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JIS K 2541-4 : 2003
(PAJ)

**Crude oil and petroleum products—
Determination of sulfur content
Part 4 : Energy-dispersive X-ray
fluorescence method**

ICS 75.080

Reference number : JIS K 2541-4 : 2003 (E)

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Foreword

This translation has been made based on the original Japanese Industrial Standard established by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee according to the proposal of establishing a Japanese Industrial Standard from the Petroleum Association of Japan (PAJ), with a draft of Industrial Standard based on the provision of Article 12 Clause 1 of the Industrial Standardization Law.

This Standard has been made based on **ISO/FDIS 8754 : 2003** *Petroleum products—Determination of sulfur content—Energy-dispersive X-ray fluorescence method* for the purposes of making it easier to compare this Standard with International Standard; to prepare Japanese Industrial Standard conforming with International Standard; and to propose a draft of an International Standard which is based on Japanese Industrial Standard.

Attention is drawn to the possibility that some parts of this Standard may conflict with a patent right, application for a patent after opening to the public, utility model right or application for registration of utility model after opening to the public which have technical properties. The relevant Minister and the Japanese Industrial Standards Committee are not responsible for identifying the patent right, application for a patent after opening to the public, utility model right or application for registration of utility model after opening to the public which have the said technical properties.

JIS K 2541 consists the following 7 parts with the general title *Crude oil and petroleum products—Determination of sulfur content*.

Part 1 : Wickbold combustion method

Part 2 : Oxidative microcoulometry

Part 3 : Quartz-tube combustion method (Air method)

Part 4 : Energy-dispersive X-ray fluorescence method

Part 5 : General bomb method

Part 6 : Ultraviolet fluorescence method

Part 7 : Wavelength-dispersive X-ray fluorescence method

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In the event of any doubts arising as to the contents,
the original JIS is to be the final authority.

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Crude oil and petroleum products— Determination of sulfur content Part 4 : Energy-dispersive X-ray fluorescence method

Introduction This Japanese Industrial Standard has been prepared based on the first edition of **ISO/FDIS 8754** *Petroleum products—Determination of sulfur content—Energy-dispersive X-ray fluorescence method*, published in 2003, with some modifications in technical contents.

Portions with sidelines or dotted underlines in this Standard are the items modified the original International Standard. The table of modification attached with their explanation is as shown in Annex (informative).

1 Scope This Standard specifies the method of quantitative determination of sulfur content in the range of 0.03 mass % to 5 mass % contained in crude oil, naphtha, unleaded gasoline, intermediate distilled oil, residual oil lubricant base oil and petroleum products of base material.

- Remarks 1 This method is also capable of measuring in the range between 0.01 mass % and 0.03 mass % by using test apparatus of high precision type excitation method or by carrying out the preparation of working curve appropriately.
- 2 When a doubt arises in the test results obtained according to this test method, light oil shall be tested according to the oxidative microcoulometry specified in **JIS K 2541-2**, and crude oil and heavy oil shall be tested according to the quartz-tube combustion method (air method) specified in **JIS K 2541-3**.
- 3 Although dangerous reagents, operation and test apparatus are sometimes used in this Standard, this Standard does not purport to address all the safely using method. Therefore, the user of this test method shall establish the cautionary safety measures for safety and health prior to the test.
- 4 The International Standard corresponding to this Standard is as follows.

In addition, symbols which denote the degree of correspondence in the contents between the relevant International Standard and **JIS** are IDT (identical), MOD (modified), and NEQ (not equivalent) according to **ISO/IEC Guide 21**.

ISO/FDIS 8754 : 2003 *Petroleum products—Determination of sulfur content—Energy-dispersive X-ray fluorescence method* (MOD)

- Information 1 The additives of heavy metal such as alkyl lead interfere the measurement in this method. If exists, not less than several hundreds ppm of silicon, phosphorus, calcium, oxygen, potassium,

zinc, molybdenum, barium, halogen will interfere. The apparatus capable of compensating the interference of matrix and spectrum by spectrodconvolution and interelemental correction by multiple regression is also found in the new test apparatus.

