

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 753

ACETIC ACID FOR INDUSTRIAL USE

METHODS OF TEST

1st EDITION

June 1968

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Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

iTeh STANDARD PREVIEW (standards.iteh.ai)

ISO/R 753:1968

<https://standards.iteh.ai/catalog/standards/sist/addd029d-586a-4086-bf15-a66a2be04533/iso-r-753-1968>

BRIEF HISTORY

The ISO Recommendation R 753, *Acetic acid for industrial use – Methods of test*, was drawn up by Technical Committee ISO/TC 47, *Chemistry*, the Secretariat of which is held by the Ente Nazionale Italiano di Unificazione (UNI).

Work on this question by the Technical Committee began in 1956 and led, in 1962, to the adoption of a Draft ISO Recommendation.

In November 1963, this Draft ISO Recommendation (No. 652) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia	Hungary	Romania
Austria	India	Spain
Belgium	Israel	Switzerland
Chile	Italy	U.A.R.
Colombia	Korea, Rep. of	United Kingdom
Czechoslovakia	Netherlands	U.S.A.
France	Poland	U.S.S.R.
Germany	Portugal	Yugoslavia

Two Member Bodies opposed the approval of the Draft :

Japan
New Zealand

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in June 1968, to accept it as an ISO RECOMMENDATION.

CONTENTS

	Page
1. Scope	5
2. Sample	5

PART I – METHODS OF TEST FOR GENERAL USE

3. Determination of the crystallizing point of glacial acetic acid	5
4. Determination of acetic acid content	6
5. Determination of residue on evaporation on a water bath	7
6. Determination of iron content	7
7. Limit test for inorganic chlorides	9
8. Limit test for inorganic sulphates	10
9. Limit test for heavy metals (including iron)	11
10. Determination of formic acid content	11
11. Determination of acetaldehyde content	13

PART II – METHODS OF TEST FOR SPECIAL PURPOSES

12. Determination of arsenic content	14
13. Determination of water content	16
14. Determination of permanganate index	17
15. Determination of total acetaldehyde content	18
16. Determination of total halogen content*	19
17. Determination of total sulphur content*	19
18. Determination of dichromate index	19
19. Determination of mercury	20
20. Test report	21

* Not included in this ISO Recommendation as these determinations are still under study.

ACETIC ACID FOR INDUSTRIAL USE

METHODS OF TEST

1. SCOPE

This ISO Recommendation describes methods of test for acetic acid for industrial use, and is divided into two parts, namely :

Part I – Methods of test for general use.

Part II – Methods of test for special purposes.

2. SAMPLE

In all cases, take the sample of acetic acid in the liquid condition. If it is solidified, melt it completely in a warm room with the temperature not above 30 °C, and thoroughly agitate it by rolling the container or by other suitable means before sampling.

Take a volume of sample that is sufficient for all analyses to be carried out so that it is representative of the bulk.

Place the sample in a clean, dry and air-tight glass stoppered bottle of such a size that it is nearly filled by the sample.

When it is necessary to seal the container, care should be taken to avoid risk of contaminating the contents in any way.

PART I – METHODS OF TEST FOR GENERAL USE

3. DETERMINATION OF THE CRYSTALLIZING POINT OF GLACIAL ACETIC ACID

3.1 Principle

Determination of the temperature to which the slightly supercooled sample in fluid form rises during crystallization.

3.2 Applicability

The method is applicable only to 98 to 100 % (m/m) acetic acid.

3.3 Apparatus

3.3.1 *Test tube*, 150 mm × 25 mm.

3.3.2 *Thermometer*, of the mercury-in-glass type, graduated for use at 100 mm immersion, certified for accuracy, and complying with the following requirements :

Thermometer range °C	Graduations °C	Length		Diameter of stem mm	Distance from bottom of bulb to main scale mm	Certificate to show necessary corrections to readings
		Main scale mm	Bulb mm			
-0.5 to about 40.5	0.1	not less than 280	10 to 15	5.5 to 7.0	not less than 30	to within ± 0.05 °C

3.4 Procedure

3.4.1 Fill the dried test tube (3.3.1) with the test sample to a depth of about 100 mm, and insert the thermometer (3.3.2). Place the test tube in water at 10 to 11 °C so that the portion occupied by the sample is completely immersed, and allow it to remain without stirring, until the thermometer indicates about 2 °C below the expected crystallizing point. Then lift the tube out of the water and stir rapidly with the thermometer to induce the formation of minute crystals. At the moment crystallization begins, the temperature will rise rapidly and then remain constant for a few minutes. As soon as the steady temperature is approached, cease stirring and suspend the thermometer so that its bulb is centrally disposed in the crystallizing mass. Read to the nearest 0.05 °C the temperature at which the thermometer reading remains constant, apply the thermometer correction and record the corrected reading as the crystallizing point.

