). Record an initial pressure loss reading at the orifice (9) and at the test fuel/water separator (14) immediately after the start.

#### 9.1.3 Preconditioning cycle

The preconditioning cycle is to saturate the test system between the point of water injection (8) and the clean- up system (19) including pipes, fuel/water separator, clean-up system, test fuel etc. with water.

Perform the preconditioning cycle in the following order:

- a) Apply the nominal fuel flow rate  $Q_T$ , bypass the fuel/water separator under investigation. Switch on the clean-up system (19).
- b) Start the water injection with a water injection flow rate  $Q_W$  [ml/min], to achieve the required concentration  $c_{U,up}$  (see 6.4.2), for a duration of:
  - 40 min for  $c_{U,up} = 1\ 500$  ppm,
  - 5 min for  $c_{U,up} = 20\ 000$  ppm.
- c) Switch in the fuel/water separator after 50 % of the duration of water injection.
- d) Continue the circulation of the test fuel through the fuel/water separator and the clean-up system until the test fuel is showing a clarity and brightness-level which is higher or equal as the fresh fuel appearance (e.g. out of the drum). Terminate the circulation when the clarity and brightness-level of a sample taken at sampling point (12) is comparable to the fresh fuel (according to ASTM D4176 or suitable method). Report the clean-up time.
- e) Take a sample at sampling point (12) to determine the base water concentration  $c_B$ :
  - If the base water concentration  $c_B$ , is below
    - $c_s + 50$  ppm in case  $c_{U,up} = 1\ 500$  ppm,
    - $c_s + 200$  ppm in case  $c_{U,up} = 20\ 000$  ppm,

the circulation can be stopped and the preconditioning cycle is completed.

  - If the base water concentration  $c_B$ , is above the specified limit, the circulation shall be continued for a further 25 min. Repeat step e). The total clean-up time shall be reported.
- f) A sample to determine the fuel properties according to Annex A (IFT, separability) shall be taken at sampling point 12 for documentation.

NOTE In case of  $c_{U,up} = 20\ 000$  ppm, the duration of the water injection might not be long enough to saturate the test fuel completely. The effect on the separation efficiency results can be neglected.

#### 9.1.4 Efficiency measurement

The efficiency measurement shall be started subsequently to the preconditioning cycle in immediate sequence.

In case an inline water concentration measurement device is used, the water concentration shall be recorded at the defined test time  $t_i$ .

Perform the following procedure in the order given:

- a) Start the water injection to achieve the required concentration  $c_{U,up}$  (see 6.4.1) and start the timing clock at the same time water begins to flow. This point is test time  $t = 0$ . Record the differential pressure  $\Delta p_0$  across the orifice (9) and test fuel/water separator (14).
- b) After 5 min:
 

Determine the water concentration  $c_{T,up}$  at sampling point (12). This is to confirm that the water injection device is adjusted correctly and the water concentration  $c_{U,up}$  is reached. This shall be evaluated immediately.
- c) After 10 min:
 

Determine the downstream water concentration  $c_{T,down,i}$ . Record the differential pressure  $\Delta p_0$  (11) across the orifice (9) and the test fuel/water separator (14).

- d) Repeat step c) every 10 min thereafter until termination of the test.
- e) Terminating criteria:
- 90 min of test duration  $t_{\text{test}}$  has been attained;
  - the differential pressure  $\Delta p_F$  across the test fuel/water separator (14) exceeds an upper limit specified by customer or fuel/water separator manufacturer; or
  - the downstream concentration  $c_{T,\text{down}}$  is above the acceptable level specified by customer or fuel/water separator manufacturer;
- f) While water injection is still running, determine the base water concentration of the fuel at sampling point (4) to verify that the clean-up system (19) is working sufficiently.
- If the base water concentration  $c_B$  has changed by more than:
- 50 ppm and the total water concentration  $\geq$  saturation level  $c_s + 50$  ppm in case  $c_{U,\text{up}} \geq 1\,500$  ppm;
  - 200 ppm and the total water concentration  $\geq$  saturation level  $c_s + 200$  ppm in case  $c_{U,\text{up}} \geq 20\,000$  ppm;
- cancel the test; check the clean-up system (20) and repeat the steps a) to e);
- g) While water injection is still running; determine the water concentration  $c_{T,\text{up}}$  at sampling point (12). This is to confirm that the water injection device was adjusted correctly during the test and the water concentration  $c_{U,\text{up}}$  was reached.
- h) Record the volume of water drained from the test fuel/water separator (14).

## 9.2 Calculation of water separation efficiency and reporting of test results

Perform the following procedures in the order given:

- a) Calculate the concentrations  $c_{U,\text{down},i}$  from the downstream samples  $c_{T,\text{down},i}$  with respect to test time  $t_i$ :
- $$c_{U,\text{down},i} = c_{T,\text{down},i} - c_B$$
- b) Report the concentration  $c_{U,\text{down},i}$  versus test time  $t$ , in min.
- c) Calculate, report and plot ([Annex F](#)-Figure 1) the instantaneous water separation efficiency  $\eta_i$ , in %, versus test time  $t$ , in min, by the following equation:

$$\eta_i = \frac{(c_{U,\text{up}} - c_{U,\text{down},i})}{c_{U,\text{up}}} * 100$$

- d) Calculate and report the average downstream water concentration  $c_{\text{av,down}}$ , in ppm by volume, by the following equation:

$$c_{\text{av,down}} = \frac{c_{U,\text{down},i}}{n}$$

$n$  is the number of samples or readings [see [9.1.4](#) c) and d)].

- e) Calculate and report the average water separation efficiency, in %, by the following equation:

$$\eta_{\text{av}} = \frac{(c_{U,\text{up}} - c_{\text{av,down}})}{c_{U,\text{up}}} * 100$$

## 10 Test report

The test report shall include:

- test fluid(s) characteristics;
- test flow rate  $Q_T$ ;
- undissolved water concentrations  $c_{U,up}$  and  $c_{U,down,I}$ ;
- calibrated orifice diameter  $d$ ;
- differential pressure  $\Delta p_O$  at orifice plate and corresponding average droplet size  $D_{3,50}$ ;
- differential pressure  $\Delta p_F$  at test fuel/water separator;
- test fuel temperature  $T$ ;
- test duration  $t_{test}$ ;
- water concentration at the saturation level  $c_S$ ;
- base water concentration  $c_B$  of the fuel at test fuel temperature  $T$ ;
- total volume of water drained from the test fuel/water separator;
- average water separation efficiency  $\eta_{av}$  including the main test conditions: test fuel, upstream water concentration  $c_{U,up}$ , droplet size  $D_{3,50}$ :  
For example:  $\eta_{av} (F2.1, 1\ 500\ \text{ppm}, 10\ \mu\text{m}) = \dots\%$
- a graph showing the instantaneous water separation efficiency  $\eta_i$  vs. test time  $t$  as shown in [Figure F.1](#);
- an explanation of reason(s) for test termination.

See [Annex F](#) for a typical fuel/water separator test report.

