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

Latex de caoutchouc — Détermination des matières solides totales

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 124 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

This fifth edition cancels and replaces the fourth edition (ISO 124:1997), of which it constitutes a minor revision to incorporate the two amendments ISO 124:1997/Amd.1:2006 and ISO 124:1997/Amd.2:2008, providing precision data and permitting the use of higher drying temperatures, respectively.

Introduction

The need in commercial practice to determine solids content rapidly has led to the introduction of higher drying temperatures. This new edition recognizes this requirement while retaining the older conventional methods.

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Latex, rubber — Determination of total solids content

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This International Standard specifies methods for the determination of the total solids content of natural rubber latex concentrate and synthetic rubber latices. These methods are not necessarily suitable for latex from natural sources other than the *Hevea Brasiliensis*, for vulcanized latex, for compounded latex or for artificial dispersions of rubber.

2 Normative references

The following referenced documents are indispensable for the application 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*

3 Principle

A test portion of the latex is dried to constant mass under specified conditions, either at atmospheric pressure or under vacuum. The total solids content is determined by weighing before and after drying to constant mass.

NOTE The determination of the residue after drying for a specific period of time is the subject of ISO 3251.

4 Apparatus

Standard laboratory apparatus, plus the following:

4.1 Flat-bottomed dishes, lipless, of diameter approximately 60 mm.

4.2 Ovens, capable of being maintained at $70\text{ °C} \pm 2\text{ °C}$, or at a selected temperature between 100 °C and 160 °C accurate to $\pm 5\text{ °C}$.

4.3 Vacuum oven, capable of being maintained at $125\text{ °C} \pm 2\text{ °C}$ and at a pressure below $20\text{ kPa}^{1)}$.

4.4 Analytical balance, capable of being read to 0,1 mg.

1) $1\text{ kPa} = 1\text{ kN/m}^2$.

5 Sampling

Carry out sampling in accordance with one of the methods specified in ISO 123.

6 Procedure

6.1 General

For natural rubber latex concentrate, proceed in accordance with 6.2 and for synthetic rubber latex proceed in accordance with 6.2, 6.3, 6.4 or 6.5. Perform the determination in duplicate.

6.2 Heating at atmospheric pressure (70 °C) — Natural and synthetic rubber latex

Weigh, to the nearest 0,1 mg, a dish (4.1). Pour into the dish $2,0 \text{ g} \pm 0,5 \text{ g}$ of latex and determine the exact mass (m_0) by weighing to the nearest 0,1 mg. Gently swirl the contents of the dish to ensure that the latex covers the bottom. If desired, approximately 1 cm^3 of distilled water or water of equivalent purity may be added and mixed with the latex by swirling.

Place the dish in the oven (4.2) so that it is horizontal, and heat it at $70 \text{ °C} \pm 2 \text{ °C}$ for 16 h or until the test portion has lost its whiteness. Remove the dish from the oven and allow it to cool to ambient temperature in a desiccator. Remove the dish and weigh. Return the dish to the oven for 30 min at $70 \text{ °C} \pm 2 \text{ °C}$. Remove the dish and allow it to cool to ambient temperature in a desiccator as before and reweigh. Repeat the drying procedure for periods of 30 min until the loss in mass between two successive weighings is less than 0,5 mg. Record the mass of the dried latex (m_1).

6.3 Heating at atmospheric pressure (105 °C) — Synthetic rubber latex

Proceed in accordance with 6.2, but heat the dish containing the latex at $105 \text{ °C} \pm 5 \text{ °C}$ for 2 h or until the test portion has lost its whiteness. After allowing to cool in a desiccator and weighing, return the dish to the oven at $105 \text{ °C} \pm 5 \text{ °C}$ for 15 min. If the loss in mass is greater than 0,5 mg, repeat the drying process.

If, after heating at $105 \text{ °C} \pm 5 \text{ °C}$, the dried deposit becomes excessively sticky, repeat the determination in accordance with 6.2.

NOTE Stickiness is symptomatic of oxidation of some rubbers when exposed to air at too high a temperature.

6.4 Heating at atmospheric pressure (up to 160 °C) — Synthetic rubber latex

By agreement between the interested parties, the drying process can be carried out at temperatures up to 160 °C to shorten drying times.

NOTE The maximum drying temperature for CR latex is 130 °C , while any rubber latex except CR in Table A.1 may be dried at up to 160 °C .

Proceed in accordance with 6.2 and 6.3, but heat the dish containing the latex at for instance $130 \text{ °C} \pm 5 \text{ °C}$ for 40 min or $160 \text{ °C} \pm 5 \text{ °C}$ for 20 min (see Annex A). After allowing to cool in a desiccator and weighing, repeat the drying for periods of 10 min until the loss in mass between two successive weighings is less than 0,5 mg. In the event of a dispute over the results, drying shall be done in accordance with 6.2.

