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R 1386

SOLVENT ACETATES FOR INDUSTRIAL USE

METHODS OF TEST

1st EDITION

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BRIEF HISTORY

The ISO Recommendation R 1386, *Solvent acetates 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 led to the adoption of Draft ISO Recommendation No. 1386, which was circulated to all the ISO Member Bodies for enquiry in February 1968. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Austria
Belgium
Brazil
Cuba
Czechoslovakia
France
Germany
Hungary
India
Iran
Ireland
Italy
Japan
Korea, Rep. of
Netherlands
New Zealand
Poland
Portugal
Romania
South Africa, Rep. of
Spain
Sweden
Switzerland
Thailand
Turkey
United Kingdom
U.S.S.R.

No Member Body opposed the approval of the Draft.

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

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ISO Recommendation R 1386 June 1970

SOLVENT ACETATES FOR INDUSTRIAL USE

METHODS OF TEST

1. SCOPE

This ISO Recommendation describes methods of test for methyl, ethyl, isopropyl, isobutyl, *n*-butyl and amyl acetates for industrial use.

2. SAMPLE*

The laboratory sample should have a volume of not less than 500 ml. It should be preserved in a clean, dry and airtight glass-stoppered bottle of such a size that it is nearly filled by the sample and should be stored in the dark.

If it has been necessary to seal the container, care should be taken to avoid contaminating the contents in any way.

3. MEASUREMENT OF COLOUR

3.1 Principle

Comparison of the colour of the sample with that of colour standards and expression in terms of Hazen colour units.

NOTE. – The Hazen colour unit is defined as the colour of a solution containing one part per million of platinum in the form of chloroplatinic acid and in the presence of two parts per million of cobalt (II) chloride hexahydrate.

3.2 Reagents

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

3.2.1 *Cobalt (II) chloride,* hexahydrate ($CoCl_2 . 6\ H_2O$).

3.2.2 *Hydrochloric acid,* ρ 1.19 (g/ml), solution approximately 38 % (m/m).

3.2.3 *Chloroplatinic acid.*

Dissolve 1.000 g of platinum in a small quantity of aqua regia contained in a glass or porcelain basin by heating on a boiling-water bath. When the metal has dissolved, evaporate the solution to dryness. Add 4 ml of the hydrochloric acid (3.2.2) and again evaporate to dryness. Repeat this operation twice more.

or

3.2.4 *Potassium chloroplatinate.*

3.3 Apparatus

Ordinary laboratory apparatus and

3.3.1 *Two matched flat-based colorimetric tubes,* of approximately 20 mm external diameter and having a graduation mark 100 mm above the base.

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* Sampling of chemical products will form the subject of a further ISO Recommendation.

3.4 Preparation of colour standards

3.4.1 Dissolve 2.0 g of cobalt (II) chloride (3.2.1) and either the chloroplatinic acid (3.2.3) or 2.490 g of potassium chloroplatinate (3.2.4) in 200 ml of hydrochloric acid (3.2.2). Warm, if necessary, to obtain a clear solution, and after cooling transfer to a 2000 ml one-mark volumetric flask. Dilute with water to the mark and mix thoroughly.

This solution has a colour of 500 Hazen units.

3.4.2 From this solution prepare a series of colour standards ranging from 0 Hazen colour units upwards, at intervals of 10 units. For each 10 units pipette 5.0 ml of the solution (3.4.1) into a 250 ml one-mark volumetric flask and dilute with water to the mark. Transfer each standard solution to a storage bottle and keep in the dark. Check these standards at intervals of 1 month against freshly prepared standards.

3.5 Procedure

Pour into one of the colorimetric tubes (3.3.1) a quantity of the laboratory sample sufficient to fill it to the graduation mark. Similarly pour the colour standard selected into the other tube to the mark. Compare the colour of the sample with that of the standard by looking down the tubes against a white background, taking care to avoid side illumination. Repeat with other colour standards if necessary until the closest match is obtained.

3.6 Expression of results

Report the result to the nearest 10 Hazen colour units. If the colour of the sample does not match that of any of the colour standards, report if possible an approximate value for colour with an appropriate note. However, should such an approximation be unobtainable, give a description of the colour of the sample.