## Annex A (normative)

### Fuel treatment to obtain test fuel F2

In this Annex, the procedure to obtain the standard test fuel F2 is defined.

The base fuel to achieve fuel F2 is CEC RF-06-03 reference fuel. CEC RF-06-03 is a reference fuel without biofuel content for European Emission Certification. There are corresponding fuels for other regions available which fulfil the CEC RF-06-03 specification. These fuels can be used alternatively as base fuel. Perform the fuel treatment in the order given below:

a) Clay treatment

Take the required volume of fuel and continuously contact the fuel with clay. This may be done by filtering the fuel through commercially available clay cartridge filters. The test fluid sump may be used.

Periodically (about every 2 h), take a sample of the fuel. Measure the interfacial tension IFT. If the IFT has reached a stable level  $>35$  mN/m, stop further contacting with clay. Report the IFT of the clay treated fuel. Remove the clay filters from the test loop, or adjust valving to isolate them from the test loop.

Particle contamination coming from the clay treatment process shall be avoided.

b) Water washing

Install an orifice plate, ensuring a droplet size of  $D_{3,50} \leq 10$   $\mu\text{m}$ . This can be realized with a differential pressure  $\Delta p_0$  over the orifice of  $\geq 3$  bar. Switch the clean-up filters in and recirculate the fuel at maximum flow rate.

Start continuous water injection at a water concentration of 2 %. Recirculate the fuel for at least two revolutions of the total fuel volume.

After the water washing step, the fuel shall be clear and bright according to ASTM-D-4176.

c) IFT adjustment<sup>1)</sup>

Add to the treated fuel an adequate amount of Monoolein, CAS-No. 25 496-72-4 or CAS-No.111-03-05, to obtain the specified IFT as defined 5.1.1:

Test fuel F2.1, high IFT fuel:  $22 \pm 2$  mN/m, Test fuel F2.2, low IFT fuel:  $13 \pm 2$  mN/m.

For reusing the fuel after a water separation test, the instructions below shall be followed:

d) After each test readjust IFT by adding additional Monoolein.

e) After 10-15 tests re-clay treat the fuel to 35 IFT and readjust the fuel to required IFT with Monoolein.

To check the fuel compliance after re-clay treating, a short duration test with a reference material/element shall be done. It is recommended to choose a reference filter in the range of 85 %–95 %

1) User Precaution: Monoolein depositing in test systems has been observed, especially when operating in the lower portion of the F.2.2 IFT range. The solubility of Monoolein in clay treated water washed fuel without free water is greater than solubility limit once free water is introduced for the filter test. When Monoolein drop out does occur deposits appear white & have a paste or wax like composition. Deposits have been reported in various portions of test benches. Common locations are reservoir tanks and cleanup filters. The minimum IFT level achievable with fuel stability can be higher than the minimum value specified for F2.2.

of water separation efficiency. The procedure shall be repeated from step e). Once reference test values become unstable the fuel shall be changed and the procedure shall be repeated from step a).

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## Annex B (normative)

### Water emulsifying device

The required characteristics of the DSD can be achieved by various water emulsifying concepts.

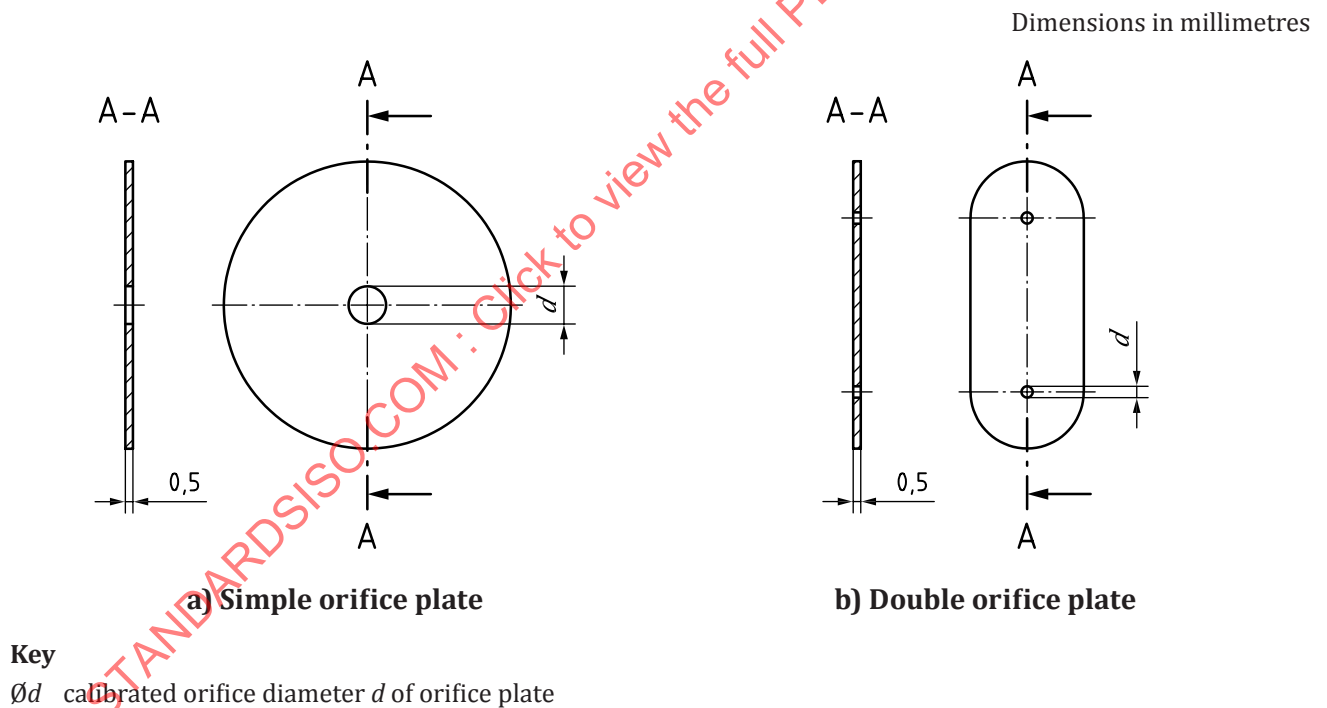
Jet emulsification with a calibrated orifice was found as one suitable concept. This is described in [B.1](#) – [B.2](#) in more detail.

#### B.1 Calibrated orifice design details

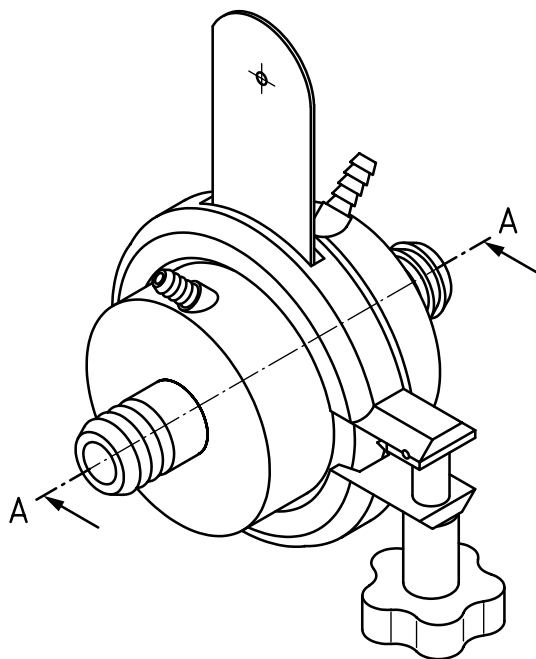
Details of the calibrated orifice (9) are shown in [Figures B.1](#) to [B.4](#).

