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(PAJ)

**Crude oil and petroleum products—
Determination of sulfur content
Part 1 : Wickbold combustion
method**

ICS 75.080

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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 4260 : 1987 *Petroleum products and hydrocarbons—Determination of sulfur content—Wickbold combustion 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 1 : Wickbold combustion method

Introduction This Japanese Industrial Standard has been prepared based on visual titration method in respect of the titration methods specified in the first edition of **ISO 4260** *Petroleum products and hydrocarbons—Determination of sulfur content—Wickbold combustion method*, published in 1987 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 1 mass ppm to 10 000 mass ppm in gasoline, kerosene and light oil by using the Wickbold combustion method.

Remarks 1 This method may be applied to products having sulfur contents in the range 1 mass ppm to 10 000 mass ppm and is particularly suitable for the qualitative determination of distilled oil with sulfur contents of less than 300 mass ppm. The sample with sulfur contents of exceeding 300 mass ppm shall be determined by any one of the following.

- 1) method to dilute the sample with a solvent and burn the whole amount.
 - 2) method to burn only the necessary amount without burning of the whole amount.
 - 3) method to take an aliquot of absorption solution and titrate.
- 2 This test method shall not apply to the samples containing halogen not less than 100 mass ppm, calcium not less than 5 mass ppm, phosphorus not less than 2 mass ppm, lead not less than 2 mass ppm and other samples containing metal which might produce insoluble sulphate.
- 3 Concerning gasoline, kerosene and light oil, when doubts arise in the test results obtained by using Wickbold combustion method, the test shall be carried out by using the oxidative microcoulometry specified in JIS K 2541-2.
- 4 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 cautionary safety measures for safety and health prior to the test.
- 5 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 4260 : 1987 *Petroleum products and hydrocarbons—Determination of sulfur content—Wickbold combustion method (MOD)*

Information : The groups of this Standard specify the test methods shown in Informative Table 1.

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 K 0512 Hydrogen

JIS K 0557 Water used for industrial water and wastewater analysis

JIS K 1101 Oxygen

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

Remarks : In the case of automatic sampling, it may be in accordance with **ISO 3171 : 1988**.

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

JIS K 8034 *Acetone*

JIS K 8223 *Perchloric acid*

JIS K 8230 *Hydrogen peroxide*

JIS K 8680 *Toluene*

JIS K 8809 *Potassium hydrogen phthalate*

JIS K 8962 *Potassium sulfate*

JIS K 9551 *Barium perchlorate*

JIS K 9703 *2,2,4-Trimethylpentane*

JIS Q 0034 *General requirements for the competence of reference material producer*

JIS Q 0035 *Certification of reference materials—General and statistical principles*

JIS R 3503 *Glass apparatus for chemical analysis*

Remarks : **ISO 641** : 1975 *Laboratory glassware—Interchangeable spherical ground joints* is equivalent to the said standard.

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 641 : 1975 *Laboratory glassware—Interchangeable spherical ground joints*

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

3 Principle of test The sample is burnt by using oxy-hydrogen flame, the resulting sulfur oxides are converted into sulfuric acid by absorption in hydrogen peroxide aqueous solution (3 %). For the quantitative determination of sulfur content in the absorption solution, the absorption solution is condensed, buffer solution and acetone are added to it and sulfur content is obtained by titrating the sulfuric acid ion in the solution with barium perchlorate standard solution using dimethylsulfonazo III as an indicator.

4 Reagents The reagents shall be as follows:

- a) **Oxygen**, specified in **JIS K 1101**.
- b) **Hydrogen**, specified in **JIS K 0512**.
- c) **Acetone**, specified in **JIS K 8034**.
- d) **Solvent** The 2,2,4-trimethylpentane specified in **JIS K 9703** or petroleum series of solvent containing sulfur content of under 1 mass ppm.
- e) **Hydrogen peroxide aqueous solution**, prepared by adding 9 volume of water into one volume of hydrogen peroxide solution (30 %) specified in **JIS K 8230**. This solution shall be preserved in a brown bottle.

- f) **Potassium hydrogen phthalate buffer solution**, prepared by dissolving 20 g of potassium hydrogen phthalate specified in **JIS K 8809** into water to make the whole amount 1 000 mL.
- g) **Dimethylsulfonazo III indicator**, prepared by dissolving 0.1 g of dimethylsulfonazo III of the most superior quality on the market [sodium salt of 2,7-bis(sulfo-4-methylphenyl) azo-1,8-dihydroxy-naphthalene-3,6-disulfonate] in 100 mL of water⁽¹⁾. This indicator shall be preserved in a brown bottle.

