

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 682

CHEMICAL ANALYSIS OF CEMENTS

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DETERMINATION OF SULPHUR AS SULPHIDE

[ISO/R 682:1968](https://standards.iteh.ai/catalog/standards/sist/8940ea59-f17e-4838-85d1-b11eb8f833e8/iso-r-682-1968)

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BRIEF HISTORY

The ISO Recommendation R 682, *Chemical analysis of cements – Determination of sulphur as sulphide*, was drawn up by Technical Committee ISO/TC 74, *Hydraulic binders*, the Secretariat of which is held by the Institut Belge de Normalisation (IBN).

Work on this question by the Technical Committee began in 1958 and led, in 1963, to the adoption of a Draft ISO Recommendation.

In February 1965, this Draft ISO Recommendation (No. 775) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Argentina	Ireland	Romania
Australia	Israel	Sweden
Austria	Italy	Turkey
Belgium	Japan	U.A.R.
Czechoslovakia	Korea, Rep. of	United Kingdom
Denmark	Netherlands	U.S.A.
France	New Zealand	U.S.S.R.
Germany	Norway	Yugoslavia
Hungary	Poland	
India	Portugal	

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council which decided, in March 1968, to accept it as an ISO RECOMMENDATION.

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CHEMICAL ANALYSIS OF CEMENTS

DETERMINATION OF SULPHUR AS SULPHIDE

1. SCOPE

This ISO Recommendation describes the method for determination of sulphur present in cements as sulphide.

2. PRINCIPLE OF METHOD

The sulphides contained in the sample are attacked by a strong acid in a reducing medium. Hydrogen sulphide formed is entrained by an air flow in an ammoniacal solution of zinc or cadmium. Zinc or cadmium sulphide precipitated is determined by iodimetry.

3. REAGENTS

3.1 *Starch solution*

To 100 ml of boiling water add 5 ml of a suspension containing 20 g of starch in 100 ml of cold water. Cool. Add 10 ml of a solution containing 10 g of sodium carbonate per 100 ml and add 3 g of potassium iodide.

3.2 *Standard potassium permanganate solution (0.03 N)*

Prepare a solution containing 0.94 g of potassium permanganate per litre, taking the usual precautions. Standardize against analytical quality sodium oxalate (about 0.150 g weighed accurately). Let this mass be *A* requiring a volume *B* of potassium permanganate solution.

3.3 *Sodium thiosulphate solution (0.03 N)*

Prepare a solution containing 7.4 g of $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ per litre.

Standardize it as follows :

Dissolve 1 g of potassium iodide in 300 ml of cold water in a 500 ml conical flask and add 10 ml of hydrochloric acid. Run in 25 ml of the standard potassium permanganate solution (3.2) from a burette.

Shake gently, stopper the flask and allow to stand for 5 min. Titrate the iodine liberated with sodium thiosulphate solution. Near the end of the reaction (colour of solution pale yellow) add 2 ml of starch solution (3.1) and continue the titration until disappearance of the blue colour, the total volume of sodium thiosulphate solution being *D*. Then add more potassium permanganate solution until a faint blue colour returns. The total volume of potassium permanganate solution used is called *C*.

The mass in grammes of sulphur equivalent to 1 ml of thiosulphate solution is :

$$e = \frac{A \times C \times 0.2392}{B \times D} \quad (1)$$

3.4 *Standard potassium iodate solution* (0.03 N)

Prepare a solution containing 1.12 g of potassium iodate and 12 g of potassium iodide per litre. Standardize it by repeating the sequence of operations employed in standardizing the thiosulphate (3.3), replacing potassium permanganate by potassium iodate solution; (a volume F of the latter requiring a volume G of sodium thiosulphate).

The mass in grammes of sulphur equivalent to 1 ml of potassium iodate solution is

$$E = \frac{A \times C \times G \times 0.2392}{B \times D \times F} = \frac{e \times G}{F} \quad (2)$$

3.5 *Stannous chloride solution*

Add 7 ml of concentrated hydrochloric acid to 10 g of $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$. Heat to aid dissolution. Cool and add 95 ml of cold water. This solution does not keep.

3.6 *Ammoniacal zinc sulphate solution*

Dissolve 50 g of $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ in 150 ml of water and 350 ml of ammonium hydroxide solution. Allow to stand for at least 24 hours and filter.

