SLOVENSKI STANDARD
kSIST-TP FprCEN/TR 17544:2020

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Goriva za motorna vozila - Poročilo o študijah o nagnjenosti k blokiranju filtra za hladno vlaženje (CS-FBT) metilnega estra maščobnih kislin (FAME) kot mešanice za dizelsko gorivo in o dizelskem gorivu, ki vsebuje do 30 % (V/V) FAME

Automotive fuels – Report on studies done on cold soak filter blocking tendency (CS-FBT) on fatty acid methyl ester (FAME) as blend component for diesel fuel, and of diesel fuel containing up to 30 % (V/V) of FAME

Kraftstoffe - Bericht über Studien zur cold soak filter blocking tendency (CS-FBT) an Fettsäuremethylester (FAME) als Mischkomponente für Dieselkraftstoff und Dieselkraftstoff, der bis zu 30% (V / V) FAME enthält

Carburants pour automobile Rapport sur les études relatives à la tendance au colmatage de filtre après macération à froid dester méthylique dacides gras (EMAG) comme composant pour le gazole et de gazole contenant jusquà 30% (V/V) dEMAG

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TECHNICAL REPORT

RAPPORT TECHNIQUE

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ICS

English Version

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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Contents

Page

European foreword

This document (FprCEN/TR 17544:2020) has been prepared by Technical Committee CEN/TC 19 "*Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin*", the secretariat of which is held by NEN.

This document is currently submitted to the Vote on TR.

Introduction

As reported in CEN/TR 16982[1], during recent past winters, a wide range of vehicles has been affected in specific European countries and there are possible links with fatty acid methyl esters (FAME) composition, base diesel quality, cold flow additives and oxidation stability effects. In order to solve these issues, some countries have introduced new additional requirements in their national specifications or "best practice" market agreements.

In the UK, developments around the Filter Blocking Tendency test (FBT) has been engaged and in particular a variant of the IP 387[2] with a Cold Soak stage (CS-FBT). This work has been exchanged with CEN/TC19 and the CEN/TC19/WG31 has started several studies in order to evaluate the interest of using this method for neat FAME and diesel fuels containing up to 30 % (V/V) of FAME.

This document reports the content of these studies.

1 Scope

This document describes the studies executed to develop a method to analyse the filter blocking tendency after a cold soak step of fatty acid methyl ester (FAME) as a blend component for diesel and of diesel fuel containing up to 30 % (*V*/*V*) of FAME, respectively.

NOTE For the purposes of this document, the term "% (*V*/*V*)" is used to represent the volume fraction, *φ*.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

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

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

— ISO Online browsing platform: available at http://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
filter blocking tendency
FBT
dimensionless value that defines the filter blocking tendency of a fuel caused by particulates

Note 1 to entry: The value is calculated using the pressure or volume attained at the end of the test. Depending on the outcome of the test, two different equations are applied (see Clause 9 of IP 387[2] for the calculation of the FBT value).

[SOURCE: IP 387]

3.2
cold soak
CS
exposure of the test portion to a constant reduced temperature for a period of time

[SOURCE: IP PM-EA[3]]

3.3
cold soak filter blocking tendency
CS-FBT
variant of an FBT determination which includes a cold soak stage before testing the sample

4 Filter blocking tendency of diesel fuels and FAME

4.1 Evolution of diesel fuels and FAME composition

In recent years diesel fuels have become more complex as FAME, hydrotreated vegetable oils (HVO), Gas-To-Liquid (GTL), etc. have been increasingly introduced into diesel blends. FAME has evolved from its origins as RME (Rapeseed Methyl Ester) into a wide variety of sources including animal sourced (TME - Tallow Methyl Ester) and used cooking oil (UCOME – Used Cooking Oil Methyl Ester). These changes to fuel composition are considered to have a possible impact to the Filter Blocking Tendency of the fuel.

4.2 Detail of field issues

The detail of field issues in different areas in Europe are well documented in CEN/TR 16982. This report includes also several works engaged in different countries to understand those field issues among which the development of FBT test and variants including the CS-FBT and the Cold FBT (C-FBT, IP 618[4]).

