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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 914

SULPHURIC ACID AND OLEUM FOR INDUSTRIAL USE

DETERMINATION OF TOTAL NITROGEN CONTENT

Volumetric method

1st EDITION
December 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 914, *Sulphuric acid and oleum for industrial use – Determination of total nitrogen content – Volumetric 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. 1184) 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	United Kingdom
Germany	Poland	U.S.S.R.
Hungary	Portugal	Yugoslavia
ICAITI *	Romania	

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 December 1968, to accept it as an ISO RECOMMENDATION.

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

ISO Recommendation R 914 December 1968

SULPHURIC ACID AND OLEUM FOR INDUSTRIAL USE

DETERMINATION OF TOTAL NITROGEN CONTENT

Volumetric method

1. SCOPE

This ISO Recommendation describes a volumetric method for the determination of total nitrogen content of sulphuric acid and oleum for industrial use.

2. FIELD OF APPLICATION

The method is applicable to the determination of total nitrogen content greater than 0.05 % (m/m) of sulphuric acid and oleum for industrial use.

3. PRINCIPLE

Conversion of the nitrogen present to ammonia by means of nascent hydrogen. Distillation and absorption of ammonia by an excess of sulphuric acid standard volumetric solution and back-titration with a sodium hydroxide standard volumetric solution in the presence of methylred-methylene blue indicator.

4. REAGENTS

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

4.1 *Devarda alloy* (45 % Al – 50 % Cu – 5 % Zn), particle size 0.2 to 0.3 mm.

4.2 *Sodium hydroxide*, 250 g/l solution.

4.3 *Potassium permanganate*, 10 g/l solution.

Dissolve 1 g of potassium permanganate in water and dilute to 100 ml.

4.4 *Sulphuric acid*, 0.1 N standard volumetric solution (see Note, section 7).

4.5 *Sodium hydroxide*, 0.1 N standard volumetric solution (see Note, section 7).

4.6 *Mixed indicator*, ethanolic solution.

Dissolve 0.1 g of methylred in 50 ml of 95 % (v/v) ethanol, add 0.05 g of methylene blue and dilute to 100 ml with the same ethanol.

4.7 *Litmus paper* (red).

5. APPARATUS

Ordinary laboratory apparatus and

5.1 *Weighing bottle*, ground glass stoppered, capacity approximately 60 ml.

5.2 *Distillation apparatus* (see Figure opposite) with ground glass joints.

6. PROCEDURE

6.1 Test portion and preparation of sample solution

Fill weighing bottle (5.1) with the test sample and weigh by difference, to the nearest 10 mg, a test portion of approximately 50 g.

Cooling to ensure that the temperature is kept below 40 °C, slowly pour the test portion onto crushed ice contained in a beaker of suitable capacity.

Transfer the solution quantitatively to a 100 ml one-mark volumetric flask. Dilute to the mark and mix thoroughly.

Transfer 50.0 ml of this solution to the distillation flask (A) through separating funnel (D).

Wash the separating funnel (D) with at least 80 ml of water, collecting the washings in the distillation flask (A).