The orifice itself (calibrated diameter  $d$ ) shall be free from any burrs.

The orifice up and downstream side shall be labelled and the mounting orientation shall be considered for emulsification and calibration.



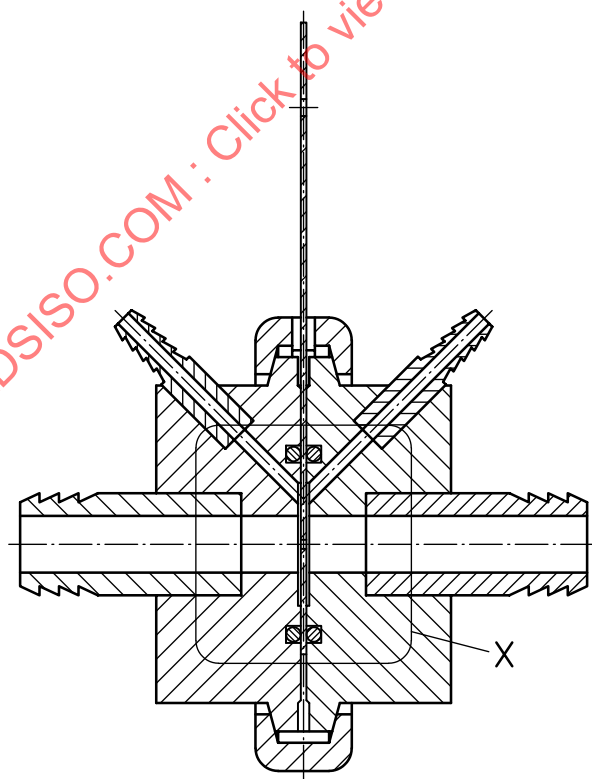
**Figure B.1 — Orifice plates with calibrated orifice diameters  $d$**



Key

A-A See [Figure B.3](#).

**Figure B.2 — Water emulsifying device (orifice plate holder) with double orifice plate and pressure taps (measurement of differential pressure  $\Delta p_0$ )**

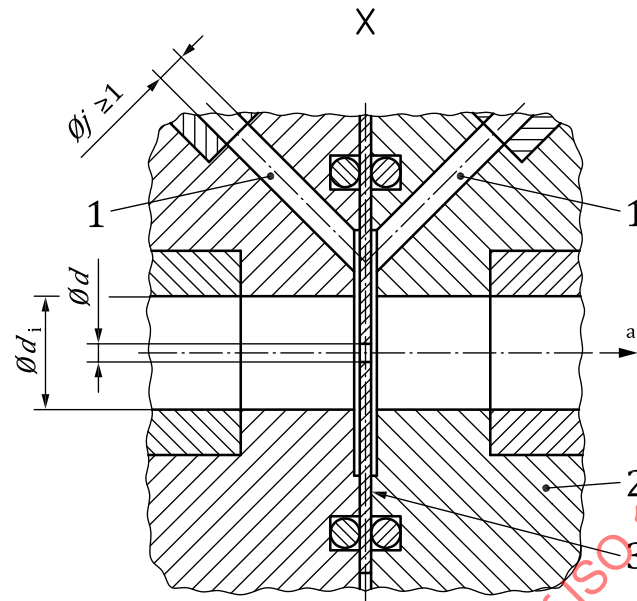


Key

X See [Figure B.4](#).

**Figure B.3 — Water emulsifying device (orifice plate holder) (cut A-A of [Figure B.2](#))**

Dimensions in millimetres

**Key**

- 1 pressure transmitting bores (to pressure tap connections for differential pressure  $\Delta p_0$  measurement)
- 2 orifice plate holder
- 3 orifice plate
- $\varnothing d$  calibrated orifice diameter of orifice plate
- $\varnothing d_i$  inner pipe diameter, (adjusted to the required flow velocity according to [5.3.2.22](#))
- $\varnothing j$  diameter of pressure transmitting bores and of pressure tap bores
- a Flow of fuel/water mixture.

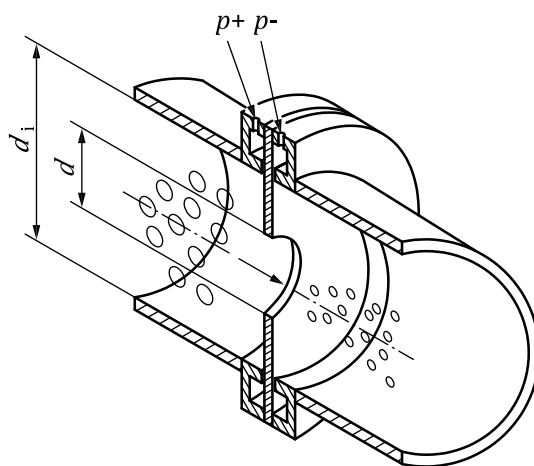
**Figure B.4 — Detail X of [Figure B.3](#): Measurement of differential pressure  $\Delta p_0$  at the orifice plate**

## B.2 Calibrated orifices concept

Because of their simplicity, orifice plates with calibrated orifice diameters  $d$  have been used to mix, under known and repeatable conditions, the water with fuel to generate droplets of known size  $D$ . The orifice plates are made of  $0,5 \pm 0,05$  mm thick stainless steel discs with a central hole of variable inside diameter  $d$  (orifice diameter). Typical stainless steel orifice plates with calibrated orifice diameters  $d$  (simple and double orifice plates) are shown in [Figure B.1](#).

The energy dissipated by a liquid flowing through the calibrated orifice diameter  $d$  is quantified by its pressure loss (or differential pressure  $\Delta p_0$  measured across the orifice plate) provided measured adequately, e.g. as shown on [Figure B.5](#).

Typical curves which are representing the correlation between differential pressure  $\Delta p_0$  and fuel flow rate  $Q_C$  are shown in [Figure B.6](#).

