SRI LANKA STANDARD 1256: PART 8: 2019
(ISO 3251: 2019)
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METHODS OF TEST FOR PAINTS AND VARNISHES
PART 8: DETERMINATION OF NON VOLATILE MATTER CONTENT
(SECOND REVISION)

SRI LANKA STANDARDS INSTITUTION

Sri Lanka Standard
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SLS 1256: Part 8: 2019
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SRI LANKA STANDARDS INSTITUTION
17, Victoria Place
Elvitigala Mawatha
Colombo - 08
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Sri Lanka Standard
METHODS OF TEST FOR PAINTS AND VARNISHES
PART 8: DETERMINATION OF NON VOLATILE MATTER CONTENT
(SECOND REVISION)

NATIONAL FOREWORD

This Standard was approved by the Sectoral Committee on Chemical and Polymer Technology and authorized for adoption and publication as a Sri Lanka Standard by the Council of the Sri Lanka Standards Institution on 2019-10-22

This Sri Lanka Standard is the Second Revision to SLS 1256: Part 8: 2008 which was an adoption of ISO 3251: 2008 Paints, varnishes and plastics- Determination of non-volatile matter content. The text of the above International Standard has been technically revised as ISO 3251: 2019 Paints, varnishes and plastics- Determination of non-volatile matter content. The International Standard ISO 3251: 2019 has been accepted for adoption as the Second Revision of SLS 1256: Part 8: 2019.

This Sri Lanka Standard is identical with ISO 3251: 2019 Paints, varnishes and plastics- Determination of non-volatile matter content, published by the International Organization for Standardization (ISO).

TERMINOLOGY AND CONVENTIONS

The text of the International Standard has been accepted as suitable for publication, without deviation, as a Sri Lanka Standard. However, certain terminology and conventions are not identical with those used in Sri Lanka Standards. Attention is therefore drawn to the following:

a) Wherever the words “International Standard” appear referring to a particular Standard they should be interpreted as “Sri Lanka Standard”.

b) The comma has been used throughout as a decimal marker. In Sri Lanka Standards it is the current practice to use the full point at the base as the decimal marker.

c) Wherever page numbers are quoted, they are ISO page numbers.

Cross References

International Standard	Corresponding Sri Lanka Standard
ISO 123, Rubber latex — Sampling	SLS 1304, Methods of testing of natural rubber latices Part 1: Sampling of latex rubber
ISO 124, Latex, rubber — Determination of total solids content	SLS 1304 Methods of testing of natural rubber latices Part 2: Determination of total solid content
ISO 1513, Paints and varnishes — Examination and preparation of test samples	SLS 1256, Methods of test for paints and varnishes Part 1: Examination and preparation of samples for testing
ISO 4618, Paints and varnishes – Terms and definitions	SLS 1541, Terms and definitions for Paints and varnishes
ISO 15528, Paints, varnishes and raw materials for paints and varnishes — Sampling	SLS 523, Methods of sampling for paints, varnishes and raw materials for paints and varnishes

SLS 1256 Part 8: 2019

INTERNATIONAL STANDARD

ISO 3251

Fifth edition
2019-05

Paints, varnishes and plastics — Determination of non-volatile-matter content

Peintures, vernis et plastiques — Détermination de la matière non volatile

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 9, *General test methods for paints and varnishes*.

This fifth edition cancels and replaces the fourth edition (ISO 3251:2008), which has been technically revised. The main changes compared to the previous edition are as follows:

— a general reference to ISO 4618 on terms and definitions has been added to Clause 3;

— the example of the desiccant in 4.5 has been changed to silica gel orange because the use of cobalt chloride as indicator is no longer allowed;

— the precision data of polymer dispersions has been corrected: the figures given in the 2008 edition were ± data which now have been converted correctly into percentages;

— the common test parameters for coating powders (powder resins) have been deleted from Table A.1 because ISO 8130-7 can be used instead;

— common test parameters for waterborne coating materials have been added to Table A.1.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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Introduction

For the method to be usable for unplasticized polymer dispersions and rubber lattices, the non-volatile residue (which consists essentially of the polymeric material and of small quantities of auxiliaries such as emulsifiers, protective colloids, stabilizers, solvents added as film-forming agents and – especially for rubber latex concentrate – preserving agents) has to be chemically stable under the test conditions. For plasticized samples, the residue, by definition, normally includes the plasticizer.

