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**GB/T 176-2017** 

Replacing GB/T 176-2008

# Methods for chemical analysis of cement

水泥化学分析方法

(ISO 29581-1:2009 Cement - Test methods -

Part 1: Analysis by wet chemistry, NEQ)

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#### **Foreword**

This standard is drafted in accordance with the rules given in GB/T 1.1-2009.

This standard replaces GB/T 176-2008 "Methods for chemical analysis of cement". Compared with GB/T 176-2008, the main changes are as follows:

- In the Scope, ADD the inductively coupled plasma emission spectroscopy (see Chapter 1; Chapter 1 of the 2008 edition).
- MODIFY that "During chemical analysis, unless otherwise specified, it shall carry out the determination of loss on ignition simultaneously" into "During chemical analysis, it is recommended to determine loss on ignition simultaneously" (see 4.1; 4.1 of the 2008 edition).
- ADD that "Unless otherwise stated, the validity period of the standard titration solution is 3 months; if it exceeds 3 months, it shall be calibrated again" (see 4.8).
- For the preparation of the specimen, MODIFY that "all passes through a square hole sieve which has an aperture of 80 μm" into "all passes through a square hole sieve which has an aperture of 150 μm". MODIFY that "The use of magnets to remove metallic iron from the sieve residue" into "if the metallic iron carried in the sample preparation process may affect the determination of related chemical properties, use the magnet to remove metallic iron from the sieve residue". In the prompt, ADD that "Before analyzing cement and cement clinker specimen, it does not require drying the specimen" (see Chapter 5; Chapter 7 of the 2008 edition).
- For the calibration of sodium thiosulfate standard titration solution, CHANGE from "potassium dichromate standard titration solution" to "potassium iodate standard titration solution" (see 6.1.85.2; 5.84.2 of the 2008 edition).
- The determination of loss on ignition is divided into "determination of loss on ignition of cement" and "determination of loss on ignition of slag Portland cement". In the determination of the loss on ignition of slag Portland cement, MAKE specific requirements for the determination method of sulfate sulfur trioxide in the sample material (see 6.3, 6.4, 6.39; Chapter 8 of the 2008 edition).
- MODIFY the "sulfur trioxide" into "sulfate sulfur trioxide" (see 6.5, 6.28, 6.29, 6.30; Chapter 10, Chapter 30, Chapter 31, Chapter 33 of 2008 edition).
- Determination of sulfate sulfur trioxide In the barium sulfate gravimetric method (reference method), MODIFY the decomposition time of the

(see 6.19).

- Determination of silicon dioxide In the potassium fluorosilicate volumetric method (alternative method), MODIFY that "Add 10 mL ~ 15 mL of nitric acid" into "Add 15 mL of nitric acid"; MODIFY that "place it under 30 °C for 15 min ~ 20 min" into "place it at 10 °C ~ 26 °C for 15 min ~ 20 min" (See 6.20; Chapter 23 of the 2008 edition).
- Determination of ferric oxide In the EDTA direct titration method (alternative method), MODIFY "pH  $1.8 \sim 2.0$ " into "pH 1.8" (See 6.21; Chapter 12 of the 2008 edition).
- Determination of aluminum trioxide In the copper sulfate back titration method (alternative method), ADD that if the consumption of the copper sulfate standard titration solution is less than 10 mL, increase the amount of EDTA standard titration solution and retest (See 6.24; Chapter 26 of the 2008 edition).
- Determination of sulfate sulfur trioxide In the coulometric titration method (alternative method), MODIFY that "When there is sulfur in other states besides sulfide (S<sup>2</sup>-) and sulfate in the specimen, it will cause an error in the determination result." into "When the sample contains a large amount of sulfide (S<sup>2</sup>-) or sulfur in other states, the sulfur or other state of sulfur may not be completely decomposed by formic acid, which will cause a positive error in the determination result, such as the cement which is mixed of a large amount of slag" (see 6.29; Chapter 33 of the 2008 edition).
- Determination of sulfate sulfur trioxide In the ion-exchange method (alternative method), ADD the requirements that "this method is only used for enterprise production control" (see 6.30; Chapter 31 of the 2008 edition).
- DELETE the determination of sulfur trioxide Barium chromate spectrophotometry (alternative method) (Chapter 32 of the 2008 edition).
- ADD the determination of chloride ion (Automatic) potentiometric titration method (alternative method) (see 6.31).
- ADD the determination of chloride ions Ion chromatography (alternative method) (see 6.32).
- DELETE the determination of chloride ion Phosphoric acid distillation-Mercury salt titration method (alternative method) (Chapter 35 of the 2008 edition).
- In the determination of free calcium oxide In the ethylene glycol method (alternative method), DELETE that "extraction filtration 4 min after heating to slight boiling" in the analytical procedures; MODIFY it into "titration

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This standard replaces the standard previously issued as follows:

- GB/T 176-1956, GB/T 176-1962, GB/T 176-1976, GB/T 176-1987, GB/T 176-1996, GB/T 176-2008;
- GB/T 19140-2003.

