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(JISF)

Iron and steel — Determination of
nitrogen — Part 3: Thermal
conductimetric method after fusion in a
current of inert gas

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Mita MT Building, 3-13-12, Mita, Minato-ku, Tokyo, 108-0073 JAPAN

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

	Page
Introduction	1
1 Scope	1
2 Normative references	1
3 Terms and definitions	2
4 General	2
5 Summary	3
6 Reagents	3
7 Apparatus and instruments	4
8 Weighing of sample	5
9 Procedure	5
9.1 Preparatory operation	5
9.2 Determination	5
10 Blank test	6
11 Preparation of calibration curve	7
11.1 General	7
11.2 Calibration curve with nitrogen standard solution	7
11.3 Calibration curve with steel and iron certified reference material	9
12 Correction of calibration curve	9
13 Calculation	10
13.1 Calibration curve with nitrogen standard solution	10
13.2 Calibration curve with steel and iron certified reference material	10
14 Precision	11
Annex JA (informative) Comparison table between JIS and corresponding International Standard	13

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 This Standard partially replaces **JIS G 1228 : 2006**, which has been withdrawn.

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JIS G 1228 series consists of the following 3 parts under the general title *Iron and steel — Determination of nitrogen* :

Part 1 : Amidosulfuric acid titrimetric method after ammonium distillation separation

Part 2 : Spectrophotometric methods after ammonium distillation separation

Part 3 : Thermal conductimetric method after fusion in a current of inert gas

Iron and steel-Determination of nitrogen-Part 3 : Thermal conductimetric method after fusion in a current of inert gas

Introduction

This Japanese Industrial Standard has been prepared based on **ISO 10720** : 1997, Edition 1, 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 the thermal conductimetric method after fusion in a current of inert gas, among methods for the determination of nitrogen in steel and iron.

The method is applicable to the determination of a nitrogen content (mass fraction) of 0.000 8 % or over up to and including 0.5 %.

NOTE 1 The determination range in **JIS G 1228** (all parts) is shown in Table 1.

Table 1 Determination range in JIS G 1228 (all parts)

Standard No.	Determination range [mass fraction (%)]
JIS G 1228-1	0.002 or over up to and incl. 0.5
JIS G 1228-2	0.000 5 or over up to and incl. 0.050
JIS G 1228-3	0.000 8 or over up to and incl. 0.5

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

ISO 10720 : 1997 *Steel and iron — Determination of nitrogen content — Thermal conductimetric method after fusion in a current of inert gas (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 (including amendments) indicated below shall be applied.

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

JIS G 1228-1 *Iron and steel — Determination of nitrogen — Part 1 : Amidosulfuric acid titrimetric method after ammonium distillation separation*

JIS G 1228-2 *Iron and steel — Determination of nitrogen — Part 2 : Spectrophotometric methods after ammonium distillation separation*

JIS K 0970 *Piston pipettes*

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 following terms and definitions, and those given in JIS G 1201, Clause 3, apply.

3.1

gas extraction

operation to fuse a steel sample at a high temperature and to release the nitride constituting nitrogen and/or dissolved nitrogen in the sample in the form of molecular nitrogen

Note 1 to entry The term “gas extraction” deviates from the definition of the term “extraction” given in JIS K 0211. It is nevertheless used in this Standard for maintaining consistency with the JISs specifying nitrogen determination method for other materials.

3.2

gas extraction temperature

temperature set as a condition for gas extraction (3.1)

3.3

degassing

operation of removing gas component in the graphite crucible or in the substance placed in the crucible by pre-burning the crucible at a temperature higher than the gas extraction temperature (3.2)

3.4

crucible degassing temperature

temperature set for performing degassing (3.3)

Note 1 to entry To be set at a temperature equal to or higher than 2 200 °C.

4 General

General requirements common to determination shall conform to JIS G 1201.

5 Summary

The sample is placed in a graphite crucible and fused in the inert gas current to extract nitrogen in molecular form. Nitrogen is separated from other gaseous extracts and transferred to a thermal conductivity cell to measure the change in thermal conductivity. A calibration curve is separately prepared using potassium nitrate or a steel and iron certified reference material, and the change in thermal conductivity is converted into the mass of nitrogen.

6 Reagents

The reagents used shall be as follows.

6.1 Water, prepared immediately before use.

6.2 Inert gas For example, helium of 99.99 % or greater volume fraction. Argon (of volume fraction equal to or higher than specified by the apparatus manufacturer) or other gases may be used. An oxidation reagent or catalyst [copper, copper (II) oxide or platinum] tube heated to a temperature above 450 °C shall be used at the inert gas purification part [7.1 a)] when the presence of organic contaminants is suspected in the inert gas.

6.3 Iron, of high purity and known nitrogen contents (mass fraction) not more than 0.000 8 %. The known content is not required to be a certified value as long as its validity has been confirmed.

6.4 Solvent, suitable for washing greasy or dirty test samples, e.g. acetone.

6.5 Nitrogen standard solution After drying at between 100 °C and 105 °C for 2 h and allowing to cool in a desiccator, weigh, to the nearest 0.1 mg, the masses of potassium nitrate (99.9 % or over in mass fraction) indicated in Table 2 and transfer to a beaker (100 ml). Dissolve the potassium nitrate in about 50 ml of water (6.1), transfer to a 100 ml one-mark volumetric flask using water and dilute to the mark with water.

Table 2 Nitrogen standard solution

Name of the standard solution	Mass of potassium nitrate g	Concentration of nitrogen standard solution mg/ml
1	9.022 8	12.5
2	7.218 2	10.0
3	5.413 8	7.5
4	3.609 1	5.0
5	1.804 6	2.50
6	0.902 3	1.25
7	0.360 9	0.50
8	Standard solution $5 \times 1/10$ ^{a)}	0.25
9	Standard solution $6 \times 1/10$ ^{a)}	0.125
10	Standard solution $7 \times 1/10$ ^{a)}	0.050
Note ^{a)} Dilute the specified standard solution exactly 10 times with water (6.1) immediately before use for preparation.		