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Iron and steel — General rules for  
analytical methods

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## Foreword

This Japanese Industrial Standard has been revised by the Minister of Economy, Trade and Industry based on the provision of Article 14, paragraph (1) of the Industrial Standardization Act applied mutatis mutandis pursuant to the provision of Article 16 of the said Act in response to a proposal for revision of Japanese Industrial Standard with a draft being attached, submitted by The Japan Iron and Steel Federation (JISF), an accredited standards development organization. This edition replaces the previous edition (**JIS G 1201** : 2014), which has been technically revised.

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# Iron and steel — General rules for analytical methods

## 1 Scope

This Japanese Industrial Standard specifies the general rules for analytical methods of iron and steel to be applied to Japanese Industrial Standards (JIS) specifying determination methods and analytical methods of individual elements of iron and steel (hereafter referred to as standards of iron and steel analytical methods).

The term “iron”, for the purpose of this Standard, refers to pig iron and cast iron, and the term “steel” refers to carbon steel, low alloy steel, high alloy steel (including stainless steel) or the like. Pure iron and soft iron are included in steel.

## 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 0203 *Glossary of terms used in iron and steel (Products and quality)*
- JIS G 0404 *Steel and steel products — General technical delivery requirements*
- JIS G 0417 *Steel and iron — Sampling and preparation of samples for the determination of chemical composition*
- JIS K 0050 *General rules for chemical analysis*
- JIS K 0067 *Test methods for loss and residue of chemical products*
- JIS K 0113 *General rules for methods of potentiometric, amperometric, coulometric, and Karl Fischer titrations*
- JIS K 0115 *General rules for molecular absorptiometric analysis*
- JIS K 0116 *General rules for atomic emission spectrometry*
- JIS K 0117 *General rules for infrared spectrophotometric analysis*
- JIS K 0119 *General rules for X-ray fluorescence analysis*
- JIS K 0121 *General rules for atomic absorption spectrometry*
- JIS K 0557 *Water used for industrial water and wastewater analysis*
- JIS K 0970 *Piston pipettes*
- JIS R 3503 *Glass apparatus for chemical analysis*
- JIS R 3505 *Volumetric glassware*
- JIS Z 2613 *General rules for determination of oxygen in metallic materials*
- JIS Z 2615 *General rules for determination of carbon in metallic materials*

- JIS Z 2616 *General rules for determination of sulfur in metallic materials*
- JIS Z 8101-1 *Statistics — Vocabulary and symbols — Part 1 : General statistical terms and terms used in probability*
- JIS Z 8401 *Rounding of numbers*
- JIS Z 8402-1 *Accuracy (trueness and precision) of measurement methods and results — Part 1 : General principles and definitions*
- JIS Z 8402-6 *Accuracy (trueness and precision) of measurement methods and results — Part 6 : Use in practice of accuracy values*
- JIS Z 8801-1 *Test sieves — Part 1 : Test sieves of metal wire cloth*

### 3 Terms and definitions

For the purpose of this Standard and standards of iron and steel analytical methods (hereafter referred to as individual standards), the terms and definitions given in the following and those given in **JIS G 0203**, **JIS G 0417**, **JIS K 0050**, **JIS Z 8101-1** and **JIS Z 8402-1** apply.

NOTE 1 The definitions of analytical chemistry terms provided in **JIS K 0211**, **JIS K 0212**, **JIS K 0213**, **JIS K 0214**, **JIS K 0215** and **JIS K 0216**, which are cited in Clause 3 of **JIS K 0050**, also apply in this Standard.

NOTE 2 Definitions of terms related to analytical procedures to be applied in individual standards are provided in **A.5**.

#### 3.1

##### chemical analysis

generic term for methods that, through causing chemical reaction in a sample, determine the amount of an analyte in the sample by means of gravimetric method, gas volumetric method, titrimetry, absorption spectrometry, atomic absorption spectrometry, inductively coupled plasma (ICP) atomic emission spectrometry, infrared absorption method, thermal conductivity method, etc.

