INTERNATIONAL STANDARD

**ISO 706**

Fourth edition
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# Rubber latex — Determination of coagulum content (sieve residue)

*Latex de caoutchouc — Détermination de la teneur en coagulum (refus sur tamis)*

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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 706 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 fourth edition cancels and replaces the third edition (ISO 706:1985), Clauses 6 and 8 of which have been technically revised. In addition, a precision statement and an informative annex concerning the applicability of the standard have been added.

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# Rubber latex — Determination of coagulum content (sieve residue)

**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.**

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## 1 Scope

This International Standard specifies a method for the determination of the coagulum content (sieve residue) of natural rubber latex concentrate and the majority of synthetic rubber latices. It is not suitable for XSBR latices intended for use in paper coating (see Annex A).

## 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*

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

ISO 4576, *Plastics — Polymer dispersions — Determination of sieve residue (gross particle and coagulum content)*

ISO/TR 9272, *Rubber and rubber products — Determination of precision for test method standards*

## 3 Terms and definitions

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

**3.1**
**laboratory sample**
a quantity of latex intended for laboratory inspection and testing that is representative of the lot

**3.2**
**coagulum content**
**sieve residue**
material, comprising foreign matter and flocculated rubber, retained under the conditions of the test on a stainless-steel wire cloth with an average aperture width of 180 μm ± 10 μm, complying with ISO 3310-1

NOTE In the context of the examination of rubber shipments, bulk deliveries, etc., this is what is generally understood by "coagulum". Pieces of latex skin and gross pieces of coagulated rubber do not constitute part of the laboratory sample and are removed by the initial straining.

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## 4 Principle

A portion of the laboratory sample taken from the bulk material is strained through a coarse filter and then mixed with surfactant and strained through a wire cloth of defined mesh. The coagulum content is determined by drying the residue after washing free from latex.

## 5 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

**5.1 Anionic surfactant**, consisting of a solution of potassium oleate or ammonium laurate containing 50 g of surfactant/$dm^3$ of solution (for use with natural rubber latices).

**5.2 Non-ionic surfactant**, consisting of a solution of water-soluble ethoxylated alkylphenol containing 50 g of surfactant/$dm^3$ of solution and having a pH of 7,0 ± 0,5 (for use with synthetic rubber latices).

**5.3 Indicator paper**.

## 6 Apparatus

Standard laboratory apparatus, plus the following:

**6.1 Filter**, consisting of a sieve with a nominal aperture of 710 μm ± 25 μm, made of stainless-steel wire or synthetic-fibre cloth, resistant to latex.

**6.2 Test filter**, consisting of a disc of stainless-steel wire cloth (preferred material), with a nominal aperture of 180 μm ± 10 μm, complying with ISO 3310-1. Synthetic-fibre cloth may also be used.

If the filter requires cleaning (e.g. for re-use), immerse it in cold 5 % (by volume) nitric acid and boil for 30 min. Wash well with water and dry to constant mass.

**WARNING — This must not be done with synthetic-fibre cloth.**

**6.3 Two stainless-steel rings**, of equal internal diameter between 25 mm and 50 mm

**6.4 Beakers**, of capacity 600 $cm^3$, with a lip.

**6.5 Oven**, capable of being maintained at 100 °C ± 5 °C (see, however, the Note to 8.6).

**6.6 Desiccator**.

**6.7 Balances**: an analytical balance capable of weighing to an accuracy of 1 mg or better and a balance capable of weighing to an accuracy of 1 g.

## 7 Sampling

Prepare a laboratory sample in accordance with one of the methods specified in ISO 123. The laboratory sample shall not include any pieces of dried latex skin or any gross pieces of coagulated rubber.

## 8 Procedure

**8.1** Carry out the test in duplicate, as follows.

**8.2** Thoroughly stir the laboratory sample to ensure that it is homogeneous.

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**8.3** Strain an adequate quantity of the laboratory sample through the 710 μm filter (6.1) into a clean beaker (6.4). Cover the beaker to ensure that there is no surface drying of the latex.

