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BSI Standards Publication

Petroleum products — Determination of sulfur content of automotive fuels — Ultraviolet fluorescence method (ISO 20846:2011)

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National foreword

This British Standard is the UK implementation of EN ISO 20846:2011. It supersedes BS EN ISO 20846:2004, which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PTI/13, Petroleum Testing and Terminology.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Petroleum products - Determination of sulfur content of automotive fuels - Ultraviolet fluorescence method (ISO 20846:2011)

Produits pétroliers - Détermination de la teneur en soufre des carburants pour automobiles - Méthode par fluorescence ultraviolette (ISO 20846:2011)

Mineralölerzeugnisse - Bestimmung des Schwefelgehaltes von Kraftstoffen für Kraftfahrzeuge - Ultraviolettfluoreszenz-Verfahren (ISO 20846:2011)

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Foreword

This document (EN ISO 20846:2011) has been prepared by Technical Committee ISO/TC 28 "Petroleum products and lubricants" in collaboration Technical Committee CEN/TC 19 “Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin” the secretariat of which is held by NEN.

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of an identical text or by endorsement, at the latest by April 2012, and conflicting national standards shall be withdrawn at the latest by April 2012.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN ISO 20846:2004.

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The text of ISO 20846:2011 has been approved by CEN as a EN ISO 20846:2011 without any modification.

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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 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.

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.

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ISO 20846 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

This second edition cancels and replaces the first edition (ISO 20846:2004), which has been technically revised.

Petroleum products — Determination of sulfur content of automotive fuels — Ultraviolet fluorescence method

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 an ultraviolet (UV) fluorescence test method for the determination of the sulfur content of motor gasolines containing up to 3,7 % (m/m) oxygen [including those blended with ethanol up to about 10 % (V/V)], and of diesel fuels, including those containing up to about 10 % (V/V) fatty acid methylester (FAME), having sulfur contents in the range 3 mg/kg to 500 mg/kg. Other products can be analysed and other sulfur contents can be determined according to this test method, however, no precision data for products other than automotive fuels and for results outside the specified range have been established for this International Standard. Halogens interfere with this detection technique at concentrations above approximately 3 500 mg/kg.

NOTE 1 Some process catalysts used in petroleum and chemical refining can be poisoned when trace amounts of sulfur-bearing materials are contained in the feedstocks.

NOTE 2 This test method can be used to determine sulfur in process feeds and can also be used to control sulfur in effluents.

NOTE 3 For the purposes of this International Standard, the terms “% (m/m)” and “% (V/V)” are used to represent the mass fraction and the volume fraction of a material respectively.

NOTE 4 Sulfate species in ethanol do not have the same conversion factor of organic sulfur in ethanol. Nevertheless, sulfates have a conversion factor close to that of organic sulfur.

NOTE 5 It is preferable to check the nitrogen interference and to take it into account, especially when sulfur content is measured on diesel blended with cetane improver containing nitrogen. For example, alkyl nitrate, as 2-ethyl hexyl nitrate (EHN), added as cetane improver to diesel fuel shows an enhancing effect on sulfur content that can range from 0 to 1,7 mg/kg when 2 000 mg/kg EHN is added to diesel fuel containing 10 mg/kg sulfur.

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 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 3170, *Petroleum liquids — Manual sampling*

ISO 3171, *Petroleum liquids — Automatic pipeline sampling*

ISO 3675, *Crude petroleum and liquid petroleum products — Laboratory determination of density — Hydrometer method*

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

3 Principle

A hydrocarbon sample is injected into a UV fluorescence detector. The sample enters a high temperature combustion tube (1 000 °C to 1 100 °C), where the sulfur is oxidized to sulfur dioxide (SO_2) in an oxygen-rich atmosphere. Water produced during the sample combustion is removed and the sample combustion gases are exposed to UV light. The SO_2 absorbs the energy from the UV light and is converted to excited sulfur dioxide (SO_2^*). The fluorescence emitted from the excited SO_2^* as it returns to a stable state SO_2 is detected by a photomultiplier tube and the resulting signal is a measure of the sulfur contained in the sample.

4 Reagents and materials

4.1 Inert gas, argon or helium, high purity grade with a minimum purity of 99,998 % (*V*/*V*).

4.2 Oxygen, high purity grade with a minimum purity of 99,75 % (*V*/*V*).

CAUTION — Vigorously accelerates combustion.

