

**INTERNATIONAL STANDARD****914***G-96-05*

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

**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.

# 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<sup>1)</sup>

### 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.

Transfer 50,0 ml of this solution to the distillation flask (A) through the dropping-funnel (C).

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

## 5.2 Blank test

Carry out a blank test at the same time as the determination, following the same procedure and using the same quantity of ice and the same reagents as used during the determination, but omitting the test portion.

## 5.3 Determination

### 5.3.1 Assembly of the apparatus

Connect the different parts of the apparatus as indicated in the figure; coat the joints of the apparatus with a silicone grease.

Transfer 50,0 ml of the standard volumetric sulphuric acid solution (3.4) to the conical flask (E).

### 5.3.2 Oxidation and neutralization of the test solution

Introduce into the distillation flask (A), through the dropping-funnel (C), a sufficient quantity of the potassium permanganate solution (3.3) to give the solution a pink coloration persistent for a few minutes.

Cool the distillation flask (A), add 2 drops of the mixed indicator solution (3.6) and neutralize the solution by adding the sodium hydroxide solution (3.2), through the dropping-funnel (C).

Disconnect the distillation flask (A), place in it approximately 1 g of the Devarda alloy (3.1) and reconnect the flask as quickly as possible to the apparatus.

### 5.3.3 Distillation

Through the dropping-funnel (C), introduce into the distillation flask (A) 25 ml of the sodium hydroxide solution (3.2), taking care to leave at least a few millimetres of liquid above the tap. Carefully bring to a gentle boil.

Distil approximately 150 ml at the rate of 1 drop per second. When the liquid in the conical flask (E) reaches a volume of about 200 ml, check the neutrality of the liquid distilling over. (For this purpose let 1 drop of the distillate fall on the litmus paper (3.7), which should not change colour.)

Stop the heating, open the tap of the dropping-funnel (C), disconnect the splash head (B) and wash the condenser (D) carefully, collecting the wash water in the flask (E). Finally disconnect the flask (E).

### 5.3.4 Titration

Carefully mix the solution contained in the flask (E) and in the two side bulbs, add a few drops of the mixed indicator (3.6), and back-titrate the excess of the standard volumetric sulphuric acid solution (3.4) with the standard volumetric sodium hydroxide solution (3.5).

During the titration, stir carefully to ensure that the solution is completely mixed.

## 6 EXPRESSION OF RESULTS

The total nitrogen content, expressed as a percentage by mass of nitrogen (N), is given by the formula

$$\begin{aligned} & (V_1 - V_2) \times 0,0014 \times \frac{100}{50} \times \frac{100}{m} \\ & = \frac{0,28 (V_1 - V_2)}{m} \end{aligned}$$

where

$V_1$  is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (3.5) used for the back-titration of the excess of the standard volumetric sulphuric acid solution (3.4) placed in the flask (E) for the blank test;

$V_2$  is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (3.5) used for the back-titration of the excess of the standard volumetric sulphuric acid solution (3.4) placed in the flask (E) for the determination;

$m$  is the mass, in grams, of the test portion (5.1);

0,0014 is the mass, in grams, of nitrogen corresponding to 1 ml of exactly 1 N sulphuric acid solution.

NOTE — If the concentrations of the standard volumetric solutions are not exactly as specified in the list of reagents, appropriate corrections should be made.

## 7 TEST REPORT

The test report shall include the following particulars :

- the reference of the method used;
- the results and the method of expression used;
- any unusual features noted during the determination;
- any operation not included in this International Standard, or regarded as optional.

