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R 912

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SULPHURIC ACID AND OLEUM FOR INDUSTRIAL USE

DETERMINATION OF SULPHUR DIOXIDE CONTENT

BARIUM SULPHATE GRAVIMETRIC METHOD

1st EDITION

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BRIEF HISTORY

The ISO Recommendation R 912, *Sulphuric acid and oleum for industrial use – Determination of sulphur dioxide content – Barium sulphate gravimetric method*, was drawn up by Technical Committee ISO/TC 47, *Chemistry*, the Secretariat of which is held by the Ente Nazionale Italiano di Unificazione (UNI).

Based on detailed work on this question carried out by the Technical Committee, a Draft ISO Recommendation was adopted in 1965.

In June 1967, this Draft ISO Recommendation (No. 1182) 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 :

Austria	India	South Africa, Rep. of
Belgium	Iran	Spain
Brazil	Ireland	Switzerland
Chile	Italy	Thailand
Cuba	Japan	Turkey
Czechoslovakia	Netherlands	U.A.R.
France	New Zealand	U.S.S.R.
Germany	Poland	Yugoslavia
Hungary	Portugal	
ICAITI*	Romania	

One Member Body opposed the approval of the Draft :

United Kingdom

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

* Instituto Centroamericano de Investigación y Tecnología Industrial (Costa Rica, Guatemala, Honduras, Nicaragua, El Salvador, Panama).

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SULPHURIC ACID AND OLEUM FOR INDUSTRIAL USE

DETERMINATION OF SULPHUR DIOXIDE CONTENT

BARIUM SULPHATE GRAVIMETRIC METHOD

1. SCOPE

This ISO Recommendation describes a gravimetric method for the determination of sulphur dioxide content of sulphuric acid and oleum for industrial use.

2. FIELD OF APPLICATION

The method is applicable to the determination of SO_2 content higher than 0.005 % (m/m) of sulphuric acid and oleum for industrial use.

3. PRINCIPLE

Displacement of the sulphur dioxide present by means of a current of pure nitrogen and absorption in a sodium hydroxide solution containing hydrogen peroxide.

Determination of the sulphuric acid by precipitation as barium sulphate in dilute hydrochloric acid.

Separation of the precipitate, ignition at 600 to 800 °C and weighing.

4. REAGENTS

Distilled water or water of equivalent purity should be used in the test.

4.1 *Nitrogen*, pure.

4.2 *Hydrogen peroxide*, 30 g/l solution, free from sulphate ions.

4.3 *Hydrochloric acid*, approximately $d = 1.18$, 37 % (m/m) or 12 N solution, free from sulphate ions.

4.4 *Barium chloride*, dihydrate ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$), 100 g/l solution.

4.5 *Silver nitrate*, 5 g/l solution.

Dissolve 0.5 g of silver nitrate in water and dilute to 100 ml.

4.6 *Potassium permanganate*, approximately 0.1 N solution.

4.7 *Sodium hydroxide*, approximately 0.1 N solution.