4.3 Solvent

4.3.1 General

Use either that specified in 4.3.2 or 4.3.3, or a solvent similar to that occurring in the sample under analysis. Correction for sulfur contribution from solvents used in standard preparation and sample dilution is required. Alternatively, use of a solvent with non-detectable sulfur contamination relative to the unknown sample makes the blank correction unnecessary.

4.3.2 Toluene, reagent grade.

4.3.3 Isooctane, reagent grade.

CAUTION — Flammable solvents.

4.4 Sulfur compounds

4.4.1 General

Compounds with a minimum purity of 99 % (*m*/*m*). Examples are given in 4.4.2 to 4.4.4. Where the purity of these compounds is less than 99 % (*m*/*m*), the concentrations and nature of all impurities shall be established.

NOTE A correction for chemical impurity can be applied when the sulfur content is known with accuracy.

Certified reference materials (CRM) from accredited suppliers are suitable alternatives to the compounds listed in 4.4.2 to 4.4.4.

4.4.2 Dibenzothiophene (DBT), of molecular mass 184,26, with a nominal sulfur content of 17,399 % (*m*/*m*).

4.4.3 Dibutyl sulfide (DBS), of molecular mass 146,29, with a nominal sulfur content of 21,915 % (*m*/*m*).

4.4.4 Thionaphthene (Benzothiophene) (TNA), of molecular mass 134,20, with a nominal sulfur content of 23,890 % (*m*/*m*).

4.5 Sulfur stock solution

Prepare a stock solution of approximately 1 000 mg/l sulfur content by accurately weighing the appropriate quantity of sulfur compound (4.4) in a volumetric flask (5.9). Ensure complete dissolution with solvent (4.3). Calculate the exact sulfur concentration of the stock solution to the nearest 1 mg/l. This stock solution is used for the preparation of calibration standards. As an alternative procedure, a sulfur stock solution of approximately 1 000 mg/kg can be prepared by accurately weighing the appropriate quantity of sulfur compound (4.4) in a volumetric flask (5.9) and reweighing the volumetric flask once it has been filled to the mark with the solvent (4.3). Take precautions to ensure that evaporation of the solvent and/or sulfur compounds is not causing weighing errors.

The appropriate mass of sulfur compound described in 4.4.2 to 4.4.4 to add to the 100 ml flask is 0,574 8 g (DBT), 0,456 3 g (DBS) and 0,418 6 g (TNA).

NOTE The shelf life of the stock solution is approximately three months when stored at low temperature, typically in a refrigerator.

4.6 Calibration standards

Prepare the calibration standards by dilution of the stock solution (4.5) with the selected solvent (4.3).

Calculate the exact sulfur content of each calibration standard.

Calibration standards with a known sulfur concentration, in milligrams per litre, (or content, in milligrams per kilogram) can be obtained with a volume/volume dilution (or mass/mass dilution, respectively) of the stock solution at 1 000 mg/l (or 1 000 mg/kg respectively). Other practices are possible, but those mentioned above avoid any density correction.

New calibration standards should be prepared on a regular basis, depending upon the frequency of use and age. When stored at low temperature, typically in a refrigerator, the calibration standards with a sulfur content above 30 mg/kg (or mg/l) have a shelf life of at least one month. Below this sulfur content (30 mg/kg), the shelf life should be reduced.

4.7 Quality control samples

Quality control samples are stable samples representative of the materials being analysed, which have a sulfur content that is known by this test method over a substantial period of time. Alternatively, there are standard materials with a certified value commercially available. Prior to use, ensure that the material is within its shelf life.

4.8 Quartz wool

Follow the manufacturer’s recommendations.

5 Apparatus

Figure 1 illustrates the basic pieces of the UVF.

5.1 Furnace, comprising an electric device, capable of maintaining a temperature sufficient to pyrolyse all of the sample and oxidize all sulfur to sulfur dioxide (SO_2).

It can be set either in a horizontal or vertical position.

5.2 Combustion tube, of quartz, constructed to allow the direct injection of the sample into the heated oxidation zone of the furnace (5.1).

The combustion tube shall have side arms for the introduction of oxygen and carrier gas. The oxidation section shall be large enough to ensure complete combustion of the sample. It can be set either in a horizontal or vertical position.

5.3 Flow controllers, capable of maintaining a constant supply of oxygen and carrier gas.

5.4 Vapour drier, capable of removing water vapour formed during combustion prior to measurement by the detector (5.5).

5.5 UV fluorescence detector, selective and quantitative, capable of measuring light emitted from the fluorescence of sulfur dioxide by UV light.