- Note 1 to entry In standards of iron and steel analytical methods, the term “gravimetric method” is used instead of “gravimetric analysis” defined in **JIS K 0211** to ensure consistency with the names of other analytical methods.
- Note 2 to entry The titrimetry, absorption spectrometry, atomic absorption spectrometry and inductively coupled plasma atomic emission spectrometry are defined in **JIS K 0211**.
- Note 3 to entry Titrimetry methods that are adopted in standards of iron and steel analytical methods are : acid-base titration, oxidation-reduction titration, complexometric titration and their back titration methods, and potentiometric titration. These titration methods are defined in **JIS K 0211**.
- Note 4 to entry ICP is defined in **JIS K 0116** as an abbreviation of inductively coupled plasma.

## 3.2

### **instrumental analysis method**

generic term for methods that determine the amount of an analyte by the spark source atomic emission spectrometry or X-ray fluorescent analysis

Note 1 to entry The spark source atomic emission spectrometry is defined in **JIS K 0212** and spark discharge is defined in **JIS K 0116**. The X-ray fluorescent analysis is defined in **JIS K 0211**.

## 3.3

### **gas volumetric method**

analytical method for determining the amount of an analyte by making it a gaseous compound or single substance and measuring the volume quantity of the product or by measuring the volume quantity reduced by reaction and absorption

## 3.4

### **visual titration**

titrimetry in which a titration end point is judged by visual observation of the colour change of an indicator

Note 1 to entry Generally, the method is simply referred to as titrimetry; the above term is used where discrimination from the potentiometric titration is necessary.

## 3.5

### **infrared absorption method**

analytical method in which the analyte in a sample is converted to gaseous compound and sent to the analysis cell, and the infrared absorption by the gaseous compound is measured to determine the amount of the analyte

## 3.6

### **thermal conductivity method**

analytical method for determining the gas composition of a sample by flowing the gas produced from the sample to the detector, and measuring the change of electric resistance of the heating filament in the detector caused by the difference in thermal conductivity from the reference gas

## 3.7

### **certified reference material, CRM**

reference material whose chemical analysis value has been certified by the standardizing body of a nation or organization

## 3.8

### **working reference material**

reference material whose chemical analysis value has been determined by a chemical

analysis method and verified for its accuracy by the use of a certified reference material

Note 1 to entry Where a corresponding certified reference material is not available, the reference value of the working reference material is verified to meet the required accuracy level according to 7.2.

### 3.9

#### blank test

test conducted using the same procedure as and in parallel with the analysis of the sample but without the use of the sample, unless otherwise specified

Note 1 to entry **JIS K 0211** defines it as “test conducted using the same procedure as the analysis of the sample but without the use of the sample, unless otherwise specified”.

Note 2 to entry In analytical methods using a calibration curve, e.g. absorption spectrometry and atomic absorption spectrometry, that are given in individual standards, blank test means a test conducted using the same procedure as and in parallel with the analysis of the sample but using high-purity iron [4.1 h)] instead of sample.

Note 3 to entry A solution prepared for blank test is referred to as blank test solution.

### 3.10

#### zero member

working solution to which the standard solution of the analyte is not added

### 3.11

#### hot (prefix)

state (or a prefix referring to the state) of a reagent solution of water, acid, etc. in which the reagent is maintained at a temperature of 60 °C or higher

Note 1 to entry For example, “hot nitric acid”.

### 3.12

#### warm (prefix)

state (or a prefix referring to the state) of a reagent solution of water, acid, etc. in which the reagent is maintained at a temperature of 40 °C or over to and excluding 60 °C

Note 1 to entry For example, “warm hydrochloric acid (2 + 100)”.

### 3.13

#### cold (prefix)

state (or a prefix referring to the state) of a reagent solution of water, acid, etc. in which the reagent is maintained at a temperature of 15 °C or under

Note 1 to entry For example, “cold hydrochloric acid (2 + 100)”.

## 4 General matters

### 4.1 General requirements

For standards of iron and steel analytical methods, the following general requirements and those given in **JIS K 0050** shall apply.

