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International Standard 753/7

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Acetic acid for industrial use — Methods of test — Part 7 : Determination of dichromate index

Acide acétique à usage industriel — Méthodes d'essai — Partie 7 : Détermination de l'indice de dichromate

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Foreword

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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 753/7 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.

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Acetic acid for industrial use — Methods of test — Part 7 : Determination of dichromate index

1 Scope and field of application

This part of ISO 753 specifies a method for the determination of the dichromate index of acetic acid for industrial use.

The method is applicable to products having dichromate indexes equal to or greater than 0,04 ml.

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

2 Reference

ISO/R 385, *Burettes.*

3 Definition

For the purposes of this International Standard, the following definition applies.

dichromate index : The number of millilitres of standard volumetric potassium dichromate solution, $c(1/6\ K_2Cr_2O_7) = 0,1$ mol/l, that are reduced by 1,0 ml of the laboratory sample under the conditions specified.

4 Principle

Heating a test portion with an excess of potassium dichromate solution in the presence of sulphuric acid. Iodometric titration of the residual potassium dichromate.

5 Reagents

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

5.1 Potassium iodide, 100 g/l solution.

5.2 Potassium dichromate, acidified standard volumetric solution, $c(1/6\ K_2Cr_2O_7) = 0,1$ mol/l.

Weigh, to the nearest 0,000 1 g, 4,903 5 g of potassium dichromate and dissolve in approximately 500 ml of water. Add slowly and carefully, while cooling, 400 ml of sulphuric acid solution, ϱ 1,84 g/ml. Transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, allow to cool to ambient temperature, dilute to the mark with water and mix.

5.3 Sodium thiosulphate, standard volumetric solution, $c(Na_2S_2O_3) = 0,1$ mol/l.

5.4 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

Clean all glassware prior to use by heating with a chromic/sulphuric acid mixture, taking the usual precautions, and then by rinsing first with running water and finally with distilled water.

Ordinary laboratory apparatus and

6.1 Two conical flasks, of capacity 500 ml, of borosilicate glass, fitted with ground glass stoppers.

6.2 Water bath, capable of being controlled at 50 ± 2 °C.

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

7 Procedure

7.1 Test portion

Measure 5,0 ml of the laboratory sample into one of the conical flasks (6.1) containing 50,0 ml of the potassium dichromate solution (5.2).

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7.2 Blank test

Carry out a blank test at the same time as the determination, using the second conical flask (6.1), 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

Loosely stopper the flask containing the test portion (7.1) and heat on the water bath (6.2), controlled at 50 ± 2 °C, for 60 min.

Cool the flask, add 100 ml of water and 10 ml of the potassium iodide solution (5.1). Titrate the mixture with the sodium thiosulphate solution (5.3) from the burette (6.3) until the solution becomes yellowish-green. 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 dichromate index is given by the formula

$$(V_0 - V_1) \times \frac{1}{5}$$

$$= 0{,}2\,(V_0 - V_1)$$

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.

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

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.

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