



SLOVENSKI STANDARD
oSIST prEN ISO 20884:2018
01-december-2018

Naftni proizvodi - Določevanje žvepla v gorivih za motorna vozila - Metoda z valovno disperzivno rentgensko fluorescenčno spektrometrijo (ISO/DIS 20884:2018)

Petroleum products - Determination of sulfur content of automotive fuels - Wavelength-dispersive X-ray fluorescence spectrometry (ISO/DIS 20884:2018)

Mineralölerzeugnisse - Bestimmung des Schwefelgehaltes in Kraftstoffen - Wellenlängendispersive Röntgenfluoreszenz-Spektrometrie (ISO/DIS 20884:2018)

Produits pétroliers - Détermination de la teneur en soufre des carburants pour automobiles - Spectrométrie de fluorescence de rayons X dispersive en longueur d'onde (ISO/DIS 20884:2018)

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Petroleum products — Determination of sulfur content of automotive fuels — Wavelength-dispersive X-ray fluorescence spectrometry

Produits pétroliers — Détermination de la teneur en soufre des carburants pour automobiles — Spectrométrie de fluorescence de rayons X dispersive en longueur d'onde

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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 Documents 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).

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).

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For an explanation of 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 www.iso.org/iso/foreword.html.

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

This third edition cancels and replaces the second edition (ISO 20884:2011), which has been technically revised following a new interlaboratory study executed by CEN/TC 19/WG 27, *Elemental analysis*, that confirmed the method precision. The main changes however compared to the previous edition are as follows:

- the scope has been extended and now includes hydrotreated vegetable oil (HVO) and the synthetic fuel Gas To Liquid (GTL);
- for instruments utilizing monochromatic excitation specific procedural steps have been included.

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.

Petroleum products — Determination of sulfur content of automotive fuels — Wavelength-dispersive X-ray fluorescence spectrometry

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

1 Scope

This document specifies a wavelength-dispersive X-ray fluorescence (WDXRF) test method for the determination of the sulfur content of liquid, homogeneous automotive fuels from 5 mg/kg to 500 mg/kg, which have a maximum oxygen content of 3,7 % (*m/m*). This product range covers diesel fuels containing up to about 30 % (*V/V*) fatty acid methyl esters (FAME) and motor gasolines containing up to about 10 % (*V/V*) ethanol, and synthetic fuels such as Hydrotreated Vegetable Oil (HVO) and Gas To Liquid (GTL) having sulfur contents in the range of 5 mg/kg to 45 mg/kg.

NOTE 1 Sulfur contents higher than 500 mg/kg can be determined after sample dilution. However, the precision was not established for diluted samples.

Products with higher oxygen content show significant matrix effects, e.g. FAME used as biodiesel. Nevertheless, FAME may be analysed when the corresponding procedures are followed (see 5.3 and 8.1).

Other products may be analysed with this test method. However, precision data for products other than those mentioned have not been established for this document.

NOTE 2 For the purposes of this document, the terms “% (*m/m*)” and “% (*V/V*)” are used to represent the mass fraction, μ , and the volume fraction, φ , of a material respectively.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3170, *Petroleum liquids — Manual sampling*

ISO 3171, *Petroleum liquids — Automatic pipeline sampling*

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>

4 Principle

The sample under analysis is exposed in a sample cell to the primary radiation of an X-ray tube. The count rates of the S K-L_{2,3} X-ray fluorescence and, where required, the count rate of the background

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radiation are measured. The sulfur content of the sample is determined from a calibration curve defined for the relevant measuring range.

NOTE The IUPAC X-ray line notation (S K-L_{2,3}) is used in this document; the corresponding Siegbahn X-ray line notation (S-K α) is being phased out.

5 Reagents

5.1 Dibutylsulfide, of nominal sulfur content 21,92 % (*m/m*), or **dibutyldisulfide**, of nominal sulfur content 35,95 % (*m/m*), used as a calibrating substance for sulfur.

Care should be taken due to volatility (see [Annex A](#)).

5.2 White oil (paraffinum perliquidum), for use as a blank solution, of high purity grade, with a sulfur content of less than 1 mg/kg. Check the blank solution prior to use with the spectrometer (6.1). A signal for sulfur shall not be detectable.

5.3 Methyl oleate, for use as a blank solution when FAME is analysed, with a sulfur content of less than 1 mg/kg. Check the blank solution prior to use with the spectrometer (6.1). A signal for sulfur shall not be detectable (i.e. the intensity shall be lower than the intensity equivalent to 1 mg/kg). Other oxygen-containing and sulfur-free blank solutions, such as octanol, can also be used.

6 Apparatus

6.1 Wavelength-dispersive X-ray fluorescence spectrometer, with the capability for measuring the count rates of the S K-L_{2,3} X-ray fluorescence radiation and the background radiation.

Due to the inherently low background radiation produced by instruments utilizing monochromatic radiation for excitation, it is not mandatory to measure the background radiation when this type of instrument is used. The minimum requirements for the spectrometer are given in Table 1.

6.2 Analytical balance, capable of weighing to the nearest 0,1 mg.

7 Sampling

Unless otherwise specified, samples shall be taken by the procedures described in ISO 3170 or ISO 3171.

