
INTERNATIONAL STANDARD



3328

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

Nitric acid for industrial use — Determination of sulphate content — Method by reduction and titrimetry

Acide nitrique à usage industriel — Dosage des sulfates — Méthode par réduction et titrimétrie

First edition — 1975-07-15

iTeh STANDARD PREVIEW
(standards.iteh.ai)

[ISO 3328:1975](#)

<https://standards.iteh.ai/catalog/standards/sist/f77d1832-72ac-4a54-99b8-3eb3b1110053/iso-3328-1975>

UDC 661.561 : 546.226 : 543.243

Ref. No. ISO 3328-1975 (E)

Descriptors : nitric acid, chemical analysis, determination of content, sulphates, volumetric analysis.

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.

International Standard ISO 3328 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in February 1974.

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

Austria	Hungary	South Africa, Rep. of
Belgium	India	Spain
Bulgaria	Ireland	Switzerland
Chile	Israel	Turkey
Czechoslovakia	Italy	United Kingdom
Egypt, Arab Rep. of	Netherlands	U.S.S.R.
France	New Zealand	Yugoslavia
Germany	Poland	

No Member Body expressed disapproval of the document.

Nitric acid for industrial use – Determination of sulphate content – Method by reduction and titrimetry

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method, by reduction and titrimetry, for the determination of the sulphate content of nitric acid for industrial use. The method is applicable to products of which the sulphate content, expressed as SO_4 , is equal to or greater than 0,000 1 % (*m/m*).

2 PRINCIPLE

Evaporation of a test portion to dryness in the presence of sodium carbonate.

Reduction of the sulphate to sulphide by a mixture of hydriodic acid and hypophosphorous acid.

Absorption of the hydrogen sulphide evolved in a mixture of acetone and sodium hydroxide solution. Titration with a standard volumetric mercury(II) acetate solution in the presence of dithizone as 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 Hydriodic acid, ρ approximately 1,71 g/ml, about 57 % (*m/m*) solution.

3.2 Hypophosphorous acid [$\text{H}(\text{H}_2\text{PO}_2)$], ρ approximately 1,21 g/ml, about 50 % (*m/m*) solution.

3.3 Hydrochloric acid, ρ approximately 1,19 g/ml, about 38 % (*m/m*) solution.

3.4 Reduction solution.

Into a 1 000 ml flask, fitted with a ground glass socket capable of accepting a reflux condenser, introduce, in the following order, under a current of nitrogen (3.11), 100 ml of the hydriodic acid solution (3.1), 25 ml of the hypophosphorous acid solution (3.2) and 100 ml of the hydrochloric acid solution (3.3). Fit the reflux condenser to the flask and, while bubbling a gentle current of nitrogen through the mixture, boil under reflux for 4 h.

Then cool to room temperature, maintaining the current of nitrogen.

Store the solution in a dark glass flask, previously purged with nitrogen, fitted with a ground glass stopper.

3.5 Sodium sulphate, 0,001 M standard reference solution.

Weigh, to the nearest 0,000 1 g, 0,142 0 g of anhydrous sodium sulphate, previously dried at 110 °C and cooled in a desiccator. Introduce into a 1 000 ml one-mark volumetric flask, dissolve in water, dilute to the mark and mix.

1 ml of this solution corresponds to 96 μg of SO_4 .

3.6 Mercury(II) acetate, 0,001 M standard volumetric solution.

Weigh, to the nearest 0,000 1 g, 0,318 7 g of mercury(II) acetate [$\text{Hg}(\text{CH}_3\text{COO})_2$]. Introduce into a 1 000 ml one-mark volumetric flask, dissolve in water, dilute to the mark and mix.

Alternatively, 0,001 M standard volumetric mercury(II) nitrate solution may be used.

3.6.1 Mercury(II) nitrate, 0,001 M standard volumetric solution. Weigh $10,85 \pm 0,01$ g of mercury(II) oxide (HgO), place in a beaker of suitable capacity (for example 100 ml) and dissolve in 10 ml of nitric acid solution, ρ approximately 1,40 g/ml, about 68 % (*m/m*) solution.

Dilute the solution, transfer it quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix. (The solution thus obtained is 0,05 M, corresponding to 0,1 N.)

Take 20,00 ml of this solution, introduce into a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

Prepare this solution just before use.

NOTES

1 In most laboratories, an exactly 0,1 N standard volumetric mercury(II) nitrate solution (corresponding to a 0,05 M solution) will be available, this solution being commonly used in the mercurimetric determination of chlorides.

2 The strengths of solutions 3.6 and 3.6.1 prepared as described above are sufficiently exact, taking into consideration the small quantities of sulphates to be determined. Standardization is therefore unnecessary.

