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Sulphuric acid and oleums for industrial use — Determination of ammoniacal nitrogen content — Spectrophotometric method

Acide sulfurique et oléums à usage industriel — Dosage de l'azote ammoniacal — Méthode spectrophotométrique

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FOREWORD

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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.

International Standard ISO 2899 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in September 1972.

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

Australia	Ireland	Spain
Austria	Israel	Sweden
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This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

No Member Body expressed disapproval of the document.

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Sulphuric acid and oleums for industrial use – Determination of ammoniacal nitrogen content – Spectrophotometric method

1 SCOPE

This International Standard specifies a spectrophotometric method using sodium phenate and sodium hypochlorite for the determination of the ammoniacal nitrogen content of sulphuric acid and oleums for industrial use.

2 FIELD OF APPLICATION

The method is applicable to products containing more than 0,000 1 % (*m/m*) of ammoniacal nitrogen. The procedure, as described, is suitable for contents between 0,000 1 and 0,000 5 % (*m/m*). If the acid analyzed contains more than 0,000 5 % (*m/m*) of ammoniacal nitrogen the mass of the test portion should be decreased accordingly.

3 PRINCIPLE

Distillation, with entrainment by steam, of the ammonia in the presence of an excess of sodium hydroxide and collection of the distillate in an excess of acid solution.

Neutralization of the excess of acid and formation of the coloured complex by treatment with sodium phenate and sodium hypochlorite in the presence of acetone.

Spectrophotometric measurement of the indophenol obtained at a wavelength of about 630 nm.

4 REAGENTS

Distilled water, or water of equivalent purity, of which the ammoniacal nitrogen content is negligible, and ice prepared from this water, shall be used in the test.

NOTE – Verify the quality of this water by means of a mixture of sodium phenate and sodium hypochlorite. Proceed as described in 6.3.1 for the preparation of the compensation solution; there should be no appreciable colour produced.

4.1 Sodium hydroxide, 350 g/l solution.

Boil this solution for 20 min to remove traces of ammoniacal nitrogen and make up to the original volume.

4.2 Sulphuric acid, approximately 0,1 N solution.

4.3 Sodium hydroxide, approximately N solution.

4.4 Sodium hydroxide, approximately 0,1 N solution.

4.5 Phenolphthalein, 10 g/l ethanolic solution.

Dissolve 1 g of phenolphthalein in 95 % (V/V) ethanol and dilute to 100 ml with the same ethanol.

4.6 Acetone.

4.7 Sodium phenate (sodium phenolate), approximately 155 g/l solution.

Dissolve 12,5 g of phenol in 27 ml of approximately 5 N sodium hydroxide solution and dilute to 100 ml.

Immediately place the solution in the dark.

Prepare this solution immediately before it is required for use.

4.8 Sodium hypochlorite, solution containing 10 g of available chlorine per litre.

Dilute a concentrated solution of sodium hypochlorite (100 to 140 g of available chlorine per litre), previously standardized against a solution of sodium arsenite. Do not use concentrated solutions containing less than 80 g of available chlorine per litre.

Store this solution in a cool place and in the absence of light.

The solution is stable for about 4 weeks.

4.9 Ammonium chloride, standard solution corresponding to 1 g of ammoniacal nitrogen per litre.

Weigh, to the nearest 0,000 1 g, 3,819 g of ammonium chloride, previously dried at 100 °C and allowed to cool in a desiccator. Place in a beaker of suitable capacity and dissolve in water. Transfer quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this solution contains 1 mg of ammoniacal nitrogen.

Renew the solution at least once a month.