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Methyl ethyl ketone, *iso*butyl methyl ketone and *iso*amyl ethyl ketone for industrial use – Determination of alcoholic impurities – Volumetric method

Méthyl-éthyl-cétone, isobutyl-méthyl-cétone et isoamyl-éthyl-cétone à usage industriel – Dosage des impuretés alcooliques – Méthode volumétrique

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FOREWORD

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It has been approved by the Member Bodies of the following countries :

Austria	Ireland	ISO 2501:1974 Spain i/catalog/standards/sist/bf397801-24c0-4c80-918a-
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France	New Zealand	United Kingdom
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Methyl ethyl ketone, *iso*butyl methyl ketone and *iso*amyl ethyl ketone for industrial use – Determination of alcoholic impurities – Volumetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a volumetric method for the determination of the content of alcoholic impurities in methyl ethyl ketone (butanone) $(CH_3COC_2H_5),$ *iso*butyl methyl ketone (4-methylpentan-2-one) $[CH_3COCH_2CH(CH_3)_2]$ and ketone *iso*amyl ethvl (5-methylheptan-3-one) [CH₃CH₂CH(CH₃)CH₂COCH₂CH₃] for industrial use.

2 REFERENCES

ISO/R 758, Method for the determination of density of liquids at 20 °C.

ISO 2887, secButyl alcohol, methyl ethyl ketone, isobutyl Add 2 ml of the pyridine (4.1) to each flask, immediately methyl ketone, isoamyl ethyl ketone, diacetone alcohol and 01:19stopper tightly and mix thoroughly, taking care not to wet hexylene glycol for industrial use — Determination of and and sthe stoppers. To one flask add the test portion (6.1), ensuring acidity to phenolphthalein — Volumetric method

3 PRINCIPLE

Acetylation of a test portion with acetyl chloride and determination of the alcohol equivalent to the acetyl groups that have reacted, by titration with standard volumetric sodium hydroxide solution in the presence of phenolphthalein, and correction for acidity in the sample.

4 REAGENTS

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

4.1 Pyridine, dry.

4.2 Acetylating reagent

To 118 ml of freshly prepared acetyl chloride, add sufficient dry toluene to give a volume of 1 000 ml.

4.3 Sodium hydroxide, N standard volumetric solution.

4.4 Phenolphthalein, 10 g/l solution in pyridine.

Dissolve 2,5 g of phenolphthalein in 250 ml of pyridine.

5 APPARATUS

Ordinary laboratory apparatus.

6 PROCEDURE

6.1 Test portion

Take 25,0 ml of the laboratory sample at 20 $^{\circ}$ C.

6.2 Determination

Place 10,0 ml of the acetylating reagent (4.2) into each of two dry 250 ml glass-stoppered conical flasks, using an automatic suction device on the pipette.

2that it all comes into contact with the reagent. Close the flask tightly again. Mix the contents of the flask thoroughly, taking care not to wet the stopper.

Place the flasks in a water bath, controlled at 60 ± 1 °C, loosening the stoppers momentarily to release any pressure and replacing them tightly. Leave the flasks in the water bath for 20 min with occasional shaking, then remove and cool.

Add 25 ml of water and 0,5 ml of the phenolphthalein solution (4.4) to each flask, and titrate with the standard volumetric sodium hydroxide solution (4.3).

7 EXPRESSION OF RESULTS

7.1 The content of alcoholic impurities, expressed as a percentage by mass, is given by the formula

$$100 \times \frac{V_1 - V_2}{1\,000} \times \frac{M}{V_0\,\rho} + \frac{C_1\,M}{60}$$

where

 V_0 is the volume at 20 °C, in millilitres, of the test portion (6.1);

 V_1 is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (4.3) used for the blank titration;

 V_2 is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (4.3) used for the titration of the test portion;

M is the relative molar mass of the alcohol in terms of which the content of alcoholic impurities is expressed :

- = 74 in the case of butanol,
- = 102 in the case of hexanol, or
- = 130 in the case of octanol;

 ρ is the density at 20 °C, in grams per millilitre, of the sample, determined by the method specified in ISO/R 758;

 C_1 is the acidity to phenolphthalein of the sample, as a percentage by mass of acetic acid, determined by the method specified in ISO 2887;

60 is the relative equivalent mass of acetic acid.

7.2 For methyl ethyl ketone, the alcohol content, expressed as a percentage by mass of butanol, C_4H_9OH , is given by the formula

7.3 For *iso*butyl methyl ketone, the alcohol content, expressed as a percentage by mass of hexanol,
$$C_6H_{13}OH$$
, is given by the formula

$$\frac{0,408 \ (V_1 - V_2)}{\rho} + 1,70 \ C_1$$

7.4 For *iso* amyl ethyl ketone, the alcohol content, expressed as a percentage by mass of octanol, $C_8H_{17}OH$, is given by the formula

$$\frac{0,520 (V_1 - V_2)}{\rho} + 2,16 C_1$$

8 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

 $\frac{0,296 (V_1 - V_2)}{\rho} + 1,23 C_1$ **iTeh STANDARS** any operation not included in this International standard or the documents to which reference is made, **istandard standard or the documents** of which reference is made, **istandard standard standard standard**

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ANNEX

This document forms part of the following series on methods of test concerning the products indicated :

Methyl ethyl ketone (butanone) for industrial use

- ISO 2497 List of methods of test.
- ISO 2498 Examination for residual odour.
- ISO 2501 Determination of alcoholic impurities Volumetric method.
- ISO 2887 Determination of acidity to phenolphthalein Volumetric method.

isoButyl methyl ketone (4-methylpentan-2-one) for industrial use

- ISO 2499 List of methods of test.
- ISO 2501 Determination of alcoholic impurities Volumetric method.
- ISO 2887 Determination of acidity to phenolphthalein Volumetric method.

isoAmyl ethyl ketone (5-methylheptan-3-one) for industrial use

- ISO 2500 List of methods of test.
- ISO 2501 Determination of alcoholic impurities Volumetric method.
- ISO 2887 Determination of acidity to phenolphthalein Volumetric method.

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