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

*Produits pétroliers — Détermination de la teneur en soufre
des carburants pour automobiles — Méthode par fluorescence
ultraviolette*

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Contents

	Page
Foreword	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions	2
4 Principle	2
5 Reagents and materials	2
6 Apparatus	4
7 Sampling and sample handling	5
8 Apparatus preparation	5
9 Apparatus calibration and verification	6
9.1 Multi-point calibration	6
9.2 One-point calibration	8
9.3 Verification	8
10 Procedure	9
11 Calculation	9
11.1 Using multi-point calibration	9
11.2 Using one-point calibration	10
11.3 Calculation	10
12 Expression of results	10
13 Precision	11
13.1 General	11
13.2 Repeatability, r	11
13.3 Reproducibility, R	11
14 Test report	12
Bibliography	13

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).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 20846:2011), which has been technically revised. The main change compared to the previous edition is the extension of the Scope to include hydrotreated vegetable oil (HVO) and the synthetic fuel “gas to liquid” (GTL).

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 — Ultraviolet fluorescence method

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 an ultraviolet (UV) fluorescence test method for the determination of the sulfur content of the following products:

- having sulfur contents in the range 3 mg/kg to 500 mg/kg,
 - motor gasolines containing up to 3,7 % (m/m) oxygen [including those blended with ethanol up to about 10 % (V/V)],
 - diesel fuels, including those containing up to about 30 % (V/V) fatty acid methyl ester (FAME),
- having sulfur contents in the range of 3 mg/kg to 45 mg/kg,
 - synthetic fuels, such as hydrotreated vegetable oil (HVO) and gas to liquid (GTL).

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 document. 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 document, “% (m/m)” and “% (V/V)” are used to represent the mass fraction, w , and the volume fraction, φ , 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 Nitrogen interference can occur, see [6.5](#) for further guidance.

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 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 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

A hydrocarbon sample is either directly injected or placed in a sample boat. Then, it enters a high temperature combustion tube (1 000 °C to 1 100 °C), where the sulfur is oxidized to sulfur dioxide (SO₂) in an oxygen-rich atmosphere. Water produced during the sample combustion is removed and the sample combustion gases are exposed to UV light. The SO₂ absorbs the energy from the UV light and is converted to excited sulfur dioxide (SO₂*). The fluorescence emitted from the excited SO₂* as it returns to a stable state SO₂ is detected by a photomultiplier tube and the resulting signal is a measure of the sulfur contained in the sample.

5 Reagents and materials

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

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

CAUTION — Oxygen vigorously accelerates combustion.

5.3 **Solvent.**

5.3.1 General

Use either that specified in 5.3.2 or 5.3.3, 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.

5.3.2 **Toluene**, reagent grade.

5.3.3 **Isooctane**, reagent grade.

CAUTION — Flammable solvents.

5.4 Sulfur compounds

5.4.1 General

Compounds with a minimum purity of 99 % (*m/m*). Examples are given in 5.4.2 to 5.4.4. Where the purity of these compounds is less than 99 % (*m/m*), the concentrations and nature of all impurities shall be established.

NOTE 1 A correction for chemical impurity can be applied when the sulfur content is known with accuracy.

NOTE 2 Certified reference materials (CRM) produced in accordance with ISO 17034^[3] are suitable alternatives to the compounds listed in 5.4.2 to 5.4.4.

5.4.2 Dibenzothiophene (DBT), of molar mass 184,26 g/mol, with a nominal sulfur content of 17,399 % (*m/m*).

5.4.3 Dibutyl sulfide (DBS), of molar mass 146,29 g/mol, with a nominal sulfur content of 21,915 % (*m/m*).

5.4.4 Thionaphthene (benzothiophene) (TNA), of molar mass 134,20 g/mol, with a nominal sulfur content of 23,890 % (*m/m*).

5.5 Sulfur stock solution

Prepare a stock solution of approximately 1 000 mg/l sulfur content by accurately weighing the appropriate quantity of sulfur compound (5.4) in a volumetric flask (6.9). Ensure complete dissolution with solvent (5.3). 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 (5.4) in a volumetric flask (6.9) and reweighing the volumetric flask once it has been filled to the mark with the solvent (5.3). 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 5.4.2 to 5.4.4 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.

