# INTERNATIONAL STANDARD



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# Nitric acid for industrial use — Determination of sulphated ash — Gravimetric method

Acide nitrique à usage industriel — Dosage du résidu fixe sulfaté — Méthode gravimétrique

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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 1983-1971 and found it technically suitable for transformation. International Standard ISO 1983 therefore replaces ISO Recommendation R 1983-1971, to which it is technically identical.

ISO Recommendation R 1983 had been approved by the member bodies of the following countries:

Australia India Austria Iran Belgium Ireland Chile Israel Czechoslovakia Italy Egypt, Arab Rep. of

Netherlands

France Germany Peru Greece Poland

Thailand Turkey New Zealand United Kingdom

> U.S.A. U.S.S.R.

Portugal

Romania

Switzerland

South Africa, Rep. of

No member body had expressed disapproval of the Recommendation.

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

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# Nitric acid for industrial use — Determination of sulphated ash — Gravimetric method

### 1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a gravimetric method for the determination of sulphated ash of nitric acid for industrial use.

#### 2 PRINCIPLE

Evaporation of a test portion; conversion of salts to sulphates by treatment with sulphuric acid; ignition at  $800\pm25\,^{\circ}\text{C}$  and weighing.

#### 3 REAGENT

During the analysis, use only reagents of recognized analytical grade.

**3.1 Sulphuric acid,**  $\rho$  approximately 1,84 g/ml, about 96 % (m/m) solution or approximately 36 N.

#### 4 APPARATUS

Ordinary laboratory apparatus and

- 4.1 Platinum dish, of capacity approximately 200 ml, flat-bottomed.
- **4.2 Electric furnace,** capable of being controlled at  $800 \pm 25$  °C.

#### **5 PROCEDURE**

## 5.1 Test portion

In the platinum dish (4.1), previously ignited at  $800 \pm 25$  °C, cooled in a desiccator and weighed to the nearest 0,000 1 g, weigh, to the nearest 0,01 g, approximately 100 g of the test sample.

#### 5.2 Determination

Evaporate most of the test portion (5.1) (the final volume should amount to about 5 to 10 ml) by carefully heating the dish containing the test portion (first on a boiling water bath and then on a sand bath, for example). Then allow to cool to ambient temperature.

Add to the dish 1 ml of the sulphuric acid solution (3.1) and heat to dryness.

Place the dish containing the residue in the electric furnace (4.2), controlled at  $800 \pm 25$  °C, and keep at this temperature for about 15 min.

Remove the dish from the furnace, place in a desiccator and, after cooling to ambient temperature, weigh to the nearest 0,000 1 g.

#### **6 EXPRESSION OF RESULTS**

The sulphated ash is given, as a percentage by mass, by the formula

$$\frac{m_1 \times 100}{m_0}$$

where

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

 $m_1$  is the mass, in grams, of the residue weighed.

#### 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.
- ${\sf ISO~2991-Determination~of~ammoniacal~nitrogen~content-Spectrophotometric~method}.$
- ISO 3328 Determination of sulphate content Method by reduction and titrimetry.
- ISO 3693 Determination of chloride ions content Potentiometric method.