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Indian Standard

METHODS OF SAMPLING AND TEST FOR NATURAL AND SYNTHETIC PERFUMERY MATERIALS

PART 4 DETERMINATION OF OPTICAL ROTATION

(Third Revision)

ICS 71.100.60
NATIONAL FOREWORD

This Indian Standard (Part 4) (Third Revision) which is identical with ISO 592:1998 'Essential oils — Determination of optical rotation' issued by the International Organization for Standardization 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.

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

<table>
<thead>
<tr>
<th>International Standard</th>
<th>Title</th>
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<tr>
<td>ISO 356 : 1996</td>
<td>Essential oils — Preparation of test samples</td>
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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 4 DETERMINATION OF OPTICAL ROTATION

(Third Revision)

1 Scope

This International Standard specifies a method for determining the optical rotation of essential oils. When dealing with solid oils, partially solid oils, oils that are highly viscous at room temperature, or highly coloured oils, this determination is carried out on a solution of the oil.

2 Normative reference

The following normative document contains 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 edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 356, Essential oils — Preparation of test samples.

3 Terms and definitions

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

3.1 optical rotation of an essential oil, \( \alpha_D \)
angle, expressed in milliradians and/or degrees of angle, described by the polarization plane of a luminous radiation whose wavelength is \( 589.3 \text{ nm} \pm 0.3 \text{ nm} \), corresponding to the D lines of sodium, when such light travels through a thickness of 100 mm of essential oil under given conditions of temperature

NOTE When the determination is carried out on different thickness, the value of \( \alpha_D \) should be computed by reference to a thickness of 100 mm. Also measurements according to the Faraday magneto-optical principle are possible. The thickness of the sample is approximately 10 mm in that case.
3.2 optical rotation of an essential oil in solution
specific rotation, $\alpha$

optical rotation $\alpha_0$, of a solution of essential oil divided by the mass of essential oil in the unit of volume

4 Reagents

Reagents shall be of analytical grade. Use distilled water or water of at least equivalent purity.

4.1 Solvent (only for essential oils that need to be tested in solution).

Use preferably 95% ethanol by volume. It is advisable to check that the optical rotation of the solvent used is nil.

5 Apparatus

5.1 Polarimeter, having a precision of at least $\pm 0.5$ mrad ($\pm 0.03^\circ$) and adjusted to give 0° and 180° with water.

The polarimeter shall be checked with a quartz plate of known optical rotation or, if that is unavailable, with an aqueous solution containing 26.00 g of anhydrous pure saccharose per 100 ml of solution. The optical rotation of this solution shall be $+60.4$ mrad ($+34.62^\circ$) in a 200 mm layer, at a temperature of 20 °C.

The instrument shall be under conditions of stability when in use, and non-electronic instruments shall be used in the dark.

5.2 Light source, comprising any device giving a light of wavelength 589.3 nm $\pm 0.3$ nm, preferably a sodium vapour lamp.

5.3 Polarimeter tubes, usually 100 mm $\pm 0.5$ mm long.

When testing slightly coloured samples of low optical rotation, tubes of length 200 mm $\pm 0.5$ mm may be used. Tubes of length 50 mm $\pm 0.05$ mm or 10 mm $\pm 0.05$ mm or even less may be used, if necessary, for strongly coloured samples.

For determination at 20 °C or at another specified temperature, use double-walled tubes, equipped with a thermometer (5.4) to ensure water circulation at the required temperature.

For determination at ambient temperature, any type of tube may be used, although it is advisable to use the type described above in this case too.

5.4 Thermometer, graduated in 0.2 °C or 0.1 °C, allowing determination of temperatures between 10 °C and 30 °C.

5.5 Thermostatically controlled device, for maintaining the temperature of the sample at 20 °C $\pm 0.2$ °C or any other specified temperature.

6 Sampling

It is important that the laboratory receive a representative sample which has not been damaged or modified during 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.\textsuperscript{1)}

7 Procedure

7.1 Preparation of test sample

See ISO 356 if it is necessary to dry the sample.

When determining specific rotation (see 3.2), prepare the oil solution in the appropriate solvent (4.1), at the concentration specified in the corresponding appropriate International Standard for the essential oil being analysed.

7.2 Determination

Switch on the light source (5.2) and wait until full luminosity is obtained.

If necessary, bring the temperature of the test sample (7.1) to 20 °C ± 1 °C or to another specified temperature (see the note), then pour the sample into the appropriate polarimeter tube (5.3), which should be at approximately the same temperature. Start water circulation under thermostatic control so as to keep the whole at the specified temperature (± 0,2 °C) during the determination.

Fill the tube with the test sample, and ensure the absence of air bubbles.

Place the tube in the polarimeter (5.1) and read the dextrorotatory (+) or laevorotatory (−) optical rotation of the oil on the scale of the instrument.

\textbf{NOTE} As a rule, the determination will be carried out at 20 °C, exceptions to that being set out in the specifications concerning a number of particular oils.

7.3 Number of determinations

Carry out at least three determinations with the same test sample.

Take as the result the mean of the values obtained for three measurements, provided that they do not differ by more than 1,4 mrad (0,08°).

8 Expression of results

8.1 Calculation and formulae

8.1.1 Optical rotation

The optical rotation, expressed in milliradians and/or degrees of angle, is given by the equation:

\[ \alpha = \frac{A}{l} \times 100 \]

where

\[ A \] is the value of the angle of rotation (see 7.3) in milliradians and/or degrees of angle;

\[ l \] is the length of the tube used, in millimetres.

Mark as positive (+) dextrorotatory optical rotations and as negative (−) laevorotatory ones.

\textsuperscript{1)} ISO 212, Essential oils — Sampling.
When polarimeter tubes with double walls for water circulation are not available, it is necessary to apply appropriate correction factors according to the oils tested (for instance, for citrus oils and for some essential oils for which correction factors are known).

NOTE These correction factors should be given in the specifications of the oils in question.

8.1.2 Optical rotation of an oil in solution, so-called "specific rotation"

The specific rotation, expressed in milliradians and/or degrees of angle, is given by the equation:

\[
[\alpha] = \frac{\alpha_0'}{c}
\]

where

\[ \alpha_0' \] is the optical rotation of the oil solution, calculated according to 8.1.1;

\[ c \] is the concentration of the oil solution, in grams of oil per millilitre of solution.

8.2 Precision

The precision of the test method is ± 3 mrad (± 0.17°).

9 Test report

The test report shall state:

- all details necessary for the complete identification of the sample;
- the sampling method used, if known;
- the test method used, with reference to this International Standard;
- whether an oil in solution was used in the test, specifying the nature of the solvent and the concentration of the oil;
- all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the test result;
- the test result obtained;
- if repeatability has been checked, the final quoted result obtained.
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Amendments Issued Since Publication

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