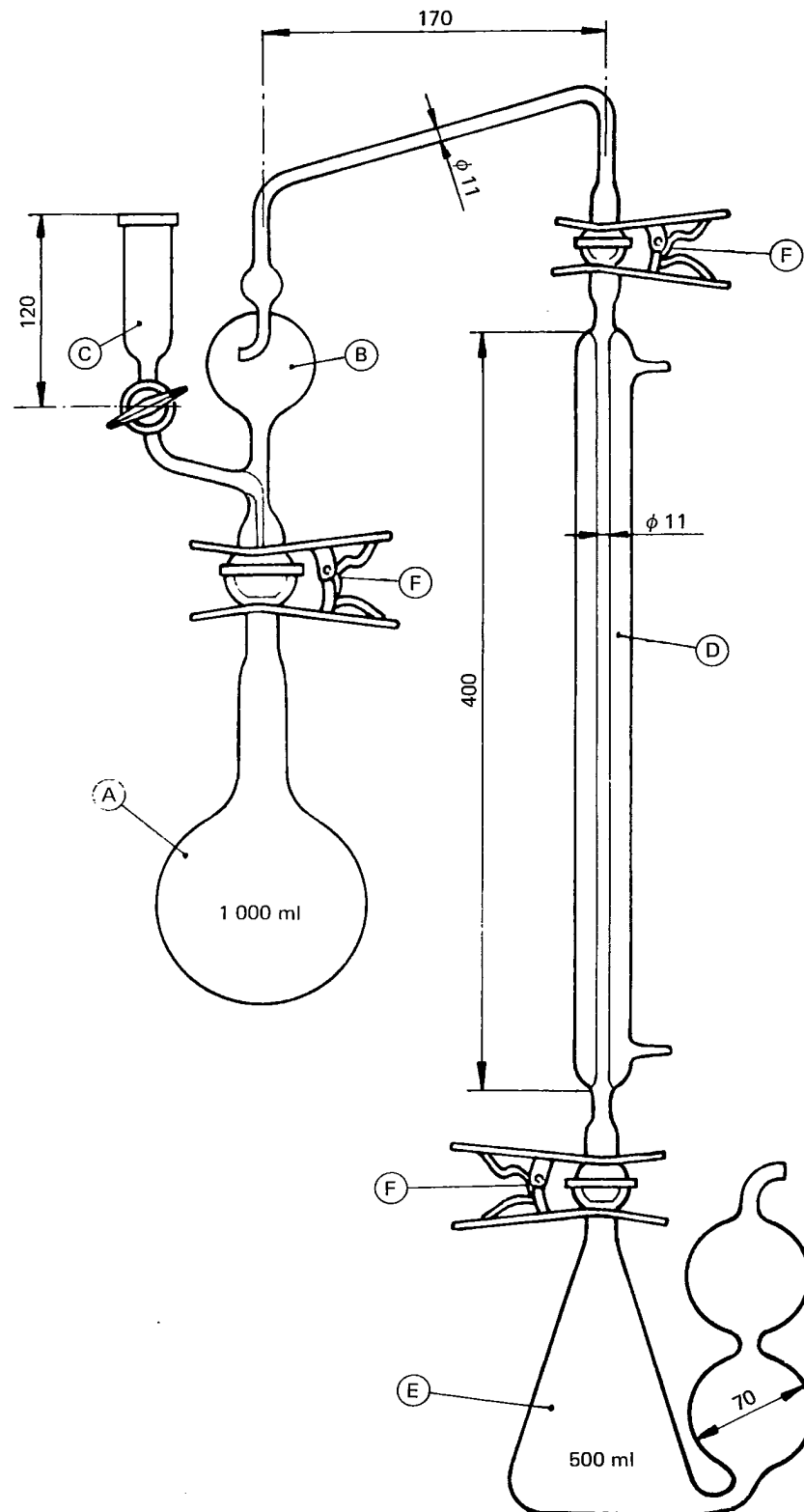


FIGURE – Typical distillation apparatus (4.2)

## ANNEX

## ISO PUBLICATIONS RELATING TO SULPHURIC ACID AND OLEUM FOR INDUSTRIAL USE

- ISO 910 – Determination of total acidity, and calculation of free sulphur trioxide content of oleum – Titrimetric method.
- ISO 911 – Evaluation of sulphuric acid concentration by measurement of density.\*
- ISO 912 – Determination of sulphur dioxide content – Barium sulphate gravimetric method.
- ISO 913 – Determination of ash – Gravimetric method.
- ISO 914 – Determination of total nitrogen content – Titrimetric method after distillation.
- ISO/R 915 – Determination of iron content – 2,2'-Bipyridyl spectrophotometric method.
- ISO 2363 – Determination of oxides of nitrogen – 2,4-Xylenol spectrophotometric method.
- ISO 2717 – Determination of lead content – Dithizone photometric method.
- ISO 2877 – Determination of chlorides content – Potentiometric method.\*
- ISO 2899 – Determination of ammoniacal nitrogen content – Spectrophotometric method.
- ISO 3423 – Determination of sulphur dioxide content – Iodometric method.
- ISO 5792 – Determination of arsenic content – Silver diethyldithiocarbamate photometric method.\*

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\* Applicable only to sulphuric acid.