- 2 For the sample of different structure such as aromatic hydrocarbon, saturated aliphatic hydrocarbon, etc., when C/H (mass ratio of carbon/hydrogen) indicating the ratio of carbon to hydrogen has changed by 1 or more as compared with the standard sample, the measured value is sometimes influenced.
- 3 The test methods shown in Informative Table 1 are included in the group of this Standard.

Informative Table 1 Classification of test method

Group of standard	Classification of test method	Kind of oil to be applied (example)	Range of measurement
K 2541-1	Wickbold combustion method	Automobile gasoline, kerosene, light oil	1 mass ppm to 10 000 mass ppm
K 2541-2	Oxidative microcoulometry	Automobile gasoline, kerosene, light oil	1 mass ppm to 1 000 mass ppm
K 2541-3	Quartz-tube combustion method (Air method)	Crude oil, light oil, heavy oil	0.01 mass % min.
	Annex (informative) Quartz-tube combustion method (Oxygen method)		
K 2541-4	Energy-dispersive X-ray fluorescence method	Crude oil, light oil, heavy oil	0.01 mass % to 5 mass %
K 2541-5	General bomb method	Crude oil, heavy oil, lubricating oil	0.1 mass % min.
	Annex (normative) Inductively coupled plasma atomic emission spectrochemical analysis	Lubricating oil	0.05 mass % min.
K 2541-6	Ultraviolet fluorescence method	Automobile gasoline, kerosene, light oil	3 mass ppm to 500 mass ppm
K 2541-7	Wavelength-dispersive X-ray fluorescence method	Automobile gasoline, kerosene, light oil	5 mass ppm to 500 mass ppm

2 Normative references The following standards contain provisions which, through reference in this Standard, constitute provisions of this Standard. The most recent editions of the standards (including amendments) indicated below shall be applied.

JIS B 7995 *Automatic analysers for sulphur in crude oil and petroleum products*

JIS K 2251 *Crude petroleum and petroleum products—Sampling*

Remarks : **ISO 3170 : 1988** *Petroleum liquids—Manual sampling* is equivalent to the said standard.

JIS K 2541-2 *Crude oil and petroleum products—Determination of sulfur content Part 2 : Oxidative microcoulometry*

JIS K 2541-3 *Crude oil and petroleum products—Determination of sulfur content Part 3 : Quartz-tube combustion method (Air method)*

JIS K 2541-7 *Crude oil and petroleum products—Determination of sulfur content Part 7 : Wavelength-dispersive X-ray fluorescence method*

JIS K 9003 *Liquid paraffin*

JIS Z 8401 *Guide to the rounding of numbers*

JIS Z 8402-6 *Accuracy (trueness and precision) of measurement methods and results—Part 6 : Use in practice of accuracy values*

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

3 Principle of test The sample is put in the beam emitted from X-ray source and the sulfur element is excited. The excitation energy is obtained from the radiation source such as ^{55}Fe or X-ray tube. The characteristic X-rays emitted from the sample are measured, and the concentration of sulfur is obtained by comparing the cumulative count numbers of sample with the count numbers of the working curve obtained by measuring the sulfur content (mass %) of a series of standard solutions for working curve including the range of sulfur concentration during measurement.

This method shall be called the energy-dispersive X-ray fluorescence method (hereafter referred to as “radiation excitation method”).

Remarks : The mass ratio of carbon/hydrogen of sample (hereafter referred to as “C/H”) influences to the test results. Therefore, it is needed that the working curve is prepared using the test apparatus equipped with automatically correcting function of C/H or using the sulfur content standard solution of the same C/H as that of sample as much as possible.

4 Reagents and others The reagents and others shall be as follows:

4.1 Liquid paraffin (white oil), being the liquid paraffin specified in **JIS K 9003** or equivalent thereto, and having not more than 20 mass ppm sulfur content.

Remarks : In the case of sulfur content of low concentration (approximately 0.1 mass %), if the diluent having similar type of composition etc. of the sample to be measured is used for the preparation of standard solution, the matrix effect, etc. can be minimized. The sulfur content of such diluent is very low and, if possible, not more than 2 mass ppm is desirable.

4.2 Sulfur compounds, having sulfur content previously determined and used for preparation of the standard solution.

Remarks : The compounds indicated in **4.2.1** to **4.2.4** are appropriate and the calculated values are given for sulfur content. When the purity of these compounds is under 99 %, the material of certified sulfur content