International Standard 1279

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Essential oils — Determination of carbonyl value — Hydroxylammonium chloride method

Huiles essentielles — Détermination de l'indice de carbonyle — Méthode au chlorure d'hydroxylammonium

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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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 1279 was prepared by Technical Committee ISO/TC 54, *Essential oils*.

ISO 1279 was first published in 1973. This second edition cancels and replaces the first edition, clauses 3, 5, 7 and 8 of the previous edition having been technically revised.

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Essential oils — Determination of carbonyl value — Hydroxylammonium chloride method

1 Scope and field of application

This International Standard specifies a method for the determination of the carbonyl value of essential oils. It is applicable to essential oils the carbonyl compounds of which are aldehydes, methylketones or certain ketones which are easy to convert to oximes.

International Standards specifying requirements for individual essential oils will specify whether this method or the free hydroxylamine method specified in ISO 1271[1] is applicable.

2 References

ISO 212, *Essential oils — Sampling.*

ISO 356, *Essential oils — Preparation of test sample.*

3 Definition

For the purpose of this International Standard, the following definition applies.

carbonyl value (of an essential oil) : Number of milligrams of potassium hydroxide, per gram of essential oil, required to neutralize the hydrochloric acid liberated in the oximation reaction with hydroxylammonium chloride.

NOTE — Oximes are the result of the reaction of carbonyl compounds with hydroxylamine.

4 Principle

Conversion of the carbonyl compounds to oximes by reaction with hydroxylammonium chloride.

Titration of the hydrochloric acid liberated during the reaction with an ethanolic potassium hydroxide solution, either colorimetrically or potentiometrically.

5 Reagents

All reagents shall be of recognized analytical grade and the water used shall be distilled water or water of at least equivalent purity.

5.1 Hydrochloric acid, standard reference solution, $c(HCl) = 0{,}5$ mol/l.

5.2 Potassium hydroxide, standard volumetric solution, $c(KOH) \approx 0{,}5$ mol/l, in 95 % (V/V) ethanol.

Standardize just before use with hydrochloric acid (5.1), using the bromophenol blue (5.3) as indicator, and adding the alkali to the acid.

5.3 Bromophenol blue, ethanolic solution.

Dissolve, by heating, 0,2 g of bromophenol blue in 3 ml of an ethanolic potassium hydroxide solution $c(KOH) = 0{,}1$ mol/l, and 10 ml of 95 % (V/V) ethanol. After cooling, dilute to 100 ml with the same ethanol.

5.4 Hydroxylammonium chloride, ethanolic solution.

Dissolve 50 g of hydroxylammonium chloride in approximately 100 ml of water, add about 800 ml of 95 % (V/V) ethanol, then 10 ml of the ethanolic bromophenol blue solution (5.3) and dilute to 1 000 ml with 95 % (V/V) ethanol. Add the ethanolic potassium hydroxide solution (5.2) until the solution is green, if the liquid is observed in a thin layer, or until red, if the layer is thick.

The solution is suitable for use if a lemon-yellow colour is obtained when 0,05 ml of the hydrochloric acid (5.1) is added to 20 ml of the solution, and a red colour is obtained when 0,05 ml of the potassium hydroxide solution (5.2) is added to another 20 ml of the solution.

The solution is stable for 1 week.

1) ISO 1271, *Essential oils — Determination of carbonyl value — Free hydroxylamine method.*

6 Apparatus

Usual laboratory equipment, and in particular

6.1 For the two techniques (colorimetric titration and potentiometric titration)

6.1.1 Glass containers (flask, bottles).

6.1.2 Measuring cylinders, of capacities 25 and 50 ml.

6.1.3 Burettes, of capacity at least 25 ml, graduated in 0,1 ml divisions.

6.1.4 Analytical balance.

6.2 For the potentiometric titration

6.2.1 Potentiometer (preferably recording potentiometer), with combined glass electrodes.

6.2.2 Magnetic stirrer.

7 Sampling

See ISO 212.

8 Procedure

8.1 Preparation of the test sample

See ISO 356.

8.2 Test portion

Weigh, to the nearest 1 mg, into a container (6.1.1) a mass, *m*, of the test sample, as specified in the International Standard specification for the essential oil concerned.

8.3 Determination

8.3.1 Colorimetric titration

Transfer, by means of a measuring cylinder (6.1.2), 25 ml of the hydroxylammonium chloride solution (5.4) into the container (6.1.1) containing the test portion (8.2), and allow to stand or heat for the time specified in the International Standard specification for the essential oil concerned. Allow to cool.

Titrate with the potassium hydroxide solution (5.2) until the colour becomes identical to that of the same volume of reagent, thus indicating that the end-point is near.

Continue the addition of the potassium hydroxide solution (5.2) until a blue colour, persisting for 5 min, is obtained.

Record the total volume, V, of the potassium hydroxide solution used.

NOTE — This method is applicable to lightly coloured essential oils. For essential oils whose intrinsic colour is likely to interfere with the colorimetric determination of the end-point, the potentiometric method (8.3.2) should be used.

8.3.2 Potentiometric titration

Transfer, by means of a measuring cylinder (6.1.2), 50 ml of the hydroxylammonium chloride solution (5.4) into the container (6.1.1) containing the test portion (8.2), and allow to stand or heat for the time specified in the International Standard specification for the essential oil concerned. Allow to cool.

Titrate potentiometrically with the potassium hydroxide solution (5.2), while stirring with the magnetic stirrer.[1] The use of a recording potentiometer will greatly simplify this operation.

Calculate the volume, *V*, of potassium hydroxide solution used at the equivalence-point from the point of inflexion of the titration-curve or from readings of the change in pH. It should be emphasized that, according to the essential oil being tested, the pH at the end-point will not always be the same so that titration to a fixed pH value is excluded.

1) The method for the potentiometric titration of essential oils forms the subject of ISO 4726, *Essential oils — Potentiometric titration*. (At present at the stage of draft.)

9 Expression of results

The carbonyl value, expressed in milligrams of potassium hydroxide per gram of essential oil, is given by the formula

$$56{,}1 \times \frac{V}{m} \times c$$

where

c is the actual concentration, in moles per litre, of the potassium hydroxide solution (5.2);

m is the mass, in grams, of the test portion (8.2);

V is the volume, in millilitres, of potassium hydroxide solution used in the determination (8.3).

The carbonyl compounds content, expressed as a specified aldehyde or ketone as a percentage by mass, is given by the formula

$$\frac{M_r \times V}{10\,m} \times c$$

where

c, m and V have the same meanings as above;

M_r is the relative molecular mass of the aldehyde or ketone specified in the International Standard specification for the essential oil concerned.

Express the result to two significant figures.

10 Test report

The test report shall show the method used and the results obtained. It shall also mention any operating details not specified in this International Standard, or regarded as optional, together with details of any incidents likely to have influenced the results.

The test report shall include all the information necessary for the complete identification of the sample.