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(JISF)

**Iron and steel—Atomic absorption
spectrometric method—Part 17:
Determination of antimony—
Section 2: Electrothermal
atomization**

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Foreword

This translation has been made based on the original Japanese Industrial Standard established by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee according to the proposal for establishment 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.

Consequently, **JIS G 1257**:2000 has been withdrawn and partially replaced with this Standard.

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JIS G 1257 series consists of the following 32 parts under the general title “*Iron and steel—Atomic absorption spectrometric method*”:

JIS G 1257-0 *Part 0: General rules*

JIS G 1257-1 *Part 1: Determination of manganese—Flame atomization after decomposition with acids*

JIS G 1257-2 *Part 2: Determination of phosphorus—Flame atomization indirect method after extraction of molybdophosphate*

JIS G 1257-3 *Part 3: Determination of nickel—Flame atomization after decomposition with acids*

JIS G 1257-4 *Part 4: Determination of chromium—Flame atomization after decomposition with acids*

JIS G 1257-5 *Part 5: Determination of molybdenum—Flame atomization after decomposition with acids*

JIS G 1257-6 *Part 6: Determination of copper—Flame atomization after decomposition with acids*

JIS G 1257-7 *Part 7: Determination of vanadium—Flame atomization after decomposition with acids*

JIS G 1257-8 *Part 8: Determination of cobalt—Flame atomization after decomposition with acids*

JIS G 1257-9 *Part 9: Determination of titanium—Flame atomization after decomposition with acids*

JIS G 1257-10-1 *Part 10: Determination of aluminium—Section 1: Flame atomization after decomposition with acids*

JIS G 1257-10-2 *Part 10: Determination of aluminium—Section 2: Determination of acid-soluble aluminium*

- JIS G 1257-10-3 *Part 10: Determination of aluminium—Section 3: Flame atomization after separation of iron*
- JIS G 1257-10-4 *Part 10: Determination of aluminium—Section 4: Electrothermal atomization*
- JIS G 1257-11-1 *Part 11: Determination of tin—Section 1: Flame atomization after extraction of iodide*
- JIS G 1257-11-2 *Part 11: Determination of tin—Section 2: Electrothermal atomization*
- JIS G 1257-12-1 *Part 12: Determination of lead—Section 1: Flame atomization after decomposition with acids*
- JIS G 1257-12-2 *Part 12: Determination of lead—Section 2: Flame atomization after extraction of iodide*
- JIS G 1257-12-3 *Part 12: Determination of lead—Section 3: Electrothermal atomization*
- JIS G 1257-13 *Part 13: Determination of magnesium—Flame atomization after decomposition with acids*
- JIS G 1257-14 *Part 14: Determination of calcium—Flame atomization after decomposition with acids*
- JIS G 1257-15-1 *Part 15: Determination of zinc—Section 1: Flame atomization after decomposition with acids*
- JIS G 1257-15-2 *Part 15: Determination of zinc—Section 2: Flame atomization after extraction of tetra-hexylammonium iodide-trioctylamine complex*
- JIS G 1257-16-1 *Part 16: Determination of bismuth—Section 1: Flame atomization after extraction of iodide*
- JIS G 1257-16-2 *Part 16: Determination of bismuth—Section 2: Electrothermal atomization*
- JIS G 1257-17-1 *Part 17: Determination of antimony—Section 1: Flame atomization after extraction of iodide*
- JIS G 1257-17-2 *Part 17: Determination of antimony—Section 2: Electrothermal atomization*
- JIS G 1257-18-1 *Part 18: Determination of tellurium—Section 1: Flame atomization after extraction of iodide*
- JIS G 1257-18-2 *Part 18: Determination of tellurium—Section 2: Electrothermal atomization*
- JIS G 1257-19-1 *Part 19: Determination of arsenic—Section 1: Electrothermal atomization*
- JIS G 1257-19-2 *Part 19: Determination of arsenic—Section 2: Hydride generation method (to be established)*
- JIS G 1257-20 *Part 20: Determination of selenium—Electrothermal atomization*

Iron and steel—Atomic absorption spectrometric method—

Part 17: Determination of antimony—

Section 2: Electrothermal atomization

Introduction

This Japanese Industrial Standard has been prepared based on the first edition of **ISO 10698** published in 1994 without any modifications of the technical contents.