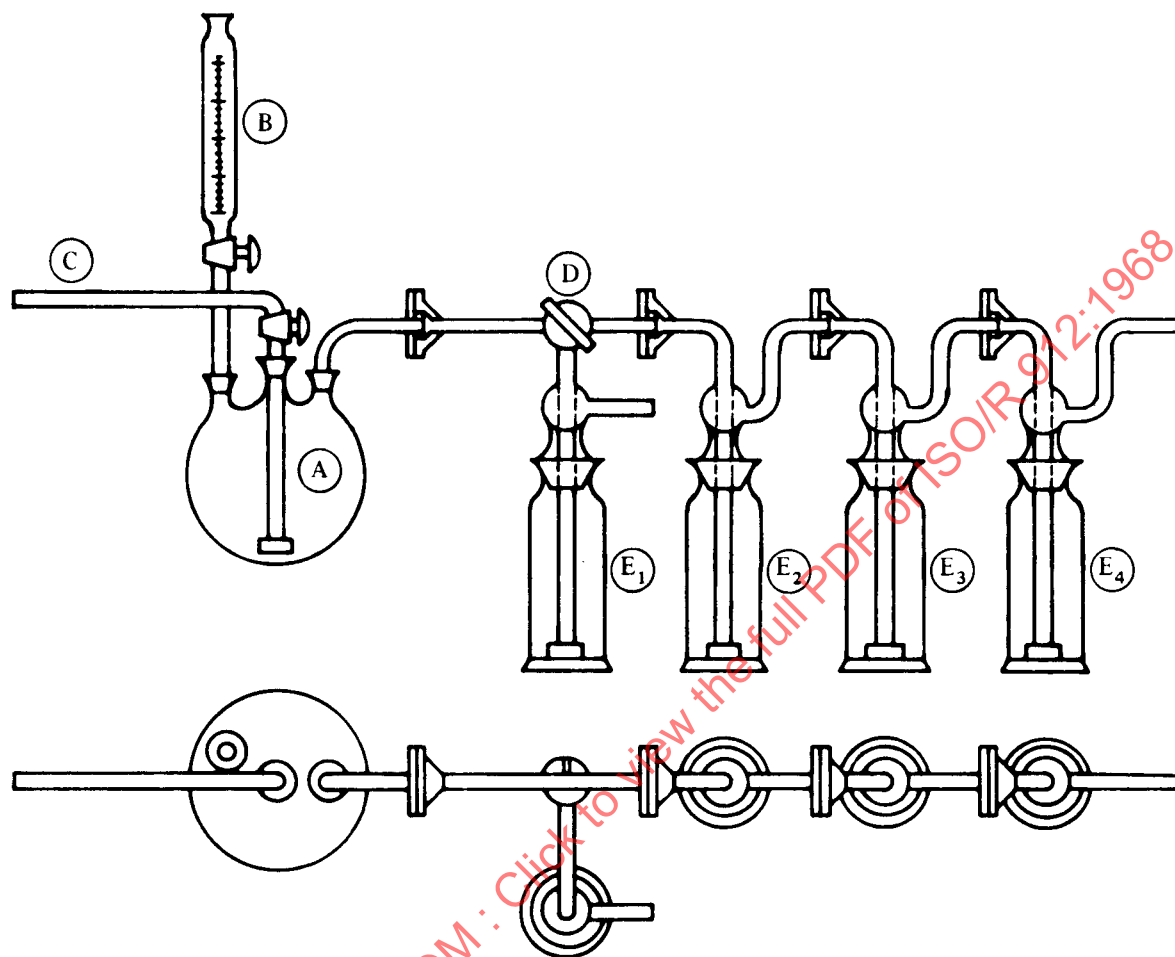
4.8 *Litmus paper*.

4.9 *Sulphuric acid*, approximately $d = 1.84$, 96 % (m/m) or 36 N solution.

5. APPARATUS

Ordinary laboratory apparatus and

5.1 Glass apparatus (see Figure below), with ground glass joints.



- (A) 500 ml, three-necked, round-bottomed flask.
- (B) 200 ml, graduated separating funnel fitting into one of the three necks of the flask.
- (C) Tube with cock, inserted into the central neck of the flask, extending about 1 cm from the bottom of the flask. The end of the tube is fitted with a sintered glass diaphragm.
- (D) Three-way cock.
- (E) Four wash-bottles (E_1 , E_2 , E_3 , E_4), capacity 100 ml, fitted with a sintered glass diaphragm (porosity 3 to 15 μm) at the end of the immersion tube, for gas distribution. One branch of the cock (D) is connected to the third neck of the round-bottomed flask (A); the second branch is connected to the first wash-bottle (E_1), which can be connected and disconnected according to requirement through the cock (D); the third branch is connected to the other wash-bottles linked in series.

FIGURE – Glass apparatus