International Standard 753/3

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Acetic acid for industrial use — Methods of test — Part 3: Determination of formic acid content — Iodometric method

Acide acétique à usage industriel — Méthodes d'essai — Partie 3: Dosage de l'acide formique — Méthode iodométrique

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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 753/3 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in August 1982.

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

Australia
Austria
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The member body of the following country expressed disapproval of the document on technical grounds:

USSR

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

Printed in Switzerland

Acetic acid for industrial use — Methods of test — Part 3: Determination of formic acid content — Iodometric method

1 Scope and field of application

This part of ISO 753 specifies an iodometric method for the determination of formic acid content of acetic acid for industrial use.

The method is applicable to products having formic acid contents, between 0,02 and 0,35 % (*m*/*m*).

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

2 Principle

2.1 Determination of total reducing matter

Oxidation of formic acid and other reducing compounds in a test portion with an excess of sodium hypobromite solution. Determination of the excess sodium hypobromite by iodometry.

2.2 Determination of reducing compound other than formic acid

Oxidation of reducing compounds other than formic acid with an excess of potassium bromide-bromate solution in an acid media. Determination of the excess potassium bromide-bromate by iodometry.

Determination of the formic acid content from the difference between the two determinations.

3 Reactions

$HCOOH + NaOBr \rightarrow NaBr + CO_2 + H_2O$

$NaOBr + 2KI + 2HCl \rightarrow 2KCl + NaBr + H_2O + I_2$

$2\ Na_2S_2O_3 + I_2 \rightarrow Na_2S_4O_6 + 2\ NaI$

4 Reagents

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

4.1 Hydrochloric acid, approximately 220 g/l solution.

4.2 Potassium iodide, approximately 250 g/l solution.

4.3 Sodium hypobromite, $c(1/2\ NaOBr) \approx 0,1$ mol/l solution.

In a 1 000 ml one-mark volumetric flask, add 500 ml of water to 100 ml of 80 g/l sodium hydroxide solution, and then carefully add 2,8 ml of bromine. Stir to a complete solution, dilute to the mark with water and mix.

WARNING — Bromine is very toxic and causes severe burns. Prevent inhalation of vapour and contact with skin and eyes.

4.4 Potassium bromide-bromate, $c(1/6\ KBrO_3) \approx 0,1$ mol/l solution.

In a 400 ml beaker, dissolve 10 g of potassium bromide and 2,78 g of potassium bromate with 200 ml of water. Quantitatively transfer the solution into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

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

4.6 Starch solution.

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

Discard the solution after 2 weeks.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Tapered-neck flasks, capacity 500 ml, resistant under vacuum (see the figure).

5.2 Dropping funnel, capacity 100 ml, resistant under vacuum (see the figure) and with tapered bottom joint matching the neck of the flask (5.1).

5.3 Vacuum pump, capable of keeping a residual pressure under 100 mbar*.

5.4 Weighing pipette, of capacity about 10 ml.

6 Procedure

6.1 Determination of total reducing matter

6.1.1 Test portion

Weigh, by difference, to the nearest 0,01 g, approximately 10 g of the laboratory sample, by means of the weighing pipette (5.4).

6.1.2 Blank test

Carry out, following the same procedure as for the determination on the sample (6.1.3), a blank test by using the same amounts of all reagents [except the sodium thiosulfate solution (4.5)] as used for the determination, but replacing the test portion with 10 ml of water.

6.1.3 Determination

Introduce 80 ml of water in a tapered flask (5.1), and place the dropping funnel (5.2) on it. Bring the flask, by means of the vacuum pump (5.3), to a residual pressure sufficient to the intake of at least 200 ml of liquid. Close the stopcock of the funnel, and disconnect the vacuum pump. Introduce, through the funnel, 25,0 ml of the sodium hypobromite solution (4.3), using two times 5 ml of water for rinsing the funnel.

Similarly, introduce the test portion (6.1.1), followed by two times 5 ml of water to rinse the funnel. Mix and allow to stand for about 10 min at room temperature to complete the reaction.

Introduce then, through the funnel, 5 ml of the potassium iodide solution (4.2), followed by 20 ml of the hydrochloric acid solution (4.1), agitating for 30 s. Open the stopcock to release the vacuum, disconnect the funnel, add 50 ml of water to the flask and titrate the liberated iodine with the sodium thiosulfate solution (4.5) to a pale yellow colour. Add about 2 ml of the starch solution (4.6), and complete the titration until the discharge of the blue colour.

6.2 Determination of reducing compounds other than formic acid

6.2.1 Test portion

Weigh, by difference, to the nearest 0,01 g, approximately 10 g of the laboratory sample, by means of the weighing pipette (5.4).

6.2.2 Blank test

Carry out, following the same procedure as for the determination on the sample (6.2.3), a blank test by using the same amounts of all reagents [except the sodium thiosulfate solution (4.5)] as used for the determination, but replacing the test portion with 10 ml of water.