Note ⁽¹⁾ When the colour change at the end point of titration has become unclear, the indicator shall be prepared again.

- h) **Barium perchlorate standard solution (0.002 5 mol/L)**, prepared as follows: weigh out approximately 0.98 g of barium perchlorate (3 hydrate) specified in **JIS K 9551**, add 4 or 5 drops of perchlorate solution (10 vol%) prepared from a small amount of water and perchlorate solution specified in **JIS K 8223**, transfer them into a 1 000 mL volumetric flask and add water to the marked line.

The rating of this solution shall be carried out as follows:

- 1) Weigh out approximately 0.44 g of potassium sulfate specified in **JIS K 8962** dried at 105 °C to 110 °C for not less than 3 h to the nearest 1 mg precisely, transfer it into a 1 000 mL volumetric flask, add water to the marked line and make it potassium sulfate solution.

Calculate the mol concentration of potassium sulfate solution according to the following formula and round off to 3 significant figures according to the provisions of **JIS Z 8401**.

$$C = \frac{K}{174.27}$$

where, C : mol concentration of potassium sulfate solution (mol/L)

K : mass of potassium sulfate weighed out (g)

- 2) Take 10 mL of potassium sulfate solution into a 200 mL beaker using a volumetric pipette precisely and add 10 mL of water.
- 3) Add approximately 50 mL of acetone and 5 mL of potassium hydrogen phthalate buffer solution, add 4 or 5 drops of dimethylsulfonazo III indicator and titrate by using barium perchlorate solution as stirring using a magnetic stirrer. When the solution has changed from purple to greenish blue and the colour change has not appeared for not less than 1 min, that shall be made the end point⁽²⁾.

Note ⁽²⁾ The end point may be easily observed by titrating against a white board.

- 4) Take 20 mL of water, titrate in the similar way to the operation of 3) as the blank test.
- 5) Calculate the mol concentration of barium perchlorate standard solution according to the following formula and round off to 3 significant figures according to the provisions of **JIS Z 8401**.

$$M = \frac{10C}{A - B}$$

where, M : mol concentration of barium perchlorate standard solution (mol/L)

A : amount of barium perchlorate standard solution required for titration (mL)

B : amount of barium perchlorate standard solution required for blank test (mL)

C : mol concentration of potassium sulfate solution (mol/L)

- i) **Barium perchlorate standard solution (0.005 mol/L)**, prepared as follows:

Weigh out approximately 1.95 g of barium perchlorate (3 hydrate), prepare in the similar way to **h)** and carry out rating.

- j) **Sulfur standard solution (250 mass ppm)**, prepared as follows:

Weigh out 0.98 g of dibutylsulfide having 21.92 mass% of sulfur content to the nearest 0.1 mg into a 100 mL volumetric flask of known mass, dissolve it by adding toluene specified in **JIS K 8680**, further add toluene to the marked line and then weigh the mass. Next, take 25 mL of this solution into a 250 mL volumetric flask using a volumetric pipette and add toluene to the marked line.

Calculate the sulfur standard value according to the following formula and round off to the nearest 0.1 according to the provisions of **JIS Z 8401**.

$$A = \frac{0.219 \ 2M}{T} \times 10^5$$

where, A : sulfur standard value (mass ppm)

M : amount of dibutylsulfide weighed out (g)

T : mass of dibutylsulfide and toluene weighed out in a 100 mL volumetric flask (g)

Remarks : Dihexyl disulfide (27.36 mass% in sulfur content) or dibenzothiophene (17.40 mass% in sulfur content) may be used as the reagent for sulfur standard solution.

- k) **Sulfur standard solution (50 mass ppm)**, prepared as follows:

Weigh out 20 mL of sulfur standard solution (250 mass ppm) into a 100 mL volumetric flask using a volumetric pipette and add toluene specified in **JIS K 8680** to the marked line. This sulfur standard value is $A/5$ and rounded off to the nearest 0.1 according to the provisions of **JIS Z 8401**.

- l) **Sulfur standard solution (5 mass ppm)**, prepared as follows:

Weigh out 2 mL of sulfur standard solution (250 mass ppm) into a 100 mL volumetric flask using a volumetric pipette and add toluene specified in **JIS K 8680** to the marked line. This sulfur standard value is $A/50$ and rounded off to the nearest 0.1 according to the provisions of **JIS Z 8401**.