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WITHDRAWAL

November 2005

Methods of Testing Petroleum and Associated Products

Sulphur in Gasoline by Energy Dispersive X-Ray Fluorescence Spectrometry (EDXRF)

This CGSB method is hereby withdrawn due to limited use and support for its revision.

RETRAIT

Novembre 2005

Méthodes d'essai des produits pétroliers et produits connexes

Soufre dans l'essence par spectrométrie de fluorescence X à dispersion d'énergie (EDXRF)

Cette méthode de l'ONGC est retirée par le présent avis en raison de son utilisation limitée et du manque de support pour sa révision.

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No. 16.1-98

Methods of Testing Petroleum and Associated Products

Sulphur in Gasoline by Energy Dispersive X-Ray Fluorescence Spectrometry (EDXRF)

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NATIONAL STANDARD OF CANADA

CAN/CGSB-3.0
No. 16.1-98

METHODS OF TESTING PETROLEUM AND ASSOCIATED PRODUCTS

SULPHUR IN GASOLINE BY ENERGY DISPERSIVE X-RAY FLUORESCENCE SPECTROMETRY (EDXRF)

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Prepared by the
Canadian General Standards Board CGSB

Approved by the
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National Standard of Canada

CGSB Ottawa Canada K1A 1G6	METHODS OF TESTING PETROLEUM AND ASSOCIATED PRODUCTS	CAN/CGSB-3.0
	Sulphur in Gasoline by Energy Dispersive X-Ray Fluorescence Spectrometry (EDXRF)	**No. 16.1-98**

1. SCOPE

1.1 This test method covers the determination of total sulphur concentrations in gasoline in the range from 0.0050 to 0.1000% mass, using energy dispersive X-ray fluorescence spectrometry.

1.2 The testing and evaluation of a product against this method may require the use of materials and/or equipment that could be hazardous. This document does not purport to address all of the safety aspects associated with its use. Anyone using this method has the responsibility to consult the appropriate authorities and to establish appropriate health and safety practices in conjunction with any regulatory requirements prior to its use. Please refer to the manufacturer's material safety data sheet (MSDS) for safety requirements for the specific chemical used in this method.

2. REFERENCED PUBLICATIONS

2.1 The following publications are referenced in this method:

2.1.1 Canadian General Standards Board (CGSB)

CAN/CGSB-3.5 — Unleaded Automotive Gasoline.

2.1.2 American Society for Testing and Materials (ASTM)

D 02-1007 — Manual in Determining Precision Data for ASTM Methods on Petroleum Products and Lubricants.

D 1266 (Annexe A1) — Sulfur in Petroleum Products (Lamp Method)

D 5453 — Determination of Total Sulfur in Light Hydrocarbons, Motor Fuels and Oils by Ultraviolet Fluorescence

D 5623 — Sulfur Compounds in Light Petroleum Liquids by Gas Chromatography and Sulfur Selective Detection

E 691 — Conducting an Interlaboratory Study to Determine the Precision of a Test Method

MNL 7 — Manual on Presentation of Data and Control Chart Analysis.

2.1.3 International Organization for Standardization (ISO)

ISO 4259 — Petroleum products — Determination and application of precision data in relation to methods of test.

2.2 A reference to a regulation is always to the latest issue. A dated reference is to the issue specified. An undated reference is to the latest issue, unless otherwise specified by the authority applying this method. The sources are given in the Notes section.

3. SUMMARY OF TEST METHOD

3.1 The liquid sample to be analyzed is transferred to a sample cup fitted with a transparent window. The cup is placed in the beam of an exciting radiation emitted from an X-ray source, which may be an X-ray tube or a radioisotope. The exciting radiation generates characteristic X-ray radiation of the elements in the fuel sample. The intensity of the resulting characteristic X-ray radiation of sulphur is compared to that measured on previously prepared calibration standards to obtain the sulphur concentration in mass percent.

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3.2 The calibration range is from 0.0001 to 0.1000% mass sulphur. It is applicable to gasoline, in which the sulphur concentration usually does not exceed 0.1000%.

4. SIGNIFICANCE AND USE

4.1 During combustion, the sulphur content in fuels results in SOx emissions and can contribute to particulate emissions. Both of these emissions degrade air quality. Certain jurisdictions may restrict the amount of sulphur in gasoline to prevent or limit pollution of the environment.

5. INTERFERENCES

5.1 Spectral interferences are those caused by the closeness of the X-ray characteristic lines of the elements present in a sample and the limited detector ability to completely resolve them. As a result, the lines produce spectral peaks that overlap with each other.

5.2 Matrix effects are caused by concentration variations of the elements in the sample. These variations directly influence X-ray absorption and change the measured intensity of each element. This type of interference is always present in X-ray fluorescence analysis and is completely unrelated to spectral interferences.