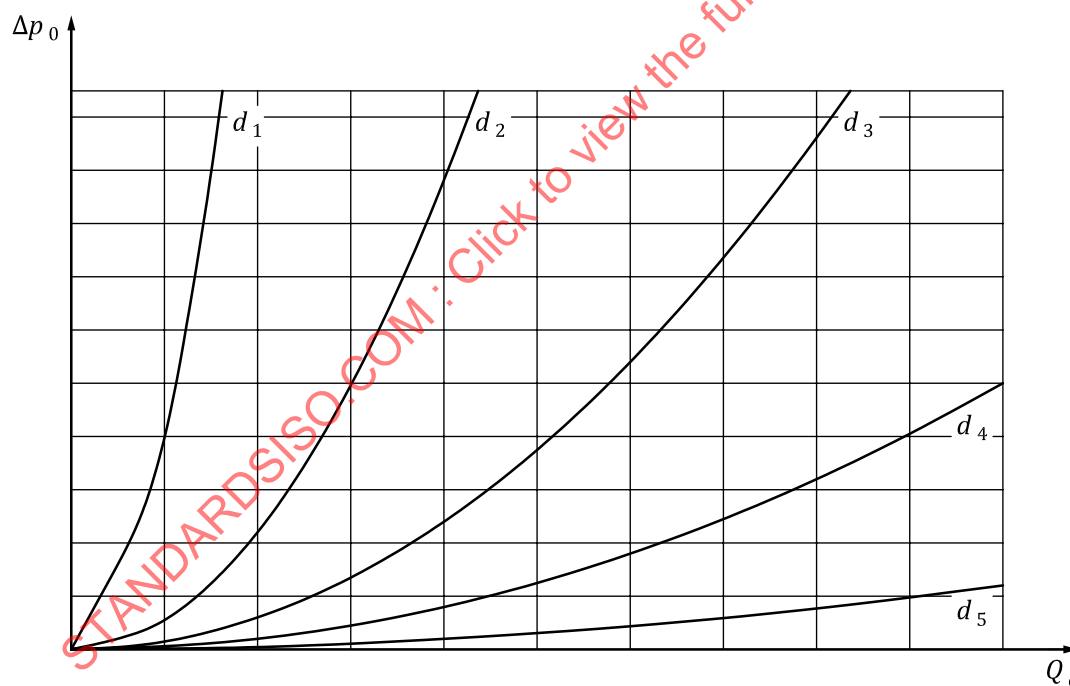
**Key**

$p+, p-$  differential pressure  $\Delta p_0$  measuring points at the orifice plate (see Figure B.4, detail X)

$\varnothing d_i$  inner pipe diameter

$\varnothing d$  calibrated orifice diameter of orifice plate

**Figure B.5 — Energy dissipated by a liquid flowing through the calibrated orifice diameter  $d$  of an orifice plate**

**Key**

$Q_c$  fuel flow rate (for calibration of orifice plates), in litres per hour (l/h)

$\Delta p_0$  orifice differential pressure, in hectoPascals (hPa)

$d_i$  calibrated orifice diameter of orifice plate,  $d_1 < d_2 < d_3 < d_4 < d_5$

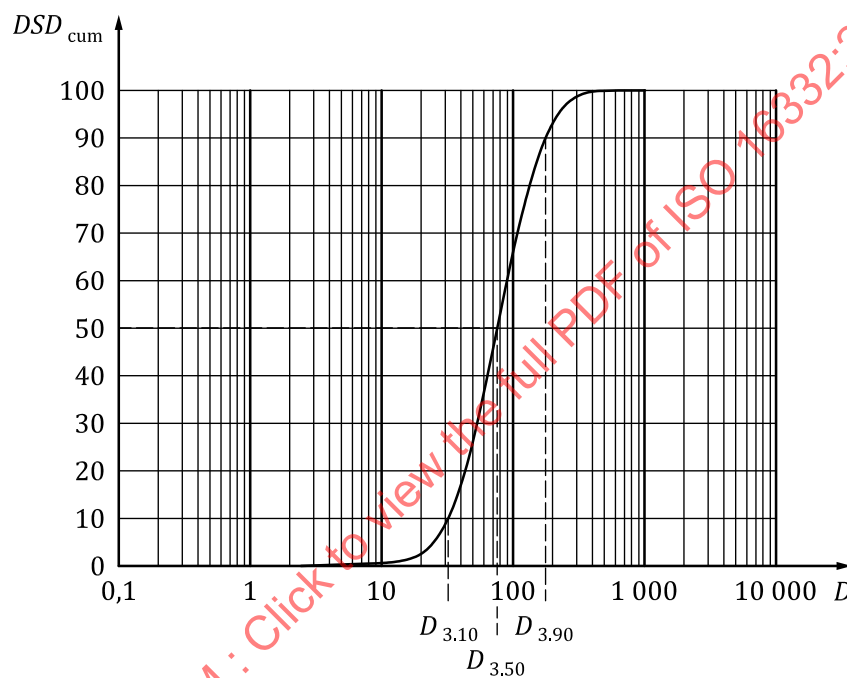
NOTE The curves of Figure B.6 are given for information only, since they strongly depend on the actual characteristics of the test fuel used.

**Figure B.6 — Typical curves of differential pressure  $\Delta p_0$  through calibrated orifice diameter  $d$**

### B.3 DSD generated by orifices

The DSD is the water droplet size distribution, which is created by the water emulsifying device (orifice), immediately after being injected, defined by the volume based distribution curve whereby the distribution curve is described by three characteristic values:

- $D_{3,10}$  is the droplet size  $D$  that only 10 Vol.-% of the droplets underpass.
- $D_{3,50}$  is the droplet size  $D$  which separates the droplets in two identical fractions, 50 Vol.-% larger and 50 Vol.-% smaller; i.e.  $D_{3,50}$  is the median diameter of the volume based droplet size distribution DSD.
- $D_{3,90}$  is the droplet size  $D$  that only 10 Vol.-% of the droplet population overpass (90 Vol.-% underpass).



#### Key

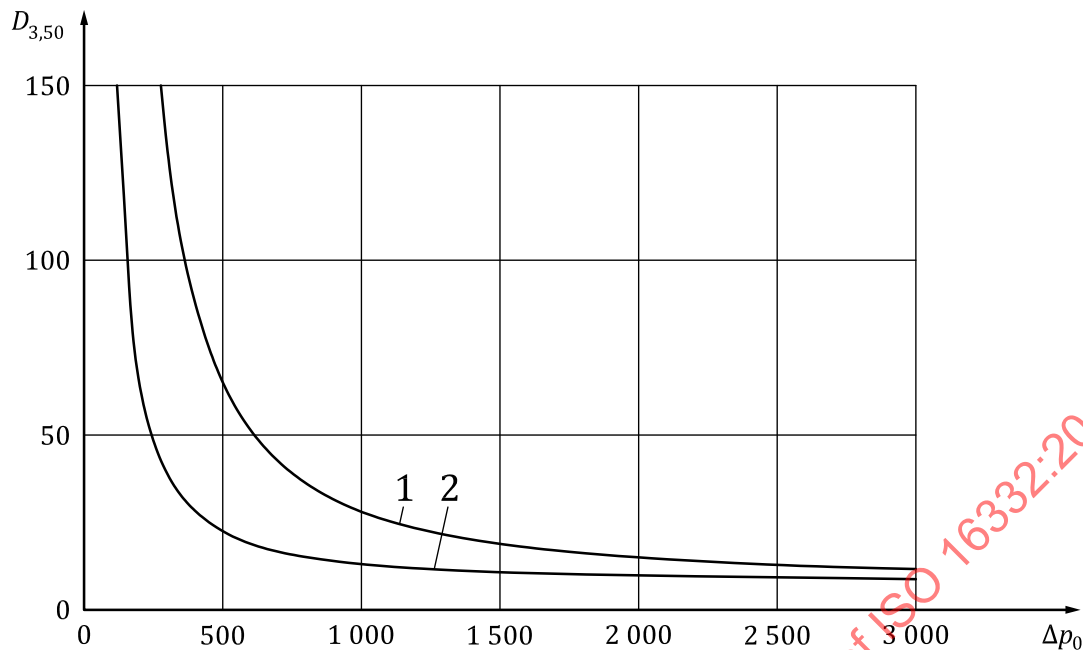
|                    |                                                 |
|--------------------|-------------------------------------------------|
| $D$                | droplet size ( $\mu\text{m}$ )                  |
| $DSD_{\text{cum}}$ | cumulative droplet size distribution, in Vol.-% |