4. DETERMINATION OF DENSITY AT 20 °C

Use the method described in ISO Recommendation R 758, *Method for the determination of density of liquids at 20 °C.*

5. DETERMINATION OF DISTILLATION YIELD

Use the method described in ISO Recommendation R 918, *Test method for distillation (distillation yield and distillation range).*

The following details, not given in ISO Recommendation R 918, apply to the particular acetate under test.

Thermometer (see clause 3.2 of ISO/R 918) of the mercury in glass type covering a convenient range, graduated at intervals of 0.2 °C and having a maximum error of 0.2 °C.

A correction for any deviation of the barometric pressure from 760 mm should be added to the specified distillation temperature for the particular acetate under test (clause 5.1 of ISO/R 918).

The appropriate correction is shown in the following table where p is the barometric pressure in standard millimetres of mercury.

Sample	Correction °C
methyl acetate	$0.04\ (p - 760)$
ethyl acetate	$0.041\ (p - 760)$
isopropyl acetate	$0.043\ (p - 760)$
isobutyl acetate	$0.046\ (p - 760)$
n-butyl acetate	$0.049\ (p - 760)$
amyl acetate	$0.05\ (p - 760)$

The interval before the first drop of distillate falls from the end of the condenser (clause 6.1 of ISO/R 918) should be as shown in the following table :

Sample	Interval
	minutes
methyl acetate	7 to 12
ethyl acetate	7 to 12
isopropyl acetate	7 to 12
isobutyl acetate	10 to 15
n-butyl acetate	10 to 15
amyl acetate	10 to 15

6. 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.*

7. DETERMINATION OF ACIDITY

7.1 For methyl and ethyl acetates

7.1.1 *Principle.* Titration at 0 °C of the acidity using a standard solution of sodium hydroxide and phenol red as indicator.

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

7.1.2.1 SODIUM HYDROXIDE, 0.1 N standard volumetric solution.

7.1.2.2 ETHANOL, 95 % (V/V).

7.1.2.3 PHENOL RED, 0.2 g/l solution of phenol red in 20 % (V/V) aqueous ethanol.

7.1.3 *Apparatus.* Ordinary laboratory apparatus and

7.1.3.1 CONICAL FLASK, of borosilicate glass, capacity 250 ml, provided with a stopper carrying a soda-lime tube.

7.1.3.2 MICROBURETTE, graduated in 0.02 ml or smaller divisions.

7.1.4 *Procedure.* Take 50 ml of ethanol (7.1.2.2), add a few drops of phenol red (7.1.2.3) and make just red by addition of the sodium hydroxide solution (7.1.2.1).

Cool to 0 °C, add 50 ml of the laboratory sample, and titrate the mixture at 0 °C with sodium hydroxide solution (7.1.2.1) placed in the microburette (7.1.3.2) until the red colour remains for 5 seconds.

7.1.5 *Expression of results.* Acidity (A), expressed as acetic acid (CH_3COOH), is given, as a percentage by mass, by the following formula :

$$\frac{0.012\, V_1}{\rho}$$

where

V_1 is the volume, in millilitres, of the sodium hydroxide solution (7.1.2.1) used;

ρ is the density, in grammes per millilitre, of the sample at 20 °C.

7.2 For isopropyl, isobutyl, *n*-butyl and amyl acetates

7.2.1 *Principle.* Titration of the acidity using a standard solution of sodium hydroxide and phenolphthalein as indicator.

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

7.2.2.1 SODIUM HYDROXIDE, 0.1 N standard volumetric solution.

7.2.2.2 ETHANOL, 95 % (V/V).

7.2.2.3 PHENOLPHTHALEIN, ethanolic solution.

Dissolve 0.5 g of phenolphthalein in 100 ml of ethanol (7.2.2.2) and make faintly pink by the addition of dilute sodium hydroxide solution.

