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Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of International Trade and Industry through deliberations at Japanese Industrial Standards Committee in accordance with the Industrial Standardization Law. Consequently JIS M 8218:1983 is replaced with JIS M 8218:1997.

In this revision, the relevant provisions of ISO Standard are described as Annex 2 and Annex 3, intending conformance with International Standard.

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Iron ores — Methods for determination of copper content

Introduction Annex 1 of this Standard is JIS wherein methods for determination of copper content of a very small amount area are newly specified. Annex 2 is JIS prepared based on **ISO 5418-1**, *Iron ores—Determination of copper content—Part 1: 2,2'-Biquinolyl spectrophotometric method* published in 1994, and Annex 3 is JIS prepared based on **ISO 4693**, *Iron ores—Determination of copper content—Flame atomic absorption spectrometric method* published in 1986 without modifying the technical content and the structure of JIS.

1 Scope This Japanese Industrial Standard specifies the methods for determination of copper content in iron ores.

2 Normative reference The following standard contains provisions which, through reference in this Standard, constitutes provisions of this Standard. The most recent edition of the standard indicated below shall be applied.

JIS M 8202 *Iron ores—General rules for chemical analysis*

3 General matters General matters common to the methods for determination shall be as specified in **JIS M 8202**.

4 The determination methods The methods for determination of copper content in iron ores shall be as prescribed in any of the following methods.

- a) **Iron separation atomic absorption method** This method is applicable to the sample of 0.001 % (m/m) or over up to and including 0.05 % (m/m) copper content, which is as prescribed in Annex 1.
- b) **2,2'-Biquinolyl spectrophotometric method [identical with International Standard (ISO 5418-1)]** This method is applicable to the sample of 0.004 % (m/m) or over up to and including 0.8 % (m/m) copper content, which is as prescribed in Annex 2.
- c) **Flame atomic absorption spectrometric method [identical with International Standard (ISO 4693)]** This method is applicable to the sample of 0.003 % (m/m) or over up to and including 1.0 % (m/m) copper content, which is as prescribed in Annex 3.

Annex 1 (normative) Iron separation atomic absorption method

1 Summary Decompose a sample with hydrochloric acid and nitric acid, and dry it up. Dissolve with hydrochloric acid, filter, extract iron in the filtrate with 4-methyl-2-pentanone to remove. Incinerate the residue, and treat it with hydrofluoric acid. Thereafter, fuse it with potassium disulfate, and combine it with the filtrate. Spray this solution into the air-acetylene flame of an atomic absorption spectrometer, and measure the absorbance.

2 Reagents The reagents shall be as follows.

- a) **Hydrochloric acid**
- b) **Hydrochloric acid [10+6⁽¹⁾, 1+1, 2+100]**
- c) **Nitric acid**
- d) **Hydrofluoric acid**
- e) **Sulfuric acid (1+1)**
- f) **Iron (III) oxide** Iron (III) oxide is of the highest possible purity containing no copper, or of which the copper content is as low as possible and known.
- g) **Potassium disulfate**
- h) **4-methyl-2-pentanone**
- i) **Standard copper solution A (100 µgCu/ml)** Decompose 0.1000 g of copper [99.9 % (m/m) or over] with 20 ml of nitric acid (1+1) by heating. After cooling to ordinary temperature, transfer it into a 1 000 ml one-mark volumetric flask by washing with water, and dilute to the mark with water to prepare standard copper solution A.
- j) **Standard copper solution B (10 µgCu/ml)** Dilute standard copper solution A accurately 10 times with water per each service to prepare standard copper solution B.

Note (1): Since the hydrochloric acid (10+6) is used for solvent extraction, it is accurately prepared.

3 Weighing out amount of sample The weighing out amount of a sample shall be 1.0 g.

4 Procedure

4.1 Preparation of sample solution Prepare sample solution as follows.

- a) Weigh out a sample, and transfer it into a beaker (300 ml).