**Figure B.7 — Example for the characterization of a droplet size distribution curve for the determination of  $D_{3,10}$ ,  $D_{3,50}$  and  $D_{3,90}$  ( $DSD_{\text{cum}}$ )**

NOTE The curve represents the cumulative droplet size distribution  $DSD_{\text{cum}}$  (i.e. percentage of droplets smaller than the indicated droplet size  $D$ ).

It has been shown with various fuel/water mixtures in a number of conditions that the average droplet size  $D_{3,50}$ , in  $\mu\text{m}$ , of the DSD is linked to the differential pressure  $\Delta p_0$  in hPa, across an orifice. There are a various number of unknown parameters which have an influence on the DSD. If no online droplet size measurement is used, for each combination of orifice, test fuel and temperature a calibration curve is mandatory.

[Figure B.8](#) shows typical curves of  $D_{3,50}$  vs.  $\Delta p_0$  for two different test fuels.

**Key**

- 1 fuel Quality A
- 2 fuel Quality B
- $\Delta p_0$  orifice differential pressure, in hectoPascals (hPa)
- $D_{3,50}$  average droplet size, in micrometers ( $\mu\text{m}$ )

**Figure B.8 — Typical curves of average droplet size  $D_{3,50}$  vs. orifice differential pressure  $\Delta p_0$  for two different fuels**

#### B.4 Water emulsifying device calibration procedure

This procedure shall be applied for each combination of orifice, batch of test fuel (independent of it being F1 or F2 fuel), flow rate and temperature.

The calibration shall be performed in the water emulsifying device, in which the orifice will be used for testing.

The calibration shall be performed with the water injection device, which will be used for testing,

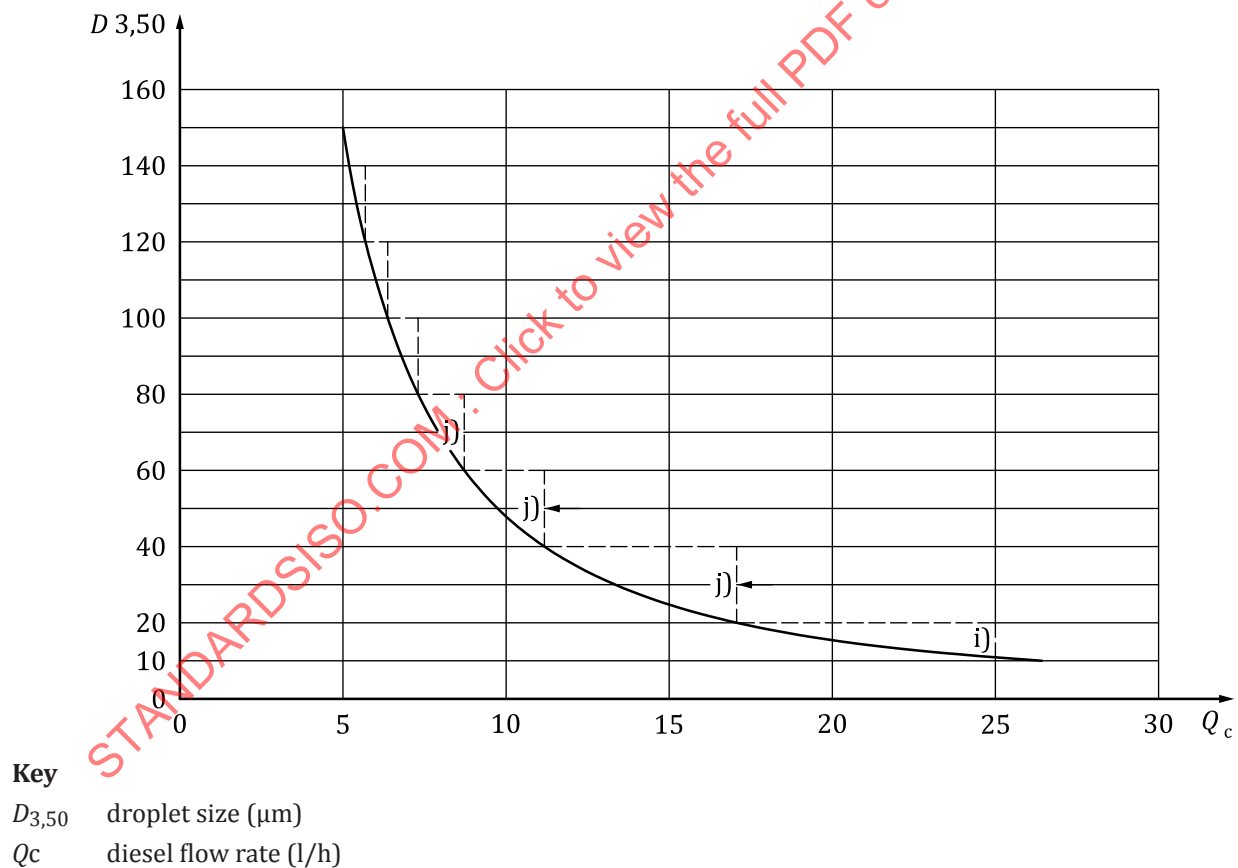
The purpose of the calibration procedure is to get a calibration curve  $D_{3,50}$  vs. flow rate  $Q_c$  for a certain combination of orifice, fuel and temperature according to [Figure B.9](#).

Perform the calibration procedure in the following order:

- a) Fill a sufficient volume of fuel (at least 15 l) for which the calibration has to be performed into the completely emptied fuel circuit.
- b) Install the orifice plate to be calibrated into the water emulsifying device (9) with the correct mounting orientation (up- and downstream side).
- c) Utilize the by-pass line (21) and the clean-up system (19) for the validation procedure.
- d) Circulate the fuel at the specified temperature and at a flow rate  $Q_c$  creating a differential pressure  $\Delta p_0$  of appr. 2,5 bar. Let the system stabilize.
- e) Start the water injection with an injection flow rate corresponding to a water concentration  $c_{U,up}$  of 1 500 ppm.

