



**Acetic acid for industrial use — Methods of test —
Part 4 : Determination of acetaldehyde monomer
content — Titrimetric method**

Acide acétique à usage industriel — Méthodes d'essai — Partie 4 : Dosage de l'acétaldéhyde monomère — Méthode titrimétrique

First edition — 1981-10-15

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UDC 661.731 : 543

Ref. No. ISO 753/4-1981 (E)

Descriptors : industrial products, acetic acid, tests, determination of content, acetaldehyde, titration.

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 753/4 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in March 1980.

It has been approved by the member bodies of the following countries :

Australia	France	Poland
Austria	Germany, F. R.	Romania
Belgium	Hungary	South Africa, Rep. of
Brazil	India	Switzerland
China	Italy	Thailand
Czechoslovakia	Korea, Rep. of	United Kingdom
Egypt, Arab Rep. of	Netherlands	USSR

No member body expressed disapproval of the document.

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

International Standards ISO 753/1 to ISO 753/11 cancel and replace ISO Recommendation R 753-1968, of which they constitute a technical revision.

Acetic acid for industrial use — Methods of test — Part 4 : Determination of acetaldehyde monomer content — Titrimetric method

1 Scope and field of application

This part of ISO 753 specifies a titrimetric method for the determination of the acetaldehyde monomer content of acetic acid for industrial use.

The method is applicable to products having acetaldehyde monomer contents equal to or greater than 0,01 % (*m/m*).

This document should be read in conjunction with ISO 753/1 (see the annex).

NOTE — For the determination of the total acetaldehyde content, including polymers, see ISO 753/5.

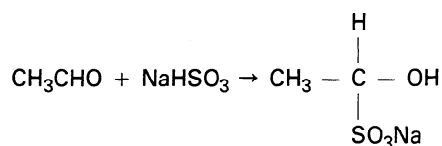
2 Reference

ISO/R 385, *Burettes*.

3 Principle

Reaction of the acetaldehyde present in a test portion with an excess of sodium hydrogen sulphite solution, and iodometric titration of the residual sodium hydrogen sulphite.

4 Reaction



5 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

5.1 Sodium hydrogen sulphite, approximately 18,2 g/l solution.

Dissolve 1,66 g of disodium disulphite ($\text{Na}_2\text{S}_2\text{O}_5$) in water and dilute to 100 ml.

Prepare this solution at the time of use.

5.2 Iodine, standard volumetric solution, $c(1/2 \text{I}_2) = 0,02 \text{ mol/l}$.

5.3 Sodium thiosulphate, standard volumetric solution, $c(\text{Na}_2\text{S}_2\text{O}_3) = 0,02 \text{ mol/l}$.

5.4 Starch solution.

Triturate 1,0 g of soluble starch with 5 ml of water and, while stirring, pour the mixture into 100 ml of boiling water. Boil for a few minutes and allow to cool.

Discard the solution after 2 weeks.

6 Apparatus

Ordinary laboratory apparatus and

6.1 Weighing pipette, of capacity 20 ml.

6.2 Two conical flasks, of capacity 250 ml, fitted with ground glass stoppers.

6.3 Burettes, of capacity 10 ml, complying with the requirements of ISO/R 385, class A.

6.4 Ice-water bath.

7 Procedure

7.1 Test portion

Using the weighing pipette (6.1), weigh, to the nearest 0,01 g, about 10 ml of the laboratory sample and transfer to a 50 ml one-mark volumetric flask containing 10 ml of water.

7.2 Blank test

Carry out a blank test at the same time as the determination, following the same procedure and using the same quantities of all the reagents [except the sodium thiosulphate solution (5.3)] as used for the determination, but omitting the test portion.

7.3 Determination

7.3.1 Preparation of test solution

Add to the volumetric flask containing the test portion (7.1) 5,0 ml of the sodium hydrogen sulphite solution (5.1) from one of the burettes (6.3). Dilute to the mark, mix and allow to stand for about 30 min.

7.3.2 Titration

At the same time as preparing the test solution, transfer 50,0 ml of the iodine solution (5.2) to each of the conical flasks (6.2) and allow them to stand in the ice-water bath (6.4).

At the end of the 30 min period, transfer 20,0 ml of the test solution (7.3.1) to one of the conical flasks and 20 ml of the blank test solution (7.2) to the other. Titrate the two solutions with the sodium thiosulphate solution (5.3) from one of the burettes (6.3) until each solution becomes pale yellow. Add 0,5 ml of the starch solution (5.4) and continue the titration until the blue colour is discharged.

8 Expression of results

The acetaldehyde (CH_3CHO) monomer content, expressed as a percentage by mass, is given by the formula

$$0,000\ 44 (V_1 - V_0) \times \frac{50}{20} \times \frac{100}{m}$$

$$= \frac{0,110 (V_1 - V_0)}{m}$$

where

V_0 is the volume, in millilitres, of the sodium thiosulphate solution (5.3) used for the blank test;

V_1 is the volume, in millilitres, of the sodium thiosulphate solution (5.3) used for the determination;

m is the mass, in grams, of the test portion (7.1);

0,000 44 is the mass, in grams, of acetaldehyde corresponding to 1 ml of sodium thiosulphate solution, $c(\text{Na}_2\text{S}_2\text{O}_3) = 0,020\ 0\ \text{mol/l}$.

NOTE — If the concentrations of the standard volumetric solutions are not exactly as specified in the list of reagents, an appropriate correction should be made.

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Annex

ISO publications relating to acetic acid for industrial use

ISO 753/1 — General.

ISO 753/2 — Determination of acetic acid content — Titrimetric method.

ISO 753/3 — Determination of low formic acid contents — Gravimetric method.

ISO 753/4 — Determination of acetaldehyde monomer content — Titrimetric method.

ISO 753/5 — Determination of total acetaldehyde content — Titrimetric method.

ISO 753/6 — Determination of permanganate index.

ISO 753/7 — Determination of dichromate index.

ISO 753/8 — Visual limit test for inorganic chlorides.

ISO 753/9 — Visual limit test for inorganic sulphates.

ISO 753/10 — Visual limit test for heavy metals (including iron).

ISO 753/11 — Determination of iron content — 1,10-Phenanthroline photometric method.