ISO 3233 (all parts) specifies test methods for determining the volume of non-volatile matter in paints, varnishes and related products.

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INTERNATIONAL STANDARD **ISO 3251:2019(E)**

Paints, varnishes and plastics — Determination of non-volatile-matter content

1 Scope

This document specifies a method for determining the non-volatile-matter content by mass of paints, varnishes, binders for paints and varnishes, polymer dispersions and condensation resins such as phenolic resins (resols, novolak solutions etc.).

The method is also applicable to formulated dispersions containing fillers, pigments and other auxiliaries (e.g. thickeners, film-forming agents).

NOTE 1 The non-volatile-matter content of a product is not an absolute quantity but depends upon the temperature and period of heating used for the determination. Consequently, when using this method, only relative and not true values for non-volatile-matter content are obtained owing to solvent retention, thermal decomposition and evaporation of low molecular mass constituents. The method is therefore primarily intended for testing different batches of the same type of product.

NOTE 2 This method is suitable for synthetic rubber lattices, provided heating for a specific period of time is considered appropriate (ISO 124 specifies heating until the loss in mass of a 2 g test portion following successive periods of heating is less than 0,5 mg).

NOTE 3 In-house methods for determining non-volatile matter often include drying with infrared or microwave radiation. Standardization of such methods is not possible, since they are not generally applicable. Several polymer compositions tend to decompose during such treatment and therefore give incorrect results.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 123, *Rubber latex — Sampling*

ISO 124, *Latex, rubber — Determination of total solids content*

ISO 1513, *Paints and varnishes — Examination and preparation of test samples*

ISO 4618, *Paints and varnishes — Terms and definitions*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 4618 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Apparatus

Ordinary laboratory apparatus, together with the following:

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4.1 Flat-bottomed dish, of metal or glass, inner diameter of base (75 ± 5) mm, height of the rim at least 5 mm, for paints, varnishes, binders for paints and varnishes and polymer dispersions.

Dishes having different diameters may be used by agreement between the interested parties. The agreed dish diameter shall be adhered to ±5 %.

For rubber latices, lipless dishes with covers are recommended.

For very viscous polymer dispersions or latices, it is recommended that aluminium foils are used which are about 0,1 mm thick, cut into rectangles of about (70 ± 10) mm × (120 ± 10) mm that can be folded in half, thus allowing the viscous liquid to be spread by gently squeezing the halves together.

4.2 Flat-bottomed dish, of metal or glass, inner diameter of base (75 ± 1) mm, height of the rim at least 5 mm, for a test portion of 3 g, for liquid crosslinking resins (phenolic resins):

For obtaining comparable film thicknesses, dishes of different diameters may be used provided the mass of the test portion m, in grams, is calculated from Formula (1):

$$m = 3 \times \left(\frac{d}{75} \right)^2 \quad (1)$$

where

d is the diameter, in millimetres, of the dish base;

3 is the nominal mass of the test portion (3 g);

75 is the nominal diameter of the dish (75 mm).

4.3 Air oven, designed to carry out the test in safe conditions, and capable of being controlled at the specified or agreed temperature (see Clause 7) ±2 °C (for temperatures up to 150 °C) or ±3,5 °C (for temperatures above 150 °C and up to 200 °C). The air oven shall be fitted with forced-ventilation equipment, except the case of phenolic resins when an oven with natural convection with a perforated metal shelf placed at one-third of the height of the oven may be used.

WARNING — To protect against explosion or fire, products containing flammable volatile substances should be handled with care.

For certain applications, drying in a vacuum might be preferable. In such cases, the conditions shall be agreed on or the method specified in ISO 124 shall be used. For referee tests, ovens of equivalent construction shall be used by all parties.

4.4 Analytical balance, capable of weighing to an accuracy of 0,000 1 g.

4.5 Desiccator, containing a suitable desiccant, for example dried silica gel orange.

5 Sampling

Take a representative sample of paints, varnishes and binders for paints and varnishes, as described in ISO 15528. Take a representative sample of polymer dispersions and rubber latices, as described in ISO 123.

