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Third edition
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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

Reference number
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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).

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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. The main changes compared to the previous edition are as follows:

- extension of the scope to include hydrotreated vegetable oil (HVO) and the synthetic fuel “gas to liquid” (GTL);
- inclusion of specific procedural steps for instruments utilizing monochromatic excitation.

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.

INTERNATIONAL STANDARD **ISO 20884:2019(E)**

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

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 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 application of the document, and fulfil other applicable requirements for this purpose.

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),
- motor gasolines containing up to about 10 % (*V*/*V*) ethanol,
- 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.

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

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

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

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

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*

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

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 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 Kα or S $K\alpha_{1,2}$) 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 for further guidance).

NOTE Certified reference materials (CRM) produced in accordance with ISO 17034[3] are suitable alternatives.

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 pure 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 that 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.

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

[a] Lower-power polychromatic systems may be used, provided they have been validated to meet the precision values 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.