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Nitric acid for industrial use — Determination of nitrous compounds — Titrimetric method

Acide nitrique à usage industriel — Dosage des composés nitreux — Méthode titrimétrique

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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 1981-1971 and found it technically suitable for transformation. International Standard ISO 1981 therefore replaces ISO Recommendation R 1981-1971, to which it is technically identical.

ISO Recommendation R 1981 had been approved by the member bodies of the following countries :

Australia	India	Portugal
Austria	Iran	Romania
Belgium	Ireland	South Africa, Rep. of
Chile	Israel	Switzerland
Czechoslovakia	Italy	Thailand
Egypt, Arab Rep. of	Netherlands	Turkey
France	New Zealand	United Kingdom
Germany	Peru	U.S.A.
Greece	Poland	U.S.S.R.

No member body had expresse disapproval of the Recommendation.

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

Printed in Switzerland

Nitric acid for industrial use — Determination of nitrous compounds — Titrimetric method

1 SCOPE

This International Standard specifies a titrimetric method for the determination of the nitrous compounds in nitric acid for industrial use, conventionally expressed as HNO_2.

2 FIELD OF APPLICATION

Two cases are considered :

a) content of nitrous compounds, expressed as HNO_2, equal to or greater than 0,01 % (*m/m*);

b) content of nitrous compounds, expressed as HNO_2, less than 0,01 % (*m/m*).

3 PRINCIPLE

Oxidation of the nitrous compounds present in a test portion by an excess of standard volumetric potassium permanganate solution. Addition of an excess of a standard volumetric iron(II) sulphate solution, and titration of this excess with a standard volumetric potassium permanganate solution.

4 REAGENTS

During the analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity, cooled to 0 °C.

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

5.2 Bath of melting ice.

5.3 Weighing pipette, capacity approximately 60 ml, 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 min.

6.2 Preliminary test

By means of the following preliminary test, 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 cooled test sample (6.1) and weigh by difference, to the nearest 0,01 g, a test portion of approximately 20 g. Transfer the test portion to one of the flasks (5.1), containing approximately 100 ml of water cooled to 0 °C. Add 20 ml of the sulphuric acid solution (4.1), cooled to 0 °C, and mix.

Titrate the nitrous compounds with the potassium permanganate solution (4.2) until a pink colour is obtained that remains stable for 1 min.

If the volume used is greater than 0,85 ml, carry out the determination as specified in 6.3.2; if not, proceed as specified in 6.3.3

6.3 Determination

6.3.1 *Test portion*

Fill the weighing pipette (5.3) with the 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, *equal to or greater than* 0,01 % *(m/m)*

In one of the flasks (5.1) place approximately 100 ml of water cooled to about 0 °C, 20 ml of the sulphuric acid solution (4.1) cooled to 0 °C, and a volume (V_0) 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 min).

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 remains stable for 1 min (V_1).

In order to establish the equivalence of the two standard volumetric solutions under the conditions of the determination, add to the same flask a further 20,0 ml of the iron(II) sulphate solution (4.4) and titrate with the potassium permanganate solution (4.2) (V_2).

6.3.3 *Content of nitrous compounds, expressed as* HNO_2, *less than* 0,01 % *(m/m)*

Carry out the determination as specified in 6.3.2 but using the potassium permanganate (4.3) and iron(II) sulphate (4.5) solutions.

7 EXPRESSION OF RESULTS

7.1 Content of nitrous compounds equal to or greater than 0,01 % (m/m)

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

$$\frac{[(V_0 + V_1) - V_2] \times 0{,}002\,35 \times 100}{m}$$

$$= \frac{0{,}235\,[(V_0 + V_1) - V_2]}{m}$$

where

V_0 is the volume, in millilitres, of the potassium permanganate solution (4.2) added at the beginning;

V_1 is the volume, in millilitres, of the potassium permanganate solution (4.2) used for the first titration;

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

m is the mass, in grams, of the test portion (6.3.1);

0,002 35 is the mass, in grams, of nitrous acid (HNO_2) corresponding to 1 ml of exactly 0,1 N potassium permanganate solution.

7.2 Content of nitrous compounds less than 0,01 % (m/m)

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

$$\frac{[(V_0 + V_1) - V_2] \times 0{,}000\,235 \times 100}{m}$$

$$= \frac{0{,}023\,5\,[(V_0 + V_1) - V_2]}{m}$$

where

V_0 is the volume, in millilitres, of the potassium permanganate solution (4.3) added at the beginning;

V_1 is the volume, in millilitres, of the potassium permanganate solution (4.3) used for the first titration;

V_2 is the volume, in millilitres, of the potassium permanganate solution (4.3) used for the second titration;

m is the mass, in grams, of the test portion (6.3.1);

0,000 235 is the mass, in grams, of nitrous acid (HNO_2) corresponding to 1 ml of exactly 0,01 N potassium permanganate solution.

NOTES

1 If the concentrations of the standard volumetric solutions used are not exactly as specified in the list of reagents, an appropriate correction should be made.

2 If it is desired to express the content of nitrous compounds as N_2O_4 instead of as HNO_2, the values in the formulae become 0,004 6 instead of 0,002 35 and 0,000 46 instead of 0,000 235.

8 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard or regarded as optional.

ANNEX

ISO PUBLICATIONS RELATING TO NITRIC ACID FOR INDUSTRIAL USE

ISO 1980 – Determination of total acidity – Titrimetric method.

ISO 1981 – Determination of nitrous compounds – Titrimetric method.

ISO/R 1982 – Determination of iron content – 2,2′-Bipyridyl photometric method.

ISO 1983 – Determination of sulphated ash – Gravimetric method.

ISO 2990 – Evaluation of the nitric acid concentration by measurement of density.

ISO 2991 – Determination of ammoniacal nitrogen content – Spectrophotometric method.

ISO 3328 – Determination of sulphate content – Method by reduction and titrimetry.

ISO 3693 – Determination of chloride ions content – Potentiometric method.

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