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Methyl ethyl ketone, *isobutyl* methyl ketone and *isoamyl* 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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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 2501 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in September 1971.

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

Austria	Ireland	Spain
Belgium	Israel	Switzerland
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France	New Zealand	United Kingdom
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No Member Body expressed disapproval of the document.

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Methyl ethyl ketone, *isobutyl* methyl ketone and *isoamyl* 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) ($\text{CH}_3\text{COC}_2\text{H}_5$), *isobutyl* methyl ketone (4-methylpentan-2-one) [$\text{CH}_3\text{COCH}_2\text{CH}(\text{CH}_3)_2$] and *isoamyl* ethyl ketone (5-methylheptan-3-one) [$\text{CH}_3\text{CH}_2\text{CH}(\text{CH}_3)\text{CH}_2\text{COCH}_2\text{CH}_3$] 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 methyl ketone, isoamyl ethyl ketone, diacetone alcohol and hexylene glycol for industrial use – Determination of 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 °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.

Add 2 ml of the pyridine (4.1) to each flask, immediately stopper tightly and mix thoroughly, taking care not to wet the stoppers. To one flask add the test portion (6.1), ensuring that 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;

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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

$$\frac{0,296 (V_1 - V_2)}{\rho} + 1,23 C_1$$

7.3 For *isobutyl* 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 *isoamyl* 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;
- d) any operation not included in this International Standard or the documents to which reference is made, or regarded as optional.

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.*