
International Standard



875

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Essential oils — Evaluation of miscibility in ethanol

Huiles essentielles — Évaluation de la miscibilité à l'éthanol

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

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.

International Standard ISO 875 was developed by Technical Committee ISO/TC 54, *Essential oils*, and was circulated to the member bodies in March 1980.

It has been approved by the member bodies of the following countries :

Australia	Egypt, Arab Rep. of	Portugal
Austria	France	South Africa, Rep. of
Bulgaria	India	Sri Lanka
Canada	Italy	USSR
Chile	Korea, Rep. of	
China	Netherlands	

No member body expressed disapproval of the document.

This International Standard cancels and replaces ISO Recommendation R 875-1968, of which it constitutes a technical revision.

Essential oils — Evaluation of miscibility in ethanol

1 Scope and field of application

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 References

ISO 212, *Essential oils — Sampling*.

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

ISO/R 385, *Burettes*.

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

3 Definitions

3.1 An essential oil is said to be miscible with V_1 volumes and more of ethanol of given strength, 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 stays so after further gradual addition of ethanol of the same strength up to a total of 20 volumes.

3.2 An essential oil is said to be miscible with V volumes of ethanol of given strength, 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 that ethanol is clear and becomes cloudy after further gradual addition of $(V' - V)$ volumes of ethanol of the same strength and remains cloudy after further addition of ethanol up to a total of 20 volumes.

3.3 An essential oil is said to be miscible with V volumes of ethanol of given strength, 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 that ethanol is clear, becomes cloudy after further gradual addition of $(V' - V)$ volumes of ethanol of the same strength, and again becomes clear after further addition of $(V'' - V')$ volumes of ethanol of the same strength.

3.4 An essential oil is said to be miscible with opalescence when the mixture of the oil with an ethanol of given strength (under the conditions as given in 3.1, 3.2 and 3.3) shows an opalescence identical with the one of the standard solution for opalescence freshly prepared according to 5.2.

NOTE — V , V' and V'' are not more than 20.

4 Principle

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

Evaluation of miscibility and possibly of opalescence.

5 Reagents

5.1 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 % (V/V) are normally used.

To prepare these mixtures, add distilled water to 95 % (V/V) ethanol of analytical grade, following the directions given in the table, and check their strengths with an alcoholmeter or a densimeter, or by determining their refractive index (see the table).

NOTE — A value for refractive index does not necessarily show a particular strength of ethanol and the precision of the determination of the strength is less than with a density measurement.

5.2 Standard solution for opalescence

Add 0,5 ml of a silver nitrate solution, $c(\text{AgNO}_3) = 0,1 \text{ mol/l}^{(1)}$, to 50 ml of sodium chloride solution, $c(\text{NaCl}) = 0,0002 \text{ mol/l}^{(2)}$; then add 1 drop of concentrated nitric acid ($\rho_{20} = 1,38 \text{ g/ml}$). Stir the solution and allow it to stand for 5 min. Keep away from direct light.

Prepare the solution freshly.

1) Previously designated "0,1 N solution".

2) Previously designated "0,000 2 N solution".

6 Apparatus

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

6.2 One-mark pipettes, capable of delivering 1 ml, conforming to the requirements of ISO 648, or **analytical balance** as appropriate (see 8.2).

6.3 Measuring cylinder or flask, of capacity 25 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 $20 \pm 0,2$ °C.

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

7 Sampling

See ISO 212.

8 Procedure

8.1 Preparation of test sample

See ISO 356.

8.2 Test sample

With a pipette (6.2)¹⁾, 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 $20 \pm 0,2$ °C.

8.3 Miscibility

Using the burette (6.1), add an ethanol of known strength (5.1), which has previously been brought to a temperature of $20 \pm 0,2$ °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 (5.1) added.

Continue adding the ethanol by increments of 0,5 ml, up to a total of 20 ml, and shaking 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 when 20 ml of solvent has been added, repeat with the next higher strength of ethanol given in the table.

8.4 Opalescence

If a clear mixture cannot be obtained but an opalescent mixture is obtained, compare its opalescence with that of the standard solution (5.2).

9 Expression of results

9.1 Miscibility

The miscibility of the essential oil with ethanol of strength t (see the table), at a temperature of 20 °C, is expressed as follows :

9.1.1 Case 3.1

1 volume of essential oil in V volumes of ethanol of strength t .

9.1.2 Case 3.2

1 volume of essential oil in V volumes of ethanol of strength t with cloudiness from V' volumes of ethanol of the same strength

9.1.3 Case 3.3

1 volume of essential oil in V volumes of ethanol of strength t with cloudiness appearing between V' and V'' volumes of ethanol of the same strength.

where

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

V' is the volume, in millilitres, of ethanol of strength t needed to produce cloudiness, following the clearness, if it occurs;

V'' is the volume, in millilitres, of ethanol of the same strength t at which cloudiness disappears, if it occurs.

9.1.4 Precision of results

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

9.2 Opalescence

If only opalescence occurs (see 8.4), report whether the opalescence is "greater than", "equal to" or "less than" that of the standard solution (5.2).

1) When the physical state of the essential oil does not permit measuring with a pipette, weigh $1 \pm 0,005$ g of the oil to the nearest 1 mg. In this case, the definition and the results are expressed as mass/volume.

10 Test report

The test report shall state the method used, the strength t of the ethanol 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 result.

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

Table — Preparation of the mixtures of ethanol and water¹⁾

Dilution : ml of ethanol in 100 ml of mixture to the nearest 0,1 % t	Volume of distilled water at 20 °C to be added to 100 ml of ethanol 95 % (V/V) at the same temperature for the prepa- ration of the correspond- ing dilutions	Mass of ethanol 95 % (V/V)	Mass of water to be added	Limits of the relative density and apparent density		Refractive index at 20 °C
				d_{20}^{20}	ρ_{20} g/ml	
% (V/V)	ml	g	g			
50	95,8	45,9	54,1	0,931 6 to 0,932 0	0,930 0 to 0,930 3	1,359 0
55	77,9	51,1	48,9	0,921 4 to 0,921 8	0,919 8 to 0,920 2	1,359 8
60	62,9	56,4	43,6	0,910 5 to 0,911 9	0,908 9 to 0,909 3	1,361 4
65	50,2	61,8	38,2	0,899 0 to 0,899 5	0,897 4 to 0,897 9	1,362 5
70	39,1	67,6	32,5	0,886 9 to 0,887 4	0,885 3 to 0,885 8	1,363 3
75	29,5	73,4	26,6	0,874 0 to 0,874 6	0,872 5 to 0,873 1	1,364 0
80	20,9	78,5	20,5	0,860 5 to 0,861 1	0,859 0 to 0,859 6	1,364 8
85	13,3	85,9	14,1	0,846 1 to 0,846 7	0,844 6 to 0,845 2	1,364 8
90	6,4	92,7	7,3	0,830 3 to 0,831 0	0,828 8 to 0,829 5	1,364 5
95	0,0	100,0	0,0	0,812 4 to 0,813 2	0,811 0 to 0,811 8	1,363 8

1) According to the tables of OIML.

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