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NITRIC ACID FOR INDUSTRIAL USE

DETERMINATION OF NITROUS COMPOUNDS

VOLUMETRIC METHOD

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NITRIC ACID FOR INDUSTRIAL USE

DETERMINATION OF NITROUS COMPOUNDS

VOLUMETRIC METHOD

1. SCOPE

This ISO Recommendation describes a volumetric method for the determination of the nitrous compounds in nitric acid for industrial use, conventionally expressed as HNO_2.

2. FIELD OF APPLICATION

The method is applicable to nitric acid for industrial use. Two cases are considered :

2.1 Content of nitrous compounds, expressed as HNO_2, greater than or equal to 0.01 % (m/m).

2.2 Content of nitrous compounds, expressed as HNO_2, less than 0.01 % (m/m).

3. PRINCIPLE

Oxidation of the nitrous compounds present by an excess of standard volumetric potassium permanganate solution. Determination of the potassium permanganate remaining by means of a standard volumetric iron (II) sulphate solution.

4. REAGENTS

Distilled water or water of equivalent purity, cooled to 0 °C, should be used in the test.

4.1 *Sulphuric acid*, approximately 4 N solution.

4.2 *Potassium permanganate*, 0.1 N standard volumetric solution.

4.3 *Potassium permanganate*, 0.01 N standard volumetric solution.

4.4 *Iron (II) sulphate*, 0. 1 N standard volumetric solution.

4.5 *Iron (II) sulphate*, 0.01 N standard volumetric solution.

5. APPARATUS

Ordinary laboratory apparatus, and

5.1 *Conical flasks*, 500 ml capacity, diameter of neck approximately 30 mm, fitted with ground glass stoppers.

5.2 *Bath of melting ice.*

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

6. PROCEDURE

6.1 Cooling of test sample

Cool the test sample to approximately 0 °C by immersion in the ice bath (5.2) for about 30 minutes.

6.2 Preliminary test

The purpose of this preliminary test is to determine whether the content of nitrous compounds, expressed as HNO_2, is less than or greater than 0.01 % (m/m).

Fill the weighing pipette (5.3) with the previously cooled test sample (6.1) and weigh by difference to the nearest 0.01 g a test portion of approximately 20 g. Collect the test portion in one of the flasks (5.1) already containing approximately 100 ml of water previously cooled to 0 °C. Add 20 ml of the sulphuric acid solution (4.1). Cool again to 0 °C and mix.

Titrate the nitrous acid with the potassium permanganate solution (4.2) until a pink colour is obtained that lasts for 1 minute.

If the volume used is greater than 0.85 ml, carry out the determination as described in clause 6.3.2; if not, proceed as described in clause 6.3.3.

6.3 Determination

6.3.1 *Test portion.* Fill the weighing pipette (5.3) with the previously cooled test sample (6.1) and weigh by difference to the nearest 0.01 g a test portion of approximately 20 g.

6.3.2 *Content of nitrous compounds, expressed as* HNO_2, *greater than or equal to 0.01 % (m/m).* In one of the flasks (5.1) place approximately 100 ml of water previously cooled to about 0 °C, 20 ml of the sulphuric acid solution (4.1) previously cooled to 0 °C, and a volume (V_1) of the potassium permanganate solution (4.2) greater by 10.0 ml than that determined in the preliminary test (6.2).

Quickly add the test portion (6.3.1). Close the flask immediately and shake until all the fumes have disappeared (approximately 5 minutes).

Add 20.0 ml of the iron (II) sulphate solution (4.4) and titrate the excess with the potassium permanganate solution (4.2) until a pink colour is obtained that lasts for 1 minute (V_2).

In order to establish the equivalence of the two solutions under the conditions of the determination, add to the same flask a further 20.0 ml of iron (II) sulphate solution (4.4) and titrate with the potassium permanganate solution (4.2) (V_3).

6.3.3 *Content of nitrous compounds, expressed as* HNO_2, *less than 0.01 % (m/m).* Carry out the determination as described in clause 6.3.2 but using standard volumetric potassium permanganate (4.3) and iron (II) sulphate (4.5) solutions instead of standard volumetric potassium permanganate (4.2) and iron (II) sulphate (4.4) solutions.

7. EXPRESSION OF RESULTS

7.1 Content of nitrous compounds greater than or equal to 0.01 % (m/m)

The content of nitrous compounds, expressed as HNO_2, is given, as a percentage by mass, by the formula

$$\frac{[(V_1 + V_2) - V_3] \times 0.002\,35}{m} \times 100$$

where

V_1 is the volume, in millilitres, of standard volumetric potassium permanganate solution (4.2) added at the beginning;

V_2 is the volume, in millilitres, of standard volumetric potassium permanganate solution (4.2) used for the first titration;

V_3 is the volume, in millilitres, of standard volumetric potassium permanganate solution (4.2) used for the second titration;

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