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Essential oils — Determination of acid value by two titration methods, manual and automatic

Huiles essentielles — Détermination de l'indice d'acide par deux méthodes de titrage, manuelle et automatique

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Contents

Page

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 of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 54, *Essential oils*.

This third edition cancels and replaces the second edition (ISO 1242:1999), which has been technically revised.

The main changes are as follows:

— the title has been changed;

— subclauses 5.2 and 9.2 have been added;

— Annex A has been added.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Essential oils — Determination of acid value by two titration methods, manual and automatic

1 Scope

This document specifies two titration methods for determining the acid value in essential oils. These two methods are not applicable to essential oils containing appreciable quantities of lactones (e.g. Massoïa essential oil).

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 385, *Laboratory glassware — Burettes*

ISO 709, *Essential oils — Determination of ester value*

3 Terms and definitions

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

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
acid value
number of milligrams of potassium hydroxide required to neutralize the free acids contained in 1 g of the essential oil

4 Principle

Neutralization of the free acids using a titrated ethanolic solution of potassium hydroxide.

5 Reagents

Use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

5.1 Ethanol 96 % (volume fraction) at 20 °C, freshly neutralized before each series of measurements with the potassium hydroxide solution (5.2), in the presence of the coloured indicator (5.3) used for the determination in the case of manual titration.

5.2 Standard ethanolic potassium hydroxide solution previously titrated at C_{KOH} = 0,05 mol/l, 0,1 mol/l or 0,5 mol/l and checked before each series of measurements.

This ethanolic potassium hydroxide solution can be titrated with, for example, a monobasic potassium hydrogen phthalate solution or sulfuric acid solution.

The choice of concentration C_{KOH} depends on the capacity of the burette used, the test portion and the target acid value in order to tend towards an optimal volume of ethanolic potassium hydroxide solution V_{KOH}.

NOTE V_{KOH} is optimal when the equivalent volume is at least equal to half the capacity of the burette used.

For information, examples of optimized analysis conditions are presented in Table 1.

Table 1 — Examples of optimized analysis conditions

Target acid value	Example of essential oils	Theoretical concentration C_{KOH} mol/l	Approximate test portions g
Maximum 1,2	*Lavandula angustifolia,* ISO 3515	0,05	2
Maximum 4,0	*Pogostemon cablin,* ISO 3757	0,05	1
Minimum 15,0	*Cinnamomum aromaticum*, China type, ISO 3216	0,10	1
30,0 to 60,0	*Vetiveria zizanioides*, Brazil type, ISO 4716	0,50	1

5.3 Coloured indicator used for manual titration.

5.3.1 Phenolphthalein or thymolphtalein 2 g/l solution in neutralized ethanol 96 % (volume fraction) (5.1);

or if the essential oil contains phenolic groups:

5.3.2 Phenol red 0,4 g/l solution in ethanol 20 % (volume fraction).

NOTE This particular case is described in the corresponding monographs.

6 Apparatus

6.1 Apparatus in the case of manual titration

6.1.1 Ordinary laboratory glassware, adapted to the kind of titration to be carried out (determination of the acid value alone or determination of the acid value followed by the determination of the ester value).

If ester value shall be determined with the same test portion, use a flask with a capacity of 100 ml to 250 ml and follow the specifications regarding the saponification device indicated in ISO 709.

6.1.2 Measuring cylinder of 5 ml capacity.

6.1.3 Burette of capacity 2 ml or 5 ml, graduated in 0,01 ml, conforming to the requirements of ISO 385, class A.

6.1.4 Analytical balance of precision 0,001 g.

6.2 Apparatus in the case of automatic titration

6.2.1 Titrator

6.2.2 **Analytical balance** of precision 0,001 g.

7 Sampling

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

The sampling is not included in this document. A recommended sampling method is given in ISO 212.

8 Preparation of test sample

Prepare the test sample in accordance with ISO 356.

9 Procedure

9.1 Manual titration

9.1.1 Test portion

Weigh to the nearest 0,001 g a test portion of the sample of 1 g minimum, depending on the target acid value and C_{KOH} concentration, in order to tend towards an optimal volume of ethanolic potassium hydroxide solution V_{KOH} if possible.

Table 1 provides examples of test portions and C_{KOH} according to the target acid value.

9.1.2 Determination

Introduce the test portion (9.1.1) into suitable glassware (6.1.1). Add 5 ml of neutralized ethanol (5.1) and no more than five drops of phenolphthalein or thymolphthalein solution (5.3.1) or of phenol red solution (5.3.2), depending on the case, as indicator. Titrate the liquid with the previously titrated potassium hydroxide solution (5.2) contained in the burette (6.1.3).

Continue the addition until a change of colour that persists for 30 s is achieved. Note the volume V_{KOH} of potassium hydroxide used.

Eventually reserve the flask and its contents in case of determination of the ester value (see ISO 709).

The colour shifts observed according to the different indicators used are:

— colourless to pink with phenolphthalein;

— colourless to blue with thymolphthalein;

— yellow-orange to red with phenol red.

9.2 Automatic titration

In a titrator cup, weigh the suitable test sample to the nearest 0,001 g. The exact mass and identity of the sample shall be entered during the creation of the sequence. Add approximately 50 ml of neutralized ethanol 96 % (volume fraction).

To confirm the result, check the shape of the curve and the equilibrium point proposed by the software. It is possible to choose another equilibrium point if it seems to be more significant. If the profile of the

curve is not complete, or if its shape is not characteristic of an acid-base assay, this can mean that the assay is not complete or that an error has occurred during the titration. In this case, repeat the analysis by reconsidering new conditions (e.g. test portion, C_{KOH}).

