INTERNATIONAL STANDARD 914

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Sulphuric acid and oleum for industrial use — Determination of total nitrogen content — Titrimetric method after distillation

Acide sulfurique et oléums à usage industriel — Dosage de l'azote total — Méthode titrimétrique après distillation

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

Prior to 1972, the results of the work of the technical committees were published as ISO Recommendations; these documents are in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 47, *Chemistry*, has reviewed ISO Recommendation R 914-1968 and found it technically suitable for transformation. International Standard ISO 914 therefore replaces ISO Recommendation R 914-1968, to which it is technically identical.

ISO Recommendation R 914 had been approved by the member bodies of the following countries :

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

No member body had expressed disapproval of the Recommendation.

No member body disapproved the transformation of the Recommendation into an International Standard.

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Sulphuric acid and oleum for industrial use — Determination of total nitrogen content — Titrimetric method after distillation

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a titrimetric method, after distillation, for the determination of the total nitrogen content of sulphuric acid and oleum for industrial use.

The method is applicable to products having a total nitrogen content equal to or greater than 0,05 % (*m/m*).

2 PRINCIPLE

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

3 REAGENTS

During the analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity.

3.1 Devarda alloy (45 % Al — 50 % Cu — 5 % Zn), particle size 0,2 to 0,3 mm.

3.2 Sodium hydroxide, 250 g/l solution.

3.3 Potassium permanganate, 10 g/l solution.

3.4 Sulphuric acid, 0,1 N standard volumetric solution.

3.5 Sodium hydroxide, 0,1 N standard volumetric solution.

3.6 Mixed indicator, ethanolic solution.

Dissolve 0,1 g of methyl red in about 50 ml of 95 % (*V/V*) ethanol and add 0,05 g of methylene blue. After dissolution, dilute to 100 ml with the same ethanol and mix.

3.7 Litmus paper (red).

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Weighing pipette, capacity approximately 60 ml, with ground glass stopper.

4.2 Distillation apparatus, with, preferably, spherical ground glass joints, or any apparatus that will ensure quantitative distillation and absorption.

The apparatus may, for example, be made up from the following items (see figure) :

4.2.1 *Distillation flask* (A), capacity 1 000 ml, with female joint.

4.2.2 *Splash head* (B), with male joints and parallel inlet and outlet into which is fused a cylindrical dropping-funnel (C), capacity 50 ml.

4.2.3 *Liebig condenser* (D), effective length about 400 mm, fitted with a female joint at the inlet and a male joint at the outlet.

4.2.4 *Conical flask* (E), capacity 500 ml, with female joint, fitted with two side bulbs.

4.2.5 *Spring clamps* (F).

5 PROCEDURE[1)]

5.1 Test portion and preparation of the test solution

Fill the weighing pipette (4.1) with the test sample and weigh by difference, to the nearest 0,01 g, 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.

1) The procedure is described in terms of the apparatus specified in 4.2 and will require modification if other apparatus is used.