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# Aggregates for concrete — Test methods for chemical properties —

## Part 2:
## Determination of soluble sulfate salts

*Granulats pour béton — Méthodes d'essai relatives aux propriétés chimiques —*

*Partie 2: Dosage des sels de sulfate solubles*

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# Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 71, *Concrete, reinforced concrete and pre-stressed concrete*, Subcommittee SC 1, *Test methods for concrete*.

A list of all parts in the ISO 24684 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

# Aggregates for concrete — Test methods for chemical properties —

## Part 2:
## Determination of soluble sulfate salts

## 1 Scope

This document describes the procedure for the determination of soluble sulfate salts which can be present in aggregates. The test is suitable for aggregates where the sulfate content derives directly from contact with, or immersion in, saline water, e.g. typical sea-dredged aggregates.

## 2 Normative references

There are no normative references in this document.

## 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

**3.1**
**test portion**
sample used as a whole in a single test

**3.2**
**test specimen**
sample used in a single determination when a test method requires more than one determination of a property

**3.3**
**laboratory sample**
reduced sample derived from a bulk sample for laboratory testing

## 4 General

An aggregate test specimen is extracted with water to remove sulfate ions. The method of analyses of the extract is based on the Volhard titration where an excess of silver nitrate solution is added to the sulfate solution and the unreacted portion is back titrated with a standardized solution of thiocyanate, using ammonium iron (III) sulfate solution as an indicator.

The sulfate are expressed in terms of, and reported as, the sulfate ion content as a percentage by mass of aggregate.

# 5 Reagents

## 5.1 General

**5.1.1** Unless otherwise stated, use only analytical grade reagents and demineralized water, or water of equivalent purity.

NOTE 1 Unless otherwise stated, "%" means "% by mass".

NOTE 2 Where no tolerances are given for reagent volumes or masses, the values quoted are approximate.

NOTE 3 Unless otherwise stated, reagent solutions can be assumed to have long-term stability.

All chemicals should be treated as potential poisons with toxic properties and appropriate precautions taken before their use. Always take time to assess possible hazards before starting any procedures and constant attention should be maintained.

**5.1.2** Concentrated liquid reagents shall have the following densities in g/cm$^3$ at 20 °C:

Hydrochloric acid: 1,18 to 1,19

Nitric acid: 1,39 to 1,42

Sulfuric acid: 1,84

Ammonium hydroxide: 0,88 to 0,91

The degree of dilution shall be indicated as a volumetric sum.

NOTE Ready-for-use solutions can be used as an alternative.

## 5.2 Reagents for determination of acid-soluble sulfates

**5.2.1 Silver nitrate ($AgNO_3$) solution**, 0,100 mol/l is prepared by drying about 20 g of silver nitrate for at least 1 h at a temperature of (100 to 115) °C, allowing it to cool in a desiccator, weighing (16,987 ± 0,001) g of the dried silver nitrate, then dissolving it in water and diluting it to 1 l in a volumetric flask. Store the solution in the amber-coloured glass reagent bottle and protect it from prolonged exposure to sunlight.

**5.2.2 Barium chloride solution**, dissolve 100 g of barium chloride in 1 l of water and filter through a medium grade filter paper before use.

**5.2.3 Methyl red indicator**, dissolve 20 mg of methyl red powder in 50 ml of ethanol, then add 50 ml of water.

## 5.3 Apparatus

**5.3.1 Well-ventilated oven**, capable of being controlled to maintain a constant temperature in the range of 40 °C to 150 °C with an accuracy of ±5 °C, equipped with a heat resistant tray made of non-corrodible material.

**5.3.2 Electric muffle furnace**, capable of being controlled to maintain a constant temperature in the range of 800 °C to 1 100 °C with an accuracy of ±25 °C.

**5.3.3 Crushing and grinding equipment** to reduce aggregates to sizes that pass through sieves suitable for particular tests while producing a minimum of fines.

**5.3.4** **Balance**, capable of weighing up to 10 kg, readable to the nearest 1 g.

**5.3.5** **Balance**, capable of weighing up to 1 kg, readable to the nearest 0,01 g.

**5.3.6** **Analytical balance**, capable of weighing up to 100 g, readable to the nearest 0,1 mg.

## 6 General requirements for testing

### 6.1 Number of tests

Unless otherwise stated, the number of single determinations is fixed at two.

### 6.2 Repeatability and reproducibility

The standard deviation of repeatability gives the closeness of agreement between successive results obtained with the same method on identical material tested under the same conditions (i.e. same operator, same apparatus, same laboratory and short time interval).

The standard deviation of reproducibility gives the closeness of agreement between individual results obtained with the same method on identical material but tested under different conditions (i.e. different operators, different apparatus, different laboratories and/or different times).

The standard deviations of repeatability and reproducibility are expressed in absolute percentage. Recognized values for some of the test methods are given in Annex A.

NOTE The values of precision are taken from previous national standards or from cross-testing exercises.

### 6.3 Expression of mass, volume, factors and results

Record the mass from an analytical balance in g to the nearest 0,1 mg and volume from the burettes in ml to the nearest 0,05 ml.