5.3 Both types of interferences may be compensated for in contemporary instruments with the use of built-in software for spectra deconvolution and inter-element correction by multiple regression.

5.4 Samples containing heavy metal additives, lead alkyls, etc., may interfere with the sulphur determination. Elements such as silicon, phosphorus, calcium, potassium and halides will interfere if present at concentrations greater than one tenth of the measured concentration of sulphur.

6. APPARATUS

6.1 **Energy Dispersive X-ray Fluorescence Analyzer** — An energy dispersive X-ray fluorescence spectrometer may be used if its design incorporates, as a minimum, the following features and if test results from these are shown to be equivalent on the samples of interest. The required design features shall include:

a. A source of exciting radiation with significant energy output above the 2.5 keV energy level.

b. A removable sample cup with a replaceable transparent plastic film window and providing a sample depth of at least 5 mm.

c. An X-ray detector with a high detection sensitivity for sulphur K-alpha radiation, at the energy level of 2.3 keV. The detector shall be able to exhibit acceptable detection capabilities at the 50 ppm level.[1]

d. An energy resolution capable of discriminating between sulphur K-alpha radiation and the X-rays from other elements. Filters of other means may be used to eliminate extraneous X-rays from striking the detector.

e. A signal conditioning and data handling capability that includes the functions of X-ray intensity counting and conversion of these counts into percent sulphur concentration.

f. A display or printer capable of direct readout of sulphur concentration.

6.1.1 The analyzer shall have the sensitivity under optimized measurement conditions to measure the concentration of sulphur at the 0.05% mass level with a demonstrated error due to counting statistics not greater than 2 (two) percent relative.

6.2 **Analytical Balance** — A balance capable of weighing to the nearest 0.1 mg.

7. REAGENTS

7.1 **Purity of Reagents** — Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society whenever such specifications are available. Other grades may be used, provided that the reagent is pure enough to permit its use without lowering the accuracy of the analysis.

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[1] *The detector exhibits acceptable detection capabilities at 50 ppm if a 50 ppm standard is analyzed 10 times in succession and the standard deviation of these runs does not exceed 5 ppm*

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7.2 **Di-n-Butyl Sulphide (DBS)** — Obtain the lot analysis of DBS and calculate the actual mass percent of total sulphur.

7.3 **Toluene** — ACS grade or equivalent grade containing no more than 0.0001% mass (1 ppm) sulphur.

7.4 **Iso-Octane** — ACS grade or equivalent grade containing no more than 0.0001% mass (1 ppm) sulphur.

8. SAMPLING

8.1 Samples shall be taken in accordance with the instructions in CAN/CGSB-3.5.

9. SAFETY PRECAUTIONS

9.1 Commercially available instruments employing radioisotopes as sources of exciting radiation are as a rule constructed to international standard requirements of radiation safety and, therefore, should not present any imminent radiation hazard, whether in use or not.

9.2 The instruments using X-ray tube(s) as excitation source(s) shall be shielded to prevent radiation hazard while in operation.

9.3 All instruments should be operated with strict adherence to the manufacturers' operational and safety instructions.

10. SAMPLE CUP PREPARATION

10.1 Sample cups must be clean and dry before use. It is strongly recommended that disposable sample cups be used whenever possible. Window material of the cup should exhibit the smallest possible absorption of sulphur X-rays; 6 µm thick polycarbonate or polyester film is usually used as the window. Samples of high aromatic content are known to react with the film. Therefore it is essential to renew the film window after each measurement and when the sample is left in the cup for an extended period of time.[2] Avoid touching the inside of the sample cup or portion of the window film that is exposed to the X-rays. Oil from fingerprints can affect the reading when analyzing low levels of sulphur. Wrinkles in the film will affect the number of sulphur X-rays transmitted. The importance of the film's smoothness and cleanliness in ensuring reliable results cannot be over-stressed.

10.2 Other materials beside polycarbonate films may be used for X-ray windows, provided that they do not contain any interfering elemental impurities.

11. CALIBRATION

11.1 **Certified Calibration Standards** — Calibration standards that are certified by a standards organization in accordance with a protocol that is technically equivalent to that used by the U.S. National Institute of Science and Technology (NIST) for certification of standard reference materials may be used.

11.1.1 ***Interlaboratory Tested Standards*** — Material with composition similar to the sample intended to be tested by this test method, and with reference values established in accordance with an interlaboratory testing protocol meeting the technical requirements of ASTM E 691, D 02-1007 or ISO 4259.[3]

11.1.2 ***Calibration Standards Prepared from Pure Compounds*** — Material prepared in accordance with procedures described in par. 11.2.