- f) Measure the  $D_{3,50}$  with an online droplet size distribution measurement device (20).
- g) Adjust the fuel flow rate  $Q_c$  until the  $D_{3,50}$  value is stabilized at 10  $\mu\text{m}$ . The water concentration  $c_{U,up}$  shall be kept constant at 1 500 ppm.
- h) Record the resulting  $D_{3,50}$  value, the orifice differential pressure  $\Delta p_0$ , the flow rate  $Q_c$  and the temperature.
- i) Reduce the fuel flow rate  $Q_c$  to a corresponding  $D_{3,50}$  of 20  $\mu\text{m}$ . The water concentration  $c_{U,up}$  shall be kept constant at 1 500 ppm.
- j) Repeat step i) in corresponding  $D_{3,50}$  steps of 20  $\mu\text{m}$  until a value of 150  $\mu\text{m}$  or the minimum test flow rate is reached.
- k) Record all data in a calibration protocol, including the location and date of calibration, fuel quality used for calibration, calibration temperature, orifice identification, identification of the water emulsifying device, identification of the online droplet size distribution measurement device.
- l) Label the orifice plate with a link to the calibration protocol.

NOTE The maximum allowed water concentration depends on the limits of the applied DSD measurement instrument.



**Figure B.9 — Typical orifice calibration curve, including calibration sequence**

## Annex C (normative)

### Conditions and parameters for the determination of the interfacial tension according to ISO 9101, drop volume method

In this Annex, the parameters for determination the interfacial tension between water and test fuel, are defined.

Interface age: 10 s

Capillary radius: <1,39 mm

NOTE The actual capillary radius is a setup parameter for the utilized IFT measurement device and will have an impact on the calculated IFT value.

Capillary geometry: According to ISO 9101

Temperature:  $23 \pm 1$  °C

For each test a new diesel sample shall be used.

The ratio of  $V_{\text{water}} / V_{\text{Diesel}}$  shall not exceed a value of 25 %.

For the determination of the interfacial tension (IFT) between fuel and water, deionised (DI) water with a surface tension of 70 mN/m to 72 mN/m at 10 s shall be used.

NOTE The defined capillary radius was proven to reach an improved precision level according to 7.2 of ISO 9101. Other capillary radii can be used if it is proven that the measurement accuracy is not negatively influenced.

## Annex D (normative)

### Validation of the sampling procedure for Karl Fischer titration and centrifuge

#### D.1 Karl Fischer titration (manual sampling)

##### D.1.1 Primary sampling procedure

The following procedure shall be executed for the upstream and downstream sampling points.

- a) Open the sampling valves at sampling points (12) and (18).
- b) Flush the volume between the sampling points (12) and (18) and discard in an appropriate repository.
- c) Immediately open the sampling bottles (prepared as described in [5.2.2](#)) and fill approximately 50 ml; the sampling time shall be between 5 s and 10 s.
- d) Close the sampling bottles directly after filling and label the sample.

##### D.1.2 Secondary sampling procedure

The sampling procedure shall be according to ISO 12937.

The volume of the samples to be analysed shall be sufficient to dose at least 50 µg of water into the titration cell.

##### D.1.3 Secondary sampling procedure validation

The validation of the secondary sampling procedure shall be done according to ISO 12937, Verification of mixing conditions.

Accept the validation test only, if the precision according to ISO 12937 is fulfilled.

#### D.2 Karl Fischer titration (automated sampling)

The validation of an automated KF system shall be done by the sample concentrations obtained according to [8.4.4](#).

#### D.3 Centrifuge

Fill a clean centrifuge tube from the sampling point (18) according to the flow rate.

Centrifuge the tube for 10 min at approximately 1 500 g centrifugal force ( $g$  = gravitational constant).

Remove the tube from the centrifuge and read off the volume of water and the total sample volume indicated on the scale of the centrifuge tube.

Based on these two volumes calculate the water concentration.

Accept the validation test only, if the measured water concentration is according to [Table 1](#).

## **Annex E** **(normative)**

### **Determination of the concentration $c_s$ of dissolved water in saturated fuel**

In this Annex, the procedure for determination of the concentration  $c_s$  at the saturation level of dissolved water in the test fuel is defined.

- Fill approximately 75 ml of test fuel into a clean, dried 100 ml sample bottle.
- Insert a magnetic stirrer.
- Inject approximately 25 ml water gently underneath the test fuel using a syringe with a long needle, gently fill the bottle completely with test fuel and close it with a rubber diaphragm cap.
- Put the bottle into a temperate water bath at  $23 \pm 2$  °C; do not shake the bottle, to ensure that the fuel/water interface is not agitated.
- Start the magnetic stirrer at the lowest speed ( $\leq 60$  rpm).

After stirring for at least 5 h take a sample with a clean, dried syringe through the rubber diaphragm from the top of the bottle. Analyse the sample with a Karl Fischer titrator validated according to [D.1](#), take three readings. The water saturation level  $c_s$  is the average of the three readings.

## Annex F (informative)

### Typical fuel/water separator test report

A typical test report sheet is formed and composed as follows.

The test report should include additional information presented in graphical form, as illustrated in [Figure F.1](#).

**Fuel/water separator efficiency test report according to ISO 16332.**

#### TEST IDENTIFICATION

|            |                |                      |
|------------|----------------|----------------------|
| TEST DATE: | TEST LOCATION: | TEST IDENTIFICATION: |
| TEST TIME: | OPERATOR:      | PROJECT:             |

#### FUEL/WATER SEPARATOR IDENTIFICATION

|                                                       |                     |
|-------------------------------------------------------|---------------------|
| FUEL/WATER SEPARATOR IDENTIFICATION:<br>HOUSING TYPE: | MANUFACTURING DATE: |
|-------------------------------------------------------|---------------------|

#### TEST FUEL

|                                                         |                      |  |                |  |
|---------------------------------------------------------|----------------------|--|----------------|--|
| Fuel category F1 ☐ F2.1 ☐ F2.2 ☐                        |                      |  |                |  |
| IFT (10 s), measured                                    | [mN/m]               |  | Identification |  |
| IFT (60 s), measured                                    | [mN/m]               |  | Supply source  |  |
| Water separability                                      | [s]                  |  | Fuel Type      |  |
| Water saturation level $c_S$                            | [ppm]                |  | Batch No.      |  |
| Base water concentration $c_B$<br>after preconditioning | [ppm]                |  | Delivery date  |  |
| Bio diesel content                                      | [%]                  |  |                |  |
| Density                                                 | [g/cm <sup>3</sup> ] |  |                |  |
| Kinematic viscosity                                     | [mm <sup>2</sup> /s] |  |                |  |
| CFPP                                                    | °C                   |  |                |  |

