SLOVENSKI STANDARD
SIST EN ISO 8754:1998

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Naftni proizvodi - Določ evanje ǵvepla - Metoda z energijsko-disperzivno rentgensko fluorescenco (ISO 8754:1992)

Petroleum products - Determination of sulfur content - Energy-dispersive-X-ray fluorescence method (ISO 8754:1992)

Mineralölerzeugnisse - Bestimmung des Schwefelgehaltes - Energiedispersives Röntgenfluoreszenz-Verfahren (ISO 8754:1992)

Produits pétroliers - Détermination de la teneur en soufre - Méthode par fluorescence X dispersive d'énergie (ISO 8754:1992)

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English version

Petroleum products - Determination of sulfur content - Energy-dispersive-X-ray fluorescence method (ISO 8754:1992)

Produits pétroliers - Détermination de la teneur en soufre - Méthode par fluorescence X dispersive d'énergie (ISO 8754:1992)

Mineralölerzeugnisse - Bestimmung des Schwefelgehaltes - Energiedispersives Röntgenfluoreszenz-Verfahren (ISO 8754:1992)

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart,36 B-1050 Brussels

Foreword

This European Standard has been taken over by the Technical Committee CEN/TC 19 "Petroleum products, lubricants and related products" from the work of ISO/TC 28 "Petroleum products and lubricants" of the International Organization for Standardization (ISO).

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by August 1995, and conflicting national standards shall be withdrawn at the latest by August 1995.

According to the CEN/CENELEC Internal Regulations, the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, United Kingdom.

Endorsement notice

The text of the International Standard ISO 8754:1992 was approved by CEN as a European Standard without any modification.

INTERNATIONAL STANDARD

ISO 8754

First edition
1992-05-01

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

Produits pétroliers — Détermination de la teneur en soufre — Méthode par fluorescence X dispersive d'énergie

Reference number
ISO 8754:1992(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards 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.

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.

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

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

1 Scope

This International Standard specifies a method for the determination of the sulfur content of hydrocarbons such as naphthas, distillates, fuel oils, residues, lubricating base oils only, unleaded gasolines and components. The method is applicable to products having sulfur contents in the range 0,01 % (*m*/*m*) to 5 % (*m*/*m*).

Samples containing heavy-metal additives, lead alkyls, etc., interfere with the method. Elements such as silicon, phosphorus, calcium, potassium and halides interfere if present in concentrations of more than a few hundred milligrams per kilogram.

2 Principle

The sample is placed in the beam emitted from a suitable low-energy radioactive source, for example ^{55}Fe source (typical radiation of 740 MBq). The resultant excited characteristic X-radiation is measured and the accumulated count is compared with counts from previously calibrated blends to obtain the sulfur concentration as a percentage by mass. Three groups of calibration samples are required to span the concentration range 0,01 % (*m*/*m*) to 5 % (*m*/*m*).

3 Reagents

3.1 Di-*n*-butyl sulfide, sulfur content 21,91 % (*m*/*m*)

3.2 White oil, high-purity grade, containing less than 20 mg of sulfur per kilogram.

4 Apparatus

Ordinary laboratory apparatus and

4.1 Energy-dispersive X-ray fluorescence analyser: any suitable model can be used, set up according to the manufacturer's instructions.[1)]

4.2 Analytical balance, accurate to 0,1 mg.

5 Procedure

5.1 Safety precautions

The X-ray 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.

Servicing of the source shall only be carried out by a fully trained and competent person using the correct shielding techniques.

NOTE 1 At least one commonly used instrument has a thin beryllium window above the radioactive source. This window is very fragile and is easily broken during cleaning of the sample area to remove oily residues.

5.2 Sample cell preparation

The cells shall be thoroughly clean and dry before use. Handling of the film shall be kept to an absolute minimum. The window shall be renewed for the measurement of each sample.

NOTES

2 Window material is usually 6 µm polyester or polycarbonate film. Normal commercially available polyester window material contains small but variable amounts of calcium which may introduce errors.

3 Samples having a high aromatic content are likely to dissolve polycarbonate films.

1) Details of suppliers of suitable equipment may be obtained from the Secretariat of ISO/TC 28.

5.3 Preparation of apparatus

Set up the apparatus according to the manufacturer's instructions. Where regulations allow, the instrument shall be continuously switched on, to maintain optimum stability. Check the calibration of the apparatus at intervals in accordance with the manufacturer's instructions.

Some instruments require a helium purge to obtain accurate measurements. Good-quality helium, at a steady pressure, as specified by the instrument manufacturer, shall be used to avoid background interference which could give variable results.

5.4 Determination

Fill the sample cell to the required depth [3 mm to 20 mm (approximately half full)]. Ensure that the thin-film window does not bow. Ensure also that there are no air bubbles between the window and the liquid. For viscous samples, it may be necessary to apply heat so that they can easily be poured into the cell. Obtain two consecutive counts using the recommended counting time for the instrument on a portion of the sample. Calculate the average count for the sample (typically acceptable counting times are 50 s to 200 s).

The analysis of a sample shall be carried out at the same period of time after its preparation in the cell as that used in the preparation of the calibration curve.

6 Calibration

6.1 Preparation of standards

6.1.1 Primary standards

Prepare primary standards having sulfur contents of 5 % (*m*/*m*), 2,5 % (*m*/*m*) and 1 % (*m*/*m*). Prepare each standard separately as follows:

Weigh, to the nearest 0,1 mg, the appropriate quantity of white oil (3.2), shown in table 1, into a suitable, narrow-necked container and then weigh in the appropriate quantity of di-*n*-butyl sulfide. Mix thoroughly (a glass-coated magnetic stirrer is advisable) at room temperature.

Calculate the sulfur content of each standard to three decimal places.

Table 1 — Composition of primary standards

Sulfur content % (*m*/*m*)	Mass of white oil g	Mass of di-*n*-butyl sulfide g
5	48,6	14,4
2,5	44,7	5,7
1	47,7	2,3

6.1.2 Calibration blends

Make up the calibration standards in three ranges by diluting primary standards with white oil (table 2).

Table 2 — Calibration standards

Range	Expected sulfur content % (*m*/*m*)	Sulfur content of standards % (*m*/*m*)			
1	0,01 to 1,0	0,0[1]	0,3	0,7	1,0
2	1,0 to 2,5	1,0[2]	1,5	2,0	2,5
3	2,0 to 5,0	2,0[3]	3,0	4,0	5,0

1) White oil (3.2), assumed to be 0 % (*m*/*m*) sulfur.
2) 1,0 % (*m*/*m*) standard from range 1 can be used.
3) 2,0 % (*m*/*m*) standard from range 2 can be used.

6.2 Storage of standards

Store the standards in dark glass-stoppered bottles in a cool, dark place until required. As soon as any sediment or change of concentration is observed, discard the standard.

6.3 Preparation of calibration curve

Proceed as described in clause 4. Obtain four readings on each calibration blend in the selected range, using the recommended counting time for the instrument. Immediately repeat the procedure using freshly prepared cells and fresh portions of each calibration blend. From the data obtained, calculate the average reading for each sulfur concentration. Prepare a calibration graph from the averaged results.

Some automated equipment ejects the test portion after two counts. Additional test portions shall then be used to obtain the required number of counts for each calibration blend.