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Ammonium sulphate for industrial use — Determination of ammoniacal nitrogen content — Titrimetric method after distillation

Sulfate d'ammonium à usage industriel — Dosage de l'azote ammoniacal — Méthode titrimétrique après distillation

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FOREWORD

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It has been approved by the Member Bodies of the following countries :

Austria	Hungary	South Africa, Rep. of
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Germany	Poland	Yugoslavia

No Member Body expressed disapproval of the document.

Ammonium sulphate for industrial use — Determination of ammoniacal 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 ammoniacal nitrogen content of ammonium sulphate for industrial use.

2 PRINCIPLE

Distillation of the ammonia after displacement by an alkali solution; absorption in an excess of standard volumetric sulphuric acid solution and back-titration with standard volumetric sodium hydroxide solution in the presence of an indicator.

3 REAGENTS

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

3.1 Sodium hydroxide, 450 g/l solution.

3.2 Sulphuric acid, 0,5 N standard volumetric solution.

3.3 Sodium hydroxide, 0,5 N standard volumetric solution.

3.4 Mixed indicator, ethanolic solution.

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

4 APPARATUS

Ordinary laboratory apparatus and

4.1 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) :

4.1.1 Distillation flask (A), capacity 1 000 ml, with a female joint.

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

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

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

4.1.5 Spring clamps (F).

5 PROCEDURE¹⁾

5.1 Test portion

Weigh, to the nearest 0,001 g, about 10 g of the test sample.

5.2 Blank test

Carry out a blank test at the same time as the determination, following the same procedure and using the same quantities of all the reagents as used for the determination.

5.3 Determination

5.3.1 Preparation of the sample solution

Place the test portion (5.1) in a 500 ml one-mark volumetric flask, dissolve in water, dilute to the mark and mix.

5.3.2 Distillation

Place 50,0 ml of the sample solution (5.3.1) in the distillation flask (A). Add about 350 ml of water and a few anti-bumping granules.

Coat the joints of the apparatus with a silicone grease. Mount the splash head (B) on the flask (A), and connect it to the condenser (D).

Place 40,0 ml of the standard volumetric sulphuric acid solution (3.2), about 80 ml of water and a few drops of the mixed indicator solution (3.4) into the flask (E). Connect the flask (E) to the condenser (D), ensuring that all the joints of the apparatus are firm by means of the spring clamps (F).

1) The procedure is described in terms of the apparatus specified in 4.1 and will require modification if other apparatus is used.