International Standard 753/10

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Acetic acid for industrial use — Methods of test — Part 10 : Visual limit test for heavy metals (including iron)

Acide acétique à usage industriel — Méthodes d'essai — Partie 10 : Essai visuel limite de contrôle des métaux lourds (y compris le fer)

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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 753/10 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 10 : Visual limit test for heavy metals (including iron)

1 Scope and field of application

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

Using a test portion of 25 g, the method is applicable directly to products having heavy metal contents, expressed as Pb, in the range 0,000 44 to 0,04 % (m/m), but this range can be extended by adjusting the mass of the test portion (see 5.1).

The method detects only the heavy metals present in non-complex form and is not specific for any one heavy metal.

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

2 Principle

Conversion of heavy metals, such as lead, copper and iron, to their sulphides by treatment with sodium sulphide in ammoniacal solution, and visual comparison of the colour produced with that of a standard lead solution similarly treated.

3 Reagents

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

3.1 Ammonia solution, ϱ 0,88 g/ml, approximately 34,3 % (m/m) solution.

3.2 Sodium sulphide nonahydrate ($Na_2S.9H_2O$), 100 g/l solution.

3.3 Lead, standard solution corresponding to 0,010 g of lead per litre.

Weigh, to the nearest 0,000 1 g, 0,016 0 g of lead nitrate [$Pb(NO_3)_2$], dissolve in water, transfer the solution to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,010 mg of Pb.

Prepare this solution at the time of use.

3.4 Litmus paper (pH range 5 to 8).

4 Apparatus

Ordinary laboratory apparatus and

4.1 Two matched Nessler cylinders, of capacity 50 ml.

5 Procedure

5.1 Test portion

If the expected heavy metals content lies within the range 0,000 44 to 0,04 % (m/m), weigh 25 ± 0,1 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 (0,02/x ml) taken in 5.4 accordingly.

5.2 Preparation of the test solution

Transfer the test portion (5.1) quantitatively to a 250 ml one-mark volumetric flask, dilute to the mark with water and mix.

5.3 Preparation of standard matching solution

Add to 20 ml of water, in one of the Nessler cylinders (4.1), 2,0 ml of the standard lead solution (3.3) and 1 ml of the ammonia solution (3.1). Dilute to the 50 ml mark with water and mix. Then add 0,1 ml (2 drops) of the sodium sulphide solution (3.2) and mix again.

5.4 Test

For a sample required to contain not more than x % (m/m) of heavy metals, expressed as Pb, transfer to the other Nessler cylinder an aliquot portion (0,02/x ml) of the test solution, not exceeding 45 ml. Add the ammonia solution (3.1) until the solution is alkaline to the litmus paper (3.4) (blue colour), dilute to the 50 ml mark with water and mix. Then add 0,1 ml (2 drops) of the sodium sulphide solution (3.2) and mix again.

Compare the depth of colour of this solution with that of the standard matching solution (5.3).

6 Expression of results

The heavy metals content does not exceed x % (m/m), expressed as Pb, if the depth of colour of the test solution does not exceed that of the standard matching solution.