

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 754

ACETIC ANHYDRIDE FOR INDUSTRIAL USE

METHODS OF TEST

1st EDITION
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BRIEF HISTORY

The ISO Recommendation R 754, *Acetic anhydride for industrial use – Methods of test*, was drawn up by Technical Committee ISO/TC 47, *Chemistry*, the Secretariat of which is held by the Ente Nazionale Italiano di Unificazione (UNI).

Work on this question by the Technical Committee began in 1956 and led, in 1962, to the adoption of a Draft ISO Recommendation.

In November 1963, this Draft ISO Recommendation (No. 653) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia	Hungary	Romania
Austria	India	Spain
Belgium	Israel	Switzerland
Chile	Italy	U.A.R.
Colombia	Korea, Rep. of	United Kingdom
Czechoslovakia	Netherlands	U.S.A.
France	Poland	U.S.S.R.
Germany	Portugal	Yugoslavia

Two Member Bodies opposed the approval of the Draft :

Japan
New Zealand

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in June 1968, to accept it as an ISO RECOMMENDATION.

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* Not included in this ISO Recommendation as these determinations are still under study.

ACETIC ANHYDRIDE FOR INDUSTRIAL USE

METHODS OF TEST

1. SCOPE

This ISO Recommendation describes methods of test for acetic anhydride for industrial use, and is divided into two parts, namely :

Part I – Methods of test for general use

Part II – Methods of test for special purposes

2. SAMPLE

Take a volume of sample that is sufficient for all analyses to be carried out so that it is representative of the bulk.

Place the sample in a clean, dry and air-tight glass stoppered bottle of such a size that it is nearly filled by the sample.

When it is necessary to seal the container, care should be taken to avoid the risk of contaminating the sample in any way.

PART I – METHODS OF TEST FOR GENERAL USE

3. DETERMINATION OF DISTILLATION YIELD

3.1 Use the method described in ISO Recommendation R 918, *Test method for distillation (Distillation yield and distillation range)*.

3.2 The following details, not given in the above ISO Recommendation, apply to acetic anhydride :

Thermometer, (see clause 3.2 of ISO Recommendation R 918) should also comply with the following requirements :

Thermometer range	Graduations	Maximum error	Values in degrees Celsius
			Maximum error in an interval of 10 degrees Celsius
98 to 152	0.2	0.4	0.4

A correction of $0.050(p - 760)$ degrees Celsius where p is the barometric pressure in standard millimetres of mercury, is added to the specified distillation temperatures (see clause 5.1 of ISO Recommendation R 918). The interval before the first drop of distillate falls from the end of the condenser should be 12 to 17 minutes (see clause 6.1 of ISO Recommendation R 918).

4. DETERMINATION OF ASH CONTENT

4.1 Apparatus

Ordinary laboratory apparatus.

4.2 Procedure

4.2.1 Evaporate to dryness a known mass (between 10 and 100 g depending on the ash content expected) of the sample in a weighed platinum or silica dish of capacity 50 ml, and gently ignite the residue until all carbonaceous matter has disappeared.

4.2.2 Cool the dish and its contents to atmospheric temperature in a desiccator containing anhydrous calcium chloride, and weigh.

4.3 Expression of results

$$\text{Ash, \% (m/m)} = \frac{100 \times M_1}{M_2}$$

where

M_1 is the mass, in grammes, of the residue,

M_2 is the mass, in grammes, of the test portion.

5. LIMIT TEST FOR INORGANIC CHLORIDES

This method is applicable when the chloride content, expressed as Cl, is not greater than 0.05 and not less than 0.0005 %. If the chloride content lies outside that range, the mass of test portion taken (5.4.1) should be reduced or increased and an appropriate adjustment made to the expression of $\frac{0.05}{x}$ ml in clause 5.4.4.

5.1 Principle

Comparison of the turbidity, obtained by the addition of silver nitrate to a solution prepared from the sample in presence of nitric acid, with that similarly obtained from a chloride solution of known concentration.

5.2 Reagents

Distilled water or water of equivalent purity should be used in the test. All reagents and filter paper should be chloride free.

5.2.1 *Nitric acid*, approximately 5 N solution.

5.2.2 *Standard chloride solution* (0.1 mg Cl/ml). Dilute 28.2 ml of 0.1 N hydrochloric acid solution to 1000 ml with water.

5.2.3 *Silver nitrate*, 50 g/l solution.

5.3 Apparatus

Ordinary laboratory apparatus.

5.4 Procedure

5.4.1 Weigh 50 ± 0.5 g of the test sample, dissolve in water, transfer to a 250 ml one-mark volumetric flask, dilute to the mark with water and mix.