WARNING — Exposure to excessive quantities of UV light is injurious to health. The operator must avoid exposing any part of his/her person, especially his/her eyes, not only to direct UV light, but also to secondary or scattered radiation that may be present.

5.6 Microlitre syringe, capable of accurately delivering between 5 µl to 50 µl quantities.

Follow the manufacturer's instructions for determining the length of the needle required. For vertical injection, syringes with a polytetrafluoroethylene (PTFE) plunger are recommended.

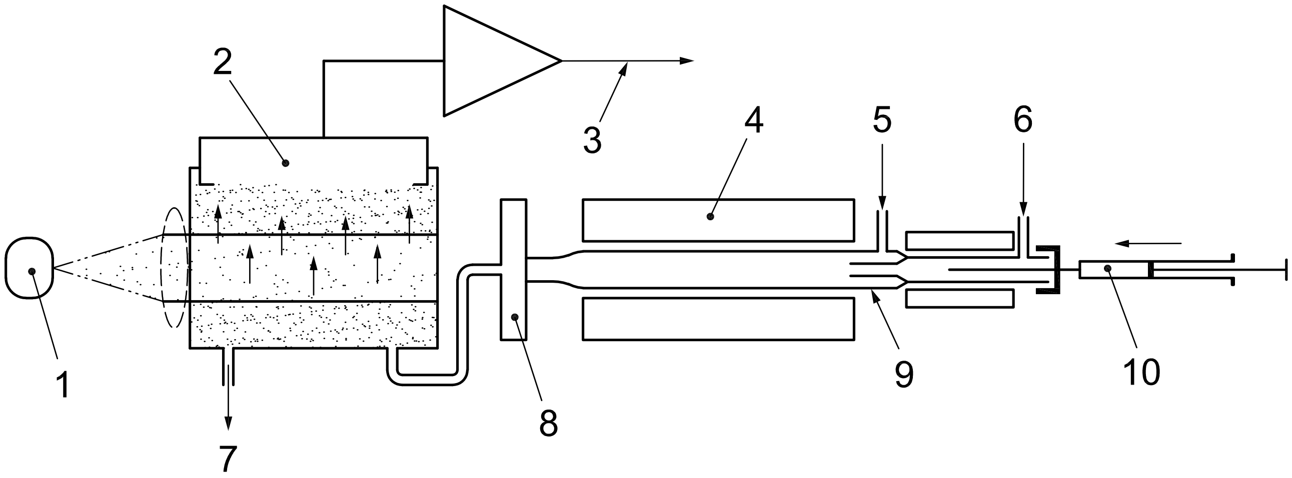
5.7 Sample inlet system, either positioned vertically or horizontally.

It shall consist of a direct injection inlet system capable of allowing the quantitative delivery of the material to be analysed into an inlet carrier stream which directs the sample into the oxidation zone at a controlled and repeatable rate. A syringe drive mechanism, which discharges the sample from the microlitre syringe at a constant rate of approximately 1 µl/s maximum, is required.

NOTE Boat injection systems can be used if they meet the performance requirements of Clause 12.

5.8 Balance, capable of weighing with an accuracy of at least 0,1 mg.

5.9 Volumetric flasks, Class A one-mark volumetric flasks, conforming to ISO 1042, of appropriate capacities, including 100 ml, for the preparation of sulfur stock solution (4.5) and calibration standards (4.6).



Key

1 UV source
2 photomultiplier
3 output signal
4 furnace, 1 000 °C to 1 100 °C
5 oxygen input
6 inert gas input
7 gases output
8 drier
9 quartz tube
10 microlitre syringe

Figure 1 — Synopsis of the apparatus

6 Sampling

6.1 Unless otherwise specified, obtain the laboratory sample by the procedures described in ISO 3170 for manual sampling, or ISO 3171 for automatic pipeline sampling. To preserve volatile components found in some samples, keep samples at as low a temperature as is practicable and do not uncover samples any longer than necessary.

Samples should be analysed as soon as possible after removal from bulk supplies, to prevent loss of sulfur or contamination due to exposure or contact with the sample container.

CAUTION — Samples that are collected at temperatures below room temperature can undergo expansion and rupture the container. For such samples, do not fill the container to the top; leave sufficient air space above the sample to allow room for expansion.

