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Iron and steel—Methods for determination of copper content

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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 G 1219:1981 (former edition) is replaced with this Standard.

In this revision, the relevant ISO Standard is laid down as Annex 1 in order to harmonize with International Standard, while "Sodium thiosulfate titration method" being not used is abolished.

JIS G 1219:1997 contains Annexes as under;

Annex 1 2,2'-Diquinolyl spectrophotometric method (ISO 4946)

Annex 2 Neocuproine extraction spectrophotometric method

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Iron and steel—Methods for determination of copper content

Introduction This Japanese Industrial Standard has been prepared on the basis of **ISO 4946**, *Steel and cast iron—Determination of copper content—2,2'-Diquinolyl spectrophotometric method* published in 1984 which are edited in Annex 1, without any modification in technical content and style thereof, but some additional requirements not specified in the corresponding International Standard are given as a part of the Japanese Industrial Standard.

Further, in this Standard, the underlined portions with dotted line are the matters not specified in the original International Standard.

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

2 Normative references The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. As to those normative references, the most recent editions thereof shall be applied.

JIS G 1201 *General rules for chemical analysis of iron and steel*

JIS K 8005 *Reference materials for volumetric analysis*

JIS Z 8402 *General rules for permissible tolerance of chemical analyses and physical tests*

ISO 4946 *Steel and cast iron—Determination of copper content—2,2'-Diquinolyl spectrophotometric method*

3 General requirements General requirements common to methods for determination shall be in accordance with **JIS G 1201**, provided that this does not cover Annex 1.

4 Division of determination methods The method for determination of copper content shall be in accordance with either of the following:

- (1) **2,2'-Diquinolyl spectrophotometric method (ISO 4946)** This method is applicable to the sample of copper contents between 0.02 % (m/m) and 5 % (m/m), and the method for determination shall be in accordance with Annex 1.
- (2) **Neocuproine extraction spectrophotometric method** This method is applicable to the sample of copper contents between 0.002 % (m/m) and 1.0 % (m/m), and the method for determination shall be in accordance with Annex 2.

Annex 1 2,2'-Diquinolyl spectrophotometric method**Foreword for Annex 1**

This Annex 1 has been prepared on the basis of the first edition of **ISO 4946 Steel and cast iron—Determination of copper content—2,2'-Diquinolyl spectrophotometric method** published in 1984, without any modification in technical content and style for Japanese Industrial Standard.

Further, in this Standard the underlined portions with dotted line are the matters not specified in the original International Standard.

1 Scope and field of application This Annex 1 specifies a 2,2'-diquinolyl spectrophotometric method for the determination of copper in steel and cast iron.

The method is applicable to copper contents between 0.02 and 5 % (m/m).

2 Normative reference

ISO/R 377 *Selection and preparation of samples and test pieces for wrought steel*

Informative reference : ISO/R 377 is superseded by the following standard:

ISO 14284:1996 Steel and iron—Sampling and preparation of samples for the determination of chemical composition

3 Principle Dissolution of a test portion with appropriate acids. Fuming with perchloric acid to remove hydrochloric and nitric acids and dehydrate silicic acid. Reduction of copper (II) to copper (I) in hydrochloric acid solution by means of L(+)-ascorbic acid (see Informative reference). Formation of a coloured compound of copper (I) with 2,2'-diquinolyl. Spectrophotometric measurement at a wavelength of about 545 nm.

Informative reference : In the text of original standard, only "ascorbic acid" is described. However, in this text, this term is modified in accordance with the nomenclature of IUPAC.

4 Reagents During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity, free from copper.

4.1 High-purity iron Containing 0.001 % (m/m) or less of copper.

4.2 Hydrochloric acid ρ approximately 1.19 g/ml.

4.3 Nitric acid ρ approximately 1.40 g/ml.

4.4 Perchloric acid ρ approximately 1.54 g/ml⁽¹⁾.

Note ⁽¹⁾ Perchloric acid, ρ approximately 1.67 g/ml, may also be used. 100 ml of perchloric acid, ρ approximately 1.54 g/ml, is equivalent to 79 ml of perchloric acid, ρ approximately 1.67 g/ml.

4.5 Perchloric acid ρ approximately 1.54 g/ml, diluted 1 + 7.

4.6 Dimethylformamide (N,N-dimethylformamide) ρ approximately 0.944 g/ml.

4.7 L(+)-ascorbic acid, (200 g/l) solution Dissolve 20 g of L(+)-ascorbic acid in water, dilute to 100 ml with water and mix. Prepare this solution immediately before use.

4.8 2,2'-Diquinolyl, solution Dissolve 0.60 g of 2,2'-diquinolyl (cuproine) in the dimethylformamide (4.6), dilute to 1 litre with the same dimethylformamide and mix. Keep this solution in a dark-coloured glass receptacle and protected from the light.

4.9 Copper, standard solutions

4.9.1 Copper, standard solution Corresponding to 1.0 g of Cu per litre. Weigh, to the nearest 0.000 1 g, 1.000 0 g of high purity copper and dissolve in a minimum of the nitric acid (4.3). Heat to boiling to remove nitrous fumes. Cool and transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 1 mg of Cu.

4.9.2 Copper, standard solution Corresponding to 0.050 g of Cu per litre. Transfer 50.0 ml of the copper standard solution (4.9.1) to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 0.05 mg of Cu.

5 Apparatus Ordinary laboratory apparatus and

5.1 Spectrophotometer.

6 Sampling Carry out sampling in accordance with **ISO/R 377** or appropriate national standards for iron and steel (see Informative reference).

Informative reference : In the text of the original standard, "cast iron" is stated. However, in this Standard, it is modified to "iron and steel", because this method is also applicable for analysis of steel.

7 Procedure

Warning

- a) **Perchloric acid vapour may cause explosions in the presence of ammonia, nitrous fumes or organic material in general.**
- b) **Dimethylformamide is a hazardous substance and should not be handled by pregnant women. It should be handled with safety gloves in a fume hood.**

7.1 Test portion Weigh, to the nearest 0.001 g, approximately 0.5 g (*m*) of the test sample.