INTERNATIONAL STANDARD 1980

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

Nitric acid for industrial use — Determination of total acidity — Titrimetric method

Acide nitrique à usage industriel — Détermination de l'acidité totale — Méthode titrimétrique

First edition — 1977-11-15

UDC 661.56 : 543.241.5 **Ref. No. ISO 1980-1977 (E)**

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

Price based on 2 pages

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 1980-1971 and found it technically suitable for transformation. International Standard ISO 1980 therefore replaces ISO Recommendation R 1980-1971, to which it is technically identical.

ISO Recommendation R 1980 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

1980-77 4851903 0008407 6

Nitric acid for industrial use — Determination of total acidity — Titrimetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a titrimetric method for the determination of the total acidity of nitric acid for industrial use, conventionally expressed as HNO_3.

2 PRINCIPLE

Addition to a test portion of an excess of a standard volumetric sodium hydroxide solution, and back-titration with a standard volumetric sulphuric acid solution in the presence of an indicator.

3 REAGENTS

During the analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity, neutral to the indicator (3.3).

3.1 Sodium hydroxide, 1 N standard volumetric solution.

3.2 Sulphuric acid, 1 N standard volumetric solution.

3.3 Indicator, having an end point within the pH range between 3,2 and 4,4; for example :

3.3.1 Methyl orange, 0,5 g/l solution.

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Flask, capacity approximately 500 ml, with neck of diameter about 30 mm, with ground glass stopper.

4.2 Spherical glass ampoule, of suitable shape and capacity, for example about 20 mm in diameter, having one capillary end of length about 50 mm (see the example shown in the figure).

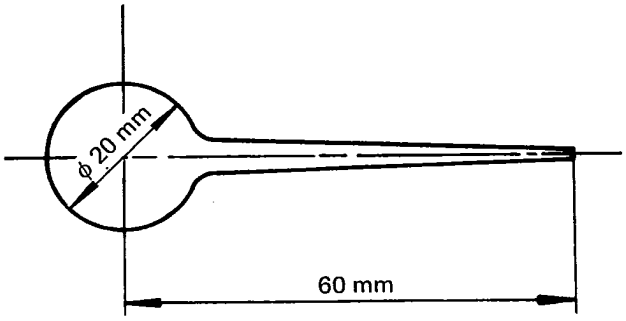

FIGURE — **Spherical glass ampoule**

4.3 Conical flask, capacity 500 ml, with ground glass stopper.

5 PROCEDURE

5.1 Test portion

Nearly fill the flask (4.1) with the test sample. Slightly heat in a flame the bulb of the glass ampoule (4.2), previously weighed to the nearest 0,000 1 g.

Immerse the capillary end of the ampoule into the flask containing the test sample and ensure that during cooling the bulb is almost completely filled (4 ml approximately) for diluted acid and half-filled (2 ml approximately) for concentrated acid.

Withdraw the ampoule and carefully wipe the capillary end with filter paper.

Seal the capillary end in an oxidizing flame, **without loss of glass.** Remove from the flame and allow to cool. Wash the capillary and wipe carefully with filter paper.

Weigh the ampoule to the nearest 0,000 1 g and calculate, by difference, the mass of the test portion.

5.2 Determination

Carefully place the ampoule containing the test portion (5.1) in the conical flask (4.3) containing 100 ml of cold water and 50,0 ml of the sodium hydroxide solution (3.1).

Stopper the flask and, while cooling, shake to break the ampoule containing the test portion.

Keep cooling and shaking until the vapours are completely absorbed.

Remove the stopper and rinse it with water, collecting the washings in the same flask. By means of a glass rod, grind the fragments of the ampoule and in particular the capillary, which may have remained intact in spite of shaking.

Withdraw the glass rod and wash it with water, collecting the washings in the same flask.

Add 2 drops of the indicator solution (3.3) and titrate the excess of sodium hydroxide solution to the end point of the indicator with the sulphuric acid solution (3.2).

6 EXPRESSION OF RESULTS

The total acidity, expressed as a percentage by mass of nitric acid (HNO_3), is given by the formula

$$\frac{(50 - V) \times 0{,}063\,0 \times 100}{m}$$

$$= \frac{6{,}3\,(50 - V)}{m}$$

where

V is the volume, in millilitres, of the sulphuric acid solution (3.2) used for the titration;

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

50 is the volume, in millilitres, of the sodium hydroxide solution (3.1) added;

0,063 0 is the mass, in grams, of nitric acid corresponding to 1 ml of exactly 1 N sodium hydroxide solution.

NOTE — 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.

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.

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.