INTERNATIONAL STANDARD

ISO 875

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Essential oils — Evaluation of miscibility in ethanol

Huiles essentielles — Évaluation de la miscibilité à l'éthanol

Reference number
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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 875 was prepared by Technical Committee ISO/TC 54, *Essential oils*.

This second edition cancels and replaces the first edition (ISO 875:1981), which has been technically revised.

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Essential oils — Evaluation of miscibility in ethanol

1 Scope

This International Standard specifies a method for the evaluation of the miscibility of essential oils with mixtures of ethanol and water of known ethanol content.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative documents referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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

ISO 385-2, *Laboratory glassware — Burettes — Part 2: Burettes for which no waiting time is specified.*

ISO 648, *Laboratory glassware — One-mark pipettes.*

3 Principle

Gradual addition to an essential oil, at a temperature of 20 °C, of an ethanol solution of suitable concentration.

Evaluation of miscibility and possibly of opalescence.

4 Classification of miscibility

4.1 An essential oil is said to be miscible with V volumes or more of ethanol of a given concentration, at a temperature of 20 °C, when the mixture of 1 volume of the oil in question with V volumes of that ethanol is clear and remains so after further gradual addition of ethanol of the same concentration up to a total of 20 volumes.

4.2 An essential oil is said to be miscible with V volumes of ethanol of a given concentration, at a temperature of 20 °C, and to become cloudy when diluted in V' volumes, when the mixture of 1 volume of the oil in question with V volumes of the ethanol is clear and becomes cloudy after further gradual addition of $(V' - V)$ volumes of ethanol of the same concentration and remains cloudy after further addition of the ethanol up to a total of 20 volumes.

4.3 An essential oil is said to be miscible with V volumes of ethanol of a given concentration, at a temperature of 20 °C, and to become cloudy when diluted in V' to V'' volumes, when the mixture of 1 volume of the oil in question with V volumes of the ethanol is clear, becomes cloudy after further gradual addition of $(V' - V)$ volumes of ethanol of the same concentration, and again becomes clear after further addition of $(V'' - V')$ volumes of ethanol of the same concentration.

4.4 An essential oil is said to be miscible with opalescence when the mixture of the oil with ethanol of a given concentration (under the conditions as given in 4.1, 4.2 and 4.3) shows an opalescence identical with the one of the standard solution for opalescence, freshly prepared in accordance with the method given in 5.3.

NOTE The numerical values of V, V' and V'' are not more than 20.

5 Reagents

Use only reagents of recognized analytical quality and distilled water.

5.1 Ethanol (95 % volume fraction)

5.2 Mixtures of ethanol and water

Mixtures of ethanol and water with an ethanol content of 50 %, 55 %, 60 %, 65 %, 70 %, 75 %, 80 %, 85 %, 90 % and 95 % (volume fraction) are normally used.

To prepare these mixtures, add distilled water to ethanol (5.1), following the directions given in Table 1, and check their concentrations with an alcoholmeter or a densimeter.

5.3 Standard solution for opalescence

Add 0,5 ml of a silver nitrate solution, $c(AgNO_3)$ = 0,1 mol/l, to 50 ml of sodium chloride solution, c(NaCl) = 0,000 2 mol/l; then add 1 drop of concentrated nitric acid (ρ_{20} = 1,38 g/ml). Stir the solution and allow it to stand for 5 min. Keep away from direct light.

Prepare the solution freshly before use.

6 Apparatus

Ordinary laboratory apparatus and, in particular, the following.

6.1 Burette, of capacity 25 ml or 50 ml, conforming to class B of ISO 385-2.

6.2 One-mark pipettes, capable of delivering 1 ml, conforming to the requirements of ISO 648, or **analytical balance,** capable of weighing to the nearest 1 mg, as appropriate (see 9.1).

6.3 Measuring cylinder or **flask**, of capacity 25 ml or 30 ml, provided with a stopper not subject to attack either by ethanol or by the essential oil to be examined.

6.4 Device, capable of maintaining a temperature of 20 °C ± 0,2 °C.

6.5 Calibrated thermometer, graduated in 0,2 °C or 0,1 °C, allowing the temperature of the device (6.4) to be checked.

7 Sampling

It is important that the laboratory receive a representative sample which has not been damaged or modified during its transportation or storage.