6.2.3 Determination

Introduce in a flask (5.1) 90 ml of water and 25,0 ml of the potassium bromide-bromate solution (4.4). Place the funnel (5.2) on the flask, and bring the apparatus by means of the vacuum pump (5.3), to a residual pressure sufficient to the intake of at least 200 ml of liquid. Close the stopcock of the funnel, and disconnect the vacuum pump. Introduce, through the funnel, the test portion (6.2.1), followed by two times 5 ml of water for rinsing the funnel, and 10 ml of the hydrochloric acid solution (4.1). Mix and allow to stand at room temperature for about 10 min.

Introduce then, through the funnel, 5 ml of the potassium iodide solution (4.2), followed by 50 ml of water. Mix, open the stopcock to release the vacuum, disconnect the funnel and titrate the free iodine so formed with the sodium thiosulfate solution (4.5) to a pale yellow colour. Add about 2 ml of the starch solution (4.6) and complete the titration until the discharge of the blue colour.

7 Expression of results

The content of formic acid, expressed as a percentage by mass, is given by the formula

$$\left(\frac{V_0 - V_1}{m_0} - \frac{V_2 - V_3}{m_1}\right) \times c \times 0{,}023 \times 100$$

where

V_0 is the volume, in millilitres, of the sodium thiosulfate solution (4.5) used for the titration of the blank (6.1.2);

V_1 is the volume, in millilitres, of the sodium thiosulfate solution (4.5) used for the determination (6.1.3);

V_2 is the volume, in millilitres, of the sodium thiosulfate solution (4.5) used for the titration of the blank (6.2.2);

V_3 is the volume, in millilitres, of the sodium thiosulfate solution (4.5) used for the determination (6.2.3);

c is the actual concentration, in moles per litre, of the sodium thiosulfate solution (4.5);

m_0 is the mass, in grams, of the test portion (6.1.1) taken for the determination of total reducing matter;

m_1 is the mass, in grams, of the test portion (6.2.1) taken for the determination of reducing compounds other than formic acid;

0,023 is the mass, in grams, of formic acid corresponding to 1,00 ml of sodium thiosulfate solution, $c(Na_2S_2O_3) = 1{,}000$ mol/l.

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* 100 mbar = 10 kPa

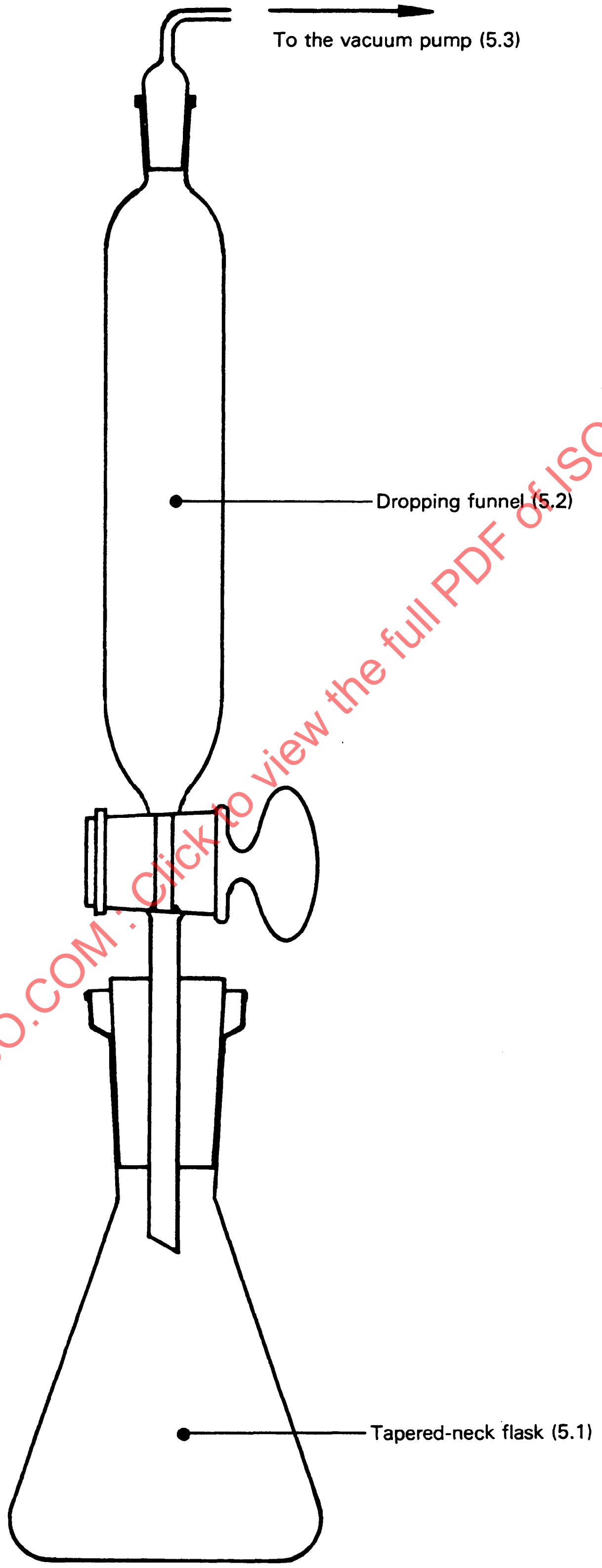

Figure — Apparatus for determination of formic acid content

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 formic acid content — Iodometric 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 sulfates.

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

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