6.2 If the sample is not used immediately, thoroughly mix it in its container prior to taking a test portion.

7 Apparatus preparation

7.1 Assemble the apparatus and check for leaks according to the manufacturer’s instructions.

7.2 Adjust the inlet pressure and flow rate of each gas according to the manufacturer’s instructions.

7.3 Refer to the manufacturer’s instructions to set the temperature of the furnace (5.1) high enough to ensure all sulfur pyrolysis and oxidation, typically 1 100 °C in the case of a one temperature zone furnace, or 750 °C for pyrolysis, and 1 000 °C to 1 100 °C for oxidation in the case of a two temperature zone furnace.

7.4 Adjust the instrument sensitivity and baseline stability, and perform instrument blanking procedures following the manufacturer’s guidelines. Ensure that the UV light is stable before measurement.

NOTE For the UV light, a warm-up time of at least 30 min is usually required.

8 Apparatus calibration and verification

8.1 Multi-point calibration

8.1.1 Select one of the suggested curves outlined in Table 1. Prepare a series of calibration standards (4.6) by carrying out dilutions of the stock solution (4.5) with the selected solvent (4.3) to cover the range of operation. The number of calibration standards used in construction of the calibration curve can vary, but it should not be less than four.

Table 1 — Calibration standards

Curve I	**Curve II**	**Curve III**
Sulfur content mg/l or mg/kg	**Sulfur content** mg/l or mg/kg	**Sulfur content** mg/l or mg/kg
0,5	5	50
2	10	150
5	30	350
10	60	500

8.1.2 Flush the microlitre syringe (5.6) several times with the solution to be analysed and ensure that the final liquid column in the syringe contains no bubbles.

8.1.3 Using one of the techniques described 8.1.3.1 or 8.1.3.2, quantitatively analyse an appropriate sample size as specified by the manufacturer, prior to injection into the combustion tube for analysis.

NOTE Injection of a constant or similar sample size for all materials analysed in a selected operating range promotes consistent combustion conditions.

8.1.3.1 For volumetric measurement, fill the syringe to the selected level and retract the plunger so that air is aspirated and the lower liquid meniscus falls on the 10 % scale mark. Record the volume of liquid in the syringe. After injection, again retract the plunger so that the lower liquid meniscus falls on the 10 % scale mark and record the volume of liquid in the syringe. The difference between the two volume readings is the volume of sample injected.

NOTE An automatic sampling and injection device can be used in place of the described manual injection procedure.

8.1.3.2 For mass measurement, weigh the syringe complete with a filled needle before injection, and the syringe and needle after injection, to determine the mass of the test portion injected.

NOTE Mass measurement can be more accurate than the volume measurement for less volatile samples, if a balance with an accuracy of at least ±0,01 mg is used.

8.1.4 Once the appropriate sample size has been measured into the microlitre syringe, promptly and quantitatively deliver the sample into the apparatus.

For direct injection, carefully insert the syringe into the inlet of the combustion tube (5.2) and the syringe drive. Allow time for sample residues to be burned from the needle (needle blank). Once a stable baseline has been re-established, promptly start the analysis. Remove the syringe once the apparatus has returned to a stable baseline.

8.1.5 Construct each calibration curve by one of the techniques described in 8.1.5.1 or 8.1.5.2.

8.1.5.1 For manual construction, analyse the calibration standards and the blank three times using the procedure described in 8.1.2 to 8.1.4. Subtract the average blank response from each analysis of the standard before determining the average integrated response. Construct a curve plotting average integrated detector response (y-axis) versus quantity, Q, expressed in nanograms, of sulfur injected (x-axis). This curve should be linear with a correlation factor of at least 0,995.

Q is calculated using Equation (1) or (2):

$$Q = m_{\mathrm{c}} \times w_{\mathrm{Sc}} \qquad (1)$$

$$Q = V_{\mathrm{c}} \times \rho_{\mathrm{S}} \qquad (2)$$

where

w_{Sc} is the sulfur content of the calibration standard, expressed in milligrams per kilogram;

V_{c} is the volume of the calibration standard injected, expressed in microlitres;

ρ_{S} is the sulfur concentration of the calibration standard, expressed in milligrams per litre;

m_{c} is the mass of the calibration standard injected, expressed in milligrams, either measured directly, or calculated from the measured volume injected and the density using Equation (3):

$$m_c = V_c \times D_c \tag{3}$$

where

D_c is the density of the calibration standard at measurement temperature, expressed in grams per millilitre.

8.1.5.2 If the apparatus features an internal calibration routine, analyse the calibration standards and the blank three times using the procedure described in 8.1.2 to 8.1.4. If blank correction is required and is not available (see 4.3), correct the analyser responses using the average response for each standard versus quantity, Q (in nanograms), of sulfur as obtained in 8.1.5.1. This curve should be linear with a correlation factor of at least 0,995.