Sampling does not constitute a part of the method specified in this International Standard. A recommended sampling method is given in ISO 212[1)].

8 Preparation of test sample

Prepare the test sample according to ISO 356.

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1) ISO 212, *Essential oils — Sampling.*

9 Procedure

9.1 Test sample

With a pipette (6.2), introduce into the measuring cylinder or flask (6.3) 1 ml of the oil. Place the cylinder and its contents in the device (6.4), maintained at a temperature of 20 °C ± 0,2 °C.

NOTE When the physical state of the essential oil does not permit the use of a pipette, weigh, to the nearest 1 mg, 1 g ± 0,005 g of essential oil. In this case, the definition and the results will be expressed in mass/volume.

9.2 Determination of miscibility

Using the burette (6.1), add a mixture of ethanol and water of known concentration (5.2), which has previously been brought to a temperature of 20 °C ± 0,2 °C, in increments of 0,1 ml until complete miscibility occurs, shaking vigorously after each addition. When the mixture is perfectly clear, record the volume of the water/ethanol mixture (5.2) added.

Continue adding the mixture of ethanol and water in increments of 0,1 ml, up to a total of 20 ml, and shake after each addition. If the mixture becomes cloudy or opalescent before the total addition is completed, record the volume added at the point where cloudiness or opalescence appears and, if applicable, the volume at which one or the other disappears.

If a clear mixture is not obtained after 20 ml of solvent has been added, repeat with the next higher concentration of the mixture of ethanol and water given in Table 1.

9.3 Opalescence

If a clear mixture cannot be obtained but an opalescent mixture is obtained, compare its opalescence with that of the standard solution (5.3), as detailed in 10.2.

10 Expression of results

10.1 Miscibility

The miscibility of the essential oil with ethanol of concentration Q (see Table 1), at a temperature of 20 °C, is expressed as follows.

a) **Case 4.1**

1 volume of essential oil in V volumes of ethanol of concentration Q;

b) **Case 4.2**

1 volume of essential oil in V volumes of ethanol of concentration Q with cloudiness from V' volumes of ethanol of the same concentration;

c) **Case 4.3**

1 volume of essential oil in V volumes of ethanol of concentration Q with cloudiness appearing between V' and V'' volumes of ethanol of the same concentration;

where

V is the volume, in millilitres, of ethanol of concentration Q needed to obtain a clear solution;

V' is the volume, in millilitres, of ethanol of concentration Q needed to produce cloudiness, following the clearness, if it occurs;

V'' is the volume, in millilitres, of ethanol of the same concentration Q at which cloudiness disappears, if it occurs.

Express the values of V, V' and V'' numerically to one decimal place.

10.2 Opalescence

If only opalescence occurs (see 4.4), report whether the opalescence is "greater than", "equal to" or "less than" that of the standard solution (5.3).

11 Test report

The test report shall state:

— the method used;

— the concentration Q of the ethanol used;

— 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.

Table 1 — Preparation of the mixtures of ethanol and water

Dilution: ml of ethanol in 100 ml of mixture, to the nearest 0,1 %	**Volume of distilled water at 20 °C to be added to 100 ml of ethanol (95 % volume fraction), at the same temperature ±0,1 °C, for preparation of the corresponding dilutions**	**Mass of ethanol (95% volume fraction)**	**Mass of water to be added**	**Values of the relative density and apparent density**	
				1)	2)
Q % (volume fraction)	ml	g	g	d_{20}^{20} ±0,000 1	ρ_{20} ±0,000 01 g/ml
50	95,76	45,9	54,1	0,931 8	0,930 14
55	77,90	51,1	48,9	0,921 6	0,919 96
60	62,92	56,4	43,6	0,910 8	0,909 11
65	50,15	61,8	38,2	0,899 3	0,897 65
70	39,12	67,5	32,5	0,887 2	0,885 56
75	29,47	73,4	26,6	0,874 4	0,872 79
80	20,94	79,5	20,5	0,860 8	0,859 27
85	13,31	85,9	14,1	0,846 4	0,844 85
90	6,40	92,7	7,3	0,830 7	0,828 18
95	0,0	100,0	0,0	0,812 9	0,811 38

1) Reference: Swiss Federal Bureau of Weights and Measurements.

2) Reference: International Organization of Legal Metrology.

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