# INTERNATIONAL STANDARD



1616

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# Glycerines for industrial use — Determination of sulphated ash — Gravimetric method

Glycérines à usage industriel – Détermination des cendres sulfatées – 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 now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 47 has reviewed ISO Recommendation R 1616 and found it technically suitable for transformation. International Standard ISO 1616 therefore replaces ISO Recommendation R 1616-1970 to which it is technically identical.

ISO Recommendation R 1616 was approved by the Member Bodies of the following countries:

Austria Hungary
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Egypt, Arab Rep. of Korea, Rep. of France Netherlands Germany New Zealand Greece Portugal

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No Member Body expressed disapproval of the Recommendation.

No Member Body disapproved the transformation of ISO/R 1616 into an International Standard.

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# Glycerines for industrial use — Determination of sulphated ash — Gravimetric method

# 1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a gravimetric method for determining sulphated ash deriving from glycerines for industrial use.

The method is applicable to products yielding a quantity of sulphated ash less than or equal to 0.5 % (m/m).

#### 2 REFERENCES

ISO 1614, Glycerines for industrial use — Samples and test methods — General.

ISO 2096, Glycerines for industrial use — Methods of sampling.

#### 3 PRINCIPLE

Combustion of a test portion and calcination of the residue in the presence of sulphuric acid, between 800 and 850 °C. Weighing of the sulphated ash thus obtained.

## 4 REAGENT

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

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

### 5 APPARATUS

Ordinary laboratory apparatus and

- **5.1 Platinum or porcelain dish**, diameter 70 to 90 mm and height 25 to 50 mm.
- **5.2** Electric furnace, capable of being controlled at 800 to  $850\,^{\circ}$ C.

# 6 PROCEDURE

## 6.1 Test portion

Heat the dish (5.1) for several minutes in the furnace (5.2) controlled between 800 and  $850\,^{\circ}$ C, allow it to cool to ambient temperature in a desiccator and weigh to the nearest 0,001 g.

Weigh  $50 \pm 0.1$  g of the test sample to the nearest 0.01 g, in the previously weighed dish (see ISO 1614).

# 6.2 Determination

Gently heat the dish containing the test portion (6.1) over a small flame, avoiding sputtering, and ignite the vapour. Stop the heating and allow to burn until a carbonaceous mass is obtained.

After cooling, moisten the residue with a few drops of the sulphuric acid solution (4.1) and remove the excess acid by heating until the white fumes disappear and combustible material is burned away. Repeat this operation and then place the dish for 5 min in the furnace (5.2) controlled between 800 and 850 °C.

Allow to cool in a desiccator to ambient temperature and weigh to the nearest 0,001 g.

## **7 EXPRESSION OF RESULTS**

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

$$(m_2 - m_1) \times \frac{100}{m_0}$$

where

 $m_0$  is the mass, in grams, of the test portion (6.1);

 $m_1$  is the mass, in grams, of the empty dish (5.1);

 $m_2$  is the mass, in grams, of the dish containing the sulphated ash.

#### 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) all operations not included in this International Standard or the International Standards to which reference is made, or regarded as optional.

#### **ANNEX**

# ISO PUBLICATIONS RELATING TO GLYCERINES FOR INDUSTRIAL USE

- ISO 1614 Samples and test methods General.
- ISO 1615 Determination of alkalinity or acidity Titrimetric method.
- ISO 1616 Determination of sulphated ash Gravimetric method.
- ISO 2096 Methods of sampling.
- ISO 2097 Determination of water content Karl Fischer method.
- ISO 2098 Determination of ash Gravimetric method.
- ISO 2099 Determination of density at 20 °C.
- ISO 2464 Calculation of Matter (Organic) Non-Glycerol (MONG).
- ${\sf ISO~2465-Determination~of~arsenic~content-Silver~diethyldithiocarbamate~photometric~method.}$
- ISO 2879 Determination of glycerol content Titrimetric method.