- b) Cover it with a watch glass, add 30 ml of hydrochloric acid, at first place the beaker at a low temperature part (60 °C to 100 °C) near the edge of hot plate, and hold it for about 1 h. Thereafter, transfer it to a high temperature part, and decompose it by heating to just before boiling for about 10 min. Then, oxidize iron or the like by adding 5 ml of nitric acid, and generate a film on the liquid surface by successively evaporating by heating. After standing to cool, add 20 ml of hydrochloric acid (1+1), and dissolve soluble salts by heating. Filter insoluble residues by using filter paper (class 5B) and a small amount of filter paper pulp, rub the inner wall of the beaker with a rubber-tipped glass rod, and transfer the adhered substance onto the filter paper. Wash the filter paper with warm hydrochloric acid (2+100) heated to about 40 °C to 60 °C until the yellow of iron (III) oxide comes not to be observed on the filter paper. Collect the filtrate and washings into a beaker (300 ml), and reserve as main solution.
- c) Transfer insoluble residues together with the filter paper into a platinum crucible (No. 30). After drying, incinerate, and let it stand to cool. Moisten the ignition residue with sulfuric acid (1+1), add 5 ml of hydrofluoric acid, and vaporize silicon dioxide and sulfuric acid by gently heating. After standing to cool, add 1.0 g of potassium disulfate thereto, cover, at first slowly heat, gradually raise temperature, heat it to a dark red heating state, and fuse the residue. Thereafter, let it stand to cool.
- d) Heat the solution obtained in b), evaporate it until a film starts to be generated on the solution surface, and add 20 ml of hydrochloric acid (10+6). After dissolving the salts, transfer it into a separatory funnel (100 ml) by washing with hydrochloric acid (10+6). Add 50 ml of 4-methyl-2-pentanone thereto, vigorously shake it for about 1 min. After standing still, transfer the lower layer aqueous phase into the original beaker. Further add 10 ml of hydrochloric acid (10+6), and mix by vigorously shaking for 30 s. After standing still, combine the lower layer aqueous phase with the previously separated aqueous phase. Gently heat the solution. After removing most of 4-methyl-2-pentanone, add 5 ml of nitric acid, and dry it up. After standing to cool, dissolve salts with 20 ml of hydrochloric acid (1+1). Put the platinum crucible obtained in c) into the solution to dissolve the melt. Wash the platinum crucible with warm water, and take it out. Concentrate the solution by heating to reduce the amount of solution to about 30 ml, cool it to ordinary temperature, transfer it into a 50 ml one-mark volumetric flask by using water, and dilute it to the mark with water.

4.2 Measurement of absorbance Aspirate a portion of the sample solution obtained in 4.1 d) into the air-acetylene flame of the atomic absorption spectrometer of which the zero point has been adjusted by using water, and measure the absorbance at 324.7 nm⁽²⁾ wavelength.

Note ⁽²⁾ When measurement at 324.7 nm is difficult according to apparatus, the absorbance may be measured at 327.4 nm.

5 Blank test Weigh out 1.0 g of iron (III) oxide [2 f)] instead of a sample, and transfer it into a beaker (300 ml). Hereafter carry out the same operation as that for the sample parallel to the sample according to the procedure of 4.1 b) to 4.2.

6 Preparation of calibration curve Prepare five beaker (300 ml) per each

copper content range of Annex 1 Table 1, weigh out 1.0 g of iron (III) oxide [2 f] per each beaker, and transfer it therein. Then, add accurately standard copper solution according to the additional amount of standard copper solution of Annex 1 Table 1. Hereafter, operate parallel to the sample according to 4.1 b) to 4.2, prepare the relation curve between the obtained absorbance and the amount of copper, and transfer the relation curve parallel so as to pass through the origin to make the calibration curve.

Annex 1 Table 1 Additional amount of standard copper solution

Copper content % (m/m)	Standard copper solution to be used	Additional amount of standard copper solution ml
0.001 or over to and excluding 0.010	B [2 j)]	0, 1, 3, 6, 10
0.010 or over up to and including 0.050	A [2 i)]	0, 1, 2, 4, 6

7 Calculation The calculation shall be as follows.

- a) Calculation of copper content** Obtain the copper amount from the absorbance obtained in 4.2 and 5, and the calibration curve prepared in 6, and calculate the copper content in a sample according to the following formula.

$$Cu = \frac{A_1 - A_2}{m} \times 100$$

where, Cu : copper content in sample [% (m/m)]

A_1 : detected amount of copper in sample solution (g)

A_2 : detected amount of copper ⁽³⁾ in blank test solution (g)

m : weighed out amount of sample (g)

Note ⁽³⁾ When copper is contained in the iron (III) oxide used for the blank test, subtract the copper amount in the weighed out iron (III) oxide.

- b) Calculation of copper oxide content** Calculate the copper oxide content in the sample from the copper content according to the following formula.

$$CuO = 1.2518 \times Cu$$

where, CuO : copper oxide content in sample [% (m/m)]

Cu : the same as in a)

8 Tolerance The tolerance shall be as specified in Annex 1 Table 2.

Annex 1 Table 2 Tolerance

Unit %(m/m)

Within-laboratory tolerance	Between-laboratories tolerance
$D(n) \times [0.0120 \times (\text{copper content}) + 0.0001]$	$2.8 \times [0.0061 \times (\text{copper content}) + 0.0003]$

When $n=2$ $D(n)=2.8$

When $n=3$ $D(n)=3.3$

When $n=4$ $D(n)=3.6$

Informative reference: Those tolerances have been obtained by using the sample of 0.0009 %(m/m) or over up to and including 0.036 %(m/m).

