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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 682

CHEMICAL ANALYSIS OF CEMENTS

DETERMINATION OF SULPHUR AS SULPHIDE

1st EDITION
March 1968

For each individual country the only valid standard is the national standard of that country.

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

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.

ISO Recommendation R 682 March 1968

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 $Na_2S_2O_3.5\,H_2O$ 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 :

$$e = \frac{A \times C \times 0.2392}{B \times D} \qquad ①$$

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

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

3.5 *Stannous chloride solution*

Add 7 ml of concentrated hydrochloric acid to 10 g of $SnCl_2.2H_2O$. 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 $ZnSO_4.7H_2O$ 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 $CdCl_2.2H_2O$ in 150 ml of water and 350 ml of ammonium hydroxide solution. Allow to stand for at least 24 hours and filter.

NOTES

1. 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.

2. 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 Woolf 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.