

# ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

## ISO RECOMMENDATION R 875

DETERMINATION OF SOLUBILITY  
OF ESSENTIAL OILS IN ETHANOL

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1st EDITION  
November 1968

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Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

## BRIEF HISTORY

The ISO Recommendation R 875, *Determination of solubility of essential oils in ethanol*, was drawn up by Technical Committee ISO/TC 54, *Essential oils*, the Secretariat of which is held by the Repartição de Normalização (IGPAI).

Work on this question by the Technical Committee led, in 1964, to the adoption of a Draft ISO Recommendation.

In November 1965, this Draft ISO Recommendation (No. 869) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Argentina	Ireland	Portugal
Belgium	Israel	Romania
Brazil	Italy	South Africa, Rep. of
Canada	Japan	Sweden
Chile	Korea, Rep. of	Turkey
France	Netherlands	United Kingdom
Germany	<u>New Zealand</u>	U.S.S.R.
India	Poland	Yugoslavia

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in November 1968, to accept it as an ISO RECOMMENDATION.

## DETERMINATION OF SOLUBILITY OF ESSENTIAL OILS IN ETHANOL

### 1. SCOPE

This ISO Recommendation describes the method to be used and the visual observations to be made in determining the solubility of essential oils in suitable dilutions of ethanol.

### 2. DEFINITIONS

- 2.1 An essential oil is said to be soluble in  $V$  volumes and more of ethanol of given strength  $t$ , when the clear solution in  $V$  volumes remains clear after further addition of ethanol of the same strength up to a total of 20 volumes of ethanol.
- 2.2 An essential oil is said to be soluble in  $V$  volumes of ethanol of given strength  $t$ , and to become cloudy when diluted, when the clear solution in  $V$  volumes becomes cloudy and stays so after further gradual addition of ethanol of the same strength up to a total of 20 volumes of ethanol.
- 2.3 An essential oil is said to be soluble in  $V$  volumes of ethanol of given strength  $t$ , and becomes cloudy in  $V'$  volumes ( $V' < 20$ ), when the clear solution in  $V$  volumes becomes cloudy and stays so after further gradual addition of ethanol of the same strength up to a total of  $V'$  volumes of ethanol.
- 2.4 An essential oil is said to be soluble with opalescence when the alcoholic solution on being diluted shows a bluish tinge, this colour being similar to that of the standard of opalescence, freshly prepared in accordance with clause 4.2.

### 3. PRINCIPLE OF METHOD

An aqueous ethanol solution of suitable known concentration is added progressively at a temperature of 20 °C to an essential oil and the degree of miscibility is observed.

#### 4. REAGENTS

- 4.1 *Dilute solutions of ethanol.* Solutions of 50, 55, 60, 65, 70, 75, 80, 85, 90 and 95 % (v/v) are normally used.
- To prepare these solutions use ethanol as pure as possible and follow the directions given in the Annex. Check their strength with an alcoholmeter or a densimeter.
- 4.2 *Standard solution for opalescence.* Add 0.5 cm<sup>3</sup> of 0.1 N silver nitrate solution to 50 cm<sup>3</sup> of 0.0002 N sodium chloride solution and one drop of 25 % (m/m) nitric acid. Stir the solution and allow it to stand for 5 minutes.

#### 5. APPARATUS

- 5.1 *Burette*, 25 or 50 ml, Class B (see ISO Recommendation R 385, *Burettes*).
- 5.2 *Cylinder*, of 25 to 30 cm<sup>3</sup> capacity, with a stopper not subject to attack either by ethanol or by the essential oil to be analysed.
- 5.3 *Constant temperature device*, maintained at a temperature of  $20 \pm 0.2$  °C.
- 5.4 *Calibrated thermometer*, graduated in fifths or tenths of a degree Celsius allowing the determination of temperatures between 10 and 30 °C.

#### 6. SAMPLING

See ISO Recommendation R 212, *Essential oils – Sampling*.

#### 7. PROCEDURE

##### 7.1 Preparation of test sample

See ISO Recommendation R 356, *Essential oils – Methods of test – Preparation of sample*.