3.7 *Ammoniacal cadmium chloride solution*

Dissolve 15 g of $\text{CdCl}_2 \cdot 2\text{H}_2\text{O}$ in 150 ml of water and 350 ml of ammonium hydroxide solution. Allow to stand for at least 24 hours and filter.

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NOTES

- Potassium permanganate and potassium iodate solutions should be checked frequently. Sodium thiosulphate solution is more stable and it is often sufficient to check potassium iodate solution against sodium thiosulphate solution.
- The factor 0.2392 of formulae 1 and 2 is derived from the ratio of the sulphur equivalent of potassium permanganate to its sodium oxalate equivalent.

4. APPARATUS

Fit the neck of a 500 ml flask with a two-hole bung holding a separating funnel and a small connecting bulb. Adjust the separating funnel so that its lower end is near the base of the flask and connect its top to a source of compressed air. The connecting bulb is connected through an L-tube to a 20 cm long straight glass tube. The latter dips into a 400 ml beaker. A bubbler (a triple-necked Wolf flask, with its centre-neck fitted with a long glass tube dipping into the wash-water) facilitates the regulation of the compressed air supply. If the compressed air is likely to contain hydrogen sulphide or sulphur dioxide, lead acetate solution should be used as the wash-water. Bungs and connecting tubes should be made of sulphur-free material (polyvinyl chloride or silicone).

NOTE. — Interchangeable conical ground glass joints in conformity with ISO Recommendation R 383, *Interchangeable conical ground glass joints*, may also be used in order to effect this assembly.

5. PROCEDURE

Place 15 ml of ammoniacal zinc sulphate solution (3.6) or 15 ml of ammoniacal cadmium chloride solution (3.7) and 285 ml of water in the beaker. Disperse 5 g of cement with 10 ml of water in the flask. Assemble the apparatus. The glass outlet tube should always be immersed in the zinc or cadmium solution. By means of the separating funnel add first 25 ml of stannous chloride solution (3.5), and then 100 ml of hydrochloric acid (1 + 3) shaking carefully after each addition.

Carefully close the tap of the separating funnel after each reagent is added. Connect the compressed air supply, open the tap of the separating funnel and adjust the air to a moderate flow. Heat and keep boiling gently for 5 or 6 min. Stop heating and wait 5 or 6 min before shutting off the air supply. Disconnect the delivery tube which then serves as a stirrer. Cool the contents of the beaker to about 20 °C and add 45 ml of standard potassium iodate solution (3.4) from a burette. Mix and add about 25 ml of concentrated hydrochloric acid. Titrate the excess of iodine with the sodium thiosulphate solution (3.3). Near the end of the reaction (pale yellow colour of solution) add 2 ml of starch solution (3.1) and continue the titration until disappearance of the blue colour. Recording the volume V of sodium thiosulphate solution used, restore a faint blue colour with the standard potassium iodate solution and note the total volume V_0 of potassium iodate solution used.

Carry out a blank test, using the same method and the same amounts of reagents as in the test on cement, so as to determine any possible correction to be made to analytical results.

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NOTES

1. Before disconnecting the delivery tube, it is advisable to pass a further moderate flow of air and test with lead acetate paper to check that liberation of hydrogen sulphide is complete.
2. When the sulphur content is more than 0.25 % use a sample smaller than 5 g.
3. Cooling the beaker and its contents is very important. Determination of the end-point of the titration is difficult when the solution is hot.
4. The presence of sulphites, thiosulphates and other compounds, intermediate between sulphides and sulphates, upsets sulphur determinations.

6. EXPRESSION OF RESULTS

The sulphur content, expressed as percent is calculated by the following formula :

$$S \% = (V_0 \times E - V \times e) \times 20$$

where

- E is the sulphur equivalent of potassium iodate solution expressed in grammes per millilitre,
- V_0 is the volume of potassium iodate solution used for the determination, expressed in millilitres,
- V is the volume of thiosulphate solution required for titration of excess iodine, expressed in millilitres,
- e is the sulphur equivalent of thiosulphate solution, expressed in grammes per millilitre,
- 20 is 100 divided by the mass of the sample (5 g).

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