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**Iron and steel — Determination of sulfur
— Part 4: Infrared absorption method
after combustion in an induction furnace**

ICS 77.040.30 ; 77.080.01

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Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of Economy, Trade and Industry, through deliberations at the Japanese Industrial Standards Committee as the result of proposal for revision of Japanese Industrial Standard submitted by The Japan Iron and Steel Federation (JISF) with the draft being attached, based on the provision of Article 12 Clause 1 of the Industrial Standardization Law applicable to the case of revision by the provision of Article 14.

Consequently **JIS G 1215-4:2017** is replaced with this Standard.

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Attention is drawn to the possibility that some parts of this Standard may conflict with patent rights, applications for a patent after opening to the public or utility model rights. The relevant Minister and the Japanese Industrial Standards Committee are not responsible for identifying any of such patent rights, applications for a patent after opening to the public or utility model rights.

JIS G 1215 series consists of the following 4 parts under the general title *Iron and steel — Determination of sulfur content* :

Part 1 : Gravimetric method after separation of iron

Part 2 : Gravimetric method after chromatographic separation

Part 3 : Methylene blue spectrophotometric method after separation of hydrosulfide

Part 4 : Infrared absorption method after combustion in an induction furnace

Iron and steel — Determination of sulfur —

Part 4 : Infrared absorption method after combustion in an induction furnace

Introduction

This Japanese Industrial Standard has been prepared based on **ISO 4935** : 1989 (Edition 1), **ISO 13902** : 1997 (Edition 1) and **ISO 15350** : 2000 (Edition 1) with some modifications of the technical contents.

The portions given sidelines or dotted underlines are the matters in which the contents of the original International Standards have been modified. A list of modifications with the explanations is given in Annex JA.

1 Scope

This Standard specifies the infrared absorption method after combustion in a high frequency induction furnace for the determination of sulfur content in iron and steel. This method is applicable to the determination of sulfur content rate (mass fraction) of 0.002 % or over to and including 0.35 % in pig iron and cast iron, and the determination of sulfur content rate (mass fraction) of 0.000 5 % or over up to and including 0.50 % in steel.

However, it is applicable to the determination of sulfur content rate (mass fraction) of 0.002 % or over up to and including 0.10 % in iron and steel in the case of preparing the calibration curve using the sulfur reference solution, to the determination of sulfur content rate (mass fraction) of 0.10 % or over up to and including 0.35 % in iron and steel in the case of preparing the calibration curve using barium sulfate, or the determination of sulfur content rate (mass fraction) of 0.003 0 % or over up to and including 0.20 % in pig iron and cast iron and the determination of sulfur content rate (mass fraction) of 0.000 5 % or over up to and including 0.50 % in steel in the case of preparing the calibration curve using steel and iron certified reference material.

NOTE 1 The determination range in **JIS G 1215** series is shown in Table 1.

Table 1 Determination range in JIS G 1215 series

Standard No.	Determination range [mass fraction (%)]
JIS G 1215-1	0.005 or over up to and incl. 0.50
JIS G 1215-2	0.003 or over up to and incl. 0.35
JIS G 1215-3	0.000 3 or over up to and incl. 0.010
JIS G 1215-4	0.000 5 or over up to and incl. 0.50

NOTE 2 The International Standards corresponding to this Standard and the symbol of degree of correspondence are as follows.

ISO 4935 : 1989 *Steel and iron — Determination of sulfur content — Infrared absorption method after combustion in an induction furnace*

ISO 13902 : 1997 *Steel and iron — Determination of high sulfur content — Infrared absorption method after combustion in an induction furnace*

ISO 15350 : 2000 *Steel and iron — Determination of total carbon and sulfur content — Infrared absorption method after combustion in an induction furnace (routine method)* (Overall evaluation : MOD)

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 21-1**.

2 Normative references

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

JIS G 1201 *Iron and steel — General rules for analytical methods*

JIS K 0970 *Piston pipettes*

JIS K 1101 *Oxygen*

JIS Z 2616 *General rules for determination of sulfur in metallic materials*

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

3 General requirements

The general requirements for the determination methods shall be in accordance with JIS G 1201 and JIS Z 2616.

4 Summary

The sample is combusted in a high frequency induction heating furnace with an accelerator in a stream of oxygen at an elevated temperature to convert sulfur to sulfur dioxide. Sulfur dioxide evolved is sent to the infrared absorption detector together with the oxygen flow, and the infrared absorption of the sulfur dioxide is measured.

5 Reagents

The reagents shall be as follows.

5.1 Oxygen, in accordance with JIS K 1101.

5.2 Iron, of high purity, in which the sulfur content rate (mass fraction) is known and is not more than 0.000 5 %. The known content rate is not required to be a certified value as long as the validity has been confirmed. For example, the sulfur content rate may be determined by the following operation.

Prepare two crucibles (6.6).

Place 0.500 g of iron in one crucible and 1.000 g in the other, and cover each with an accelerator (5.5). In the case of analysis using the calibration curve prepared with the sulfur reference solution, prior to placing iron and the accelerator, transfer to the crucible a tin capsule (6.5) previously crushed with tweezers, etc. and folded into a small size, and press it lightly against the bottom of the crucible.

Treat the crucible and contents as specified in b) and c) of 8.2. Convert the obtained reading values into mass (mg) of sulfur by means of the calibration curve (Clause 10).

Obtain the mass (m_2) (mg) of sulfur in 0.500 g of iron by subtracting the mass (m_3) (mg) of sulfur measured by placing 0.500 g of iron in the crucible from mass (m_4) (mg) of sulfur measured by placing 1.000 g of iron in the crucible. The mass (m_5) (mg) of sulfur in 1.000 g of iron shall be twice the mass (m_2) (mg) of sulfur in 0.500 g of iron.

$$m_5 = 2 \times m_2 = 2 \times (m_4 - m_3)$$

5.3 Inert ceramic (sintered clay particles), with a particle size suitable for the apparatus to be used, for example, 0.7 mm to 1.2 mm, and impregnated with sodium hydroxide.