4.3 FBT test development

As described in CEN/TR 16884[5], the FBT test (IP 387, ASTM D2068[6]) was originally developed as the “Navy Rig Test” in the 1980s by the UK Ministry of Defence (National Gas Turbine Establishment) to predict operability of warships after the Falklands War. Warship filters were being blocked by rust, sand, microbiological growth and insoluble gums, and a need was identified to develop a lab test with a direct correlation to filter blocking of filter/coalescer elements. The test was later standardized as IP 387 following a Ministry of Defence request to establish test precision. The NATO F-76 naval distillate fuel specification contains a requirement for FBT, and in Australia and New Zealand, legislation requires that diesel fuel shall meet a maximum FBT limit.

Today, the FBT test is used to determine the filterability of middle distillate fuels, biofuels such as FAME and diesel / biofuel blends. This method is not necessarily a cold flow test, however it has been found to be effective at detecting poorly blended MDFI additives and is sensitive to a number of other solid contaminants that can be found in modern diesel fuels. The filter pore size is also representative of modern diesel vehicle fuel filter technology.

For the Filter Blocking Tendency Test, a 300 ml sample portion of the fuel is passed at a constant flow rate of 20 ml/min through a specified filter medium. There are several different procedures contained within the FBT test method. In Procedures A and B a 13 mm diameter, 1,6 µm glass fibre filter is used; whereas in Procedure C a 22 mm diameter, 5 µm nylon filter is used. The pressure difference across the filter and the volume of fuel passing through the filter are monitored until the pressure reaches 105 kPa or the volume of fuel passing through the filter medium reaches 300 ml, at which point the test is terminated.

Cold flow tests such as Cloud Point (CP) [7], Cold Flow Plugging Point (CFPP) [8] and Pour Point (PP) [9] are designed to detect and test the impact of paraffin wax deposition once a distillate fuel reaches and drops below its cloud point. The field issues mentioned in 4.2 were encountered at temperatures above the CP of the fuel.

The FBT test was developed to detect potential filter blocking above the cloud point of the fuel. It’s important to note that it should not be used at, or below, the cloud point.

5 CS-FBT studies

5.1 General

The CS-FBT is a variant of FBT test developed by Energy Institute (IP PM-EA[3]). The CS-FBT test adopts a low temperature sample pre-conditioning step which involves cold soaking the fuel sample for 16 h to accelerate the precipitation of the insoluble impurities, before allowing the fuel to warm to ambient temperature and performing a filtration step identical to the FBT test. By allowing the sample to warm to ambient temperature after the cold soak, the test only measures those impurities that do not re-dissolve easily after they have formed.

Using the FBT test only may not detect impurities such as saturated monoglycerides (SMG) and sterol glucosides (SG) which only manifest themselves at lower temperatures, but above the cloud point of the fuel. Therefore, a “cold” version of IP387 was investigated to detect these impurities which can precipitate out of the fuel above its cloud point

This type of approach has been studied in northern America. In Canada, the CGSB (Canadian General Standards Board) has already developed its own CS-FBT test method (CGSB-3.0 – No 142.0[10]) that is also based on the FBT test. This test has been adopted in the CGSB 3.524[11] specification for FAME. In the USA, the Cold Soak Filtration Test (ASTM D7501[12]) has also been developed and is required in the ASTM specification for FAME (ASTM D6751[13]).

NOTE Details of the different methods can be found in Table A.1.

5.2 Ruggedness studies – 2011 - 2013

5.2.1 Ruggedness study 2011

5.2.1.1 Origin

A joint meeting with CEN/TC 19/WG 31 and Energy Institute was held in London on February 1st 2011 as a “Cold Filterability Workshop”.

Plans were discussed to organize a ruggedness study. The study procedure was defined in June 2011 and testing to be carried out in September 2011 with results to be available for WG24 meeting in November 2011.