11.2 **Preparation of Calibration Standards Using Pure Compounds** — If pure compounds are used to prepare calibration standards, the sample matrix shall be prepared from a mixture of 25% toluene and 75% iso-octane. These

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[2] The measured sulphur concentration may vary with the time that the sample/standard contacts the film covering the sample cup. To reduce such possible variation, the length of time that the sample/standard contacts the film should be minimized and consistent.

[3] Materials are available from the Alberta Research Council, Fuels and Lubricants Group, P.O. Box 8330, Postal Station F, Edmonton, Alberta T6H 5X2.

chemicals must contain a combined sulphur content of less than 0.0005% mass.[3] Calibration standards are made up in two ranges:

Range #1: 0.0001 to 0.0200% mass sulphur

Range #2: 0.0200 to 0.1000% mass sulphur.

The target concentrations for the two calibration ranges are listed in Table 1.

TABLE 1

Target Concentrations for the Calibration Curves

Range Number	Sulphur Content Range (% Mass)	Sulphur Content (% Mass)
1	0.0001 — 0.0200	0.0001 0.0010 0.0050 0.0100 0.0200
2	0.0200 — 0.1000	0.0200 0.0350 0.0500 0.0750 0.1000

Note: *See Table 2 for suggested masses to prepare the standards.*

11.3 **Calculations** — To calculate the exact concentration of the prepared standards, use the following equation:

$D = [(A \times E) + (B \times C)]/(A + B)$

where:

A = actual mass of DBS (grams)

B = actual mass of toluene/iso-octane (grams)

C = the sulphur content of the toluene/iso-octane mixture (% mass)

D = sulphur concentration of the prepared standard (% mass)

E = the percentage of sulphur (% mass sulphur) in the DBS

For proper calibration of the instrument, standards meeting the requirements of section 11 and the nominal values in Table 1 should be used.

11.4 **Quality Control Standards** — Several additional standards (quality control standards), having sulphur concentration other than those found in Table 1, may prove useful. (See Table 3 for the recommended quality control standards.) Calibration standards prepared in accordance with par. 11.2, as well as any appropriate certified standard prepared in accordance with par. 11.1, may be used as quality control standards. Control samples may be calibration standards with sulphur levels close to those typically encountered in the normal use of this test method, or aliquot samples prepared from a larger batch of material representative of that normally encountered both in composition and sulphur level. It is recommended that at least one control sample be of the latter. Samples should be

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stored in an environment suitable for long-term storage, without degradation. Analysis of results from these samples can be carried out using control chart techniques.[4]

TABLE 2

Calibration Standards, Recommended Target Sulphur Concentrations Preparation Table[5 and 6]

Range Number	Sulphur Content (% Mass)	Mass of Toluene/iso-octane (grams)	Mass DBS (grams)
1	0.0001	50.0000	0.0000
	0.0010	50.0000	0.0021
	0.0050	50.0000	0.0112
	0.0100	50.0000	0.0226
	0.0200	50.0000	0.0454
2	0.0200	50.0000	0.0454
	0.0350	50.0000	0.0797
	0.0500	50.0000	0.1141
	0.0750	50.0000	0.1714
	0.1000	50.0000	0.2289

11.4.1 It is recommended to discard the test aliquot of the standard after measurement, therefore the lower costs prepared calibration standards or the prepared quality control standards are recommended for daily use. (See Tables 3 and 4 for recommendations on choosing and preparing the quality control standards.)

TABLE 3

Recommended Quality Control Standards

Range Number	Sulphur Content Range (% Mass)	Sulphur Content (% Mass)
1	0.0001 — 0.0200	0.0025
		0.0075
		0.0125
		0.0175
2	0.0200 — 0.1000	0.0250
		0.0450
		0.0675
		0.0800

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[4] Reference ASTM MNL 7, Manual on Presentation of Data and Control Chart Analysis, section 3,"Control Charts for Individuals."

*[5] Weigh the DBS to the recommended mass as close as possible. It is important that the **exact** mass is known and thus the exact concentration of the prepared standards can be calculated and entered into the instrument for calibration purposes.*

[6] The toluene/iso-octane mixture shall not be assumed to be 0.0000% mass sulphur. Consult your supplier for an accurate sulphur concentration or test the toluene/iso-octane mixture using ASTM D 5453, D 5623 or D 1266 (Annexe A1). If the toluene/iso-octane mixture being used for the preparation of standards contains sulphur, this value should be included in the calculated sulphur content of the prepared standards in accordance with the equation in par. 11.3.