3.4.2 Prevent contamination of the sample with moisture during the test.

3.4.3 If, after cooling and stirring as described above, the temperature rise exceeds 3 °C the observed crystallizing point is liable to be below the true figure, and the operation should be repeated with less supercooling.

3.4.4 If crystallization will not begin after removal of the test tube from the cold water and vigorous stirring, the thermometer should be withdrawn and touched against some solid acetic acid previously prepared, then quickly re-inserted in the sample under test and the stirring resumed.

4. DETERMINATION OF ACETIC ACID CONTENT

4.1 Principle

Titration of acidity with a standard volumetric solution of sodium hydroxide using phenolphthalein as indicator, making allowance for any formic acid present.

4.2 Reagents

Distilled water or water of equivalent purity should be used in the test.

4.2.1 *Sodium hydroxide*, N standard volumetric solution.

4.2.2 *Phenolphthalein*, 5 g/l ethanolic solution. Dissolve 0.5 g of phenolphthalein in 100 ml of 95 % (v/v) ethanol and make faintly pink by the addition of dilute sodium hydroxide solution.

4.3 Apparatus

Ordinary laboratory apparatus and

4.3.1 *Weighing pipette*, capacity 10 ml.

4.4 Procedure

4.4.1 Transfer, by means of the weighing pipette (4.3.1), an accurately weighed quantity of the test sample, equivalent to 2 to 3 g of glacial acid, to a 250 ml conical flask containing about 50 ml of recently boiled and cooled water. Suitable quantities based on various nominal strengths of the acid are given in the following Table :

Nominal strength acetic acid %	Mass of test sample to be taken g
98 to 100	2.5
80	3.0
60	4.0
40	6.0

4.4.2 Add 0.5 ml of phenolphthalein solution (4.2.2) and titrate with sodium hydroxide solution (4.2.1).

4.5 Expression of results

$$\text{Acetic acid content (CH}_3\text{COOH) per cent by mass} = \frac{6.0 \times V}{M} - 1.3 A$$

where

V is the volume in millilitres, of N sodium hydroxide solution (4.2.1) used,

M is the mass, in grammes, of the test portion,

A is the formic acid content, per cent by mass, determined by the method described in section 10.

5. DETERMINATION OF RESIDUE ON EVAPORATION ON A WATER BATH

Use the method described in ISO Recommendation R 759, *Method for the determination of residue on evaporation on a water bath*.

6. DETERMINATION OF IRON CONTENT

6.1 Principle

Conversion of any iron present in the sample into the sulphate by evaporation to dryness of the specimen with sulphuric acid, and colorimetric determination of the iron using 2,2'-bipyridyl.

NOTE. — Although this method specifies the use of a spectrophotometer or photometer, it is permissible to employ, as an alternative procedure, a visual method comparing the test solution with a series of standard matching solutions (see clause 6.5.5).

6.2 Reagents

Distilled water or water of equivalent purity should be used in the test.

6.2.1 *Sulphuric acid*, $d = 1.84$, diluted 1 + 6 by volume.

6.2.2 *Nitric acid*, $d = 1.4$, diluted 1 + 3 by volume.

6.2.3 *Urea solution*. Dissolve 100 g of urea in 100 ml of water.

6.2.4 *Hydroxylammonium chloride*, 100 g/l solution.

6.2.5 *Ammonium acetate*, 500 g/l solution.

6.2.6 *2,2'-bipyridyl*, 5 g/l hydrochloric acid solution. Dissolve 0.5 g of 2,2'-bipyridyl in 100 ml of N hydrochloric acid solution.

6.2.7 *Standard iron solution* (10 µg Fe/ml). Dissolve 0.7022 g of pure iron (II) ammonium sulphate hexahydrate ($\text{FeSO}_4 \cdot (\text{NH}_4)_2 \text{SO}_4 \cdot 6\text{H}_2\text{O}$) in 50 ml of sulphuric acid solution (6.2.1) and dilute to 1000 ml with water. Dilute 100 ml of the solution thus obtained to 1000 ml with water.

6.3 Apparatus

Ordinary laboratory apparatus, and

6.3.1 *Spectrophotometer or photometer*.

6.4 Calibration charts

6.4.1 Place in 100 ml one-mark volumetric flasks the following quantities of standard iron solution (6.2.7):

0 – 2.0 – 4.0 – 7.0 – 10.0 – 15.0 and 20.0 ml.

To each add 20 ml of nitric acid solution (6.2.2), 2 ml of urea solution (6.2.3) and 2 ml of hydroxylammonium chloride solution (6.2.4). Mix and allow to stand for 2 minutes. Then add 30 ml of ammonium acetate solution (6.2.5) and 5 ml of 2,2'-bipyridyl solution (6.2.6). Dilute to the mark with water.