5.6 Calibration standards

Prepare the calibration standards by dilution of the stock solution (5.5) with the selected solvent (5.3).

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.

5.7 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

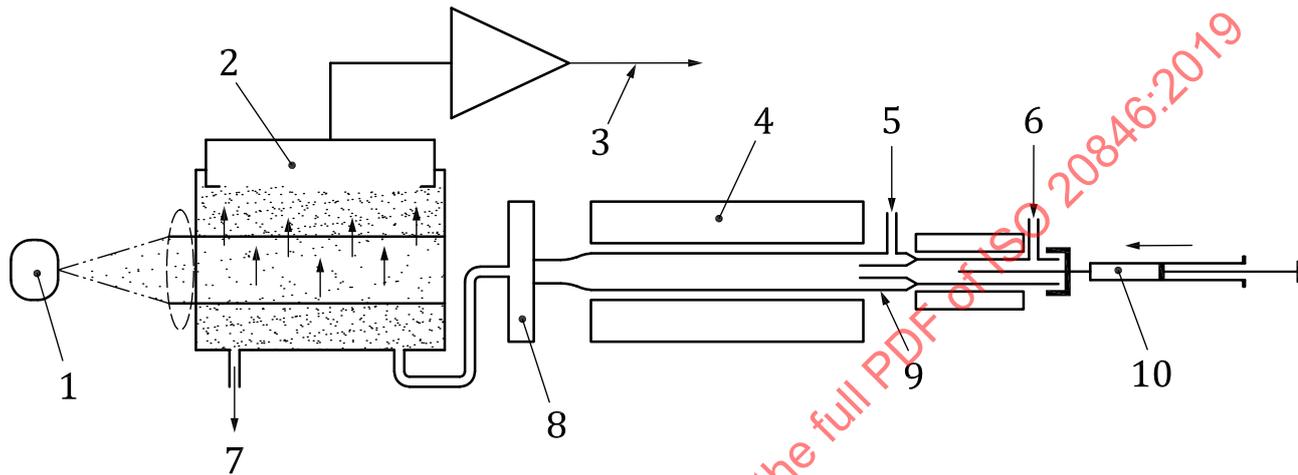
are standard materials with a certified value commercially available. Prior to use, ensure that the material is within its shelf life.

5.8 Quartz wool

Follow the manufacturer's recommendations.

6 Apparatus

Figure 1 illustrates the basic pieces of the ultraviolet fluorescence equipment.



Key

- | | |
|-------------------|-----------------------|
| 1 UV source | 6 inert gas input |
| 2 photomultiplier | 7 gases output |
| 3 output signal | 8 vapour drier (6.4) |
| 4 furnace (6.1) | 9 quartz tube |
| 5 oxygen input | 10 microlitre syringe |

Figure 1 — Synopsis of the apparatus

6.1 Furnace, comprising an electric device, capable of maintaining a temperature sufficient, 1 000 °C to 1 100 °C, to pyrolyze all of the sample and oxidize all sulfur to sulfur dioxide (SO₂).

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

6.2 Combustion tube, of quartz, constructed to allow the direct injection of the sample into the heated oxidation zone of the furnace (6.1).

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.

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

6.4 Vapour drier, capable of removing water vapour formed during combustion prior to measurement by the detector (6.5).

6.5 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 should 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 might be present.

It is recommended to verify that the nitrogen interference has been compensated for. If in doubt check with the manufacturer.

NOTE 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.

6.6 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.

6.7 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 (6.6) at a constant rate of approximately 1 µl/s maximum, is required.

Boat injection systems may be used if they meet the performance requirements of [Clause 13](#).

6.8 Balance, capable of weighing to the nearest 0,1 mg.

6.9 Volumetric flasks, Class A one-mark volumetric flasks, which shall conform to ISO 1042, of appropriate capacities, including 100 ml, for the preparation of sulfur stock solution (5.5) and calibration standards (5.6).