TABLE 4

Suggested Quality Control Standards Preparation Table[5 and 6]

Range Number	Sulphur Content (% Mass)	Mass of Toluene/ Iso-octane (grams)	Mass DBS (grams)
1	0.0025	50.0000	0.0055
	0.0075	50.0000	0.0169
	0.0125	50.0000	0.0283
	0.0175	50.0000	0.0397
2	0.0250	50.0000	0.0569
	0.0450	50.0000	0.1026
	0.0675	50.0000	0.1542
	0.0800	50.0000	0.1829

11.5 **Storage of Standards** — Store standards in dark (amber) tightly closed glass bottles having Teflon-lined caps, in a cool, dark, place. After a prolonged period of storage, mix each standard in its storage bottle before use. The standard must be free of air bubbles before it is analyzed. Discard the standard if any sediment, phase separation or discoloration is observed.

12. PREPARATION OF INSTRUMENT

12.1 Set up the instrument according to the manufacturer's instructions. (It is recommended that whenever possible the instrument should be left on continuously to maintain optimum stability.)

13. PROCEDURE

13.1 Prepare the sample cup (sample cell) in accordance with section 10 and fill it with sample to a minimum depth of 5 mm. Provide adequate head space. Provide a vent hole in the side opposite the window to reduce bowing of the cup window during the measurement of samples.

13.2 **Instrument Calibration** — Calibrate the instrument for each of the two ranges as listed in Table 1, following the manufacturer's instructions. The calibration procedure involves setting up the instrument for the recording of the net sulphur X-ray intensity, followed by measurement of known standards. Measure each standard three times using a counting time of 200 to 300 s. The average values are used to develop a calibration curve having 5 to 6 data points for each range.

13.3 **Sensitivity Check** — Measure the appropriate certified calibration standard three times immediately after calibration at a given concentration range. Record the individual results and their average. Use the average value obtained as the reference for any future measurements using this calibration curve.

13.3.1 Quality control standards are used to verify instrument stability. These quality control standards must be prepared using the same techniques as the preparation of the calibration standards. An industry-accepted practice to establish measurement process stability is to use control charts on results obtained from testing quality control materials as if they were normal production samples to verify instrument stability. Under this scheme, the control limits are generated by control chart data.[7]

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[7] *The principal factors to consider when determining the frequency of testing the quality control samples are:*

1. *Utilization factor of the analytical measurement system*
2. *Criticality of the quality being measured*
3. *Established system stability and precision performance based on historical data.*
4. *Analytical measurement, system method requirements*
5. *Contractual/regulatory obligations*
6. *Business economics*
7. *Instrument design.*

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13.4 **Analysis of Unknown Samples** — Fill the cup with the sample to be measured to at least 5 mm depth. Ensure that no air bubbles are present between the cup window and the liquid sample. Measure each sample three times (see Table 5 for the recommended counting times for the specific type of instrument). Obtain the average of the readings for the sulphur content in the unknown sample.

TABLE 5

Counting Times for Sulphur Content Analysis

Sulphur Content Range (% mass)	Counting Times (seconds)
0.0001 — 0.1000	200 — 300

14. RESULTS

14.1 Report the results to the nearest 0.0001% mass.

15. PRECISION AND BIAS

15.1 **Precision** — The precision of this test as obtained by statistical analysis of interlaboraty test results is as follows:

15.1.1 ***Repeatability*** — The difference between successive test results obtained by same operator with the same operating conditions on identical test materials would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty.

Repeatability $(r) = 0.0046(S + 0.0009)^{0.25}$

where:

S = % mass of sulphur

15.1.2 ***Reproducibility*** — The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, exceed the following values only in one case in twenty.

Reproducibility $(R) = 0.0138(S + 0.0009)^{0.25}$

Where:

S = % mass of sulphur

15.2 **Bias** — Since there is no accepted reference material suitable for measuring bias for this test method, no statement of bias can be made.

16. NOTES

16.1 Related Publication

16.1.1 Canadian General Standards Board (CGSB)

CAN/CGSB-3.0 No. 16.0 — Sulphur in Diesel Fuel by Energy Dispersive X-Ray Fluorescence Spectrometry (EDXRF).

16.2 Sources of Referenced Publications

16.2.1 The publications referred to in par. 2.1.1 and 16.1.1 may be obtained from the Canadian General Standards Board, Sales Centre, Ottawa, Canada K1A 1G6. Telephone (819) 956-0425 or 1-800-665-CGSB (Canada only). Fax (819) 956-5644.

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16.2.2 The publications referred to in par. 2.1.2 may be obtained from the American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, U.S.A., or from the Global Info Centre Canada (par. 16.2.3).

16.2.3 The publication referred to in par. 2.1.3 may be obtained from the Global Info Centre Canada, 240 Catherine Street, Suite 305, Ottawa, Ontario K2P 2G8. Telephone (613) 237-4250 or 1-800-854-7179. Fax (613) 237-4251.

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