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IS 326-6 (2005): Methods of sampling and test for natural and synthetic perfumery materials (Part 6) Determination of solubility [PCD 18: Natural and Synthetic Fragrance Materials]



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भारतीय मानक

प्राकृतिक और संश्लेषित सुगन्ध सामग्री के नमूने लेने और  
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भाग 6 एथेनॉल में मिश्रणीयता का अंश ज्ञात करना  
( तीसरा पुनरीक्षण )

*Indian Standard*

**METHODS OF SAMPLING AND TEST FOR  
NATURAL AND SYNTHETIC PERFUMERY  
MATERIALS**

**PART 6 EVALUATION OF MISCIBILITY IN ETHANOL**  
( *Third Revision* )

ICS 71.100.60

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**BUREAU OF INDIAN STANDARDS**  
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## NATIONAL FOREWORD

This Indian Standard (Part 6) (Third Revision) which is identical with ISO 875 : 1999 'Essential oils — Evaluation of miscibility in ethanol' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendations of the Natural and Synthetic Fragrance Materials Sectional Committee and approval of the Petroleum, Coal and Related Products Division Council.

The text of ISO Standard has been proposed to be approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

- a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.
- b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

In this adopted standard, reference appears to the following International Standard for which Indian Standard also exists. The corresponding Indian Standard which is to be substituted in its place, is listed below along with its degree of equivalence for the edition indicated. However, that International Standard cross-referred in this adopted ISO Standard, which has subsequently been revised, position in respect of that latest ISO Standard has been given:

<i>International Standard</i>	<i>Corresponding Indian Standard</i>	<i>Degree of Equivalence</i>
ISO 648 : 1977 Laboratory glassware — One-mark pipettes	IS 1117 : 1975 One-mark pipettes ( <i>first revision</i> )	Technically equivalent

The Technical Committee responsible for the preparation of this standard has reviewed the provisions of following International Standards and decided that they are acceptable for use in conjunction with this standard:

<i>International Standard</i>	<i>Title</i>
ISO 356 : 1996	Essential oils — Preparation of test sample
ISO 385-2 : 1984	Laboratory glassware — Burettes — Part 2 : Burettes for which no waiting time is specified

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

## Indian Standard

# METHODS OF SAMPLING AND TEST FOR NATURAL AND SYNTHETIC PERFUMERY MATERIALS

## PART 6 EVALUATION OF MISCIBILITY IN ETHANOL

### ( *Third Revision* )

#### 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,  $c(\text{AgNO}_3) = 0,1 \text{ mol/l}$ , to 50 ml of sodium chloride solution,  $c(\text{NaCl}) = 0,000 2 \text{ mol/l}$ ; 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 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  $20 \text{ }^\circ\text{C} \pm 0,2 \text{ }^\circ\text{C}$ .

**6.5 Calibrated thermometer**, graduated in  $0,2 \text{ }^\circ\text{C}$  or  $0,1 \text{ }^\circ\text{C}$ , 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<sup>1)</sup>.

## 8 Preparation of test sample

Prepare the test sample according to ISO 356.

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<sup>1)</sup> 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  $20\text{ }^{\circ}\text{C} \pm 0,2\text{ }^{\circ}\text{C}$ .

NOTE When the physical state of the essential oil does not permit the use of a pipette, weigh, to the nearest 1 mg,  $1\text{ g} \pm 0,005\text{ g}$  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  $20\text{ }^{\circ}\text{C} \pm 0,2\text{ }^{\circ}\text{C}$ , 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  $20\text{ }^{\circ}\text{C}$ , 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 $\pm 0,1$ °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)
				$d_{20}^{20}$ $\pm 0,000 1$	$\rho_{20}$ $\pm 0,000 01$ g/ml
$Q$ % (volume fraction)	ml	g	g		
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.

## Bureau of Indian Standards

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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

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