The mini ILS test on CS-FBT method was carried out in September 2011:

— ten FAME samples tested by 8 laboratories using the method developed by CEN/TC 19/WG 31 (main parameters presented in Table 1),

— Each FAME sample tested as a B10 blend in a EN 590[14] compliant fuel (GO PSA4 blanc) and de-aromatized kerosene as described in the IP PM-EA test, hereafter:

o A FAME sample is treated to delete its thermal history by keeping it at elevated temperature (60 °C) for (2 – 3) hours and then allowing to cool to room temperature. Then, it is blended as B10 and it is cooled to 5 °C, kept at this temperature for 16 h and again allowed to reach room temperature (see Figure 1). The objective of this process is to favour the precipitation of any compounds that can cause filtration problems.

o The appearance of the sample is then evaluated.

o The filter blocking tendency (FBT according to IP 387-procedure B) is determined by passing a constant flow of the prepared sample through a specific filter.

o The FBT value and the appearance of the sample (AR) (see Table B.2) are used to calculate the Filterability Factor (FF) (see Formula (B.1) which determines the acceptability of the sample.

— Testing was carried out in duplicate.

NOTE Details for this Ruggedness study 2011 can be found in Annex B

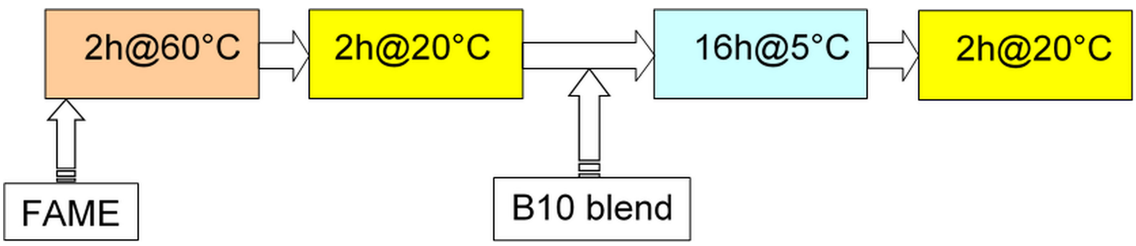


Figure 1 — Sample preparation scheme

Table 1 — Ruggedness studies, procedure details

Parameter	Ruggedness Study 2011	Ruggedness Study 2013
Glassware	Glassware not defined	Unscratched Glassware for FBT
FAME sample homogenization	Yes (shaking for 30s)	Yes (shaking for 30s)
FAME heating	60 °C ± 2 °C for 120 min ± 10 min	60 °C ± 2 °C for 120 min ± 10 min
FAME cooling	At ambient T in air or water bath	At ambient T in air
Mixing with DAK	Stirring during 1min	Shaking approx. 2min
Cold Soak	16h@5 °C, cooling chamber or water bath	16h@5 °C, cooling chamber
Warming-up	20 °C ± 5 °C for a period no longer than 2 h in air or water batch	20 °C ± 5 °C for a period no longer than 2 h in air
Homogenization	Stir the sample vigorously, using a magnetic stirrer, for 120 s ± 5s, and allow to stand for 300 s	shake 5 min, allow to stand for 2 min
Beaker	Keep in same beaker for FBT test	Transfer to FBT beaker (unscratched)

5.2.1.2 Outcome

Main results following the 2011 study are:

— Evaluation using FBT discriminated between good and bad samples but 2R limit not reached,

— Evaluation on volume gave even better discriminated between good and bad samples,

— All test conditions (for both solvents) in general gave comparable results,

— However, precision remained an issue when comparing results from different laboratories; sample homogeneity or sample preparation (B10 blending) may be a reason.

Main outcomes are:

— New project group set up to optimize the precision of proposed IP PM-EA test,

— Detailed investigation of sample preparation and filtration,

— Work on calibration for FBT methods and explore reasons for test variability. Explore opportunities for developing a suitable calibration material for “higher” FBT values,

— Work on modifications of the method (e.g. stirring during filtration).

