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Fatty acid methyl esters (FAME) — Determination of sulfur content — Inductively coupled plasma optical emission spectrometry (ICP-OES) method

Esters méthyliques d'acides gras — Détermination de la teneur en soufre — Méthode par spectroscopie d'émission optique par plasma à couplage inductif (ICP-OES)

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Foreword

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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).

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This document was prepared by Technical Committee ISO/TC 28, *Petroleum and related products, fuels and lubricants from natural or synthetic sources*, Subcommittee SC 7, *Liquid Biofuels*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

This document seeks to evaluate the quality of fatty acid methyl esters (FAME) in terms of sulfur.

Though FAME itself does not contain sulfur, sulfur might occur as contaminant either in feedstock, due to the use of fertilizers, or in production processes using sulfuric acid. The presence of sulfur in FAME can be caused by the production process of FAME and/or possible contaminations by diesel fuel. Above certain levels of sulfur concentration, it can be harmful to use FAME as fuel. The test method provided in this document offers a simple and effective way to check and control the sulfur level of FAME, which is used as pure fuel or as blend component.

Fatty acid methyl esters (FAME) — Determination of sulfur content — Inductively coupled plasma optical emission spectrometry (ICP-OES) method

WARNING — The use of this document can involve hazardous materials, operations and equipment. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of users of this document to take appropriate measures to ensure the safety and health of personnel prior to the application of this document, and to determine the applicability of any other restrictions.

1 Scope

This document specifies a test method for inductively coupled plasma optical emission spectrometry (ICP-OES) for the detection of the sulfur content from 2 mg/kg to 21 mg/kg in fatty acid methyl esters (FAME).

NOTE 1 For the purposes of this document, the term "% (*m/m*)" is used to represent the mass fraction (μ) of the material.

NOTE 2 The method can also be used for the determination of concentrations outside the given limits. The precision statement, however, is only valid for the concentration range given in the scope.

NOTE 3 The method described in the document was tested with FAME derived from soybean oil and beef tallow. FAME derived from other feedstock, in particular aged oils, may behave different due to the different nature of sulfur compounds.

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 3170, *Petroleum liquids — Manual sampling*

ISO 3171, *Petroleum liquids — Automatic pipeline sampling*

ISO 12185, *Crude petroleum and petroleum products — Determination of density — Oscillating U-tube method*

3 Terms and definitions

No terms and definitions are listed in this document.

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

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

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

4 Principle

An exactly weighed test portion is diluted with kerosene to allow the proper introduction of the aerosol into the plasma. The resulting solution is directly injected into the plasma of the ICP-OES spectrometer. For reference and calibration purposes, calibration samples with a known sulfur content in the range of <1 mg/kg to 10 mg/kg are used.

The sulfur content is obtained by linear regression of the sulfur emission in the sample solution and the emission of the calibration solutions in the respective wavelength.

5 Reagents and materials

Use only reagents of recognized analytical grade.

5.1 Diluent Solvent, solvent with a sulfur content below 1 mg/kg and suited to completely dissolve the samples. Kerosene (CAS 8008-20-6) was successfully used as dilution solvent in the interlaboratory study on precision.

WARNING — Flammable and harmful product.

5.2 Low viscosity oil, mineral oil (liquid vaseline). Oils with a viscosity between 10,8 mm^2/s and 13,6 mm^2/s at 40 °C and a density between 0,828 g/ml and 0,856 g/ml were found suitable. The sulfur content shall be less than 1 mg/kg. Check the sulfur content using the spectrometer (6.1). A signal for sulfur shall not be detectable.

NOTE Other oils can be used, for example: paraffin oil with a viscosity of 33,5 mm^2/s at 40 °C or base oil with a viscosity from 14 mm^2/s to 18 mm^2/s at 40 °C, as well as other base oils, as long as the viscosity at 40 °C is known and as long as they are sulfur free.

5.3 Standard solution, organic sulfur compound in base oil or in FAME. A sulfur concentration of 500 mg/kg was found suitable, other concentrations may also be used.

Individual organic standard sulfur solutions with other concentrations, commercially available, can also be used. In this case, the masses and the volumes detailed below shall be recalculated.

5.4 Argon, minimum purity of 99,999 %.

Other auxiliary gases as well as argon may be used, for example nitrogen, oxygen and synthetic air, as required by the equipment manufacturer's specifications.

6 Apparatus

6.1 Inductively coupled plasma optical emission spectrometer (ICP-OES), equipped with the following components.

6.1.1 Adequate set for the introduction of organic solvents (nebulizer, spray chamber, injector and tubes).

6.1.2 Peristaltic pump, able to provide flow between 0,5 ml/min and 3 ml/min.

6.2 Cooling chamber, with adjustable temperature (optional).

6.3 Balance, with a minimum resolution of 0,000 1 g.

6.4 Conical flasks, borosilicate glass.

6.5 Pipettes, for volumetric preparation of the solutions (8.1).

7 Sampling

Samples shall be taken as described in ISO 3170 or ISO 3171.

8 Preparation of stock solution and calibration solution

8.1 Stock solution

Sulfur solution with a concentration of 50 mg/kg: add to an appropriate conical flask (6.4) approximately 3,0 g of the standard sulfur solution (5.3), fill up to 30 g with diluent (5.1).

Weigh the masses with an accuracy of 0,000 1 g and homogenize the solution. Calculate the exact concentration of this solution, which shall be used to prepare the calibration solutions.

It is also permitted to prepare the dilution on volumetric basis. In this case, use appropriate pipettes (6.5) to transfer the volumes. In this case the concentration is expressed as mass/volume.

Other concentrations and quantities can be used for the stock solution but the precision data in Clause 10 were obtained from analysis with the given concentration above.

8.2 Preparation of the calibration solutions

The calibration solutions are prepared from an organic sulfur compound which is commercially available. The sulfur compound is diluted in a mixture of diluent (6.1) and base oil (6.2). The addition of the base oil has the objective to reduce the differences in viscosity between the samples and the calibration solutions.

8.2.1 Blank solution for sulfur

Prepare an adequate amount of a solution of 10 % (*m/m*) low viscosity oil (5.2) in the solvent (5.1).

WARNING — When using other oils (see Note to 5.2) the concentration of low viscosity oil in the blank solution shall be calculated as described in Annex A.

8.2.2 Calibration sample set

Table 1 shows the recommended calibration set for sulfur (0,0 mg/kg, 1,0 mg/kg, 2,5 mg/kg, 5,0 mg/kg and 10 mg/kg).

Weigh the masses with an accuracy of 0,000 1 g into the flask and fill up to the given amount with blank solution (8.2.1). Stir the samples until the homogenization is complete.

Calculate the exact concentrations of the calibration solutions for given in Table 1.

When the concentrations of the solutions are expressed in mass/volume percentages (8.1), the calibration solutions shall be expressed in the same way.

For each calibration, freshly prepared calibration samples shall be used.

Table 1 — Calibration solutions

Sulfur content mg/kg	Stock solution g	Total quantity g
0	0	10
1,0	0,2	10
2,5	0,5	10
5,0	1,0	10
10,0	2,0	10

NOTE Depending on the instrument larger calibration samples might be necessary in order to have suitable amounts for repeat measurement.