
**Petroleum products — Determination of
sulfur content — Energy-dispersive X-ray
fluorescence spectrometry**

*Produits pétroliers — Détermination de la teneur en soufre —
Spectrométrie de fluorescence de rayons X dispersive en énergie*

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Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 8754 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

This second edition cancels and replaces the first edition (ISO 8754:1992), which has been technically revised.

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Introduction

Specialized procedures, using the analytical technique described in this International Standard, for automotive fuels with sulfur contents below 0,20 % (*m/m*), are under development.

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Petroleum products — Determination of sulfur content — Energy-dispersive X-ray fluorescence spectrometry

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

1 Scope

This International Standard specifies a method for the determination of the sulfur content of petroleum products, such as naphthas, unleaded motor gasolines, middle distillates, residual fuel oils, base lubricating oils and components. The method is applicable to products having sulfur contents in the range 0,03 % (*m/m*) to 5,00 % (*m/m*).

NOTE For the purposes of this International Standard, the term "% (*m/m*)" is used to represent the mass fraction of a material.

Heavy metal additives, such as lead alkyls, may interfere with the determination. Elements such as silicon, phosphorus, calcium, oxygen, potassium, zinc, molybdenum, barium and halogens interfere, if present in concentrations of more than a few hundred milligrams per kilogram. Some modern instruments allow the analyst to compensate for matrix and spectral interferences by spectra deconvolution and inter-element correction by multiple regression.

For samples varying in composition of aromatic hydrocarbons and paraffinic hydrocarbons, the ratio of carbon to hydrocarbon in a sample (*C/H* ratio) may also interfere with the determination, when the ratio of the sample differs by one or more from that of the reference materials from which the calibration is obtained.

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:—¹⁾, *Petroleum liquids — Manual sampling*

ISO 3171:1988, *Petroleum liquids — Automatic pipeline sampling*

3 Principle

The test portion is placed in the beam emitted from an X-ray source. The excitation energy may be derived from a radioactive source, such as ⁵⁵Fe, or from an X-ray tube. The resultant excited characteristic X radiation is measured, and the accumulated count is compared with a calibration graph plotting counts against sulfur content as percentage by mass [% (*m/m*)], on a series of calibration samples covering the range of sulfur contents under examination.

1) To be published. (Revision of ISO 3170:1988)

4 Reagents and materials

4.1 White oil (light paraffin oil), of high purity grade, with a maximum sulfur content of 20 mg/kg.

For measurements at very low levels of sulfur content [below approximately 0,1 % (*m/m*)], matrix effects are minimized if a diluent of a type similar to the product being analysed is used for the production of the calibration standards described in Clause 8. Such a diluent should be of very low sulfur content, preferably below 2 mg/kg.

4.2 Sulfur compounds, of known sulfur content, used for the preparation of the primary standards.

NOTE The compounds given in 4.2.1 to 4.2.3 are suitable, and their nominal sulfur contents are given. Where the purity of these compounds is less than 99 %, certified materials are required, or the concentrations and nature of all impurities are to be known.

4.2.1 Dibenzothiophene (DBT), with a nominal sulfur content of 17,399 % (*m/m*).

4.2.2 Dibutylsulfide (DBS), with a nominal sulfur content of 21,915 % (*m/m*).

4.2.3 Thionaphthene (benzothiophene) (TNA), with a nominal sulfur content of 23,89 % (*m/m*).

4.3 Certified reference materials: use materials from a national standards body or accredited supplier, with a range of certified sulfur contents for the production of calibration curves for routine analysis. However, particularly for some heavier materials, the excitation is slightly affected by the background matrix. Therefore, in cases of dispute, the parties shall agree on a common range of certified standards, or shall both prepare standards from the materials described in 4.1 and 4.2.

5 Apparatus

5.1 Energy-dispersive X-ray fluorescence analyser: use any suitable model, provided that the design incorporates the features given in 5.1.1 to 5.1.6. It shall be set up according to the manufacturer's instructions.

5.1.1 Source of X-ray excitation, with significant X-ray flux at energies above 2,5 keV.

5.1.2 Removable sample cup, providing a sample depth of at least 3 mm, and equipped with replaceable X-ray transparent film.

NOTE Window material is normally 6 µm polyester, polypropylene or polycarbonate film. Commercial polyester film may contain small but variable amounts of calcium, which may interfere. Samples of very high aromatic content may dissolve polycarbonate film.

5.1.3 X-ray detector, with high sensitivity at 2,3 keV.

5.1.4 Filters, or other means of discriminating between sulfur K α radiation and other X-rays.

5.1.5 Signal-conditioning electronics, that include the functions of pulse counting and pulse-height analysis.

5.1.6 Display or printer, that provides a readout in counts, sulfur content as a percentage by mass [% (*m/m*)], or both.

CAUTION — If the analyser contains a radioactive source, the equipment and manner of use shall comply with the regulations governing the use of ionizing radiation and/or recommendations of the International Commission on Radiological Protection. The radiation source shall be checked for radiation leakage at intervals as required by the regulations. All attention to the source shall only be carried out by fully trained and competent persons, using the correct shielding techniques.

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