6.5 Heating at reduced pressure — Synthetic rubber latex

Weigh, to the nearest 0,1 mg, a dish (4.1). Pour into the dish $1,0 \text{ g} \pm 0,2 \text{ g}$ of latex and weigh to the nearest 0,1 mg. Add approximately 1 cm^3 of distilled water or water of equivalent purity and mix by swirling, ensuring that the latex covers the bottom of the dish.

Place the dish in the vacuum oven (4.3) so that it is horizontal. Reduce the pressure slowly to avoid foaming and splattering, and heat at 125 °C for 45 min to 60 min at a pressure below 20 kPa. Release the vacuum slowly, remove the dish from the oven and allow to cool in a desiccator. Remove the dish and weigh. Repeat the above drying procedure for periods of 15 min until the loss in mass between two successive weighings is less than 0,5 mg.

7 Expression of results

Calculate the total solids content, TSC, expressed as a percentage by mass of the latex, using the equation

$$\text{TSC} = \frac{m_1}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion before drying;

m_1 is the mass, in grams, of the final dried material.

The results of the duplicate determinations shall not differ by more than 0,2 % (mass/mass).

NOTE Over a large number of determinations, the vacuum method (6.5) tends to give marginally lower values, but does not differ by more than 0,1 %.

8 Precision

See Annex B.

9 Test report

The test report shall include the following:

- a) a reference to this International Standard;
- b) details of the drying method and temperature used;
- c) all details necessary for identification of the test sample;
- d) the average value of the results and the units in which they have been expressed;
- e) details of any unusual features noted during the determination;
- f) details of any operation not included in this International Standard or in the International Standard to which reference is made, as well as any operation regarded as optional.

Annex A (informative)

Drying conditions for synthetic latices at atmospheric pressure

A.1 Suitable drying conditions for various synthetic latices have been determined, i.e. conditions which give a constant mass. These are summarized in Table A.1. The conditions given for each latex are not to be considered as required conditions, but as recommended conditions for the measurement of total solids content.

A.2 CR (chloroprene rubber) latex should not be heated at more than 130 °C, because of possible decomposition.

Table A.1 — Drying conditions at 130 °C and 160 °C

Latex ^a	Drying time min	
	130 °C	160 °C
X-SBR	40	20
CR	30	Not applicable ^b
VP	40	20
SBR	40	20
X-SBR (with antidegradant)	40	20
NBR (with antidegradant)	40	20
X-NBR	40	20
X-NBR (with antidegradant)	40	20
X-MBR	40	20
^a "X-" means "carboxylated". ^b See Clause A.2.		

Annex B (informative)

Precision

B.1 The precision data presented in Tables B.1 and B.2 were obtained in separate inter-laboratory test programmes (ITPs) carried out at different times employing the test methods specified in 6.2 and 6.4, respectively.

B.2 The precision was determined in accordance with ISO/TR 9272. Refer to ISO/TR 9272 for terminology and other statistical details.

B.3 The precision details in this annex give an estimate of the precision of these test methods with the materials used in the particular ITP as described below. The precision parameters should not be used for acceptance/rejection testing of any group of materials without documentation that the parameters are applicable to those particular materials and the specific test protocols that include these test methods.

B.4 The precision results are given in Tables B.1 and B.2. The precision is expressed on the basis of a 95 % confidence level for the values established for repeatability r and reproducibility R .

NOTE Bias is not applicable. In test method terminology, bias is the difference between an average test value and the reference (or true) test property value. Reference values do not exist for this test method, since the value (of the test property) is exclusively defined by the test method. Bias, therefore, cannot be determined for this particular test method.

B.4.1 The results contained in Table B.1 are average values and give an estimate of the precision of this test method as determined in an ITP in 2001, in which 13 laboratories performed triplicate analyses on two samples, A and B, which were prepared from high-ammonia natural-rubber latex concentrate. The bulk latex was strained and then homogenized by thorough blending and stirring prior to being sub-sampled into 1-litre bottles labelled A and B. Thus, essentially, samples A and B were the same and treated as such in the statistical computations. Each participating laboratory was required to carry out the test using these two samples on the dates which had been given to the participants in the ITP.

B.4.2 The results contained in Table B.2 are average values and give an estimate of the precision of this test method as determined in an ITP conducted in 2004. 11 laboratories performed triplicate analyses on three materials, X-SBR-1, X-SBR-2 and CR. Each participating laboratory was required to carry out the test using these three materials, which had been given to the participants in the ITP, using the drying temperatures and times given in Table B.2.

B.5 In each case, a type 1 precision was determined, based on the sampling method used for the latex samples in the ITP in 2001 and 2004.

B.6 Repeatability: The repeatability r (in measurement units) of each test method has been established as the appropriate value tabulated in Table B.1 or B.2. Two single test results, obtained in the same laboratory under normal test conditions, that differ by more than the tabulated value of r (for any given level) should be considered to have come from different (non-identical) sample populations.

B.7 Reproducibility: The reproducibility R (in measurement units) of each test method has been established as the appropriate value tabulated in Table B.1 or B.2. Two single test results, obtained under normal test conditions, that differ by more than the tabulated value of R (for any given level) should be considered to have come from different (non-identical) sample populations.