The portions with dotted underlines are the matters not given in the original International Standard.

1 Scope

This Standard specifies an electrothermal atomic absorption spectrometric method for the determination of the antimony content in steel. The method is applicable to antimony contents (mass fraction) of 0.000 5 % or over up to and including 0.010 %.

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

ISO 10698:1994 *Steel—Determination of antimony content—Electrothermal atomic absorption spectrometric method* (IDT)

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. For standards with the year indication, only the editions of the indicated year shall be applied and the revisions (including amendments) made thereafter shall not be applied. For those without the indication of the year, the most recent edition (including amendments) shall be applied.

JIS G 0417 *Steel and iron—Sampling and preparation of samples for the determination of chemical composition*

NOTE : Instead of **ISO 377-2:1989** *Selection and preparation of samples and test pieces of wrought steels—Part 2: Samples for the determination of the chemical composition*, which the corresponding International Standard cites as a normative reference, this Standard cites **JIS G 0417**, an identical **JIS** standard to **ISO 14284** *Steel and iron—Sampling and preparation of samples for the determination of chemical composition*, which has replaced **ISO 377-2**.

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

JIS K 0557 *Water used for industrial water and wastewater analysis*

JIS R 3505 Volumetric glassware

ISO 385-1:1984 Laboratory glassware—Burettes—Part 1: General requirements

ISO 648:1977 Laboratory glassware—One-mark pipettes

ISO 1042 Laboratory glassware—One-mark volumetric flasks

ISO 3696 Water for analytical laboratory use—Specification and test methods

ISO 5725:1986 Precision of test methods—Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests

3 Principle

Dissolution of a test portion in hydrochloric and nitric acids and dilution of the solution to a known volume. Introduction of a known volume of the solution into an electrothermal atomizer of an atomic absorption spectrometer. Measurement of the atomic absorption of the 217.6 nm spectral resonance line energy emitted by an antimony hollow cathode lamp or an antimony electrodeless discharge lamp, using background correction.

Calibration by the standard additions technique.

NOTE : In this Standard, solutions prepared (or have been prepared) by standard additions to the test solution are referred to as the test addition solutions, and those prepared by standard additions to the blank solutions, as the blank addition solutions.

4 Reagents

During analysis, unless otherwise stated, use only reagents of recognized analytical grade and Grade 2 water as specified in **ISO 3696**. Water used may alternatively be Grade A3 or A4 water specified in **JIS K 0557**.

4.1 Nitric acid, density about 1.40 g/ml.

4.2 Hydrochloric acid, density about 1.19 g/ml.

4.3 Phosphoric acid, density about 1.71 g/ml.

4.4 Nitric acid, density about 1.40 g/ml, diluted 1+1.

4.5 Antimony standard solutions

4.5.1 Antimony stock solution (Sb: 1.00 g/L)

Weigh, to the nearest 0.1 mg, 0.100 g of high purity antimony metal (mass fraction 99.9 % min.). Transfer to a beaker (100 ml) and dissolve in 30 ml of hydrochloric acid (4.2) and 5 ml of nitric acid (4.1). Cover with a watch glass, and heat until it is completely dissolved and boil gently to expel oxides of nitrogen. Cool to the ordinary temperature and remove the watch glass after rinsing its bottom surface with nitric acid (4.4) and collecting the washings in the beaker. Transfer the solution quantitatively to a 100 ml volumetric flask using nitric acid (4.4). Dilute to the mark with nitric acid (4.4). Store in a polyethylene bottle.

1 ml of this stock solution contains 1.00 mg of antimony.