Examine and prepare samples of paints and varnishes for testing, as described in ISO 1513.

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6 Procedure

6.1 Number of determinations

Carry out the determination in duplicate.

6.2 Preparation and weighing of dish

Degrease and clean a dish (4.1 or 4.2).

For better precision, it is recommended that the dish is dried in the oven (4.3) at the specified or agreed temperature for the specified or agreed period (see Clause 7) and stored in the desiccator (4.5) until used.

Determine the mass of the clean, dry dish (m_0) to the nearest 1 mg.

6.3 Weighing of samples

Weigh a test portion (see Clause 7), to the nearest 1 mg, into the dish (m_1) and distribute it evenly.

In the case of products that are highly viscous ($\nu \geq 500$ mPa · s or flow time $t \geq 74$ s measured with a 6 mm flow cup in accordance with ISO 2431) or that form skins, distribute the test portion uniformly with a tared metal wire (for example an uncoated, bent paper-clip), if necessary after the addition of 2 ml of a suitable solvent.

Condensation resins as used for paints and varnishes and other common applications (for example abrasives, friction linings, foundry binders, moulding materials) require higher test-portion masses since materials used for these applications need to be tested in thicker layers so that the monomers of the condensation resins can react during crosslinking. For comparative tests, the thickness of the layer of test portion in the dish shall be constant. Therefore the diameter of the dishes shall be (75 ± 1) mm or the formula given in 4.2 shall be used.

NOTE The non-volatile-matter content of a test portion is influenced greatly by how well and for how long the test portion is distributed in the dish. If a test portion is poorly distributed, e.g. because of high viscosity, the apparent non-volatile-matter content will be higher.

For better precision when testing paints, varnishes and binders for paints and varnishes, it is recommended that 2 ml of a suitable highly volatile solvent is always added.

It is also recommended that the dish is covered during the weighing procedure.

In the case of highly volatile products, it is recommended that a portion of the thoroughly mixed sample is placed in a stoppered bottle or, alternatively, in a weighing pipette or a 10 ml syringe without a needle.

From this, the test portion is weighed by difference, to the nearest 1 mg, into the dish and distributed evenly over the bottom of the dish.

If solvent is added, it is recommended that the dish with the test portion is allowed to stand at room temperature for 10 min to 15 min.

Aqueous systems such as polymer dispersions and rubber latices splash when heated, due to surface skinning which could also be influenced by temperature, airflow in the oven and possibly relative humidity. In such cases, the thickness of the layer of material in the dish shall therefore be kept as low as possible.

6.4 Heating

Transfer the dish to the oven, previously brought to the specified or agreed temperature (see Clause 7). Leave the dish in the oven for the specified or agreed period (see Clause 7).

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6.5 Weighing after heating

When the period of heating is completed, transfer the dish to the desiccator and allow to cool to room temperature, or optionally, place the dish in a dust-free atmosphere to cool down.

NOTE The precision of the method can be affected by not using a desiccator.

Weigh the dish and residue (m_2) to the nearest 1 mg.

7 Supplementary test conditions

For any particular application of the method specified in this document more details in addition to those in the preceding clauses might need to be given.

To enable the method to be carried out, the following test parameters shall be specified, as appropriate:

a) the test temperature;

b) the period of heating;

c) the mass of the test portion.

Common test parameters are given in Annex A for information.

8 Expression of results

Calculate the non-volatile-matter content, NV, expressed as a mass fraction, in per cent, using Formula (2):

$$\mathrm{NV} = \frac{(m_2 - m_0)}{(m_1 - m_0)} \times 100 \qquad (2)$$

where

m_0 is the mass, in grams, of the empty dish;

m_1 is the mass, in grams, of the dish with the test portion;

m_2 is the mass, in grams, of the dish with the residue.

If the two results (duplicates) differ by more than 2 % to the mean (absolute) for paints, varnishes and binders or by more than 0,5 % for polymer dispersions, e.g. if they are 53,7 % and 53,1 %, repeat the procedure described in Clause 6.

Calculate the mean of two valid results (replicates) and report the test result to the nearest 0,1 % (by mass).