##### 7.2 Determination

Measure into the cylinder (5.2) 1 cm<sup>3</sup> of essential oil, either with a pipette or by weighing to an accuracy of  $\pm 5$  mg. Place the cylinder and its contents in the constant temperature device (5.3) maintained at a temperature of  $20 \pm 0.2$  °C.

Using the burette (5.1), add the diluted solution of ethanol of known strength (4.1), which has previously been brought to a temperature of  $20 \pm 0.2$  °C, by increments of 0.1 cm<sup>3</sup> until the oil is completely dissolved, shaking frequently and vigorously during the addition of the solvent. When the solution is perfectly clear, record the volume of ethanol solution (4.1) added.

Continue to add the solvent by increments of 0.5 cm<sup>3</sup>, up to 20 cm<sup>3</sup>, and keep shaking after each addition. If the solution becomes cloudy or opalescent before a total volume of 20 cm<sup>3</sup> of ethanol has been added, record the volume added at the point where cloudiness or opalescence appears and, if applicable, the volume at which it disappears.

If a clear solution is not obtained when 20 cm<sup>3</sup> of solvent have been added, repeat with the next higher concentration of ethanol solution given in the Table (see Annex).

## 8. EXPRESSION OF RESULTS

### 8.1 Method of calculation

If

$V$  is the volume, in cubic centimetres, of dilute solution of ethanol of dilution  $t$  to obtain a clear solution;

$V'$  is the volume, in cubic centimetres, of dilute solution of ethanol of dilution  $t$  producing cloudiness, following the clearness, if it occurs;

$V''$  is the volume, in cubic centimetres of dilute solution of ethanol of dilution  $t$  at which cloudiness disappears;

the solubility of the essential oil in the dilute solution of ethanol of strength  $t$  will be :

1 volume in  $V$  volumes, with cloudiness between  $V'$  and  $V''$  volumes.

### 8.2 Accuracy of results

Express the values  $V$ ,  $V'$  and  $V''$  by numbers to one decimal place.

If the solution is not entirely clear, but only opalescent, record whether the opalescence is "greater than", "equal to", or "less than" that of the standard solution (see clause 4.2).

## 9. TEST REPORT

State with the test result the method used, the method of expression of results, any characteristics noted during the determinations, and any operations not laid down in this ISO Recommendation or regarded as optional, that might have affected the results. —

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ISO/R 875:1968

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ANNEX

TABLE — Dilutions of ethanol by volume and by mass

Dilutions : cm <sup>3</sup> of ethanol in 100 cm <sup>3</sup> of the mixture (% v/v) to ± 0.1 %	Volume of distilled water at 20 °C, to be added to 100 cm <sup>3</sup> of ethanol 95 % (v/v) at the same temperature within ± 0.1 °C, for the preparation of the corresponding dilutions  cm <sup>3</sup>	Mass of ethanol 95 % (v/v)  g	Mass of water to be added  g	Limits of the relative density and the apparent density	
				$d_{20}^{20}$	$d_{20}^{20}$
50	95.8	46	54	0.9316 to 0.9320	0.9300 to 0.9304
55	77.9	51.1	48.9	0.9214 to 0.9218	0.9198 to 0.9202
60	62.9	56.4	43.6	0.9105 to 0.9110	0.9089 to 0.9094
65	50.2	61.9	38.1	0.8990 to 0.8994	0.8974 to 0.8978
70	39.1	67.6	32.4	0.8868 to 0.8873	0.8852 to 0.8857
75	29.5	73.4	26.6	0.8740 to 0.8745	0.8725 to 0.8730
80	20.9	79.6	20.4	0.8604 to 0.8610	0.8589 to 0.8595
85	13.3	86	14	0.8460 to 0.8466	0.8445 to 0.8451
90	6.4	92.7	7.3	0.8303 to 0.8309	0.8288 to 0.8294
95	0.0	100	0	0.8123 to 0.8131	0.8109 to 0.8117