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### (JCA)

# Methods for chemical analysis of cements

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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 Japan Cement Association (JCA) 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 R 5202:1999 is replaced with this Standard.

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#### Methods for chemical analysis of cements

#### Introduction

This Japanese Industrial Standard was established in 1950 and has been revised ten times up to the present, and the last revision was made in 1999. This Standard specifies the methods for the analysis of cement by wet chemistry.

The International Standard for analysis methods of cements by wet analysis, **ISO 29581-1**, which was published as the first edition on March 15th, 2009, specifies the procedure of complete decomposition of cement for chemical analysis of major elements of cement [silicon dioxide, aluminium oxide, iron (III) oxide, calcium oxide and magnesium oxide].

On the other hand, this **JIS** Standard specifies the procedure for dissolution of cement by acid and determination of contents of the dissolved components, and is to be cited and used for determination of components in each of the quality standards of cement (**JIS R 5210**, **JIS R 5211**, **JIS R 5212**, **JIS R 5213** and **JIS R 5214**).

Therefore, this **JIS** Standard only adopts, from among the methods specified in the said International Standard, the analysis method in which cement is dissolved by acid, while adding some analysis methods that are not given in the International Standard.

Portions with continuous sidelines or dotted underlines are the matters in which the contents of the corresponding International Standard have been modified. A list of modifications with explanations is given in Annex JA. Further, a comparison table between the previous and current editions of this JIS on technically significant revisions is shown in Annex JB.

#### 1 Scope

This Standard specifies the methods for the analysis of cement $\frac{1}{2}$  by wet chemistry.

It is also applicable to analysis of clinkers and blast-furnace slag to be used for manufacture of blast-furnace slag cement.

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

ISO 29581:2009 Cement—Test methods—Part 1: Analysis by wet chemistry (MOD)

The symbols which denote the degree of correspondence in the contents between **JIS** and the corresponding International Standard are IDT (identical), MOD (modified) and NEQ (not equivalent) according to **ISO/IEC Guide 21-1**.

Note <sup>1)</sup> "Cement", for the purpose of this Standard, is portland cement specified in **JIS R 5210**, portland blast-furnace slag cement specified in **JIS R 5211**, portland pozzolan cement specified in **JIS R 5212**, portland fly-ash cement specified in **JIS R 5213**, and ecocement specified in **JIS R 5214**. 2 R 5202 : 2010

#### 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) indicated below shall be applied.

JIS K 0050	General rules for chemical analysis
JIS K 0113	General rules for methods of potentiometric, amperometric, coulomet- ric, and Karl Fischer titrations
JIS K 0115	General rules for molecular absorptiometric analysis
JIS K 0121	General rules for atomic absorption spectrometry
JIS K 8005	Reference materials for volumetric analysis
JIS K 8102	Ethanol (95) (Reagent)
JIS P 3801	Filter paper (for chemical analysis)
JIS R 3503	Glass apparatus for chemical analysis
JIS R 3505	Volumetric glassware
JIS R 5204	Chemical analysis method of cement by x-ray fluorescence
JIS Z 8401	Guide to the rounding of numbers
JIS Z 8801-1	Test sieves—Part 1: Test sieves of metal wire cloth

#### **3** General requirements for testing

#### 3.1 Number of tests

The number of tests performed on one identical sample shall be two, except where the analysis is one of a series subject to statistical control, in which case a single test shall be the minimum required.

#### 3.2 Tolerance

When the difference between the two tests exceeds the specified tolerance, a retest shall be conducted, and two most adjacent test results shall be averaged and reported as the analysis results (see **3.3**).

#### 3.3 Expression of masses, volumes, factors and results

Express masses in grams to the nearest 0.000 1 g and volumes from burettes in milliliters to the nearest 0.01 ml.

Express the factors of solutions given by the mean of three measurements.

Express each analysis result, except for that of chlorine (Cl), as a percentage generally to two decimal places according to 2 b) of **JIS Z 8401**. The analysis result of chlorine (Cl) shall be expressed as a percentage to three decimal places.

Express the results, where two test results have been obtained, as the mean of the results, as a percentage generally to two decimal places according to Rule A in 2 c) of **JIS Z 8401**, except for analytical items of chlorine (Cl). Express the results of chlorine (Cl) as a percentage to three decimal places.