7.2.3 *Apparatus.* Ordinary laboratory apparatus and

7.2.3.1 CONICAL FLASK, of borosilicate glass, capacity 250 ml, provided with a stopper carrying a soda-lime tube.

7.2.3.2 MICROBURETTE, graduated in 0.02 ml or smaller divisions.

7.2.4 *Procedure.* Take 50 ml of ethanol (7.2.2.2), add 0.5 ml of phenolphthalein (7.2.2.3) and make faintly pink by the addition of the sodium hydroxide solution (7.2.2.1). Add 50 ml of the laboratory sample and titrate the mixture with the sodium hydroxide solution (7.2.2.1) placed in the microburette (7.2.3.2) until the pink colour remains for 5 seconds.

7.2.5 *Expression of results.* Acidity (A), expressed as acetic acid (CH_3COOH), is given, as a percentage by mass, by the following formula :

$$\frac{0.012\,V_2}{\rho}$$

where

V_2 is the volume, in millilitres, of the sodium hydroxide solution (7.2.2.1) used;

ρ is the density, in grammes per millilitre, of the sample at 20 °C.

8. DETERMINATION OF ESTER CONTENT

8.1 Principle

Saponification of the ester, using potassium hydroxide in ethanolic solution, followed by titration with standard hydrochloric acid.

8.2 Reagents

Distilled water or water of equivalent purity, freshly boiled and cooled, should be used in the test.

8.2.1 *Potassium hydroxide,* approximately N solution in ethanol (95 % V/V).

8.2.2 *Hydrochloric acid,* N standard volumetric solution.

8.2.3 *Phenolphthalein,* ethanolic solution.

Dissolve 0.5 g of phenolphthalein in 100 ml of ethanol (95 % V/V) and make faintly pink by addition of dilute sodium hydroxide solution.

8.3 Apparatus

Ordinary laboratory apparatus and

8.3.1 *Two conical flasks* of borosilicate glass, capacity 250 ml, provided with ground glass stoppers.

8.3.2 *Water-cooled reflux condensers,* with ground glass joints to fit the flasks.

8.3.3 *Weighing pipette,* capable of delivering up to 5 g of the sample.

8.4 Procedure

8.4.1 Introduce successively from the same one-mark pipette 50 ml of the potassium hydroxide solution (8.2.1) into each of the two conical flasks (8.3.1).

By means of the weighing pipette (8.3.3) transfer immediately a weighed amount (see Table – clause 8.6) of the laboratory sample, into one of the flasks.

8.4.2 For methyl and ethyl acetates, close the two flasks with ground glass stoppers and leave them at a room temperature of not less than 15 °C for 4 hours. A shorter reaction time may be employed at higher room temperatures.

8.4.3 For propyl, butyl and amyl acetates, attach the flasks to the condensers (8.3.2) and heat for 1 hour in a boiling-water bath.

Withdraw the flasks, still carrying their condensers, and immerse them in cold running water. When cold, wash down the inside of each condenser with two 20 ml portions of water.

Disconnect the flasks and wash each joint with a further 20 ml of water.

8.4.4 Add 0.5 ml of the phenolphthalein solution (8.2.3) to each flask and immediately titrate the mixture and the blank successively with the hydrochloric acid solution (8.2.2) until the pink colour is discharged.

8.5 Expression of results

Ester content, expressed as the acetate CH_3COOR, where R is the alkyl radical present in the acetate (see Table – clause 8.6), is given, as a percentage by mass, by the following formula :

$$\frac{M\,(V_3 - V_4)}{10\,m} - \frac{M \times A}{60}$$

where

V_3 is the volume, in millilitres, of the hydrochloric acid solution (8.2.2) required by the blank;

V_4 is the volume, in millilitres, of the hydrochloric acid solution (8.2.2) required by the test portion;

m is the mass, in grammes, of the test portion;

A is the acidity, expressed as a percentage by mass, of acetic acid (see clause 7.1.5 or 7.2.5 above);

M is the molecular mass of the acetate (see Table – clause 8.6).

8.6 **Table** – Mass of test portion

Sample	R	Mass of test portion	M
		g	
methyl acetate	CH_3	1.6 to 2.1	74.1
ethyl acetate	C_2H_5	2.0 to 2.4	88.1
isopropyl acetate	C_3H_7	2.4 to 2.8	102.1
butyl acetates	C_4H_9	2.7 to 3.2	116.2
amyl acetates	C_5H_{11}	3.0 to 3.5	130.2