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(PAJ)

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

ICS 75.080

Reference number : JIS K 2541-3 : 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 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.

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

1 Scope This Japanese Industrial Standard specifies the method of quantitative determination of the sulfur content of not less than 0.01 mass % in crude oil, light oil and heavy oil by the quartz-tube combustion method (air method).

Remarks 1 This method can not be applied to the sample compounded with additives containing the following:

- a) Metal to produce insoluble sulfate (barium, calcium, etc.)
- b) Elements to produce acid by combustion (phosphorus, nitrogen, chlorine)

2 Although dangerous reagents, operation and test apparatus are sometimes used in this Standard, this Standard does not purport to address all the safety precautions. Therefore, the user of this Standard shall establish the precautionary measures for safety and health prior to the test.

Information : The group of standards including 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 C 1601 *Indicating thermoelectric thermometers*
- JIS C 1602 *Thermocouples*
- JIS K 0557 *Water used for industrial water and wastewater analysis*
- JIS K 1101 *Oxygen*
- JIS K 2251 *Crude petroleum and petroleum products—Sampling*
- JIS K 2601 *Testing methods for crude petroleum*
- JIS K 2839 *Glasswares for testing apparatus of petroleum products*
- JIS K 8001 *General rule for test methods of reagents*
- JIS K 8005 *Reference materials for volumetric analysis*
- JIS K 8102 *Ethanol (95)*
- JIS K 8230 *Hydrogen peroxide*
- JIS K 8517 *Potassium dichromate*
- JIS K 8576 *Sodium hydroxide*
- JIS K 8896 *Methyl red*
- JIS K 8897 *Methylene blue*
- JIS K 8951 *Sulfuric acid*
- 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*
- 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*

3 Principle of test The sample is burnt by introducing air into the quartz combustion tube heated at 950 °C to 1 100 °C. The produced sulfur oxides are absorbed in hydrogen peroxide aqueous solution (3 %) to make sulfuric acid, and the sulfur content is obtained by the neutralization titration of this sulfuric acid by using sodium hydroxide standard solution.

4 Reagents The reagents shall be as follows:

- a) **Hydrogen peroxide aqueous solution (3 %)**, prepared by adding nine volumes of water into one volume of hydrogen peroxide aqueous solution (30 %) specified in **JIS K 8230**. This solution shall be preserved in a brown bottle.
- b) **The 0.05 mol/L sodium hydroxide standard solution**, prepared by diluting approximately 2 g of sodium hydroxide, specified in **JIS K 8576**, in water to

make 1 000 mL. This solution shall be preserved in a brown bottle fixed with a protective tube filled with soda-lime so as not to absorb the carbon dioxide from the air.

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

- 1) Weigh out 1 g to 1.25 g of dry sulfamic acid specified in **JIS K 8005** to the nearest 0.1 mg and dilute it with water to the marked line by using a 250 mL volumetric flask.
- 2) Take 25 mL of sulfamic acid solution prepared in 1) into an Erlenmeyer flask by using a volumetric pipette and titrate it with sodium hydroxide solution by using bromothymol blue indicator solution specified in **JIS K 8001**. Regard the point where the colour of the solution has changed from yellow to blue as the end point, calculate the mol concentration of sodium hydroxide standard solution from the amount of sodium hydroxide solution required for titration according to the following formula and round off to the nearest 0.000 1 according to the requirements in **JIS Z 8401**.

$$N = \frac{1.030A}{V}$$

where, N : mol concentration of sodium hydroxide standard solution (mol/L)

 A : amount of sulfamic acid weighed out (g)

 V : amount of sodium hydroxide solution required for titration (mL)

Remarks : In the case of the sample with a small amount of sulfur content, 0.02 mol/L sodium hydroxide standard solution shall be used.

- c) **Mixed indicator**⁽¹⁾, prepared by mixing the following two indicators with equal amounts. This mixed indicator shall be preserved in a brown bottle and shall not be used after one week or longer has elapsed.

Note (1) A 0.1 % solution of methyl purple may be used instead of a mixed indicator.

