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Acetic acid for industrial use — Methods of test — Part 9 : Visual limit test for inorganic sulphates

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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 753/9 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 9 : Visual limit test for inorganic sulphates

1 Scope and field of application

This part of ISO 753 specifies a visual limit test for inorganic sulphates in acetic acid for industrial use.

Using a test portion of 100 g, the method is applicable directly to products having inorganic sulphate contents, expressed as SO_4^{2-} , in the range 0,001 to 0,1 % (*m/m*), but this range can be extended by adjusting the mass of the test portion (see 5.1).

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

2 Principle

Visual comparison of the turbidity obtained by adding a barium chloride solution to a solution of a test portion acidified with hydrochloric acid, with that similarly obtained from a sulphate solution of known concentration.

3 Reagents

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

3.1 Sodium carbonate, anhydrous, 53 g/l solution.

3.2 Hydrochloric acid, 36,5 g/l solution.

3.3 Barium chloride dihydrate, 100 g/l solution.

3.4 Sulphate, standard solution corresponding to 0,1 g of SO_4^{2-} per litre.

Transfer 20,8 ml of a standard volumetric sulphuric acid solution, $c(1/2 \text{H}_2\text{SO}_4) = 0,1 \text{ mol/l}$, to a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.

1 ml of this standard solution contains 0,1 mg of SO_4^{2-} .

4 Apparatus

Ordinary laboratory apparatus and

4.1 Filter papers, sulphate-free.

4.2 Two matched Nessler cylinders, of capacity 100 ml.

5 Procedure

5.1 Test portion

If the expected inorganic sulphates content lies within the range 0,001 to 0,1 % (*m/m*), weigh $100 \pm 1 \text{ g}$ of the laboratory sample. If the content is outside this range, weigh an appropriately reduced or increased mass and adjust the volume of the aliquot portion (0,1/*x* ml) taken in 5.4 accordingly.

5.2 Preparation of the test solution

Transfer the test portion (5.1) quantitatively to a porcelain evaporating dish of suitable capacity. Add 0,2 ml of the sodium carbonate solution (3.1) and evaporate to dryness on a boiling water bath in a fume cupboard. Dissolve the residue in water containing 1 ml of the hydrochloric acid solution (3.2), transfer the solution quantitatively to a 250 ml one-mark volumetric flask, dilute to the mark with water and mix.

If the solution is cloudy, filter it through one of the filter papers (4.1) to remove turbidity due to aluminium. Remove any residual turbidity, due to contamination with wax, by extraction with a suitable solvent, for example light petroleum.

5.3 Preparation of standard turbidimetric solution

Transfer 4,0 ml of the standard sulphate solution (3.4) to one of the Nessler cylinders (4.1), dilute to the mark with water, add 2 ml of the hydrochloric acid solution (3.2) and mix.

5.4 Test

For a sample required to contain not more than x % (m/m) of inorganic sulphates, expressed as SO_4^{2-} , transfer to the other Nessler cylinder (4.1) an aliquot portion ($0,1/x$ ml) of the test solution (5.2), dilute to the 100 ml mark with water, add 2 ml of the hydrochloric acid solution (3.2) and mix.

Add to each Nessler cylinder 2 ml of the barium chloride solution (3.3) and mix. Allow the cylinders to stand for 5 min, mix

again and compare the turbidity produced by the aliquot portion of the test solution with that produced by the standard turbidimetric solution (5.3).

6 Expression of results

The inorganic sulphates content does not exceed x % (m/m) of SO_4^{2-} if the turbidity produced by the test solution does not exceed that produced by the standard turbidimetric solution.

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.