6.4.2 Measure the optical densities of the solutions in the spectrophotometer or photometer (6.3.1), determining the optical density at a wave length between 510 and 520 nm.

6.4.3 Draw a graph plotting optical densities as a function of the quantities of iron (in microgrammes) in 100 ml of the solutions.

6.5 Procedure

6.5.1 Weigh 100 g of the test sample in a platinum basin of capacity about 150 ml and evaporate to dryness on a water bath under a hood having a good draught. Allow to cool and add 10 ml of sulphuric acid solution (6.2.1). Evaporate, first on a water bath and finally on a sand bath, until white fumes are just evolved.

6.5.2 Allow to cool, add a few drops of nitric acid solution (6.2.2), and re-evaporate until white fumes just cease to be evolved. If tarry products remain, add a few further drops of nitric acid solution (6.2.2) and again evaporate on the sand bath.

6.5.3 Take up the residue with 20 ml of nitric acid solution (6.2.2) warming to assist solution of salts. Transfer the solution quantitatively to a 100 ml one-mark volumetric flask rinsing the platinum basin. Add 2 ml of urea solution (6.2.3), stir and add 2 ml of hydroxylammonium chloride solution (6.2.4), mix and allow to stand for 2 minutes. Then add 30 ml of ammonium acetate solution (6.2.5) and 5 ml of 2,2'-bipyridyl solution (6.2.6), and dilute to the mark with water.

6.5.4 Measure the optical density of the solution in the spectrophotometer or photometer (6.3.1) at a wave length between 510 and 520 nm using a cell with the same optical path length as those used in the preparation of the calibration chart and, by reference to the calibration chart prepared as indicated in paragraph 6.4, read the iron content (in microgrammes per 100 ml) corresponding to this optical density.

6.5.5 As an alternative to measurement of optical density using a spectrophotometer or photometer, the test solution prepared as in clause 6.5.3 may be compared visually with a series of standard matching solutions prepared under similar conditions, and its iron content (in microgrammes per 100 ml) deduced.

6.6 Expression of results

Express the iron content of the sample in parts per million, by mass, calculated by dividing by 100 the iron content determined according to clause 6.5.4 or clause 6.5.5.

7. LIMIT TEST FOR INORGANIC CHLORIDES

This method is applicable when the chloride content, expressed as C1, is not greater than 0.05 % and not less than 0.0005 %. If the chloride content lies outside that range, the mass of test portion taken (7.4.1) should be reduced or increased and an appropriate adjustment made to the expression $\frac{0.05}{x}$ ml in clause 7.4.4.

7.1 Principle

Comparison of the turbidity, obtained by the addition of silver nitrate to a solution prepared from the test sample in presence of nitric acid, with that similarly obtained from a chloride solution of known concentration.

7.2 Reagents

Distilled water or water of equivalent purity should be used in the test. All reagents and filter paper should be chloride free.

7.2.1 *Nitric acid*, approximately 5 N solution.

7.2.2 *Standard chloride solution (0.1 mg Cl/ml)*. Dilute 28.2 ml of 0.1 N hydrochloric acid solution to 1000 ml with water.

7.2.3 *Silver nitrate*, 50 g/l solution.

7.3 Apparatus

Ordinary laboratory apparatus.

7.4 Procedure

7.4.1 Weigh 50 ± 0.5 g of the test sample, transfer to a 250 ml one-mark volumetric flask, dilute to the mark with water and mix.

7.4.2 If the solution is not clear, pass it through a filter paper. This should remove turbidity due to aluminium. If any turbidity remains in the filtrate due to contamination with wax, remove it by shaking with a suitable solvent, for example, light petroleum.

7.4.3 To prepare the chloride solution of known concentration, add to a 100 ml Nessler cylinder 1.0 ml of the standard chloride solution (7.2.2), dilute to the mark with water, add 2 ml of nitric acid solution (7.2.1) and mix.

7.4.4 For a sample required to contain not more than x % of chloride, expressed as C1, transfer to a 100 ml Nessler cylinder an aliquot, $\frac{0.05}{x}$ ml, of the solution prepared from the test sample (7.4.1), dilute to the mark with water, add 2 ml of nitric acid solution (7.2.1), and mix.

7.4.5 Add to each Nessler cylinder 1 ml of silver nitrate solution (7.2.3) and mix. Allow the cylinders to stand in the dark for 5 minutes then compare the turbidity produced by the test sample with that produced by the chloride solution of known concentration.

7.5 Expression of results

A sample required to contain not more than x % of C1 does so if the turbidity produced from its solution (7.4.4) is equal to or less than that produced from the chloride solution of known concentration (7.4.3).