3.7 Acetone.

3.8 Dithizone, 0,5 g/l solution in acetone.

3.9 Sodium hydroxide, approximately 1 N solution.

3.10 Sodium carbonate, anhydrous.

3.11 Nitrogen.

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Microburette, graduated in 0,01 ml.

4.2 Apparatus for reduction and distillation (see figure for a typical example), of which all the components are fitted together by means of ground glass joints.

5 PROCEDURE

5.1 Test portion and preparation of the test solution

Weigh, to the nearest 0,000 1 g, a mass of the test sample containing between 0,5 and 2 mg of SO₄.

Place in a beaker of convenient capacity, add 20 to 30 mg of the sodium carbonate (3.10) and evaporate to dryness in a fume-cupboard. Dissolve the residue in 10,00 ml of water, slightly acidified with the hydrochloric acid solution (3.3).

Prepare this solution at the time of use.

5.2 Check test

The purpose of this test is to check the gas-tightness and functioning of the apparatus (reduction of sulphur compounds and quantitative recovery of the hydrogen sulphide liberated).

Introduce 5 ml of the sodium hydroxide solution (3.9), 5 ml of the acetone (3.7) and 0,1 ml of the dithizone solution (3.8) into the test tube of the apparatus (4.2).

Mix and add, drop by drop, the mercury(II) acetate solution (3.6) till the colour changes from yellow to pink.

Introduce 2,00 ml of the standard reference sodium sulphate solution (3.5) into the clean and dry reduction flask. Fit the components of the apparatus (4.2) together as indicated in the figure, taking care to smear the ground joints lightly with a silicone grease and to tighten them with suitable spring-clips in order to ensure perfect gas-tightness. Pass a gentle flow of nitrogen (3.11) through the inlet tube (2 to 3 bubbles per second). Then run 15 ml of the reduction solution (3.4) into the reduction flask through the dropping funnel, under a slight pressure of nitrogen.

Maintaining the current of nitrogen through the inlet tube, immerse the reduction flask partially (about 70 mm) in a boiling water bath for at least 30 min. Then allow the nitrogen to flow through the dropping funnel for 5 min.

The hydrogen sulphide liberated by the reaction is absorbed in the solution contained in the test tube which becomes yellow.

NOTE — Development of a blue-green coloration indicates that a large quantity of hydrochloric acid has been entrained and the test must be repeated.

Disconnect the apparatus (4.2) and wash the connection tube with several millilitres of a mixture of equal volumes of water and the acetone (3.7), collecting the washings in the test tube. Titrate the solution obtained with the standard volumetric mercury(II) acetate solution (3.6), contained in the microburette (4.1), until a new pink coloration is obtained.

The result of the check test is considered satisfactory if the volume of the standard volumetric mercury(II) acetate solution (3.6) used for the titration is between 1,90 and 2,10 ml, after subtraction of the blank test result.

Otherwise, check the apparatus for leaks.

5.3 Determination

Place 1,00 ml of the test solution (5.1) in the reduction flask of the apparatus and connect the different parts of the apparatus (4.2). Introduce 5 ml of the sodium hydroxide solution (3.9), 5 ml of the acetone (3.7) and 0,1 ml of the dithizone solution (3.8) into the test tube. Mix and add, drop by drop, the standard volumetric mercury(II) acetate solution (3.6) until the colour changes from yellow to pink.

Pass a current of nitrogen through the apparatus and run in through the dropping funnel, 15 ml of the reduction solution (3.4), under a slight pressure of nitrogen.

Continue as specified in 5.2 for the check test starting from the fifth paragraph.

5.4 Blank test

Carry out a blank test under the same conditions as the determination (5.3) but using 1,00 ml of water instead of the 1,00 ml of the test solution (5.1).

6 EXPRESSION OF RESULTS

The sulphate content, expressed as a percentage by mass of sulphate (SO₄), is given by the formula

$$\frac{(V_1 - V_0) \times 96 \times 10 \times 100}{1\ 000\ 000\ m_0} = \frac{(V_1 - V_0) \times 0,096}{m_0}$$

where

V₀ is the volume, in millilitres, of the standard volumetric mercury(II) acetate solution (3.6) used for the blank test;

V₁ is the volume, in millilitres, of the standard volumetric mercury(II) acetate solution (3.6) used for the determination;

m_0 is the mass, in grams, of the test portion (5.1);

96 is the mass, in micrograms, of SO_4 corresponding to 1 ml of the standard volumetric mercury(II) acetate solution (3.6).

7 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.