- 1) The indicator prepared by dissolving 0.125 g of methyl red specified in **JIS K 8896** in 100 mL of ethanol (95) specified in **JIS K 8102**.
- 2) The indicator prepared by dissolving 0.083 g of methylene blue specified in **JIS K 8897** in 100 mL of ethanol (95) specified in **JIS K 8102**.

- d) **Activated carbon** It shall be granular activated carbon.

- e) **Sulfuric acid (1+1)**, prepared by adding 50 mL of sulfuric acid specified in **JIS K 8951** to 50 mL of water.

Moreover, the adding of sulfuric acid shall be carried out little by little while stirring the water.

- f) **Sodium hydroxide solution**, prepared by dissolving 30 g of sodium hydroxide specified in **JIS K 8576** in 100 mL of water.

- g) **Sulfur content certified reference material** That certified according to **JIS Q 0034** and **JIS Q 0035**.

Information : It is supplied from the Japan Petroleum Institute.

- h) **Water**, being A3 specified in **JIS K 0557**.

5 Test apparatus The test apparatus shall be as follows:

- a) **Construction of test apparatus** The test apparatus shall be composed of 1) to 11) and an example of the construction is shown in Fig. 1.

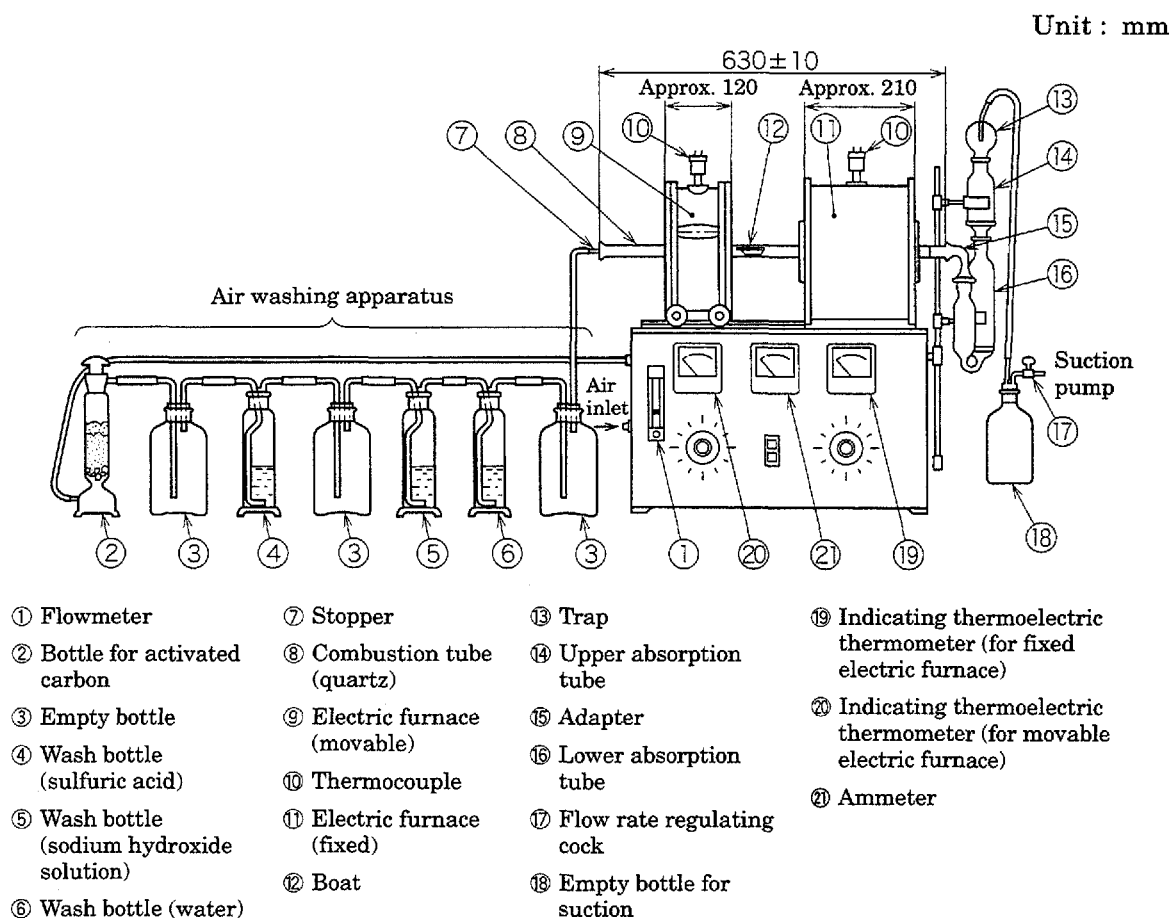


Fig. 1 Construction of test apparatus for quartz-tube combustion method (Air method) (example)