5.2.2 Ruggedness study 2013

5.2.2.1 Origin

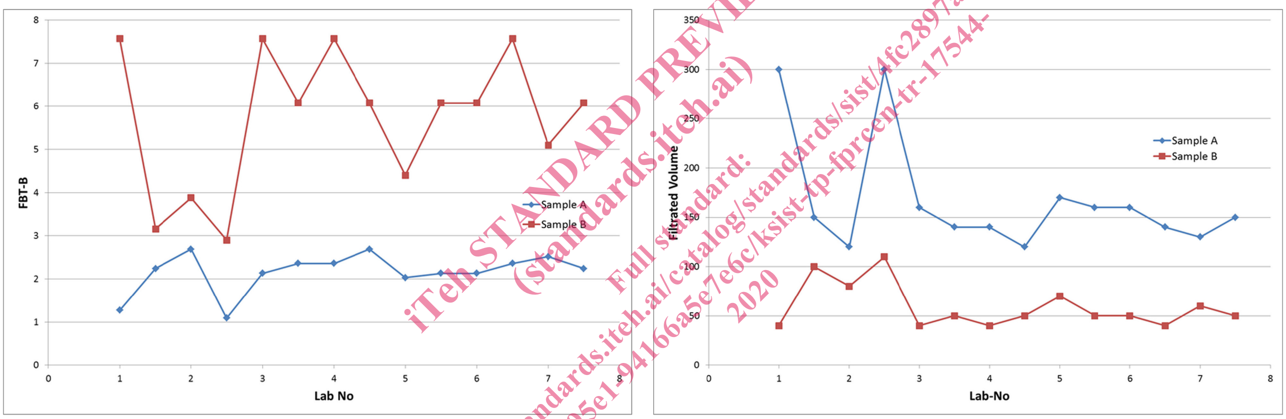
At the 18th meeting of WG 31 (7th February 2013), the group felt that main issue with original ruggedness test was poor precision and the correlation with field filterability issues.

NOTE The details of Ruggedness study 2013 are given in Annex D.

5.2.2.2 Preliminary testing

It was reported preliminary tests carried out in 2 FBT verification fluids to understand if such fluids can be used in future ILS to check the validity of the IP 387 FBT apparatus. The fluids were EN 590 diesel products with a contaminant added: two samples with FBT of approx. 2,5 and two others with FBT of approx. 5,0 and Type B filters were supplied to the seven laboratories taking part in the study (see Table D.1).

A detailed sample preparation procedure was provided to the participants as well (samples are shaken for 120 s and then allowed to stand for 5 min in order to simulate real life, i.e. let large particles drop down to the bottom).



a) b)

Figure 2– Main results of preliminary testing

Main results of the pre-study were (see Figure 2):

- The results were overall encouraging.
- Reproducible results can be obtained on the same sample. Deviations found for some laboratories may have been due to technical reasons.
- It was concluded that it would be appropriate to have a verification material for future ILS.

Proposals discussed to improve precision in the group were then:

- Stirring,
- Evaluation of pressure curves,
- Cooling rate,
- Container material – Feedback from one Lab mentioned some influence on FBT,

— Grounding of IP 387 apparatus – Canadian work on CS-FBT showed that grounding has an influence,

— More defined sample preparation – Possible reason for poor results in the 2011 ruggedness study.

5.2.2.3 Ruggedness study 1

The scope of the new ruggedness study was defined to identify if proposed modifications of the method were successful and to give preliminary evaluation of the test precision.

The set-up of the study was defined as follow:

— a new draft method has been defined referenced as WG 24/N403 (see right column in Table 1),

  o Method has two parts for testing FAME and diesel,

  ▪ Procedure A – For testing of FAME as B10 in de-aromatized kerosene,

  ▪ Procedure B – For testing of finished diesel fuels.

— four laboratories,

— three FAME samples for Method A testing,

— three B10 finished blends (using the same FAME samples) for Method B testing.

Protocol used:

— Three different types of preparation were requested on the samples to the laboratories:

  o With FAME samples received: Prepare two different B10 blends as per procedure A,

  o With FAME samples received: Prepare 0,8 l of one B10 sample, but separate the samples before cold soak, carry out the rest of the procedure A 2 times,

  o With two B10 samples per FAME as received: carry out procedure B.