Annex 2 (normative) 2,2'-Biquinolyl spectrophotometric method

Introduction This Annex has been prepared based on the first edition of **ISO 5418-1** (*Iron ores—Determination of copper content—Part 1: 2,2'-Biquinolyl spectrophotometric method*) published in 1994, without modifying the technical content and structure of JIS.

Further, "Informative reference" to which side lines or underline (dotted lines) are applied in this Annex is an item which is not specified in the original International Standard.

1 Scope This Annex specifies a 2,2'-biquinolyl spectrophotometric method for the determination of the copper content of iron ores.

This method is applicable to copper contents between 0.004 % (m/m) and 0.8 % (m/m) in natural iron ores, iron ore concentrates and agglomerates, including sinter products.

Informative reference: This method is also applicable to iron raw materials such as sintered sulfide iron ore, scale and dust or briquettes manufactured from those powdery and/or granular materials, etc.

2 Normative references The following standards contain provisions which, through reference in this text, constitute provisions of this part of **JIS M 8218**. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of **JIS M 8218** are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of **IEC** and **ISO** maintain registers of currently valid International Standards.

ISO 648 : 1977	<i>Laboratory glassware—One-mark pipettes</i>
ISO 1042 : 1983	<i>Laboratory glassware—One-mark volumetric flasks</i>
ISO 3081 : 1986	<i>Iron ores—Increment sampling—Manual method</i>
ISO 3082 : 1987	<i>Iron ores—Increment sampling and sample preparation—Mechanical method</i>
ISO 3083 : 1986	<i>Iron ores—Preparation of samples—Manual method</i>
ISO 3696 : 1987	<i>Water for analytical laboratory use—Specification and test methods</i>
ISO 7764 : 1985	<i>Iron ores—Preparation of predried test samples for chemical analysis</i>

3 Principle Decomposition of the test portion by treatment with hydrochloric, nitric and perchloric acids.

Dehydration of silica, dilution and filtration. Ignition of the residue, treatment with hydrofluoric and sulfuric acids, and fusion with sodium carbonate. Dissolution of the cooled melt in the filtrate.

Reduction of copper (II) with ascorbic acid. Addition of 2,2'-biquinolyl in the presence of *N,N*-dimethylformamide to form the red-violet complex of copper (I).

Spectrophotometric measurement of the absorbance of the coloured complex at a wavelength of approximately 545 nm.

4 Reagents During the analysis, use only reagents of recognized analytical grade and water that complies with grade 3 of **ISO 3696**.

Note 1: The distillation apparatus used should not contain any copper, and deionized water should not come into contact with copper tubing or taps.

4.1 Sodium carbonate (Na_2CO_3) anhydrous powder

4.2 Iron (III) oxide, minimum purity: 99.9 % (m/m), copper content less than 0.000 2 % (m/m)

4.3 Hydrochloric acid, ρ 1.16 g/ml to 1.19 g/ml

4.4 Hydrochloric acid, ρ 1.16 g/ml to 1.19 g/ml, diluted 1+2

4.5 Hydrochloric acid, ρ 1.16 g/ml to 1.19 g/ml, diluted 1+10

4.6 Nitric acid, ρ 1.4 g/ml

4.7 Nitric acid, ρ 1.4 g/ml, diluted 1+1

4.8 Perchloric acid, ρ 1.54 g/ml, 60 % (m/m), or ρ 1.67 g/ml, 70 % (m/m)

4.9 Sulfuric acid, ρ 1.84 g/ml, diluted 1+1

4.10 Hydrofluoric acid, ρ 1.13 g/ml, 40 % (m/m), or ρ 1.85 g/ml, 48 % (m/m)

Informative reference: The ρ 1.85 g/ml herein is a mistake of ρ 1.185 g/ml.

4.11 Ascorbic acid ($\text{C}_6\text{H}_8\text{O}_6$), solution, 200 g/l Prepare this solution at the time of use.

4.12 *N,N*-Dimethylformamide [$\text{HCON}(\text{CH}_3)_2$] Warning—Take care not to inhale toxic fumes.

4.13 2,2'-Biquinolyl ($\text{C}_{18}\text{H}_{12}\text{N}_2$), solution Dissolve 0.15 g of 2,2'-biquinolyl in 250 ml of *N,N*-dimethylformamide. Protect the solution from light and store in a brown bottle.

