



SLOVENSKI STANDARD
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Automotive diesel fuels - Determination of filtrability - AGELFI method

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CEN REPORT
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CEN BERICHT

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ICS

English version

**Automotive diesel fuels - Determination of filtrability - AGELFI
method**

Combustibles pour moteurs diesel (gazole) - Détermination
de la filtrabilité - Méthode AGELFI

This CEN Report was approved by CEN on 1 December 1999. It has been drawn up by the Technical Committee CEN/TC 19.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

Foreword

This CEN Report has been prepared by Technical Committee CEN/TC 19 "Petroleum products, lubricants and related products" the Secretariat of which is held by the Netherlands Standardization Institute (NNI).

1. Scope

This CEN Report describes a method of test which can be used to determine the AGELFI filtrability temperature of automotive diesel fuels.

The method described is applicable to distillate fuels, including those containing flow-improving or other additives, intended for use in automotive diesel engines and may be used to estimate the lowest temperature at which a fuel will give trouble-free operation in a diesel-engined vehicle.

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

2. Normative references

This CEN Report incorporates, by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

EN ISO 3170, *Petroleum liquids — Manual sampling.*

EN ISO 3171, *Petroleum liquids — Automatic pipeline sampling.*

EN ISO 3696, *Water for analytical laboratory use - Specification and test methods.*

3. Definitions

For the purposes of this Report, the following definition applies:

3.1

AGELFI filtrability temperature

lowest temperature at which a given volume of fuel, cooled under standardized conditions, will pass through a standardized filtration device in a specified time.

4. Principle

A test portion of fuel is cooled under controlled conditions and, at a selected temperature, a fraction corresponding to one sixth by mass of 90 % of the total mass of the test portion is filtered through a 25 μm paper filter under controlled pressure. The procedure is repeated at 1 $^{\circ}\text{C}$ intervals below the first test temperature, using a new fraction of the test portion for each filtration, until the first temperature at which the time required to filter a fraction exceeds 5 min is reached, this is the plugging temperature.

The AGELFI filtrability temperature is recorded as being 1 $^{\circ}\text{C}$ above the determined plugging temperature.

5. Reagents and materials

Use only reagents of recognized analytical grade and water conforming to grade 3 of EN ISO 3696.

5.1 Solvent, technical grade n-heptane or equivalent.

5.2 Filter paper, 25 μm retention.

5.3 Lintless filter paper, 5 ± 1 μm retention.

6. Apparatus

Usual laboratory apparatus and glassware, together with the following:

6.1 AGELFI apparatus, comprising a pneumatic unit and a measuring unit, connected to a cryostat and monitored by a micro-computer. The components of the apparatus are detailed in 6.2 to 6.14 and a general arrangement of the apparatus is shown in figure 1.

6.2 Pneumatic unit, (see figure 2), comprising a vacuum/pressure pump, with the characteristics shown in figure 3, a vacuum system (reservoir, gauge, solenoid valve) for suction of the sample and a pressure system (high pressure reservoir, regulated pressure reservoir, gauges, solenoid valves) for filtration of the sample.

6.3 Measuring unit, comprising four identical test chambers which allow fully independent and simultaneous testing of four different fuel samples. Each test chamber includes an enclosure for the test tube, positioned horizontally, with dimensions and shape such that the test tube is held in a stable position. A general arrangement of a suitable test chamber is shown in figure 4.

6.4 Regulation unit, for the test tube enclosure, having a temperature control capacity ranging from + 45 $^{\circ}\text{C}$ to -40 $^{\circ}\text{C}$, comprising a heating element, a cooling system (by circulation of refrigerant) and a turbine (to ensure the homogeneous distribution of temperature throughout the enclosure).

6.5 Pneumatic jack, to tilt the test tube in the enclosure.

6.6 Temperature probe, to measure the chamber temperature.

6.7 Filtration system, as shown in in figures 5, 6 & 7, comprising a cylindrical clear glass test tube of internal diameter 22 mm, external diameter 26 mm and height 270 mm with a threaded brass tip. The test tube is fitted with a piston of diameter 22 mm and length 36 mm, made from Voltalef with two nitrile rubber O-rings (R = 13; 70 Shore), and a polytetrafluoroethylene stopper with two nitrile rubber O-rings (R = 13; 70 Shore).

6.8 Filter assembly, made up of an aluminium filter holder with a threaded internal cavity (the test tube cap screwed on to its threaded tip), a stainless steel filter grid and its corresponding O-ring, a paper filter (diameter 26 mm, porosity 25 μm), a stainless steel filter restrictor (external diameter 26 mm, central hole diameter 10 mm, thickness 0,4 mm), and a platinum resistor temperature probe with an accuracy $\pm 0,1$ $^{\circ}\text{C}$ (PT 100), and a corresponding O-ring (R = 4; 70 Shore).

6.9 Sample outlet tube, silicone, internal diameter 2 mm, external diameter 4 mm, allowing aspiration and recovery of the sample.

6.10 Pneumatic jack, made from Voltalef, positioned at the test tube cap outlet, to either block or allow the passage of the substance.

