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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.

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).

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Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

This seventh edition cancels and replaces the sixth edition (ISO 124:2011) which has been technically revised to introduce the following modifications:

— the introduction has been deleted;

— the scope has been extended to cover field latex;

— Subclause 6.1 now states the preferred method in case of dispute;

— the precision data in Annex B have been updated to cover field latex.

Latex, rubber — Determination of total solids content

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This International 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 field and concentrated latices and synthetic rubber latex. 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 documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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.[1]

4 Apparatus

Usual laboratory equipment, and in particular, the following.

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

4.2 Ovens, capable of being maintained at 70 °C ± 5 °C, 105 °C ± 5 °C, or at another selected temperature between 100 °C and 160 °C accurate to ±5 °C.

4.3 Vacuum oven, capable of being maintained at 125 °C ± 2 °C and at a pressure below 20 kPa[1)].

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

5 Sampling

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

1) 1 kPa = 1 kN/m^2.

6 Procedure

6.1 General

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

In case of dispute, heating the latex at 70 °C is the preferred method.

6.2 Heating at atmospheric pressure (70 °C and 105 °C) — Natural and synthetic rubber latex

Weigh, to the nearest 0,1 mg, a dish (4.1). Pour into the dish 2,0 g ± 0,5 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 can 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 °C ± 5 °C for 16 h, or at 105 °C ± 5 °C for 2 h, or until the test portion has lost its whiteness.

NOTE 1 The disappearance of whiteness is the first indication of dryness. Dry latex film is translucent.

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 °C ± 5 °C or 15 min at 105 °C ± 5 °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 or 15 min, as appropriate until the loss in mass between two successive weightings is less than 0,5 mg.

Record the mass of the dried latex (m_1).

If after heating at 105 °C ± 5 °C, the dried deposit becomes excessively sticky, repeat the determination at 70 °C ± 5 °C.

NOTE 2 Stickiness is symptomatic of oxidation of some rubbers when exposed to air at too high a temperature.

6.3 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 can be dried at up to 160 °C.

Proceed in accordance with 6.2, but heat the dish containing the latex at for instance 130 °C ± 5 °C for 40 min or 160 °C ± 5 °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 weightings is less than 0,5 mg.

6.4 Heating at reduced pressure — Synthetic rubber latex

Weigh, to the nearest 0,1 mg, a dish (4.1). Pour into the dish 1,0 g ± 0,2 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 mass fraction of the latex, using Formula (1):

$$TSC = \frac{m_1}{m_0} \times 100 \quad (1)$$

where

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

m_1 is the mass of the final dried material, expressed in g.

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

NOTE Over a large number of determinations, the vacuum method (6.4) tends to give marginally lower values, but does not differ by more than 0,1 % mass fraction.

8 Precision

See Annex B.

9 Test report

The test report shall include the following:

a) a reference to this International Standard (i.e. ISO 124:2014);

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 Chloroprene rubber (CR) 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 A.2.

Annex B
(informative)

Precision statement

B.1 The precision data presented in Tables B.1, B.2, and B.3 were obtained in separate interlaboratory test programmes (ITPs) carried out at different times employing the test methods specified in 6.2 and 6.3, respectively.

B.2 The precision was determined in accordance with ISO/TR 9272.[2] Refer to ISO/TR 9272[2] 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, B.2, and B.3. 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 2012, in which seven laboratories performed triplicate analyses on three samples, FL/1, FL/2, and FL/3, which were prepared from fresh field natural rubber latex. The bulk latex of each sample was homogenized and stirred prior to being sub-sampled into 1 l bottles labelled FL/1, FL/2, and FL/3. Each participating laboratory was required to carry out the test using these three 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 in 2010, in which 10 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 l 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.4.3 The results contained in Table B.3 are average values and give an estimate of the precision of this test method as determined in an ITP conducted in 2004. Triplicate analyses on three materials, X-SBR-1, X-SBR-2, and CR were performed by 11 laboratories. 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.3.

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 2004, 2010, and 2012.

B.6 The repeatability, *r* (in measurement units), of each test method has been established as the appropriate value tabulated in Table B.1, B.2, or B.3. 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 The reproducibility, *R* (in measurement units), of each test method has been established as the appropriate value tabulated in Table B.1, B.2, or B.3. Two single test results, obtained under normal test