#### OPERATING CONDITIONS

|                   |                                      |             |                                             |     |
|-------------------|--------------------------------------|-------------|---------------------------------------------|-----|
| TEST WATER        | Surface tension:                     | 72 ± 2 mN/m |                                             |     |
| TEST SYSTEM       | Test flow rate $Q_T$ :               | l/h         | Volume of test fuel:                        | l   |
|                   | Calibrated orifice<br>diameter $d$ : | mm          | Test fuel temperature $T$ :                 | °C  |
|                   | droplet size $D_{3,50}$ :            | µm          | droplet size $D_{3,90}$ :                   | µm  |
|                   |                                      |             | droplet size $D_{3,10}$ ,<br>(informative): | µm  |
| INJECTION SYSTEM: | Water injection flow rate<br>$Q_W$ : | ml/min      | Water concentration $c_{U,up}$<br>(calc.):  | ppm |

TEST RESULTS

|                                                                       |   |    |    |    |    |    |    |    |    |    |
|-----------------------------------------------------------------------|---|----|----|----|----|----|----|----|----|----|
| Test time $t$ (min)                                                   | 0 | 10 | 20 | 30 | 40 | 50 | 60 | 70 | 80 | 90 |
| Differential pressure $\Delta p_F$ at test fuel/water separator (hPa) |   |    |    |    |    |    |    |    |    |    |
| Differential pressure $\Delta p_O$ at orifice plate (hPa)             |   |    |    |    |    |    |    |    |    |    |
| Downstream water concentration $c_{U,down,i}$ (ppm)                   |   |    |    |    |    |    |    |    |    |    |
| Water separation efficiency $\eta_i$ (%)                              |   |    |    |    |    |    |    |    |    |    |

Test duration  $t_{test}$ : [min]

Average downstream water concentration  $c_{av,down}$ : [ppm]

Average water separation efficiency  $\eta_{av}(test\ fuel, C_{U,up}, D_{3,50})$  [%]

Total volume of water drained from test fuel/water separator: [l]

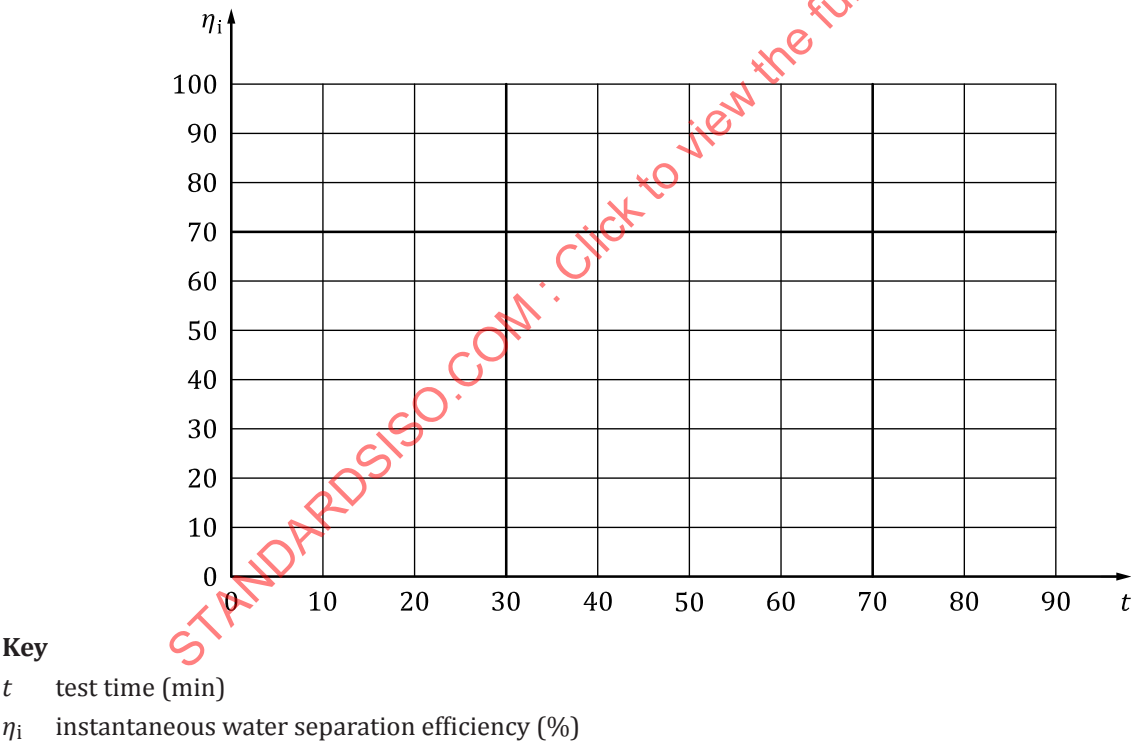


Figure F.1 — Typical test diagram

## Annex G (informative)

### Report round robin

#### Summary of international inter-laboratory trial (“round robin”) to validate ISO 16332 protocol

The following are conclusions drawn from the ISO 16332 round robin studies conducted in 2014 and 2016. Data and comments are given on each major step and a summary is included at the end.

The objectives of the round robin studies were:

- evaluation of the feasibility of the test procedure;
- evaluation of the sensitivity of the procedure regarding filter performance, with respect to water separation efficiency; and
- evaluation of the comparability between laboratories.

#### Round robin test conditions

ISO 16332 round robin 2014:

The 2014 round robin test conditions were defined to ensure an objective evaluation of the procedure itself, including the validation procedure in [Clause 8](#) and the test procedure in [Clause 9](#), and to avoid variation caused by influences which are not related to the procedure (such as fuel quality).

|                            |                                                                                                                                                                                                                            |
|----------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Filters to be tested:      | Filter 1: “high efficiency filter” out of one production batch<br>Filter 2: “medium efficiency filter” out of one production batch                                                                                         |
| Test fuel:                 | Fuel F1 according to <a href="#">5.1</a> : Total EN 590 B7 out of one production batch, with an Interfacial tension (IFT) after 10 s of 18 mN/m, as an average, measured value of all participating laboratories           |
| Flow rate:                 | 150 l/h                                                                                                                                                                                                                    |
| Water concentration:       | 1 500 ppm                                                                                                                                                                                                                  |
| Droplet size distribution: | The orifices to create the defined droplet size distribution ( $D_{3,50} = 10 \mu\text{m}$ ) were all produced and calibrated by one lab.                                                                                  |
| Water detection system:    | For the determination of the water concentration, each lab used direct KF titration. In addition, an inline water concentration measurement device according to <a href="#">5.3.2.21</a> was used and reported by one lab. |

ISO 16332 round robin 2016:

The 2016 ISO 16332 round robin study was performed in addition to evaluate the procedure using test fuel F2.2, including the process to produce the fuel described in Annex A.

The 2016 ISO 16332 round robin test conditions were the same as for the ISO 16332 2014 conditions, with the following deviations:

- Test fuel: Fuel F2.2 according to 5.1 with a nominal IFT after 10 s of  $14 \pm 1$  mN/m Each lab has itself produced the test fuel, following the procedure described in Annex A.
- Droplet size distribution: The orifices to create the defined droplet size distribution ( $D_{3,50} = 10 \mu\text{m}$ ) were chosen by the laboratories themselves.