4.14 Copper standard solutions

4.14.1 Standard solution A, 1000 $\mu\text{gCu/ml}$ Dissolve 0.500 g of copper metal [of minimum purity 99.9 % (m/m)] in 20 ml of dilute nitric acid (4.7) in a 250 ml tall-form beaker. After elimination of the nitrous fumes by boiling, cool, transfer to a 500 ml one-mark volumetric flask, dilute to volume with water and mix.

4.14.2 Standard solution B, 50 µgCu/ml Transfer 25.0 ml of standard solution A (4.14.1) to a 500 ml one-mark volumetric flask and dilute to volume with water.

5 Apparatus Ordinary laboratory equipment, including one-mark pipettes and one-mark volumetric flasks complying with the specifications of ISO 648 and ISO 1042 respectively (unless otherwise indicated).

5.1 Platinum crucible, of capacity 25 ml to 30 ml.

5.2 Muffle furnace, suitable for heating at 1 000 °C.

5.3 Spectrophotometer, suitable for measurement of absorbance at approximately 545 nm.

6 Sampling and samples

6.1 General For analysis, use a laboratory sample of minus 100 µm particle size which has been taken in accordance with ISO 3081 or ISO 3082 and prepared in accordance with ISO 3082 or ISO 3083. In the case of ores having significant contents of combined water or oxidizable compounds, use a particle size of minus 160 µm.

Note 2: A guideline on significant contents of combined water and oxidizable compounds is incorporated in ISO 7764.

Informative reference: As for contents of combined water and oxidizable compounds are incorporated in JIS M 8202.

6.2 Preparation of predried test samples Thoroughly mix the laboratory sample and, taking multiple increments, extract a test sample in such a manner that it is representative of the whole contents of the container. Dry the test sample at 105 °C±2 °C as specified in ISO 7764. (This is the predried test sample.)

7 Procedure

7.1 Number of determinations Carry out the analysis at least in duplicate in accordance with Annex 2A, independently, on one predried test sample.

Note 3: The expression "independently" means that the second and any subsequent result is not affected by the previous result(s). For this particular analytical method, this condition implies that the repetition of the procedure is carried out either by the same operator at a different time or by a different operator, including appropriate recalibration in either case.

7.2 Test portion Taking several increments, weigh, to the nearest 0.000 2 g, approximately 0.5 g or 1 g of the predried test sample (see Annex 2 Table 1) obtained in accordance with 6.2.

Note 4: The test portion should be taken and weighed quickly, to avoid reabsorption of moisture.

Annex 2 Table 1 Measurement guide for test solution

Copper content of test sample % (m/m)	Mass of test portion g	Volumetric flask ml	Cell cm
0.004 to 0.05	1.0	50	5
0.05 to 0.4	0.5	100	2
0.4 to 0.8	0.5	100	1

7.3 Blank test and check test In each run, one blank test and one analysis of a certified reference material of the same type of ore shall be carried out in parallel with the analysis of the ore sample(s) under the same conditions. A predried test sample of the certified reference material shall be prepared as specified in 6.2.

Note 5: The certified reference material should be of the same type as the sample to be analysed and the properties of the two materials should be sufficiently similar to ensure that in either case no significant changes in the analytical procedure will become necessary.

Where the analysis is carried out on several samples at the same time, the blank value may be represented by one test, provided that the procedure is the same and the reagents used are from the same reagent bottles.

Where the analysis is carried out on several samples of the same type of ore at the same time, the analytical value of one certified reference material may be used.

7.4 Determination

7.4.1 Decomposition of the test portion Place the test portion (7.2) in a 250 ml tall-form beaker, and moisten with 5 ml of water. Add 20 ml of hydrochloric acid (4.3), cover the beaker with a watch glass, and heat the solution gently without boiling until decomposition of the test portion is complete. Add 5 ml of nitric acid (4.6), followed by 10 ml of perchloric acid (4.8) and 0.2 ml of sulfuric acid (4.9). Cover the beaker with a watch glass, and heat until perchloric acid fumes are evolved. Continue heating for a further 3 min to 5 min.

Allow the beaker to cool and add 20 ml of hydrochloric acid (4.4). Boil for 1 min to remove chlorine, and dilute with 10 ml of water.

Filter the solution through a medium-texture filter paper, collecting the filtrate in a 300 ml beaker. Wash the paper with hydrochloric acid (4.5), using as small a volume as possible, until the yellow colour due to iron (III) can no longer be detected. Finally, wash with hot water until the washings are free from acid. Reserve the filtrate and washings as the main solution. Transfer the filter paper containing the residue to a platinum crucible (5.1).

Informative reference: Class 5C corresponds to "the medium-texture filter paper".