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(JISF)

**Iron and steel—ICP atomic
emission spectrometric method—
Part 2: Determination of various
elements—Decomposition with
phosphoric and sulfuric acids**

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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 1258-2:2007** is replaced with this Standard.

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JIS G 1258 series consists of the following 8 parts under the general title “*Iron and steel—ICP atomic emission spectrometric method*”:

Part 0: General rules

Part 1: Determination of various elements—Decomposition with acids and fusion with potassium disulfate

Part 2: Determination of various elements—Decomposition with phosphoric and sulfuric acids

Part 3: Determination of various elements—Decomposition with acids and fusion with sodium carbonate

Part 4: Determination of niobium content—Dissolution in phosphoric and sulfuric acids or Dissolution in acids and fusion with potassium disulfate

Part 5: Determination of boron content—Dissolution in phosphoric and sulfuric acids

Part 6: Determination of boron content—Dissolution in acids and fusion with sodium carbonate

Part 7: Determination of boron content—Distillation as trimethyl borate

Iron and steel—ICP atomic emission spectrometric method—Part 2: Determination of various elements— Decomposition with phosphoric and sulfuric acids

Introduction

This Japanese Industrial Standard has been established in 2007, and the revision at this time is to respond to the recent expansion of applicable elements.

No corresponding International Standard has been established at this point.

1 Scope

This Standard specifies ICP atomic emission spectrometric method using decomposition in phosphoric and sulfuric acids to determine the content rate of 14 components specified in table 1 in steel. This method applies to the determination of each component within the range specified in table 1, however, it is not applicable to steels when they contain one or more components of content rate over the maximum limit of determination specified in table 1. In the case of arsenic, this method is not applicable to steels of iron content rate under 90 % (mass fraction).

Table 1 Applicable element and determination range

Applicable element	Determination range [mass fraction (%)]
Manganese	≥ 0.01 ≤ 20.0
Nickel	≥ 0.01 ≤ 30.0
Chromium	≥ 0.01 ≤ 35.0
Molybdenum	≥ 0.01 ≤ 10.0
Copper	≥ 0.01 ≤ 5.0
Tungsten	≥ 0.10 ≤ 10.0
Vanadium	≥ 0.01 ≤ 5.0
Cobalt	≥ 0.01 ≤ 20.0
Titanium	≥ 0.001 ≤ 3.0
Niobium	≥ 0.01 ≤ 5.0
Calcium	≥ 0.001 ≤ 0.005
Magnesium	≥ 0.0005 ≤ 0.011
Arsenic	≥ 0.003 ≤ 0.012
Zirconium	≥ 0.010 ≤ 0.060

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) shall be applied.

JIS G 1258-0 *Iron and steel—ICP atomic emission spectrometric method—Part 0: General rules*

JIS H 1363 *Methods for determination of zirconium in aluminium alloys*

JIS K 0116 *General rules for atomic emission spectrometry*

JIS K 8001 *General rule 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 General rules

General rules common to determination methods shall be in accordance with **JIS G 1258-0** and **JIS K 0116**.

4 Summary

Add sulfuric and phosphoric acids to the sample, and heat until the sulfur trioxide creates the white smoke. Add hydrogen peroxide, water and hydrochloric acid to dissolve salt. Add yttrium to the solution as an internal reference element¹⁾. Spray part of this solution into the argon plasma of ICP atomic emission spectrometer. Measure the emission intensity of the analytical lines of the analyte and yttrium. Calculate the emission intensity ratio of each analyte to that of yttrium.

NOTE : For the analytical line to measure the emission intensity, the specifications common to **JIS G 1258** series are described in 5.1 of **JIS G 1258-0**. An example of analytical line is shown in table 2.

Note ¹⁾ For the internal reference element and the amount to be added, the specifications common to **JIS G 1258** series are described in 5.2 of **JIS G 1258-0**.