Validation procedures according to Clause 8

The validation procedures reveal the effectiveness of the test circuit and the water injection system in maintaining the required DSD and water concentration.

Validation of the emulsifying device, according to 8.3

Four laboratories participated, three laboratories using laser diffraction instruments from the same supplier, one lab using a laser diffraction instrument from a different supplier. The hydraulic setups for the laser diffraction instruments were different.

- Acceptance criteria for the orifice validation:  $D_{3,50} = 10 \mu\text{m} \pm 15 \%$  at 150 l/h  
 $D_{3,90} \leq 30 \mu\text{m}$

Figures G.1 and G.2 show the orifice calibration curves obtained for  $D_{3,50}$  and  $D_{3,90}$ .

The droplet sizes and calibration curves of all laboratories are comparable and within the tolerance defined in Table 1 of this document. The validation is confirmed.

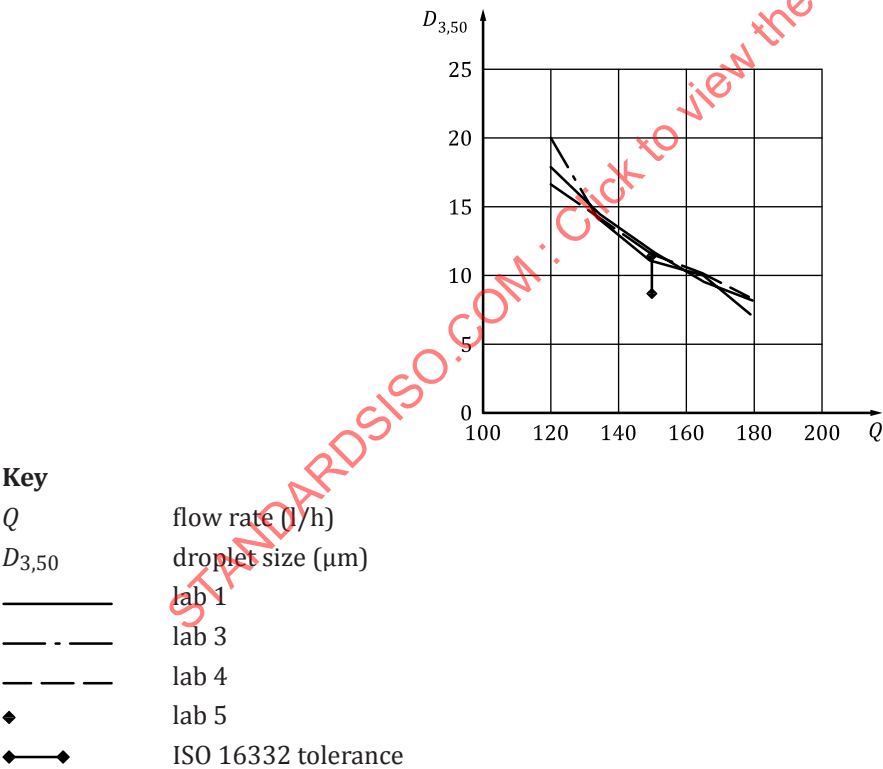


Figure G.1 — Orifice calibration curves (I)

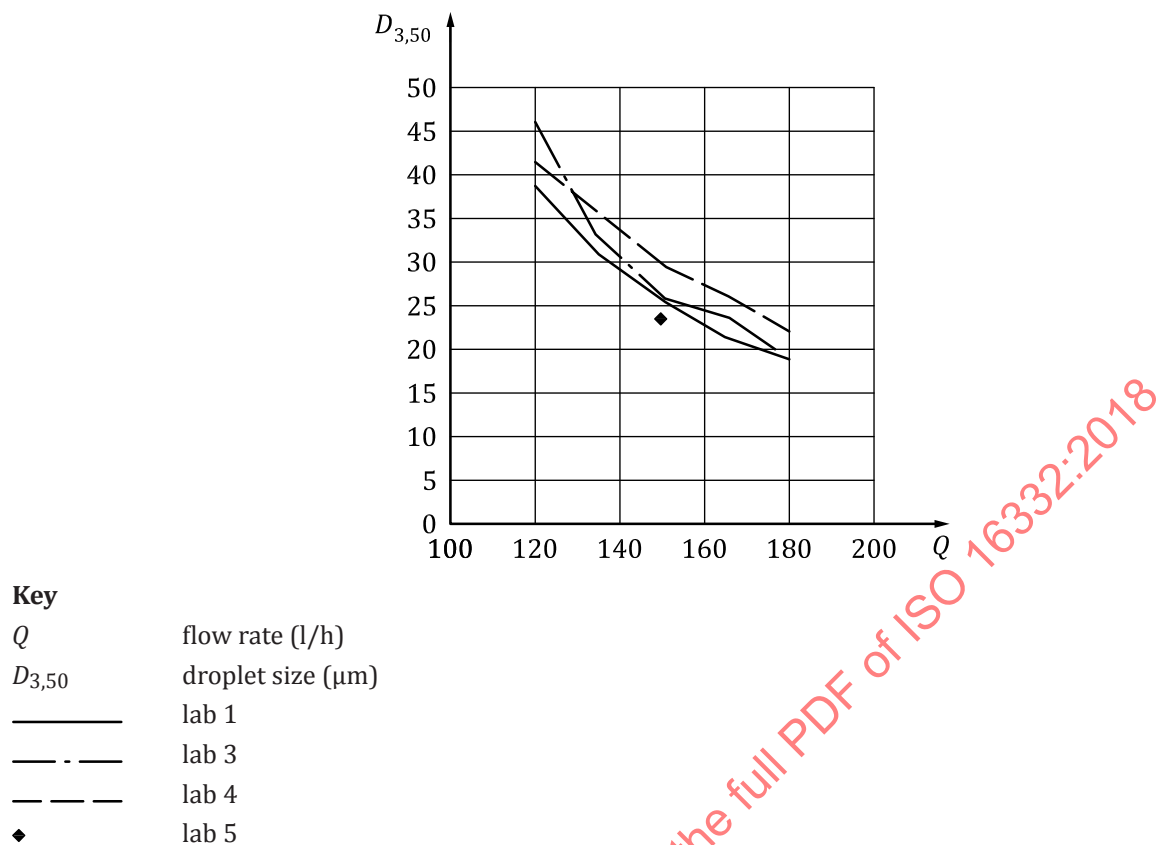


Figure G.2 — Orifice calibration curves (II)

#### Validation of the filter test circuit and the water injection system, according to 8.4

Figures G.3 and G.4 show the validation curves for the downstream water concentration  $c_u$  at sampling point (18); 5 participating laboratories, 2 laboratories with inline sensor.

Acceptance criterion:  $c_u \in 1\,500 \pm 100$  ppm

The reported downstream water concentration curves are fulfilling the acceptance criteria, one lab at a higher injection rate with a nominal downstream water concentration of 1 550 ppm, due to test rig limitations.