8. LIMIT TEST FOR INORGANIC SULPHATES

This method is applicable when the sulphate content, expressed as SO_4 , is not greater than 0.1 % and not less than 0.001 %. If the sulphate content lies outside that range the mass of test portion taken (8.4.1) should be reduced or increased and an appropriate adjustment made to the expression $\frac{0.1}{x}$ ml in clause 8.4.4.

8.1 Principle

Comparison of the turbidity, obtained by the addition of barium chloride to a solution prepared from the sample in presence of hydrochloric acid, with that similarly obtained from a sulphate solution of known concentration.

8.2 Reagents

Distilled water or water of equivalent purity should be used in the test.

8.2.1 *Sodium carbonate*, N solution.

8.2.2 *Hydrochloric acid*, N solution.

8.2.3 *Barium chloride*, $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$, 100 g/l solution.

8.2.4 *Standard sulphate solution* (0.1 mg SO_4 /ml). Dilute 20.8 ml of 0.1 N standard volumetric solution of sulphuric acid to 1000 ml with water and mix thoroughly.

8.3 Apparatus

Ordinary laboratory apparatus.

8.4 Procedure

8.4.1 Weigh 100 ± 1 g of the test sample, add 0.2 ml of sodium carbonate solution (8.2.1) and evaporate to dryness in an evaporating basin on a boiling water bath. Dissolve the residue in water containing 1 ml of hydrochloric acid solution (8.2.2), transfer to a 250 ml one-mark volumetric flask, dilute to the mark with water, and mix.

8.4.2 If the solution is not clear, pass it through a filter paper. This should remove turbidity due to aluminium. If any turbidity remains in the filtrate due to contamination with wax, remove it by shaking with a suitable solvent, for example, light petroleum.

8.4.3 To prepare the sulphate solution of known concentration, add to a 100 ml Nessler cylinder 4.0 ml of the standard sulphate solution (8.2.4), dilute to the mark with water, add 2 ml of hydrochloric acid solution (8.2.2) and mix.

8.4.4 For a sample required to contain not more than x % of SO_4 , transfer to a 100 ml Nessler cylinder an aliquot, $\frac{0.1}{x}$ ml, of the solution prepared from the test sample (8.4.1). Dilute to the mark with water, add 2 ml of hydrochloric acid solution (8.2.2) and mix.

8.4.5 Add to each Nessler cylinder 2 ml of barium chloride solution (8.2.3) and mix. Allow the cylinders to stand for 5 minutes, mix again, and compare the turbidity produced by the test sample with that produced by the sulphate solution of known concentration.

8.5 Expression of results

A sample required to contain not more than x % of SO_4 does so if the turbidity produced from its solution (8.4.4) is equal to or less than that produced from the sulphate solution of known concentration (8.4.3).

9. LIMIT TEST FOR HEAVY METALS (INCLUDING IRON)

9.1 Principle

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

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

9.2 Reagents

Distilled water or water of equivalent purity should be used in the test.

9.2.1 *Aqueous ammonia*, $d = 0.88$.

9.2.2 *Sodium sulphide*, 100 g/l solution.

9.2.3 *Standard lead solution* (10 μg Pb/ml), freshly prepared. Dissolve 0.0160 g of lead nitrate in water and make up to 1000 ml.

9.3 Apparatus

Ordinary laboratory apparatus.

9.4 Procedure

9.4.1 Pipette 25 ml of the test sample into a 250 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

9.4.2 Transfer a 10 ml aliquot to a Nessler cylinder. Add aqueous ammonia (9.2.1) until the solution is alkaline to litmus paper, and dilute to 50 ml with water. Add 0.1 ml (two drops) of sodium sulphide solution (9.2.2) and mix well.

9.4.3 Preparation of agreed standard matching solution. To 20 ml of water contained in a second Nessler cylinder add an agreed volume of standard lead solution (9.2.3) and 1 ml of aqueous ammonia (9.2.1). Dilute to 50 ml with water and mix well. Add 0.1 ml (two drops) of sodium sulphide solution (9.2.2) and again mix well.

9.4.4 Compare the darkening of the test solution (9.4.2) with that of the standard matching solution (9.4.3).

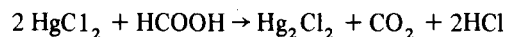
9.5 Expression of results

Report the darkening produced in the test solution as greater than, equal to, or less than that of the agreed standard matching solution, mentioning the lead content of the latter.

10. DETERMINATION OF FORMIC ACID CONTENT

10.1 Principle

Oxidation of the formic acid in the sample to carbon dioxide by mercuric chloride according to the reaction.



Iodometric determination of the mercurous chloride produced.