6.11 Beaker, 150 ml, with a protective lid and stopper.

6.12 Tray, for sample weighing.

6.13 Cryostat, having the following characteristics:

- Operating temperature range ($^{\circ}\text{C}$): [-70 to 100]
- Temperature setting/resolution ($^{\circ}\text{C}$): digital/0,1
- Fine settings of temperature, range/solution ($^{\circ}\text{C}$): $\pm 0,2/0,01$
- Temperature control at -10 $^{\circ}\text{C}$ ($^{\circ}\text{C}$): [0,02 to 0,05]
- Effective cooling capacity at 0 $^{\circ}\text{C}$ (kW): 0,32
- Simplex pump:
 - flow rate at pressure head 0, (pump capacity) with connector 11/13 (l/min): 11/15
 - max. discharge pressure (bar) 0,25

6.14 Micro-computer, having the following characteristics:

- AT 100 % IBM compatible
- VGA screen
- 20 Mb minimum HD
- 640 Kb minimum Ram
- free serial port RS232C (COM 1 or COM 2)
- floppy disk drive 3.5 in high density
- centronics parallel port
- printer

7. Sampling

Unless otherwise specified in the commodity specification, samples shall be taken as described in EN ISO 3170 or EN ISO 3171, and/or in accordance with the requirements of national standards or regulations for the sampling of the product under test.

8. Preparation of the test sample

Filter approximately 120 ml of the sample, at ambient laboratory temperature but at not less than 15 °C and at least 10 °C above the cloud point, through dry filter paper (5.3).

Where a fuel has previously been subjected to temperatures at or below its cloud point, additional requirements for test sample preparation are required, these are given in annex A.

9. Preparation of apparatus

9.1 Prepare the AGELFI apparatus (6.1) in accordance with the manufacturer's instructions. In addition, carry out the following calibration checks:

- a) Verification of correct cooling bath temperature (at least 4 times per year)
- b) Measurements of the temperature probe, the pressures and the weighing unit (once a month).

NOTE: This can be achieved by using the subsection "Measured Display" of the section "Service" in the main group menu of the analyser.

9.2 Before each test, wash the filter holders, the grids, the restrictors, the outlet tubes, the beakers and the test tubes with solvent (5.1) and dry them in an air stream. Ensure that all parts are clean and dry, and leave at room temperature for 4 h to prevent solvent attack of the O-rings. Check the stainless steel grids and replace these if damaged.

9.3 Before each test, wash the pistons and their O-rings. Replace these if any problem occurs during filling of the tube.

10. Procedure

10.1 Place the stainless steel grid, the filter paper (5.2), the filter restrictor and the O-ring successively into the aluminium filter holder and screw the holder onto the test tube. Insert the piston, lubricated with the fuel to be tested, into the test tube and place the stopper at the open end of the test tube. Position the lubricated outlet tube and the temperature probe into the tube cap.

10.2 Place the test tube, with its cap in a stable position, in the enclosure and secure to the stand. Place a beaker, filled with the fuel to be tested, on the tray and arrange the outlet tube so that it is dipping into the fuel; position the lid and stopper.

10.3 Choose the appropriate cooling program (see annex B) from the list of already recorded programs.

NOTE: If there is no suitable program already recorded, create a new programme by using the subsection "Creation/Modification" of the section "Programs" in the main group menu.

10.5 Start the test by validating the subsection "Start of test" of the section "Tests" in the main group menu and allow the filling of the tube, regulation of the temperature and pressure, filtration and warming sequences to run automatically.

NOTE: The following program of filtration is recommended, where CP is the approximate cloud point of the fuel under test:

Allocate two test chambers of the apparatus to the same sample.

In the first chamber, carry out the first filtration at a temperature corresponding to CP-3 °C, the second filtration at CP-4 °C, the third titration at CP-5 °C, the fourth filtration at CP-6 °C, the fifth titration at CP-7 °C and the sixth and last filtration at CP-8 °C.

In the second chamber, carry out the first filtration at a temperature corresponding to CP-9 °C, the second filtration at CP-10 °C, the third filtration at CP-11 °C, the fourth filtration at CP-12 °C, the fifth filtration at CP-13 °C, and the sixth and last filtration at CP-14 °C.

If plugging occurs at the temperature corresponding to CP-3 °C, a further test should be carried out with filtrations ranged from CP to CP-5 °C.

If no failure has occurred at the temperature corresponding to CP-14 °C, a further test should be carried out with filtrations ranged from CP-15 °C to CP-20 °C, and so on until plugging occurs.

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10.6 Repeat 10.1 to 10.5 for each of the other three test chambers.

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10.7 At the completion of each test procedure, the computer will produce a report sheet, quoting either the plugging temperature, if plugging occurred, or the last temperature of filtration, if no plugging occurred.

11. Expression of results

Report the AGELFI filtrability temperature as 1 °C above the plugging temperature (see 10.6).

12. Precision

12.1 Repeatability

The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the 2 °C only in one case in twenty.

12.2 Reproducibility

The difference between two single and independent results, obtained by different operators working in different laboratories on identical test material, would in the long run, in the normal and correct operation of the test method, exceed 3,9 °C only in one case in twenty.

13. Test report

The test report shall contain at least the following information:

- a) the type and identification of the product under test;
- b) a reference to this CEN Report;
- c) the sampling procedure used (see clause 7);
- d) the results of the test (see clause 11);
- e) any deviation from the procedure described;
- f) the date of the test.

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