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(JISF)

**Iron and steel — Determination of nickel
— Part 2: Disodium dihydrogen
ethylenediamine tetraacetic acid back
titration by zinc after precipitation
separation of dimethylglyoximate
complex**

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In the event of any doubts arising as to the contents,
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Foreword

This Japanese Industrial Standard has been established by the Minister of Economy, Trade and Industry based on the provision of Article 14, paragraph (1) of the Industrial Standardization Act in response to a proposal for establishment of Japanese Industrial Standard with a draft being attached, submitted by The Japan Iron and Steel Federation (JISF), an accredited standards development organization. This Standard partially replaces **JIS G 1216** : 2017, which has been withdrawn.

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JIS G 1216 series consists of the following 3 parts under the general title *Iron and nickel — Determination of nickel*:

Part 1: Nickel dimethylglyoxime gravimetric method

Part 2: Disodium dihydrogen ethylenediamine tetraacetic acid back titration by zinc after precipitation separation of dimethylglyoximate complex

Part 3: Dimethylglyoxime spectrophotometric methods

Iron and steel — Determination of nickel — Part 2 : Disodium dihydrogen ethylenediamine tetraacetic acid back titration by zinc after precipitation separation of dimethylglyoximato complex

Introduction

This Japanese Industrial Standard has been prepared based on **ISO 4938** : 2016, Edition 2, with some modifications of the technical contents.

The vertical lines on both sides and dotted underlines indicate changes from the corresponding International Standard. A list of modifications with the explanations is given in Annex JA.

1 Scope

This Standard specifies, among the nickel determination methods for iron and steel, the disodium dihydrogen ethylenediamine tetraacetic acid back titration by zinc after precipitation separation of dimethylglyoximato complex.

This method is applicable to nickel contents (mass fraction) from 0.1 % to 30 %.

NOTE 1 Table 1 shows the applicable determination ranges of standards in **JIS G 1216** series.

Table 1 Determination ranges of standards in JIS G 1216 series

Standard number	Determination range [mass fraction (%)]
JIS G 1216-1	<u>0.1</u> or over up to and incl. 30
JIS G 1216-2	<u>0.1</u> or over up to and incl. 30
JIS G 1216-3	<u>0.01</u> or over up to and incl. <u>5.0</u>

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

ISO 4938 : 2016 *Steel and iron — Determination of nickel content — Gravimetric or titrimetric method* (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

Part or all of the provisions of the following standards, through reference in this text, constitute provisions of this Standard. The most recent editions of the standards (in-

cluding amendments) indicated below shall be applied. For those without the indication of the year, the most recent editions (including amendments) shall be applied.

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

JIS K 0121 *General rules for atomic absorption spectrometry*

JIS K 8001 *General rules for test methods of reagents*

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

3 Terms and definitions

For the purpose of this Standard, the terms and definitions given in Clause 3 of JIS G 1201 apply.

4 General requirements

General requirements for analyses shall be in accordance with JIS G 1201 and JIS K 0121.

5 Summary

Dissolution of a test portion with appropriate acids. Addition of citric acid or L(+)-tartaric acid, and then of aqueous ammonia to make alkaline. Addition of dimethylglyoxime and precipitation of nickel dimethylglyoxime generated. If copper and/or cobalt is present in the sample, oxidization, masking, reduction deposition or other necessary pretreatment is performed prior to precipitation.

Separation of the precipitate by filtration and dissolution with nitric acid. Addition of a specified amount of disodium dihydrogen ethylenediamine tetraacetic acid (hereafter referred to as EDTA2Na) to generate complex with nickel. After adjustment of pH, titration of excess EDTA2Na using zinc solution. Determination of the amount of residual nickel in the filtrate(s) by atomic absorption spectrometry and addition of the determined amount to the amount of nickel determined by titration.

6 Reagents

The following reagents shall be used.

6.1 Hydrochloric acid

6.2 Hydrochloric acid (1+1, 2+100)

6.3 Nitric acid

6.4 Nitric acid (1+2)

6.5 Perchloric acid

6.6 Perchloric acid (2+100)

6.7 Hydrofluoric acid

6.8 Aqueous ammonia

6.9 Aqueous ammonia (1+1)

6.10 Ammoniacal washing water Add one or two drops of aqueous ammonia to approximately 500 ml of warm water, and mix. This washing water shall be prepared at each time of use.

6.11 Aluminium chips Thin shaven chips of aluminium 99.99 % or greater in mass fraction and 1.7 mm to 3 mm in thickness.

6.12 Potassium bromate

6.13 Sodium disulfate ($\text{Na}_2\text{S}_2\text{O}_7$)

6.14 Potassium disulfate

6.15 Acetic acid

6.16 Citric acid solution Weigh 500 g of citric acid monohydrate, dissolve in water, and dilute to 1 000 ml with water.

6.17 L(+)-tartaric acid solution (500 g/L)

6.18 L(+)-ascorbic acid solution (10 g/L) This solution shall be prepared at each time of use.

6.19 Ammonium acetate solution (200 g/L)

6.20 Diammonium hydrogen citrate solution (100 g/L)

6.21 (+)-Sodium tartrate solution Weigh 10 g of (+)-sodium tartrate dihydrate, dissolve in water and dilute to 1 000 ml with water.

6.22 Dimethylglyoxime solution Weigh 10 g of dimethylglyoxime, and dissolve in 1 000 ml of ethanol (95). Otherwise, weigh 5 g of dimethylglyoxime, dissolve in 250 ml of sodium hydroxide solution (20 g/L), and dilute to 500 ml with water.

6.23 Hydrazinium sulfate solution (100 g/L)

6.24 0.02 mol/L EDTA2Na solution (7.445 g/L) Preparation, standardization and calculation of factor of this solution shall be in accordance with **JA.6.4 c) 3)** of **JIS K 8001**.

6.25 0.02 mol/L zinc solution (1.307 6 g/L) Preparation and calculation of factor of this solution shall be in accordance with **JA.6.4 a) 3)** of **JIS K 8001**.

6.26 Xylenol orange solution Preparation of this solution shall be in accordance with Table JA.7 of **JIS K 8001**.

7 Apparatus

The following apparatus shall be used.

7.1 Apparatus for controlled-potential electrolysis, with a reference electrode (saturated calomel reference electrode or silver-silver chloride electrode) and a platinum electrode.

NOTE The procedure in Clause 9 uses a saturated calomel reference electrode. Figure 1 illustrates an example of apparatus construction.