International Standard 1271

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Essential oils — Determination of carbonyl value — Free hydroxylamine method

Huiles essentielles — Détermination de l'indice de carbonyle — Méthode à l'hydroxylamine libre

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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.

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Essential oils — Determination of carbonyl value — Free hydroxylamine 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 which contain carbonyl compounds (especially ketones, excluding methylketones) which are difficult to convert to oximes by the method specified in ISO 1279.

The method is not applicable to essential oils which contain substantial amounts of esters or other alkali-sensitive constituents.

International Standards specifying requirements for individual essential oils will specify whether this method or the hydroxylammonium chloride method specified in ISO 1279[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) : The 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 free hydroxylamine liberated in a mixture of hydroxylammonium chloride and potassium hydroxide.

Titration of the excess alkali with hydrochloric acid 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 volumetric solution, $c(HCl) \approx 0{,}5$ mol/l.

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

5.3 Bromophenol blue, ethanolic solution.

Dissolve, by heating, 0,2 g of bromophenol blue in 3 ml of 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.

A lemon-yellow colour shall be obtained when 0,05 ml of the hydrochloric acid solution (5.1) is added to 20 ml of the solution, and a red colour shall be 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 one week.

1) ISO 1279, *Essential oils — Determination of carbonyl value — Hydroxylammonium chloride method.*

6 Apparatus

Usual laboratory equipment, and in particular

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

6.1.1 Glass flasks, alkali-resistant, of capacity 100 to 200 ml, with ground necks, fitted with either ground glass stoppers or with glass tubes at least 1 m long and at least 10 mm in internal diameter to serve as reflux condensers.

6.1.2 Pipettes, of capacities 20 and 50 ml.

6.1.3 Burettes, of capacity 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 flask (6.1.1) a mass m of the test sample, as specified in the International Standard specification for the essential oil concerned.

8.3 Blank test

Simultaneously with the determination, carry out a blank test using the same reagents and following the same procedure, but omitting the test portion.

If the technique by potentiometric titration is used (see 8.4.2) it is important that the blank test immediately precedes the determination, in order to operate at the same temperature.

8.4 Determination

8.4.1 Colorimetric titration

Transfer, by means of a pipette (6.1.2), 20 ml of the hydroxylammonium chloride solution (5.4) into the flask (6.1.1) containing the test portion (8.2). Add 15 ml of the potassium hydroxide solution (5.2) measured from a pipette or a burette and mix. Allow the flask containing the mixture to stand, or boil under reflux, for the time specified in the International Standard specification for the essential oil concerned. If boiling is carried out, cool rapidly before removing the reflux condenser.

Titrate with the hydrochloric acid solution (5.1) until the greenish-yellow end-point is observed. The titration should be carried out in a location that is well illuminated by natural daylight.

NOTE — This method is applicable to lightly coloured essential oils. For strongly coloured essential oils, the potentiometric method specified in 8.4.2 or, if a potentiometer is not available, the modified procedure described in the annex should be used.

8.4.2 Potentiometric titration

Transfer, by means of a pipette (6.1.2), 50 ml of the hydroxylammonium chloride solution (5.4) into the flask containing the test portion (8.2) and immediately add, by means of another pipette, 25 ml of the ethanolic potassium hydroxide solution (5.2). Close the flask with its glass stopper and mix the contents well by swirling. Allow the mixture to stand at room temperature, or boil under reflux, for the time specified in the International Standard specification for the essential oil concerned.

Titrate potentiometrically with the hydrochloric acid solution (5.1), while stirring with the magnetic stirrer (6.2.2).[1] The use of a recording potentiometer will greatly simplify this operation.

Calculate the volume of hydrochloric acid solution used at the equivalence-point from 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.

9 Expression of results

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

$$56{,}1 \times \frac{(V_0 - V_1)}{m} \times c$$

where

c is the exact concentration, in moles per litre, of the hydrochloric acid solution (5.1);

1) The method for the potentiometric titration of essential oils will form the subject of ISO 4726.

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

V_0 is the volume, in millilitres, of hydrochloric acid solution (5.1) used in the blank test (8.3);

V_1 is the volume, in millilitres, of hydrochloric acid solution (5.1) used in the determination (8.4).

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

$$\frac{M_r\,(V_0 - V_1)}{10\,m} \times c$$

where

c, m, V_0 and V_1 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 state the method used and the result obtained. It shall also mention any operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that might have influenced the results.

The test report shall include all details required for the complete identification of the sample.

Annex

Modified procedure to be used for strongly coloured essentials oils when the potentiometric method cannot be applied

(This annex forms an integral part of the Standard.)

Transfer, by means of a pipette (6.1.2), 20 ml of the hydroxylammonium chloride solution (5.4) into a flask (6.1.1). Add 15 ml of the potassium hydroxide solution (5.2) from a pipette or a burette and mix.

Transfer the mixture into the flask containing the test portion and retain the emptied conical flask without washing it. Allow the flask containing the mixture to stand, or boil under reflux, for the time specified in the International Standard specification for the essential oil concerned. If boiling is carried out, cool rapidly before removing the reflux condenser.

Titrate with the hydrochloric acid solution (5.1) until the greenish-yellow end-point is observed.

Transfer about half of the content to the original flask, neutralize until the solution takes on a lemon-yellow colour, transfer to the other flask, mix, and again return one half of the solution to the original flask.

Repeat this operation until the addition of two drops of the hydrochloric acid solution (5.1) to the solution contained in one of the two flasks causes no further change of colour when compared with the solution contained in the other flask.