Dimensions in millimetres

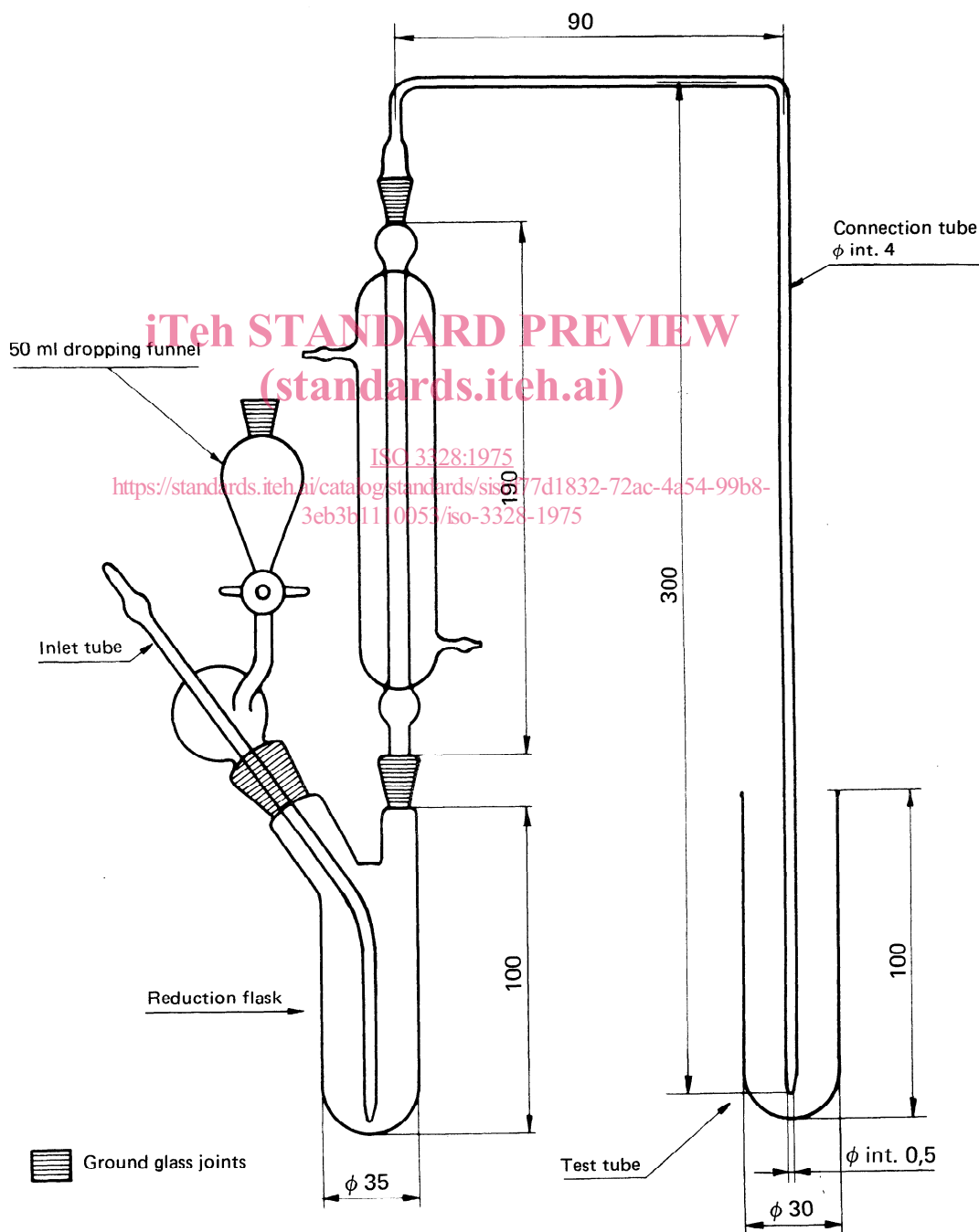


FIGURE – Typical apparatus for reduction and distillation

ANNEX

ISO PUBLICATIONS RELATING TO NITRIC ACID FOR INDUSTRIAL USE

- ISO/R 1980 – Determination of total acidity – Volumetric method.
- ISO/R 1981 – Determination of nitrous compounds – Volumetric method.
- ISO/R 1982 – Determination of iron content – 2,2'-bipyridyl photometric method.
- ISO/R 1983 – Determination of sulphated residue on ignition – 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 chlorides content – Potentiometric method.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

[ISO 3328:1975](https://standards.iteh.ai/catalog/standards/sist/f77d1832-72ac-4a54-99b8-3eb3b1110053/iso-3328-1975)

<https://standards.iteh.ai/catalog/standards/sist/f77d1832-72ac-4a54-99b8-3eb3b1110053/iso-3328-1975>

iTeh STANDARD PREVIEW
(standards.iteh.ai)

This page intentionally left blank

[ISO 3328:1975](#)

<https://standards.iteh.ai/catalog/standards/sist/f77d1832-72ac-4a54-99b8-3eb3b1110053/iso-3328-1975>

iTeh STANDARD PREVIEW
(standards.iteh.ai)

This page intentionally left blank

[ISO 3328:1975](#)

<https://standards.iteh.ai/catalog/standards/sist/f77d1832-72ac-4a54-99b8-3eb3b1110053/iso-3328-1975>