International Standard



753/5

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION MEXALYHAPODHAR OPFAHUSALUUR NO CTAHDAPTUSALUUMOORGANISATION INTERNATIONALE DE NORMALISATION

Acetic acid for industrial use - Methods of test -Part 5 : Determination of total acetaldehyde content — **Titrimetric method**

Acide acétique à usage industriel — Méthodes d'essai — Partie 5 : Dosage de l'acétaldéhyde total — Méthode titrimétrique

First edition - 1981-10-15

UDC 661.731:543

Ref. No. ISO 753/5-1981 (E)

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

Foreword

3.50%

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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 753/5 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 Austria Belgium Brazil China Czechoslovakia Egypt, Arab Rep. of France

Germany, F. R. Hungary India Italy Korea, Rep. of Netherlands Poland Romania

South Africa, Rep. of Switzerland Thailand United Kingdom USSR

No member body expressed disapproval of the document.

Pure smmen-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.

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Acetic acid for industrial use — Methods of test — Part 5 : Determination of total acetaldehyde content — Titrimetric method

1 Scope and field of application

This part of ISO 753 specifies a titrimetric method for the determination of the total acetaldehyde content (polymerized and monomeric acetaldehyde) of acetic acid for industrial use.

The method is applicable to products having total acetaldehyde contents equal to or greater than 0,004 % (m/m).

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

NOTE — For the determination of the acetaldehyde monomer content, see ISO 753/4.

2 Reference

ISO/R 385, Burettes.

3 Principle

Heating a test portion in an acid medium to depolymerize any paraldehyde present and to entrain, by distillation, both regenerated acetaldehyde and the monomeric acetaldehyde originally present. Reaction of the acetaldehyde in the distillate with an excess of sodium hydrogen sulphite solution. lodometric titration of the residual sodium hydrogen sulphite solution.

4 Reaction

$$H$$

CH₃CHO + NaHSO₃ → CH₃ - C - OH
|
SO₃Na

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, 12,6 g/l solution.

Dissolve 1,15 g of disodium disulphite $(Na_2S_2O_5)$ in water and dilute to 100 ml.

Prepare this solution at the time of use.

5.2 Phosphoric acid, ρ approximately 1,70 g/ml, about 85 % (m/m) solution.

5.3 Iodine, standard volumetric solution, $c(1/2 \mid_2) = 0,1 \text{ mol/l}.$

5.4 Sodium thiosulphate, standard volumetric solution, $c(Na_2S_2O_3) = 0,1 \text{ mol/l}.$

5.5 Starch solution.

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

Discard the solution after 2 weeks.

6 Apparatus

Ordinary laboratory apparatus and

6.1 Weighing pipette, of capacity 20 ml.

6.2 Distillation apparatus, with ground glass joints, assembled as shown in the figure and consisting of the following items.

6.2.1 Distillation flask, of capacity 250 ml, of borosilicate glass.

6.2.2 Splash-head adapter, fitted with a recovery bend.

6.2.3 Water condenser, carrying a

6.2.4 Receiver adapter.

6.3 Conical flask, of capacity 200 ml, graduated at 50 and 100 ml.

6.4 Burette, of capacity 50 ml, complying with the requirements of ISO/R 385, class A.

6.5 Ice-water bath.