Table 2 Example of analytical line

Unit: nm

Component to be measured	Analytical line
Manganese	257.61, 293.31
Nickel	231.60, 341.68
Chromium	267.72, 276.65
Molybdenum	202.03, 281.62, 386.41
Copper	324.75, 327.40
Tungsten	207.91, 220.45
Vanadium	292.40, 311.07
Cobalt	228.62, 345.35
Titanium	334.94, 337.28
Niobium	309.42, 313.08, 319.50
Calcium	393.37
Magnesium	279.55
Arsenic	189.04, 193.70
Zirconium	339.20, 343.82
Yttrium	371.03

5 Reagents

The reagents shall be as follows.

To determine calcium, reagents having the lowest possible calcium content shall be selected. To prepare a series of blank, calibration and sample solutions, all reagents to be used shall be taken from the same batch. All water to be used shall be purified within the same time period.

NOTE : For the standard solutions of reagents, the specifications common to **JIS G 1258** series are described in 5.3 of **JIS G 1258-0**.

5.1 Hydrochloric acid

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

5.3 Nitric acid

5.4 Aqua regia (hydrochloric acid 3, nitric acid 1)

5.5 Acid mixture (sulfuric acid 1, phosphoric acid 3, water 2)

5.6 Iron Iron of high purity, in which the content rate of the analyte is under 1/10 of its minimum limit of determination, or in which the content rate of the analyte is known and is not more than its minimum limit of determination. The known content rate is not required to be a certified value as long as the validity has been confirmed.

5.7 Hydrogen peroxide (1+1)

5.8 Yttrium solution (Y: 1 mg/ml)

Weigh 1.270 g of diyttrium trioxide (mass fraction 99.9 % min.), transfer into a beaker (200 ml), cover with a watch glass, add 50 ml of hydrochloric acid (1+1) and heat gently until the yttrium decomposes. Cool to ordinary temperature, rinse the bottom surface of the watch glass with water and remove the watch glass. Transfer the solution into a 1 000 ml volumetric flask using water and dilute to the mark with water.

5.9 Manganese standard solution A (Mn: 5 mg/ml)

Weigh 5.000 g of manganese (mass fraction 99.9 % min.), transfer into a beaker (300 ml), cover with a watch glass, add 100 ml of nitric acid (1+1) by slow degrees, and heat gently until the manganese decomposes. Cool to ordinary temperature, rinse the bottom surface of the watch glass with water and remove the watch glass. Transfer the solution into a 1 000 ml volumetric flask using water and dilute to the mark with water.

5.10 Manganese standard solution B (Mn: 500 µg/ml)

Dilute manganese standard solution A (5.9) exactly 10 times with water immediately before use.

5.11 Manganese standard solution C (Mn: 50 µg/ml)

Dilute manganese standard solution A (5.9) exactly 100 times with water immediately before use, or dilute manganese standard solution B (5.10) exactly 10 times with water immediately before use.

5.12 Nickel standard solution A (Ni: 25 mg/ml)

Weigh 25.00 g of nickel (mass fraction 99.9 % min.), transfer into a beaker (500 ml), cover with a watch glass, add 200 ml of nitric acid (1+1) by slow degrees, and heat gently until the nickel decomposes. Cool to ordinary temperature, rinse the bottom surface of the watch glass with water and remove the watch glass. Transfer the solution into a 1 000 ml volumetric flask using water and dilute to the mark with water.

5.13 Nickel standard solution B (Ni: 5 mg/ml)

Dilute nickel standard solution A (5.12) exactly five times with water immediately before use.

5.14 Nickel standard solution C (Ni: 500 µg/ml)

Dilute nickel standard solution A (5.12) exactly 50 times with water immediately before use, or dilute nickel standard solution B (5.13) exactly 10 times with water immediately before use.

5.15 Nickel standard solution D (Ni: 50 µg/ml)

Dilute nickel standard solution B (5.13) exactly 100 times with water immediately before use, or dilute nickel standard solution C (5.14) exactly 10 times with water immediately before use.