5.4 Magnesium perchlorate $[\text{Mg}(\text{ClO}_4)_2]$, with a particle size suitable for the apparatus to be used, for example, 0.3 mm to 2.3 mm.

5.5 Accelerator Tungsten in accordance with 8.12 c) of JIS Z 2616, or the mixture of tungsten in c) and tin in b), with a sulfur content rate (mass fraction) of not more than 0.000 5 %.

The optimum type and quantity of accelerator for the apparatus and samples used shall be determined based on an analysis of a sample with a known sulfur content.

5.6 Sulfur reference solutions (potassium sulfate solution)

Weigh out, to the nearest 0.1 mg, the masses described in Table 2 of potassium sulfate (99.9 % or over in mass fraction) previously dried at 110 °C for approximately 2 h and allowed to cool in a desiccator to the room temperature.

Transfer each of these into eight separate 100 ml beakers and add water to each to dissolve.

Transfer into eight 100 ml volumetric flasks by using water, and dilute up to the mark with water.

Table 2 Preparation of sulfur reference solutions

Reference of sulfur reference solution	Mass of potassium sulfate g	Concentration of sulfur mg/ml
5.6.0 (water)	0	0
5.6.1	0.217 4	0.40
5.6.2	0.380 5	0.70
5.6.3	0.543 5	1.00
5.6.4	1.087 0	2.00
5.6.5	1.902 3	3.50
5.6.6	2.717 6	5.00
5.6.7	4.348 2	8.00

5.7 Barium sulfate (99.5 % or over in mass fraction), dried for approximately 3 h at 105 °C to 110 °C, and left to cool to room temperature in a desiccator prior to the test.

5.8 Steel and iron certified reference material, with a certified value of sulfur content rate, and having a composition (except for sulfur) similar to that of the test portion.

5.9 Reference material for correction of calibration curve The steel and iron certified reference material of which the value of sulfur content rate is close to the upper limit of the calibration curve range. The steel and iron certified reference material which was used for the preparation of calibration curve may be used.

6 Apparatus, instruments and materials

The apparatus required for combustion in the high frequency induction heating furnace and the subsequent infrared absorption measurement of the evolved sulfur dioxide may be obtained commercially from a number of manufacturers. Operate the apparatus in accordance with the manufacturer's instructions.

6.1 High frequency induction heating furnace, in accordance with 8.6 b) of JIS Z 2616.

6.2 Determination part of sulfur oxide, connected to the high frequency induction heating furnace to determine the evolved sulfur dioxide in accordance with 9.5.2 of JIS Z 2616.

6.3 Micro balance, weighing to the nearest 0.001 mg.

6.4 Piston pipettes, of 50 µl and 100 µl, and conforming to JIS K 0970.

6.5 Tin capsule, 6 mm in diameter, 18 mm in height and about 0.3 g in mass, about 0.4 ml in volume, and in which the sulfur content rate (mass fraction) is not more than 0.000 5 %.

6.6 Porcelain combustion crucible (hereafter referred to as crucible), in accordance with 8.10 of JIS Z 2616.

Prior to use, ignite the crucible in an electric furnace in air or in a current of oxygen under the conditions suitable for the crucible to be used (for example, for not less than 2 h at 1 000 °C or over). When having ignited multiple crucibles at the same time, al-

low to cool to a temperature where they are still warm, transfer to a desiccator (not coated with grease, etc.) until immediately before use.

6.7 Glass-fibre filter, cut to the diameter of the crucible and ignited for 12 h or more at 450 °C.

7 Test portion

Weigh out the following amount of the analytical sample specified for each calibration curve to use, to the nearest 1 mg.

- a) When using a calibration curve prepared with the use of sulfur reference solution of under 0.040 % in sulfur content rate (mass fraction) : 1 g
- b) When using a calibration curve prepared with the use of sulfur reference solution of 0.040 % or over in sulfur content (mass fraction) : 0.5 g
- c) When using a calibration curve prepared with the use of barium sulfate : 0.5 g

Add 0.500 g $\pm$ 0.001 g of iron (5.2) to the above weighed out sample.

- d) When using calibration curves prepared with the use of the steel and iron certified reference material : approximately equivalent to the amount of the steel and iron certified reference material used for the preparation of this calibration curve.

NOTE Typically, 0.5 g or 1 g of the steel and iron certified reference material is used.

When preparing the calibration curve of the steel and iron certified reference material with an apparatus having the mechanism to carry out the simultaneous determination of sulfur and carbon, and when the preparation range of calibration curves defined by the weighing amount overlaps, the weighing amount may be determined in consideration of the determination of carbon.

8 Operation

Warning 1 The risks related to combustion analysis are mainly burns in pre-igniting the crucibles or porcelain combustion boats or in the fusion. At all times, use tongs for combustion crucible etc. The used crucibles or porcelain combustion boats are hot; therefore, they shall be transferred to and left to cool in an adequate container capable of withstanding a high temperature and insulating its contents from external heat.

Warning 2 Normal precautions for handling oxygen cylinders shall be taken. Oxygen from the combustion process shall be removed effectively from the apparatus, since a high concentration of oxygen in a confined space can present a fire hazard.

8.1 Apparatus conditioning

The oxygen (5.1) supply shall be purified in tubes packed with the inert ceramic (sintered clay particles) (5.3) and magnesium perchlorate (5.4), but the oxygen flow shall be suspended whilst on standby. A glass-wool filter or a stainless steel net shall