#### 3.4 Ignition

Carry out the ignition as follows:

Place the filter paper and its precipitate (residue) into a crucible which has been previously ignited and tared. Dry it, then incinerate slowly in an oxidizing atmosphere in order to avoid immediate flaming, while ensuring completer incineration. Ignite the crucible and its contents at the stated temperature, then allow to cool to the laboratory temperature in a desiccator. Weigh the crucible and its contents.

#### 3.5 Glass apparatus

Glass apparatus used for performing the analysis shall be in accordance with **JIS R 3503** and **JIS R 3505**.

#### 3.6 Other general requirements

General requirements other than stated above shall be in accordance with JIS K 0050, JIS K 0113, JIS K 0115 and JIS K 0121.

#### 4 Preparation of a test sample of cement

Take, for preparing test sample, cement from a unit of inspection such that the taken portion represents the average quality of the unit, and divide it to obtain representative sample of about 5 kg. The sampling and dividing method shall be agreed upon between the purchaser and the supplier. Take approximately 100 g of the laboratory sample by means of a sample divider or by quartering. Sieve this portion on a 150  $\mu$ m or 125  $\mu$ m sieve as specified in **JIS Z 8801-1**. Remove metallic iron from the material retained on the sieve by means of a magnet. Then grind the iron-free fraction of the retained material so that it completely passes the 150  $\mu$ m or 125  $\mu$ m sieve. Transfer the sample to a clean, dry container with an airtight closure and shake vigorously to mix it thoroughly.

Carry out all operations as quickly as possible to ensure that the test sample is exposed to ambient air only for the minimum time.

#### 5 Determination of loss on ignition

#### 5.1 Principle

The loss on ignition is determined in an oxidizing atmosphere (air). By igniting the sample in air at 950 °C  $\pm$  25 °C, the carbon dioxide and water are driven off and any oxidizable elements present are oxidized to some extent. The error resulting from the oxidation of metallic iron, iron (II) oxide or bivalent manganese (II) oxide is usually considered negligible and only the correction for the extent of oxidation of any sulfides is applied.

#### 5.2 In the case of other than portland blast-furnace slag cement and blastfurnace slag

#### 5.2.1 Procedure

a) Weigh approximately  $1 g^{2}$  of sample, correctly to 0.000 1 g, into a platinum or porcelain crucible which has been previously ignited and tared.

- b) Place the cover on the crucible, sliding off a little to make some clearance, and place the crucible in the electric furnace controlled at 950 °C  $\pm$  25 °C, and ignite for 15 min.
- c) Allow the crucible to cool to room temperature in a desiccator and weigh the mass.
- d) Repeat this ignition, of 15 min each, to determine the constant mass. The constant mass is known to be reached when the mass difference between before and after ignition is less than 0.000 5 g.

If any gain in mass has occurred in the determination of the constant mass, the mass before the gain shall be employed. If the mass has gained at the first 15 min ignition, it shall be noted simply as "+".

Note <sup>2)</sup> In the corresponding International Standard, the sampling range is specified to be  $1.00 \text{ g} \pm 0.05 \text{ g}$ .

#### 5.2.2 Calculation

Calculate the loss on ignition from the following formula:

$$ig.loss = \frac{m-m'}{m} \times 100$$
  
where,  $ig.loss$ : loss on ignition (%)  
 $m$ : mass of sample (g)  
 $m'$ : mass of sample after ignition (g)

#### 5.3 In the case of portland blast-furnace slag cement and blast-furnace slag

#### 5.3.1 Procedure

- a) Perform the procedures in **5.2.1** a) to d) and determine the apparent loss on ignition [*ig.loss*(ap)] by the formula in **5.2.2**.
- b) Determine the content of sulfur trioxide in sample before ignition  $[SO_3(B)]$  according to clause 12.
- c) Determine the content of sulfur trioxide in sample after ignition  $[SO_3(A)]$  according to the following procedure.

Weigh 2 g to 3 g of sample into a crucible, and ignite it according to **5.2.1** b) to  $\mathbf{d}$ ).

The constant mass shall be confirmed when the mass difference between before and after the ignition is less than 0.05 %. Using this ignited sample<sup>3)</sup>, determine the sulfur trioxide content according to the procedure in clause **12**.

For the sulfur trioxide content after ignition, the value of sulfur trioxide of glass bead determined according to **JIS R 5204** may be adopted.

Note <sup>3)</sup> For weighing out the sample after ignition, the lump of sample should be broken down into uniform structure, and then quickly weighed out.

#### 5.3.2 Calculation

Calculate the loss on ignition in the sample from the following formula: