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Essential oils — Determination of peroxide value

Huiles essentielles — Détermination de l'indice de peroxyde

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: Foreword — Supplementary information.

The committee responsible for this document is ISO/TC 54, *Essential oils*.

Essential oils — Determination of peroxide value

1 Scope

This International Standard specifies a method for the determination of the peroxide value in an essential oil. The peroxide value is a measure of the oxidation present.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 212, *Essential oils — Sampling*

ISO 356, *Essential oils — Preparation of test samples*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

3 Terms and definitions

For the purpose of this document, the following terms and definitions apply.

3.1
peroxide value
Ip

number that expresses, in millimoles (or milliequivalents), the quantity of peroxide contained in 1 000 ml of the substance

4 Principle

It is a redox titration of the iodometry type. The iodide ions added to the essential oil oxidize when reacting with peroxides, obtaining iodine which is titrated with thiosulphate. It can be carried out by volumetric or potientiometric titration.

Potientiometric titration is particularly recommended for highly coloured essential oils for which are difficult to appreciate the end point of the coloured indicator (e.g. vetiver essential oil).

5 Reagents

During the analysis, only reagents of recognized analytical grade and reverse osmosis or distilled or deionized water of Grade 3, as defined in ISO 3696, should be used.

5.1 Trichloromethane (chloroform), 99 % (volume/fraction), or cyclohexane, 99,5 % (volume/fraction) for laboratories with restrictions on the use of chloroform.

5.2 Glacial acetic acid, 99,5 % (volume/fraction). Degassed in an ultrasonic bath or by purging with a current pure and dry inert gas (carbon dioxide or nitrogen).

5.3 Potassium iodide, saturated solution of potassium iodide in deionised water, freshly prepared. The solution must remain saturated (undissolved crystals must be present). The solution has to be kept protected from light.

5.4 Sodium thiosulfate solution, 0,01 mol/l (0,01 N) or 0,1 mol/l (0,1 N).

5.5 Coloured standard indicator, starch solution 1 % (volume/fraction). It is not necessary for potentiometric titration.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 Balance, ±1 mg.

6.2 Erlenmeyer flask, of capacity 250 ml.

6.3 Shaker.

6.4 Pipettes, of capacity 1 ml, 10 ml, graduated in 0,1 ml.

6.5 Burette, of capacity 10 ml graduated in 0,05 ml.

6.6 Test tubes, of capacity 50 ml, 100 ml.

6.7 Potentiometer.

7 Sampling

Sampling is not included in the method specified in this International Standard. A recommended sampling method is given in ISO 212.

It is important that the laboratory receives a representative sample, not damaged or modified during the transport or storage, before the arrival at the laboratory.

8 Preparation of test sample

The test sample shall be prepared according to ISO 356.

The test sample for the determination of peroxide value shall be taken first and the peroxide value shall be determined immediately. Homogenize the sample, preferably without heating and without aireation. Avoid direct solar radiation. Heat solid samples carefully to 10 °C above their melting point. Samples with visible impurities shall be filtered.

For some products, the amount of extracted essential oil can be lower than 5 g, or the peroxide value of the essential oil can be over 30 meq of active oxygen per kilogram. In these cases, the user should choose a smaller sample mass.

9 Procedure

9.1 Test portion

Introduce in an Erlenmeyer flask 10 ml of essential oil to be titrated (see 6.1, 6.3).

9.2 Determination

Add with a test tube (see 6.6) 20 ml of trichloromethane or cyclohexane, 30 ml of glacial acetic acid and 1 ml of potassium iodide, saturated solution (see 5.1, 5.2, 5.3) and add 2 drops starch solution if volumetric titration is carried out.

Shake for approximately 1 min. The dissolution acquires an orangey colour.

Add about 100 ml of distilled water.

Tritate with sodium thiosulfate solution. Use 0,1 mol/l when the expected peroxide value are over 20 mmol/l. Use 0,01 mol/l sodium thiosulfate solution when the expected peroxide value are less than 20 mmol/l.

The end point is obtained when the dissolution becomes white.

Carry out a blank titration under the same conditions. No more than 0,5 ml of 0,01 mol/l sodium thiosulfate solution should be consumed for this purpose.

9.3 Automated potentiometric titration

Many laboratories often use automated equipment for titration. In this case, some points have to be taken into consideration.

— If the system works with autosampler, it is strictly necessary to use amber beaker glasses in order to stop the formation of Iodine while the sample remains in the tray.

— If the addition of potassium iodide wants to be automated, prepare 70 % (volume/fraction) solution in distilled water instead of saturated solution, fill an opaque bottle, and replace it every week. Therefore, the addition of potassium iodide in 9.2 shall be 10 ml instead of 1 ml.

10 Expression of results

10.1 Calculation

The peroxide value, *Ip,* in mmol/l, is given by Formula (1):

$$Ip = (V_1 - V_0) \times (N_{titrant}) \times 50 \quad (1)$$

where

V_1 is the volume, in millilitres, of titrant sodium thiosulphate, used in the main test;

V_0 is the volume, in millilitres, of titrant sodium thiosulphate, used in the blank test;

$N_{titrant}$ is the concentration of titrant, sodium thiosulphate, 0,1 N (see 5.4).

10.2 Conversion from mmol/l to meq/kg

The conversion from mmol/l to meq/kg is calculated by Formula (2):

$$\text{meq/kg} = \text{mmol/l} \times 2/\text{density (kg/l)} \quad (2)$$

11 Precision

11.1 Repeatability

The absolute difference between two independent single test results, obtained using this method on the same essential oil tested in the same laboratory by the same operator using the same equipment within a short period of time, was not, in more than 5 % in absolute values or 0,1 in relative values

11.2 Reproducibility

The absolute difference between two single test results, obtained using the same method on the same essential oil tested in different laboratories with different operators using different equipment, was not, in more than 10 % in absolute values or 0,3 in relative values.

Bibliography

[1] ISO 385-1[1)], *Laboratory glassware — Burettes — Part 1: General requirements*

1) This standard has been replaced by ISO 385.