7 Sampling and sample handling

7.1 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.

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

8 Apparatus preparation

8.1 Ensure that the instrument has stabilized, and check for leaks according to the manufacturer's instructions.

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

8.3 Refer to the manufacturer's instructions to set the temperature of the furnace (6.1) 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.

8.4 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.

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

9 Apparatus calibration and verification

9.1 Multi-point calibration

9.1.1 Select one of the suggested curves outlined in Table 1. Prepare a series of calibration standards (5.6) by carrying out dilutions of the stock solution (5.5) with the selected solvent (5.3) 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

9.1.2 Flush the microlitre syringe (6.6) several times with the solution to be analysed and ensure that the final liquid column in the syringe contains no bubbles.

9.1.3 Using one of the techniques described 9.1.3.1 or 9.1.3.2, use an appropriate sample size as recommended 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.

9.1.3.1 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.

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

9.1.3.2 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 $\pm 0,01$ mg is used.

9.1.4 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 (6.2) 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.

9.1.5 Construct each calibration curve by one of the techniques described in 9.1.5.1 or 9.1.5.2.

9.1.5.1 For manual construction, analyse the calibration standards and the blank three times using the procedure described in 9.1.2 to 9.1.4. 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 coefficient of at least 0,995.

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

$$Q = m_c \times w_{Sc} \quad (1)$$

$$Q = V_c \times \rho_S \quad (2)$$

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 Formula (3):

$$m_c = V_c \times D_c \quad (3)$$

where

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

9.1.5.2 If the apparatus features an internal calibration routine, analyse the calibration standards and the blank three times using the procedure described in 9.1.2 to 9.1.4. If blank correction is required (see 5.3.1) and is not an available instrument option, correct the analyser responses using the average response for the blanks using the procedure as described in 9.1.5.1. This curve should be linear with a correlation coefficient of at least 0,995.

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

9.1.6 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.

9.2 One-point calibration

9.2.1 Prepare a calibration standard (5.6) with a sulfur content close to that of the sample under analysis ($\pm 50\%$ maximum), by carrying out dilutions of the stock solution (5.5). 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 (5.3).

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

9.2.2 Using the procedure described in 9.1.2 to 9.1.4, analyse a minimum of three times the calibration standard using an appropriate sample size as specified by the manufacturer.

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

$$K = \frac{A_c}{m_c \times w_{Sc}} \quad (4)$$

$$K = \frac{A_c}{V_c \times \rho_S} \quad (5)$$

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 Formula (3).

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.

9.3 Verification

9.3.1 Check the calibration validity with a quality control sample of known sulfur content (5.7), at the beginning of a series of analyses and at least every 20 analyses during a long series of analyses.

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

9.3.3 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.

10 Procedure

10.1 Determine the density of the sample in accordance with the hydrometer method following ISO 3675 or the oscillating U-tube method following 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.

10.2 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 (5.3).

NOTE If the sulfur content of the sample is above 500 mg/kg, the sample can be diluted to a practical level. The precision has not been established for diluted samples.

10.3 Using the procedure described in 9.1.2 to 9.1.4, analyse an appropriate sample size three times, as specified by the manufacturer.

10.4 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 10.4.1 and 10.4.2.

10.4.1 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.

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

11 Calculation

11.1 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 Formula (6) or (7):

$$w_s = \frac{A - Y}{m \times S_s \times F_g} \quad (6)$$

$$w_s = \frac{A - Y}{V \times S_s \times F_v} \quad (7)$$

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 [Formula \(8\)](#):

$$m = V \times D \quad (8)$$

where

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

11.2 Using one-point calibration

Calculate the sulfur content, w_s , of the sample, expressed in milligrams per kilogram, using [Formula \(9\)](#) or [\(10\)](#):

$$w_s = \frac{A}{m \times K \times F_g} \quad (9)$$

$$w_s = \frac{A}{V \times K \times F_v} \quad (10)$$

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 [Formula \(8\)](#).

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

11.3 Calculation

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

12 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.