## Methods for chemical analysis of cement

## 1 Scope

This standard specifies methods for chemical analysis of cement, X-ray fluorescence analysis methods, inductively coupled plasma emission spectrometry for the determination of loss on ignition (LOI), SO<sub>3</sub>, insoluble (IR), SiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub>, CaO, MgO, TiO<sub>2</sub>, Cl<sup>-</sup>, K<sub>2</sub>O, Na<sub>2</sub>O, S<sup>2-</sup>, MnO, P<sub>2</sub>O<sub>5</sub>, CO<sub>2</sub>, ZnO, F<sup>-</sup>, free calcium oxide (fCaO), SrO. Chemical analysis methods of cement are further divided into reference methods and alternative methods. If multiple determination methods are listed for the same component, the reference method shall prevail in case of dispute.

This standard applies to general Portland cement, clinker, raw material for the preparation of the above cement, other cements and materials designated to use this standard.

#### 2 Normative references

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) are applicable to this standard.

GB/T 5762 Methods for chemical analysis of limestone, quicklime and hydrated lime for building materials industry

GB/T 6682 Water for analytical laboratory use - Specifications and test methods

GB/T 8170 Rules for rounding off for numerical values & expression and judgement of limiting values

GB/T 12573 Sampling method for cement

GB/T 15000 (all parts) Directives for the work of reference materials

GSB 08-1110 Standard sample of cement raw materials for X-ray fluorescence analysis

GSB 08-1355 Standard sample for analysis of cement clinker composition

GSB 08-1356 Standard sample for composition analysis of ordinary Portland

A set of standard samples used for calibrating analytical instruments such as X-ray fluorescence analyzer which is related to chemical composition.

# 4 Basic requirements for test

#### 4.1 Number of tests and requirements

The number of tests for each determination is specified as two; the absolute difference between the two results is within the repeatability limit (Table 3, Table 4, Table 5). The average value of the two test results is used to indicate the determination result.

During routine production control analysis, the number of tests for each determination may be one.

In chemical analysis, it is recommended to determine the loss on ignition at the same time.

Except for the loss on ignition, it shall perform other blank tests simultaneously and correct the determination results.

#### 4.2 Representation of mass, volume, titer and results

Mass is expressed in "grams (g)" and is accurate to 0.0001 g. The volume of the burette is expressed in "milliliter (mL)"; the reading is accurate to 0.01 mL. The titer is expressed in "milligrams per milliliter (mg/mL)".

The titer of benzoic acid-anhydrous ethanol standard titration solution for the titer of calcium oxide retains three significant figures; the concentration, titer and volume ratio of other standard titration solutions retain four significant figures.

Unless otherwise stated, all analysis results are based on mass fraction. The results of chloride ion analysis are expressed as % to three digits after the decimal point; the other analysis results are expressed as % to two digits after the decimal point.

The rounding of the value is performed according to GB/T 8170.

## 4.3 Repeatability limit and reproducibility limit

The repeatability limits and reproducibility limits listed in this standard are absolute deviations, expressed in terms of mass fraction (%).

by the use of silver nitrate.

#### 4.8 General requirements for reagents

Unless otherwise specified, the reagents used shall not be lower than analytical grade; the reagents used for calibration shall be reference reagents. The water used shall not be lower than the requirements of the third-grade water as specified in GB/T 6682.

The density of the commercially available concentrated liquid reagents listed in this standard refers to the density ( $\rho$ ) at 20 °C, in the unit of grams per cubic centimeter (g/cm<sup>3</sup>).

In chemical analysis, the acid or ammonia used, where no concentration is indicated, refers to commercially available concentrated acid or ammonia.

Use the volume ratio to indicate the degree of dilution of the reagent, for example: hydrochloric acid (1 + 2) means that 1 part of concentrated hydrochloric acid is mixed with 2 parts of water.

Unless otherwise stated, the validity period of the standard titration solution is 3 months. If it exceeds 3 months, recalibrate it.

#### 4.9 Verification of test methods

The test methods listed in this standard can be compared with certified standard samples / standard materials (such as GSB 08-1110, GSB 08-1355, GSB 08-1356, GSB 08-1357, GSB 08-2985) to verify the accuracy of the method.

# **5 Preparation of specimens**

Take sample according to the method of GB/T 12573. The sample sent to the laboratory shall be a representative uniform sample. The sample is divided to about 100 g by a quartering method or a divider. After sieving through a 150  $\mu$ m square hole sieve, the debris is removed. After the sieve residue is ground, it is passed through a 150  $\mu$ m square hole sieve. Make it thoroughly mixed and contained into a clean, dry sample bottle. Seal it. Mix it further to prepare for the use of determination.

If the metallic iron carried in the sample preparation process may affect the determination of related chemical properties, use a magnet to remove the metallic iron from the sieve residue.

Tip: Prepare the specimen as quickly as possible to prevent moisture

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