NOTE For curve III (Table 1), the blank correction can be omitted.

8.1.6 The analyser calibration can be performed using a different calibration curve from those listed in Table 1. It is good practice to construct a calibration curve in such a way that the sulfur content of the sample under analysis is in the middle of the calibration curve.

8.2 One-point calibration

8.2.1 Prepare a calibration standard (4.6) with a sulfur content close to that of the sample under analysis (±50 % maximum), by carrying out dilutions of the stock solution (4.5). A calibration standard with a sulfur concentration greater than 50 % from that of the sample may be used if the linearity of the apparatus has previously been checked.

If necessary, the sulfur content of the calibration standard should be corrected by the sulfur content of the selected solvent (4.3).

NOTE In order to measure the sulfur content of the selected solvent, the method of standard additions of the solvent gives satisfactory results.

8.2.2 Using the procedure described in 8.1.2 to 8.1.4, analyse a minimum of three times the calibration standard using an appropriate sample size as specified by the manufacturer.

8.2.3 Calculate a calibration factor, K, expressed in number of counts per nanogram of sulfur, using Equation (4) or (5):

$$K = \frac{A_c}{m_c \times w_{Sc}} \tag{4}$$

$$K = \frac{A_c}{V_c \times \rho_S} \tag{5}$$

where

A_c is the integrated detector response for calibration standard, in number of counts;

w_{Sc} is the sulfur content of the calibration standard, expressed in milligrams per kilogram;

V_c is the volume of the calibration standard injected, expressed in microlitres;

ρ_S is the sulfur concentration of the calibration standard, expressed in milligrams per litre;

m_c is the mass of the calibration standard injected, expressed in milligrams, either measured directly or calculated from the measured volume injected and the density using Equation (6):

$$m_\mathrm{c} = V_\mathrm{c} \times D_\mathrm{c} \qquad (6)$$

where

D_c is the density of the calibration standard at measurement temperature, expressed in grams per millilitre.

Calculate the average of the calibration factor and check that the standard deviation is within the accepted tolerance.

When the concentration of calibration standards is known in milligrams per kilogram, and volume injections are performed, a correction for the difference of density between standards and samples is necessary.

8.3 Verification

8.3.1 Check the calibration validity with a quality control sample of known sulfur content, at the beginning of a series of analyses and at least every twenty analyses during a long series of analyses.

8.3.2 Compare the results to the known values and associated uncertainties. The out of limit results shall be investigated for root cause(s).

NOTE Control limits are established from the laboratory statistical control charts, but initial values should be set before experience is established. Limits of the repeatability of this method, or 0,7 times the reproducibility, are reasonable starting points.

9 Procedure

9.1 Determine the density of the sample, in accordance with the hydrometer method specified in ISO 3675 or the oscillating U-tube method specified in ISO 12185.

If the temperature of sample injection is more than 3 °C from the temperature of determination of density, or if the density used is at a reference temperature, ISO 91-1 may be used to calculate the density at the injection temperature.

9.2 Estimate the sulfur content of the sample, and prepare a test portion that contains a quantity of sulfur within the range of the calibration curve selected, if the multi-point calibration is chosen, or close to the sulfur content of the calibration standard (±50 % maximum), if one-point calibration is chosen. If the expected sulfur content is above the content of the calibration standards, prepare either a volumetric or mass-diluted solution with a selected solvent (4.3).

When the sulfur content of the sample is above 500 mg/kg, dilute to the most convenient level.

9.3 Using the procedure described in 8.1.2 to 8.1.4, analyse an appropriate sample size three times, as specified by the manufacturer.

9.4 Inspect the combustion tube and other flow path components to verify complete oxidation of the test portion. If deposits are noted, follow the procedures given in 9.4.1 and 9.4.2.

9.4.1 If coke or soot is observed, clean any parts of the combustion tube in accordance with the manufacturer's instructions. After cleaning and/or adjustment, assemble the apparatus and check for leaks. Carry out a check on the calibration quality prior to reanalysis.

9.4.2 Use a reduced sample size or a reduction of injection rate, or both.

10 Calculation

10.1 Using multi-point calibration

For analysers calibrated using a standard curve, calculate the sulfur content, w_S, of the sample, expressed in milligrams per kilogram, using Equation (7) or (8):

$$w_\mathrm{S} = \frac{A - Y}{m \times S_\mathrm{s} \times F_\mathrm{g}} \tag{7}$$

$$w_\mathrm{S} = \frac{A - Y}{V \times S_\mathrm{s} \times F_\mathrm{v}} \tag{8}$$

where

A is the integrated detector response for the sample, expressed in number of counts;

Y is the y-intercept of the standard curve, expressed in number of counts;

S_s is the slope of the standard curve, expressed in counts per nanogram of sulfur;

F_g is the gravimetric dilution factor, mass of test portion/mass of test portion and solvent, expressed in grams per gram;

V is the volume of the test portion solution injected, expressed in microlitres;

F_v is the volumetric dilution factor, mass of test portion/volume of test portion and solvent, expressed in grams per millilitre;

m is the mass of the test portion solution injected, expressed in milligrams, either measured directly or calculated from the measured volume injected and density using Equation (9):

$$m = V \times D \tag{9}$$

where

D is the density of the test portion solution at measurement temperature, expressed in grams per millilitre.

10.2 Using one-point calibration

Calculate the sulfur content, w_S, of the sample, expressed in milligrams per kilogram, using Equation (10) or (11):

$$w_\mathrm{S} = \frac{A}{m \times K \times F_\mathrm{g}} \tag{10}$$

$$w_S = \frac{A}{V \times K \times F_v} \quad (11)$$

where

K is the calibration factor, expressed in counts per nanogram of sulfur;

V is the volume of the test portion solution injected, expressed in microlitres;

A is the integrated detector response for the sample, expressed in number of counts;

F_g is the gravimetric dilution factor, mass of test portion/mass of test portion and solvent, expressed in grams per gram;

F_v is the volumetric dilution factor, mass of test portion/volume of test portion and solvent, expressed in grams per millilitre;

m is the mass of the test portion solution injected, expressed in milligrams, either measured directly or calculated from the measured volume injected and density using Equation (12):

$$m = V \times D \quad (12)$$

where

D is the density of the test portion solution at measurement temperature, expressed in grams per millilitre.

If there is a dilution in low sulfur content, the calculation shall take into account the correction with the sulfur content of the solvent.

10.3 Calculation

Calculate the average sulfur content of the sample from three determinations.

11 Expression of results

Report the sulfur content of the sample, in milligrams per kilogram (mg/kg), to the nearest 1 mg/kg above 60 mg/kg and to the nearest 0,1 mg/kg below 60 mg/kg.

12 Precision

12.1 General

The precision, as determined by statistical examination in accordance with ISO 4259, is given in 12.2 and 12.3.

12.2 Repeatability, r

The difference between two test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the values given in Table 2 in only one case in 20.

Table 2 — Repeatability

Sulfur content mg/kg	Gasolines	Gas oils
3 to 60	$r = 0{,}063\,1X + 0{,}35$	$r = 0{,}055\,3X + 0{,}55$
>60 to 500	$r = 0{,}041\,7X + 1$	$r = 0{,}028\,5X + 2$
NOTE X is the average of the results being compared, in milligrams per kilogram.		

12.3 Reproducibility, R

The difference between two single and independent test results obtained by different operators working in different laboratories on identical material would, in the long run, in the normal and correct operation of the test method, exceed the values given in Table 3 in only one case in 20.

Table 3 — Reproducibility

Sulfur content mg/kg	Gasolines	Gas oils
3 to 60	$R = 0{,}174\,9X + 0{,}96$	$R = 0{,}112\,0X + 1{,}12$
>60 to 500	$R = 0{,}157\,3X + 2$	$R = 0{,}108\,0X + 2$
NOTE X is the average of the results being compared, in milligrams per kilogram.		

Examples of the above precision estimates are given in Table 4.

Table 4 — Repeatability and reproducibility

Sulfur content mg/kg	Repeatability r		Reproducibility R	
	Gasoline	Gas oil	Gasoline	Gas oil
10	1,0	1,1	2,7	2,2
30	2,2	2,2	6,2	4,5
50	3,5	3,3	9,7	6,7
100	5	5	18	13
350	16	12	57	40
500	22	16	81	56

13 Test report

The test report shall contain at least the following information:

a) a reference to this International Standard (ISO 20846:2011);

b) the type and complete identification of the product tested;

c) the result of the test (see Clause 11);

d) any deviation, by agreement or otherwise, from the procedure specified;

e) the date of the test.

Bibliography

[1] ISO 91-1, *Petroleum measurement tables — Part 1: Tables based on reference temperatures of 15 °C and 60 °F*

[2] ISO 4259, *Petroleum products — Determination and application of precision data in relation to methods of test*

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