9 Precision

9.1 Repeatability limit *r*

The repeatability limit *r* is the value below which the absolute difference between two single test results, each the mean of duplicates, can be expected to lie when this method is used under repeatability conditions. In such cases, the test results are obtained on identical material by one operator in one laboratory within a short interval of time using the standardized test method. In this document, *r* is

— 2 % (absolute) for paints, varnishes and binders,

— 1,2 % (absolute) for polymer dispersions,

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with a 95 % probability.

9.2 Reproducibility limit *R*

The reproducibility limit *R* is the value below which the absolute difference between two single test results, each the mean of duplicates, can be expected to lie when this test method is used under reproducibility conditions. In such cases, the test results obtained on identical material by operators in different laboratories using the standardized test method. In this document, *R* is

— 4 % (absolute) for paints, varnishes and binders,

— 2 % (absolute) for polymer dispersions,

with a 95 % probability.

10 Test report

The test report shall contain at least the following information:

a) all details necessary for complete identification of the product tested;

b) a reference to this document (ISO 3251:2019);

c) the type of dish used;

d) the type of oven used;

e) the oven temperature and the period of heating;

f) the type of solvent added (if applicable);

g) the result of the test, as indicated in Clause 8;

h) any deviation from the method specified;

i) any unsusual features (anomalies) observed during the test;

j) the date of the test.

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Annex A
(informative)

Common test parameters

Common test parameters for paints, varnishes, binders for paints and varnishes and liquid phenolic resins are listed in Table A.1. Common test parameters for polymer dispersions are listed in Table A.2.

Table A.1 — Test parameters for paints, varnishes, binders for paints and varnishes and liquid phenolic resins

Period of heating min	Temperature °C	Mass of test portion g	Examples of product classes
60	80	1,0 ± 0,1[a]	Cellulose nitrate, cellulose nitrate lacquers, polyisocyanate resins[b]
60	105	1,0 ± 0,1[a]	Cellulose derivatives, cellulose paints and lacquers, air-drying paints, polyisocyanate resins[b]
60	125	1,0 ± 0,1[a]	Synthetic resins (including polyisocyanate resins[b]), stoving (baking) paints, acrylate resins (preferred conditions)
60	150	1,0 ± 0,1[a]	Stoving (baking) priming paints, acrylate resins
30	180	1,0 ± 0,1[a]	Paints for electrocoating
120	80	1,0 ± 0,1[a]	Reactive paint systems; e.g. car refinish paints
120	130	1,0 ± 0,1	waterbased coating materials
60	135[c]	3,0 ± 0,5	Liquid phenolic resins

[a] Test portions other than 1 g may be used by agreement between the interested parties. If this is the case, a test portion of not more than (2 ± 0,2) g is recommended. For resins containing solvents with boiling points of 160 °C to 200 °C, an oven temperature of 160 °C is recommended. If even higher boiling solvents are present, the conditions shall be agreed between the interested parties.

[b] The test parameters will depend on the individual type of polyisocyanate resin under test.

[c] An alternative temperature may be used. Recommended alternative temperatures are 120 °C and 150 °C.

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Table A.2 — Test parameters for polymer dispersions

Period of heating min	**Temperature** °C	**Mass of test portion** g	**Method**[a]
120	80	1 ± 0,2[b]	A
60	105	1 ± 0,2[b]	B
60	125	1 ± 0,2[b]	C
30	140	1 ± 0,2[b]	D

[a] The conditions to be used will depend on the type of polymer dispersion or latex under test and shall be selected by agreement between the interested parties.

[b] Test portions other than 1 g may be used by agreement between the interested parties. However, the size of the test portion shall not exceed 2,5 g. Test portions of 0,2 g to 0,4 g, weighed to the nearest 0,1 mg, may also be used. In this case, the period of heating can be reduced provided it has been established (by measurements on the type of dispersion under test) that the same results are obtained as under the conditions given in this table.

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Bibliography

[1] ISO 3233 (all parts), *Paints and varnishes — Determination of percentage volume of non-volatile matter*

[2] ISO 2431, *Paints and varnishes — Determination of flow time by use of flow cups*

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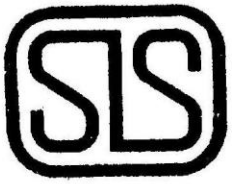

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