Table 1 — Spectrometer requirements

Component	Polychromatic excitation	Monochromatic excitation
Anode	Rhodium, scandium or chromium	Rhodium, scandium, chromium or titanium
Voltage ^a	No less than 30 kV	No less than 30 kV
Current ^a	No less than 50 mA	No less than 0,1 mA
Collimator or optic	Coarse collimator	Monochromator
Analysing crystal	Germanium, pentaerythrite or graphite	Germanium, pentaerythrite or graphite
Optical path ^b	Vacuum or helium	Vacuum or helium

^a Lower-power systems may be used, provided they have been validated to meet the precision requirements specified in [Clause 13](#).

^b In instruments using vacuum, the sample is not exposed directly to the vacuum of the optical path, but is separated by a secondary window.

^c Polyester film is the preferred choice as samples of very high aromatic content may dissolve polycarbonate film. There are possibly trace amounts of silicon, calcium and sulfur in some types of film. However, the effects should be cancelled out when samples and standards are analysed using the same batch of film. It is important that samples, standards and blanks be measured using the same batch of film to avoid bias. Other sample window materials with the same or better absorptivity, purity and stability can also be used.

Table 1 (continued)

Component	Polychromatic excitation	Monochromatic excitation
Sample cup window ^c	Polyester film, 4 µm maximum	Polyester film, 4 µm maximum
Detector	Proportional counter with pulse-height analyser	Proportional counter with single channel analyser
Wavelengths	S K-L _{2,3} at 0,537 3 nm Background radiation at 0,545 nm	S K-L _{2,3} at 0,537 3 nm
<p>^a Lower-power systems may be used, provided they have been validated to meet the precision requirements specified in Clause 13.</p> <p>^b In instruments using vacuum, the sample is not exposed directly to the vacuum of the optical path, but is separated by a secondary window.</p> <p>^c Polyester film is the preferred choice as samples of very high aromatic content may dissolve polycarbonate film. There are possibly trace amounts of silicon, calcium and sulfur in some types of film. However, the effects should be cancelled out when samples and standards are analysed using the same batch of film. It is important that samples, standards and blanks be measured using the same batch of film to avoid bias. Other sample window materials with the same or better absorptivity, purity and stability can also be used.</p>		

8 Calibration solutions

8.1 Blank solution

Use white oil ([5.2](#)) as a blank solution.

C074314efig1.EPSIf FAME is under analysis, use a blank solution in accordance with 4.3 to minimize potential matrix effects.

8.2 Stock solution

Weigh a quantity of the required calibrating substance ([5.1](#)) to the nearest 0,1 mg, and dilute with blank solution ([5.2](#) or 5.3, as appropriate) at room temperature so that a stock solution with an accurately known sulfur content (to the nearest 1 mg/kg) of approximately 1 000 mg/kg is obtained.

Care should be taken due to volatility of the calibrating substance (see [Annex A](#)).

8.3 Calibration solutions

Weigh an appropriate quantity of the stock solution ([8.2](#)) to the nearest 0,1 mg into bottles and dilute with blank solution ([5.2](#) or 5.3, as appropriate) so that standard solutions with sulfur contents in accordance with Tables 2 and 3 are obtained by stirring at room temperature. The sulfur contents in the calibration solutions are indicated in milligrams per kilogram, rounded to the nearest 0,1 mg/kg.

Table 2 — Calibration solutions — Low sulfur range

Number	Sulfur content mg/kg
Blank solution 0	0,0
2,1	5,0
2,2	10,0
2,3	25,0
2,4	50,0

Table 3 — Calibration solutions — High sulfur range

Number	Sulfur content mg/kg
Blank solution 0	0,0
3,1	50,0
3,2	100,0
3,3	200,0
3,4	350,0
3,5	500,0

8.4 Storage and stability of the calibration solutions

Calibration solutions prepared in accordance with Table 2 have a limited stability and shall be used on the day they are prepared.

Calibration solutions prepared in accordance with Table 3 have a stability of no more than one week if stored in a cool location (refrigerator).

9 Settings

9.1 Measuring parameters

For optimum measuring parameters, refer to Table 1.

9.2 Optimization

The spectrometer shall be optimized in accordance with the manufacturer's specifications in order to achieve the optimum signal-to-noise ratio. The calibration solution with 50 mg/kg sulfur is recommended for optimization.

For polychromatic excitation, the counting time shall be set so that the total net count from a 50 mg/kg solution is W 40 000. This counting time shall be used for both calibration (see [Clause 10](#)) and measurement (see [Clause 11](#)).

9.3 Performance check of the spectrometer

Prior to a measurement series (calibration and/or measurement), but in any case at least once daily, the manufacturer's specifications shall be used to check that the spectrometer is operating correctly, so that an optimum performance and a consistent standard of highest possible quality is ensured.

Performance checks should be carried out on a regular basis, since such checks give valuable information about the status and stability of the spectrometer.

10 Calibration

10.1 General

Ensure that the spectrometer is in an optimized condition after having executed all provisions given in [Clause 9](#) and operating optimally before calibration (see [9.3](#)).

For the range from 5 mg/kg to 60 mg/kg sulfur, and for the range 60 mg/kg to 500 mg/kg sulfur, separate calibrations shall be conducted.