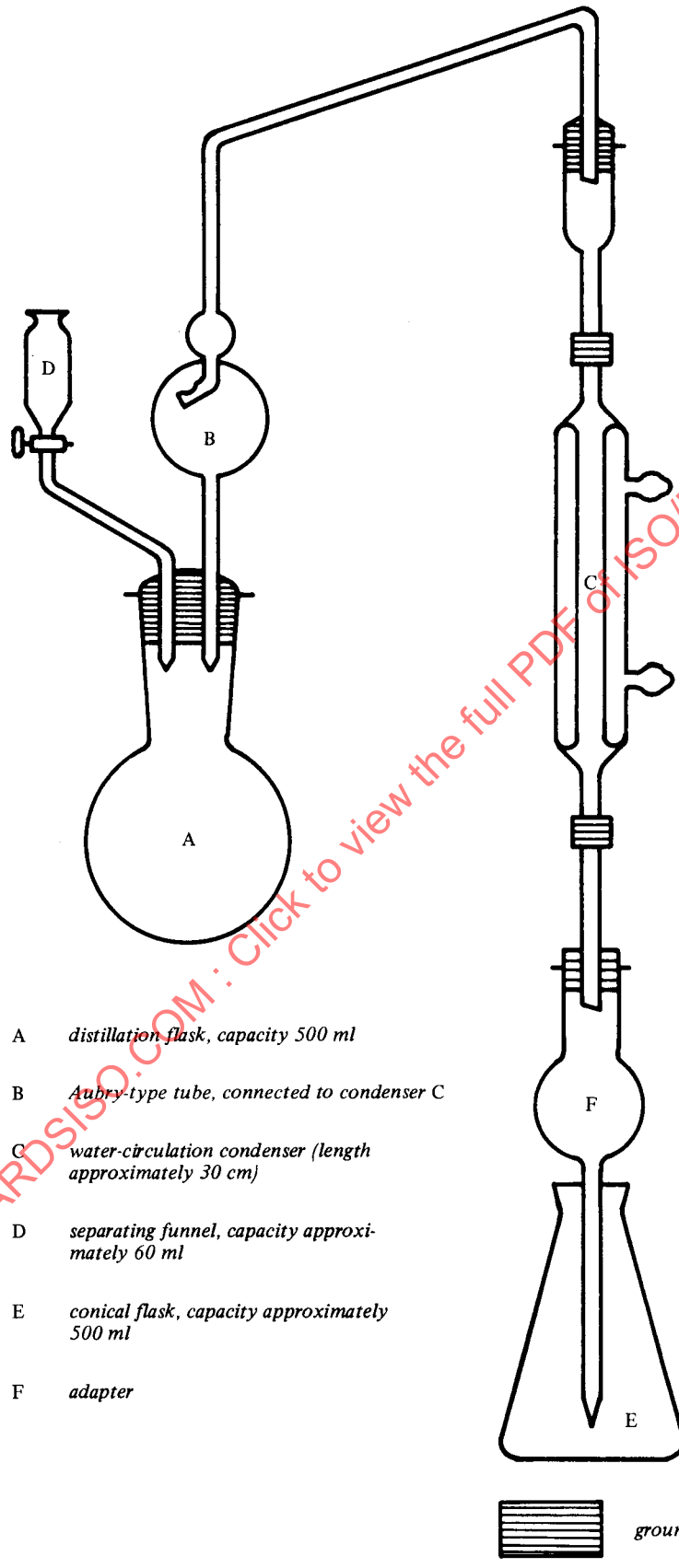
6.2 Blank test

At the same time as the analysis and following the same procedure, carry out a blank test using the same quantity of ice and reagents as used in the test.

6.3 Determination

6.3.1 *Assembly of the apparatus.* Connect the different parts of the apparatus as indicated in the Figure opposite.

Transfer 50.0 ml of the sulphuric acid standard volumetric solution (4.4) to the conical flask (E).



A *distillation flask, capacity 500 ml*

B *Aubry-type tube, connected to condenser C*

C *water-circulation condenser (length approximately 30 cm)*

D *separating funnel, capacity approximately 60 ml*

E *conical flask, capacity approximately 500 ml*

F *adapter*

FIGURE – **Distillation apparatus**

6.3.2 *Oxidation and neutralization of the sample solution.* Introduce into the distillation flask (A), through separating funnel (D), a sufficient quantity of the potassium permanganate solution (4.3) to give the solution a pink colouration persistent for a few minutes.

Cool the distillation flask (A), add two drops of the mixed indicator solution (4.6) and neutralize the solution by adding sodium hydroxide solution (4.2), through the separating funnel (D).

Disconnect the distillation flask, place in it approximately 1 g of the Devarda alloy (4.1) and reconnect as quickly as possible to the apparatus.

6.3.3 *Distillation of ammonia.* Through the separating funnel (D), pour into the distillation flask (A) 25 ml of the sodium hydroxide solution (4.2) and carefully bring to a gentle boil.

Distil approximately 150 ml at the rate of one drop per second. When the liquid in the conical flask reaches a volume of about 200 ml, check the neutrality of the liquid distilling over. (For this purpose let a drop of the distillate fall on the litmus paper (4.7), which should not change colour.)

Stop distilling at this point. Wash condenser (C) and collect the washing in conical flask (E).

6.3.4 *Titration.* Add to the solution a few drops of the mixed indicator solution (4.6) and back-titrate the excess of acid by means of the sodium hydroxide solution (4.5).

7. EXPRESSION OF RESULTS

Total nitrogen content (N) is given as a percentage, by mass, by the following formula :

$$\frac{[(V_1 - V_2) - (V_{B1} - V_{B2})] \times A \times 200}{E}$$

where

V_1 is the volume, in millilitres, of the sulphuric acid solution (4.4) placed in conical flask (E) for the test (see Note below),

V_2 is the volume, in millilitres, of the sodium hydroxide solution (4.5) used for the back-titration of the excess of the sulphuric acid solution (V_1) (see Note below),

V_{B1} is the volume, in millilitres, of the sulphuric acid solution (4.4) placed in conical flask (E) for the blank test (see Note below),

V_{B2} is the volume, in millilitres, of the sodium hydroxide solution (4.5) used for the back-titration of the excess of sulphuric acid (V_{B1}) (see Note below),

A is the mass, in grammes, of nitrogen corresponding to 1 ml of 0.1 N sulphuric acid solution (theoretical value 1 ml $\triangleq$ 0.0014 g of N) (see Note below).

E is the mass, in grammes, of the test portion.

NOTE. – If the sulphuric acid (4.4) and sodium hydroxide (4.5) standard volumetric solutions are not of exactly the strength indicated in the list of reagents, a suitable correction factor should be employed in calculating the results.