- 1) **Combustion tube**, made of transparent quartz glass and having the shape and dimensions as shown in Fig. 2⁽²⁾.

Note (2) That of Fig. 154 specified in **JIS K 2839** corresponds to this.

Unit : mm

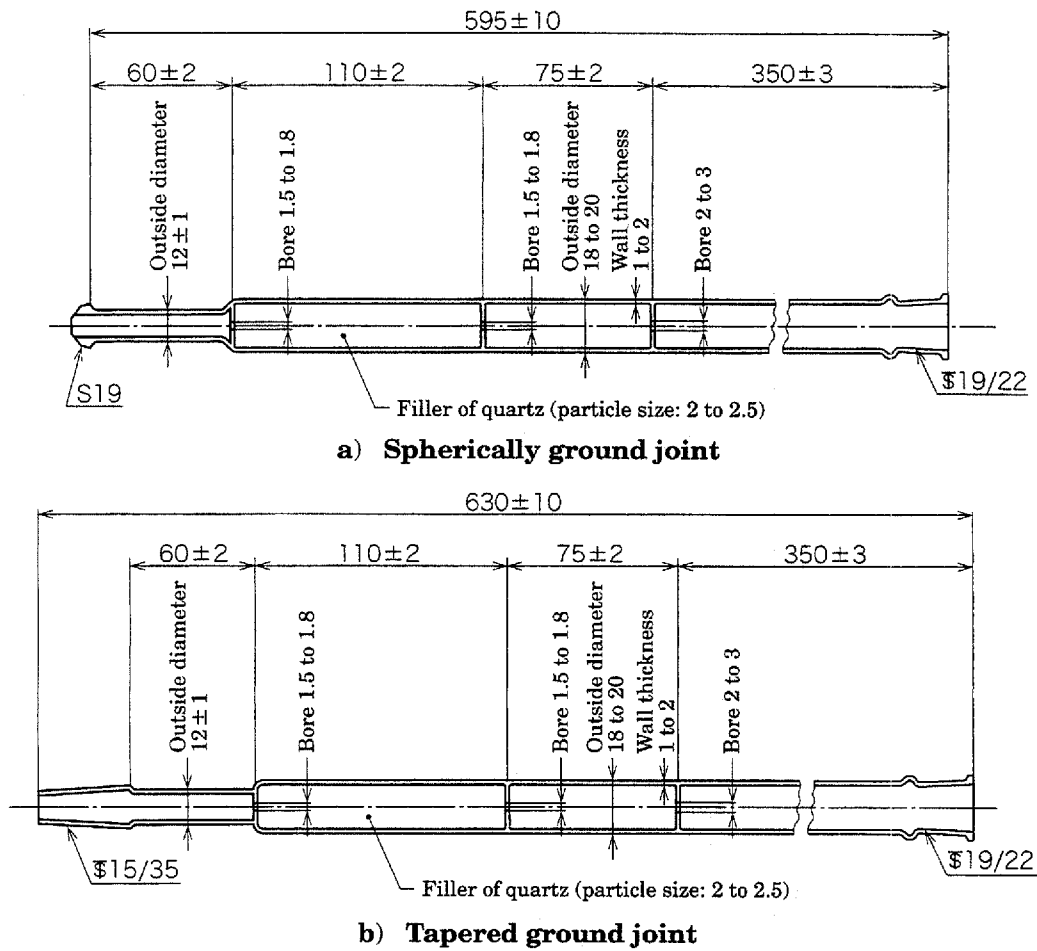
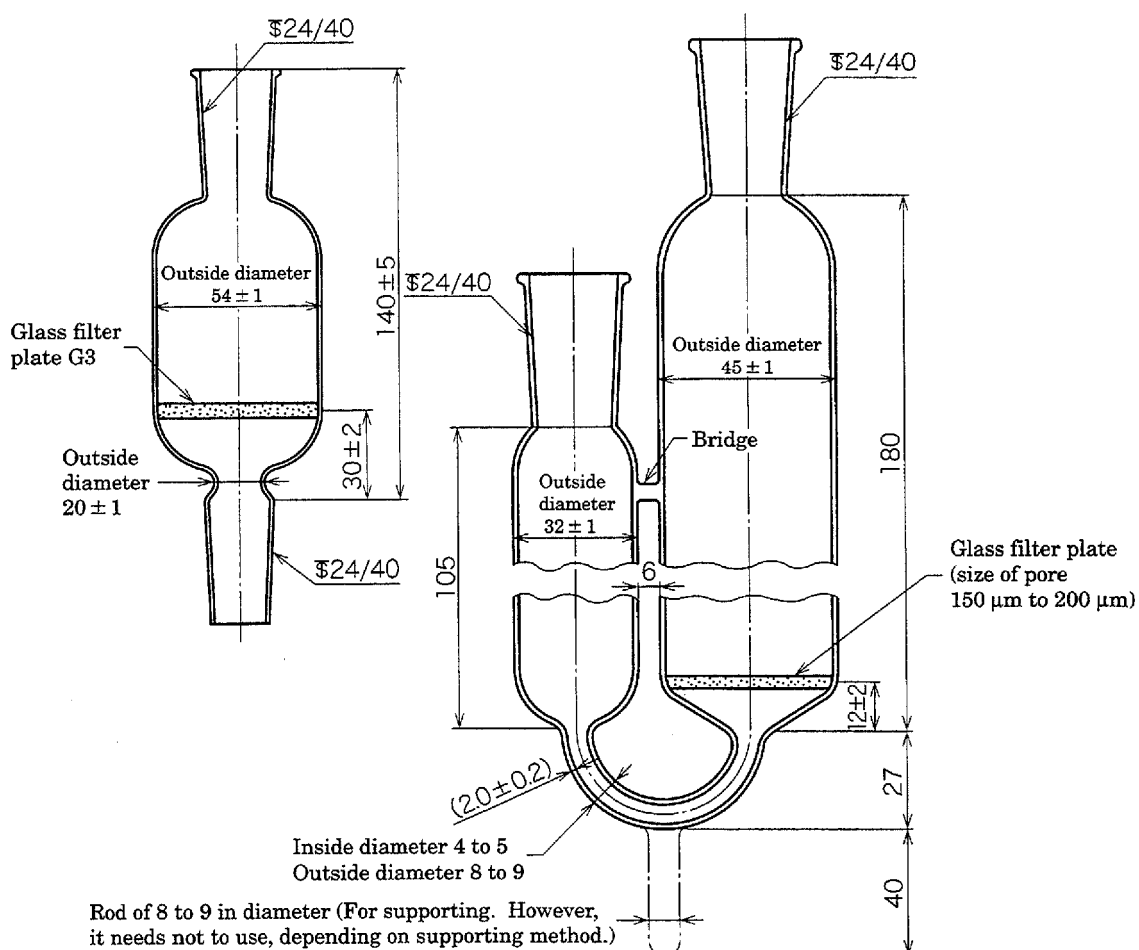


Fig. 2 Combustion tube

- 2) **Absorption tube**, composed of the upper absorption tube and the lower absorption tube, being made of borosilicate glass-1 specified in **JIS R 3503** and having the shape and dimensions as shown in Fig. 3⁽³⁾.

Note (3) Those of Fig. 64 and Fig. 79 specified in **JIS K 2839** correspond to these.

Unit : mm



Upper absorption tube

Lower absorption tube

Fig. 3 Absorption tube

- 3) **Trap**, made of borosilicate glass-1 specified in **JIS R 3503** and capable of connecting to the absorption tube by interchangeable ground joint. An example is shown in Fig. 4⁽⁴⁾.

Note (4) That of Fig. 65 specified in **JIS K 2839** corresponds to this.