- a) **Treatment of requirements of referred ISO Standards** For treatment of requirements of **ISO Standards** referred to in the **JISs** of iron and steel analytical methods that are developed as **ISO-identical standards**, Annex B shall apply.
- b) **Volumetric pipette and burette** The volumetric pipettes and burettes to be used in standards of iron and steel analytical methods, unless otherwise specified, shall conform to Class A specified in **JIS R 3505**. They may conform to Class B if they have been calibrated according to Annex I of **JIS K 0050**.

An automatic burette may be used in place of a volumetric pipette and/or burette provided that the value that is twice the standard deviation of the repeated measurements of the specified drip amount (volume-converted value) of the automatic burette is within Class A tolerance for that specified drip amount (volume) given in **JIS R 3505**.

- c) **Piston pipette** The piston pipette to be used in standards of iron and steel analytical methods, unless otherwise specified, shall be of air displacement type (Type A) specified in **JIS K 0970**.
- d) **Volumetric flask** The volumetric flask to be used in standards of iron and steel analytical methods, unless otherwise specified, shall be a volumetric flask (for receiving) of Class A specified in **JIS R 3505**. It may conform to Class B if it has been calibrated according to Annex I of **JIS K 0050**.
- e) **Balance** The balance to be used for weighing analytical samples, etc. for chemical analysis, unless otherwise specified, shall be a chemical balance or an electronic balance of which the minimum reading is 0.1 mg or less, and which has been calibrated using weights traceable to a national standard. The balance to be used for infrared absorption method and thermal conductivity method may have a minimum reading of 1 mg.
- f) **Sieve** The sieve to be used for the size control of samples, unless otherwise specified, shall be in accordance with **JIS Z 8801-1**.
- g) **Water** The water to be used for determination operations in standards of iron and steel analytical methods, unless otherwise specified, shall be the water of Classification A3 or A4 specified in **JIS K 0557**.
- h) **Iron** The iron used in the preparation of working solutions or in blank tests shall have a guaranteed concentration (mass fraction) of the analyte of less than  $1/10$  <sup>1)</sup> of the minimum limit of determination <sup>2)</sup> or have a specified concentration of the analyte that is equal to or below the minimum limit of determination <sup>3)</sup>. The guaranteed concentration should be a certified value that may or may not include an inequality sign. The specified concentration may be other value <sup>4)</sup> than the certified

value as long as its validity has been verified.

The validity is considered verified if the analysis of a certified reference material with a certified value of analyte concentration (mass fraction) close to the minimum limit of determination of the analyte performed in parallel with the analysis of the iron yields a quantitative value equal to the certified value. The verified validity automatically guarantees that the concentration is less than of 1/10 the minimum limit of determination.

Note <sup>1)</sup> Some individual standards indicate specific concentration values (mass fraction).

Note <sup>2)</sup> Some individual standards require that the iron shall not contain the analyte.

Note <sup>3)</sup> Some individual standards require that the iron shall have an analyte concentration that is as low as possible.

Note <sup>4)</sup> For example, an informative value of the certified reference material, a nominal value indicated by the iron reagent manufacturer of which the validity has been verified.

- i) **Stock solutions and standard solutions** The stock solutions and/or standard solutions of each element to be used in individual standards shall normally be prepared using high-purity metal, high-purity metallic oxide or high-purity compound decomposed or dissolved in suitable acid, alkali, flux or water, as specified in the individual standards.

Alternatively, commercially available metal standard solutions may be used provided that :

- they have the same level of concentration as the stock solution or standard solution specified in the individual standard;
- they have a guaranteed traceability of measurements;
- they are free from contaminants of substances other than from the reagents used for decomposition and preparation, and do not use any reagents in preparation that may affect the analysis.

The concentration specified in the individual standards shall be either the concentration of the commercially available standard solutions or corrected by multiplication by a factor.

The standard solutions shall be prepared by diluting at least 2 ml, and preferably 5 ml or more of the stock solution or other standard solutions.

- j) **Use of watch glass** The watch glass should be of the same material as the beaker to be employed, and used according to the following instructions. Since detailed instructions are provided here, they need not be included in individual standards. These instructions also apply to preparation of the stock solution, standard solution and working solution of each component.

- 1) Prepare as many beakers covered with watch glasses as the number of samples to