Examples of curves are given in Figures A.1 and A.2.

10 Calculation

The acid value, A_V, is given by Formula (1):

$$A_V = 56{,}11 \times C_{KOH} \times V_{KOH} / m \quad (1)$$

where

C_{KOH} is the concentration, in moles per litre, of potassium hydroxide solution (5.2) used;

V_{KOH} is the volume, in millilitres, of potassium hydroxide solution (5.2) used;

m is the mass, in grams, of the test portion (9.1.1).

Express the result to one decimal place.

NOTE In the case of automatic titration, the neutralization of ethanol and titration of potassium hydroxide (5.2) can take place simultaneously. In this case, an additional factor can be added to Formula (1).

11 Precision

11.1 Repeatability

The difference between two independent test results obtained by means of this method, with the same essential oil tested in the same laboratory and by the same operator using the same apparatus in a short period of time, shall not be greater than 0,05 in absolute terms or not greater than 2,5 % in relative terms, taking into account the greatest value for a given result.

11.2 Reproducibility

The difference between two individual test results obtained by means of this method, with the same essential oil tested in different laboratories and by different operators using different apparatus, shall not be greater than 0,1 in absolute terms or not greater than 5 % in relative terms, taking into account the greatest value for a given result.

12 Test report

The test report shall include:

a) the sample

b) the International Standard used (including its year of publication) i.e. ISO 1242:2023;

c) the reference of the method used;

d) the test result(s) obtained;

e) if the repeatability has been verified, the final result obtained;

f) any deviations from the procedure;

g) any unusual features observed;

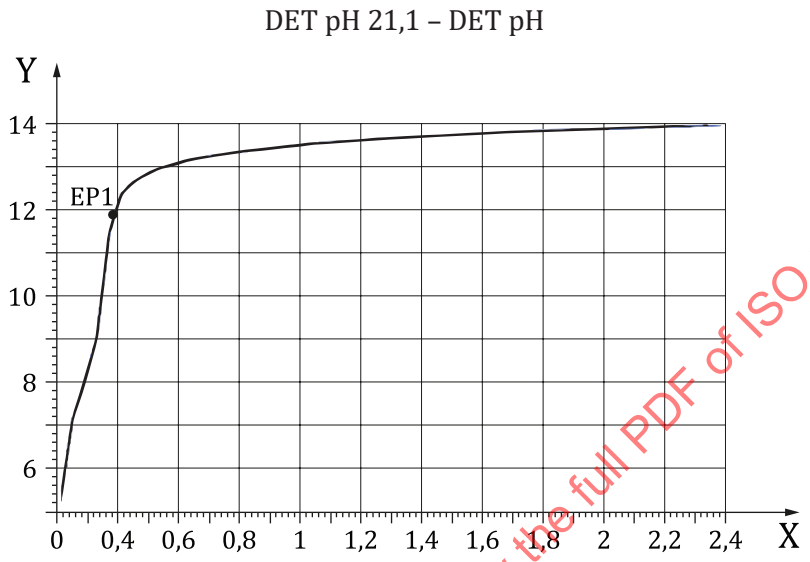
h) the date of the test.

It shall also mention any operating conditions not specified in this document, 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 A
(informative)

Typical curves obtained with a titrator



Parameters and results:

— test portion: 1,083 8 g

— blank volume: 0,025 ml

— KOH exact concentration: 0,101 mol/l

— equivalent point: 0,182 0 ml for 11,879 pH

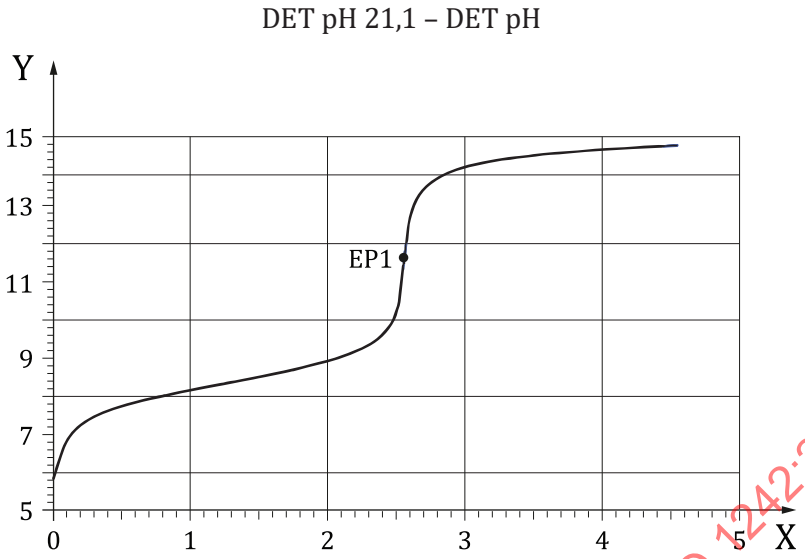
— calculated acid index: 0,82

Key

X volume (ml)

Y pH

Figure A.1 — Acid value for *Lavandula sp.* essential oil obtained with $C_{KOH} \approx 0{,}1$ mol/l



Parameters and results:

— test portion: 1,471 4 g

— KOH concentration: 0,498 mol/l

— equivalent point: 2,558 7 ml for 11,468 pH

— calculated acid index: 48,6

Key

X volume (ml)

Y pH

Figure A.2 — Acid value for *Vetiveria zizanioides* essential oil obtained with C_{KOH} ≈ 0,5 mol/l