Record the mass from the ordinary balance specified to the nearest 1 g or from the balance specified to the nearest 0,01 g.

Express the factors of solutions, given by the mean of three determinations, to three decimal places.

Express the results of the tests, given by the mean of two determinations, as a percentage, to the nearest 0,01 %, unless otherwise stated.

If an accepted value of repeatability is available (see Annex A), the results for the two determinations shall be reviewed. If the difference between two determinations is more than twice the repeatability standard deviation, repeat the test and take the mean of the two closest values.

### 6.4 Drying of materials

Unless otherwise stated in a test method, materials shall be dried in a well-ventilated oven, at a temperature of 100 °C to 115 °C.

### 6.5 Ignitions of precipitates

Ignite precipitates as follows:

Place the filter paper and its contents into a crucible, which has been previously ignited and tarred. Dry and then incinerate the filter paper slowly in an oxidizing atmosphere without flaming, while ensuring complete combustion.

Ignite the precipitate for at least 1 h at the stated temperature.

Allow the crucible and its contents to cool to the room temperature in a desiccator. Weigh the crucible and its contents.

### 6.6 Check for the absence of chloride ions (silver nitrate test)

After five or six washes of a precipitate, rinse the base of the filter stem with a few drops of water.

Wash the filter and its contents with several ml of water and collect this in a test tube. Add several drops of concentrated nitric acid and of silver nitrate solution. Check for the absence of turbidity or precipitate in the solution. If necessary, continue washing.

The absence of turbidity in the silver nitrate test confirms that the washings are free from chloride ions.

## 7 Determination of acid soluble sulfates

### 7.1 Principle

Sulfates are extracted from a test portion of the aggregate by dilute hydrochloric acid. The sulfate ion content is determined by gravimetry and expressed as a percentage by mass of the aggregate.

The laboratory sample shall be taken in accordance with the procedures specified.

The laboratory sample should be representative of the moisture content of the batch.

### 7.2 Preparation of test portion

Reduce the laboratory sample by the procedures specified to an amount not less than the mass specified in Table 1 appropriate to the upper (*D*) sieve size of the aggregate.

If necessary, dry the sample at a temperature of (100 to 115) °C.

NOTE Drying at a higher temperature will oxidize any sulfides.

**Table 1 — Minimum mass of preliminary sub-sample**

| Nominal maximum particle size of aggregate mm | Minimum mass of sub-sample kg |
|---|---|
| 63 | 50 |
| 45 | 35 |
| 31,5 | 15 |
| 22,4 or less | 5 |

Stepwise crush and reduce the sub-sample to a mass of approximately 20 g and then grind the particles until they all pass the 125 µm sieve. Take approximately 8 g of this material as the test portion.

### 7.3 Procedure

Weigh the test portion to the nearest 0,1 mg ($m_6$, see 7.4). Place 360 ml water and 40 ml of concentrated hydrochloric acid (see 5.1.2) in a 1 000 ml conical flask and heat to boiling point. Remove from the source of heat and, while stirring, sprinkle the test portion on to the acid solution. Break up the solids with the flattened end of a glass-stirring rod. Allow the solution to digest for (15 ± 1) min at a temperature just below boiling.

Filter the residue through a medium filter paper into a 1 000 ml beaker. Thoroughly wash the contents of the filter with hot demineralized water. Check the washings to ensure the absence of chloride ions using the silver nitrate test.

Adjust the volume to about 600 ml with demineralized water and warm up the content of the beaker to below the boiling point.

Make the solution alkaline by adding ammonium hydroxide solution. Check the alkalinity by using methyl red as an indicator, or by a pH value of 6 or higher when using a pH-meter. Simmer for 30 s and then filter under gentle suction using a medium porosity filter paper. Wash the contents of the filter at least three times with a little hot, demineralised water, reserve the filtrates and reject the precipitate if any. Acidify the filtrates and washings by means of hydrochloric acid (1+11) to red coloration of methyl red indicator.

Bring to the boil and boil for (5 ± 0,5) min and then check that the solution is clear. If it is not clear, start the test again using a new test portion.

While maintaining the solution at boiling point and stirring vigorously, slowly add 40 ml of the barium chloride solution heated to just below boiling. Mature, filter and ignite the barium sulfate precipitate.

Weigh to the nearest 0,1 mg and calculate the mass of precipitate ($m_7$, see 7.4).

## 7.4 Calculation and expression of results

Calculate the acid soluble sulfate content of the aggregate, expressed as $SO_3$ to the nearest 0,1 %, using Formula (1):

$$SO_3 = m_7/m_6 \times 34{,}30 \text{ (in \%)} \qquad (1)$$

# 8 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 24684-2:2023;

b) the method used;

c) the source of the sample;

d) the designation of the sample;

e) the mass of dry sample tested, (in grams);

f) the result(s), including a reference to the clause which explains how the results were calculated;

g) any deviations from the procedure;

h) any unusual features observed;

i) the date of the test.