**8.4** Dry the test filter (6.2) to constant mass in the oven (6.5) at 100 °C ± 5 °C (see Note to 8.6) and record the mass to the nearest 1 mg ($m_1$). Firmly clamp the test filter between the stainless-steel rings (6.3).

Weigh, to the nearest gram, 200 g ± 1 g ($m_0$) of the strained laboratory sample prepared in 8.3 into a beaker (6.4). Mix thoroughly while adding 200 $cm^3$ of anionic surfactant solution (5.1) in the case of natural rubber latex concentrate or 200 $cm^3$ of non-ionic surfactant solution (5.2) in the case of synthetic rubber latex.

Wet the clamped test filter (6.2) with the surfactant solution used (5.1 or 5.2), then pour the latex/surfactant mixture through it. Without delay, rinse out the beaker with a small amount of the surfactant solution, pouring the washings through the filter. Continue to wash the residue on the filter with small amounts of surfactant solution until the washings are clear and free of latex.

With natural rubber latex, continue to wash the residue with water until the washings are neutral to indicator paper (5.3).

With synthetic rubber latex, wash the residue with a further 200 $cm^3$ of water.

**8.5** Carefully remove the filter containing the wet coagulum from the clamp and blot the underside with filter paper.

**8.6** Dry the filter and coagulum for 30 min in the oven at 100 °C ± 5 °C (see Note), transfer to the desiccator (6.6) and allow to cool. Weigh to the nearest 1 mg. Return to the oven at 100 °C ± 5 °C (see Note) for a further period of 15 min, allow to cool in the desiccator and weigh as before. Repeat the 15 min drying period until the loss in mass between successive weighings is less than 1 mg. Record the mass of the dried filter and coagulum ($m_2$).

NOTE Other drying temperatures may be used if considered suitable (see ISO 4576).

## 9 Expression of results

The coagulum content, expressed as a percentage by mass of the latex, is given by the equation:

$$\% \text{ coagulum content} = \frac{m_2 - m_1}{m_0} \times 100$$

where

$m_0$ is the mass, in grams, of the test portion;

$m_1$ is the mass, in grams, of the test filter;

$m_2$ is the mass, in grams, of the test filter plus dried coagulum.

Report the mean of the duplicate determinations. If the individual results differ from the mean by more than 0,001 units, repeat the determination.

## 10 Precision

**10.1** The precision of this method was determined in accordance with ISO/TR 9272. Refer to this document for terminology and other statistical details.

**10.2** The precision data are given in Table 1. The precision parameters shall not be used for acceptance or rejection of any group of materials without documentation that the parameters are applicable to those particular materials and specific test protocols of the test method. The precision is expressed on the basis of a 95 % confidence level for the values established for repeatability $r$ and reproducibility $R$.

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**10.3** The results contained in Table 1 are average values and give an estimate of the precision of this test method as determined in an interlaboratory test programme carried out in 2001 and including 13 laboratories performing triplicate analyses on two samples A and B which were prepared from high-ammonia latex. Before the bulk was sub-sampled into 1 l bottles labelled A and B, it was filtered and homogenized by thorough stirring. Thus, essentially, samples A and B were the same and were treated as such in the statistical computations. Each participating laboratory was required to carry out the test using these two samples, on the dates given to them.

**10.4** A Type 1 precision was evaluated based on the method of sampling used for the interlaboratory test programme.

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

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

**10.7 Bias:** 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 cannot therefore be determined for this particular method.

**Table 1 — Precision data**

| Average result | Within laboratory | | Between laboratories | |
|---|---|---|---|---|
| | $s_r$ | $r$ | $s_R$ | $R$ |
| 0,001 | 0,000 4 | 0,001 | 0,000 5 | 0,002 |

$r = 2{,}83 \times s_r$

where

$r$ is the repeatability (in measurement units);

$s_r$ is the within-laboratory standard deviation;

and

$R = 2{,}83 \times s_R$

where

$R$ is the reproducibility (in measurement units);

$s_R$ is the between-laboratory standard deviation.

## 11 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) all details necessary for the identification of the material tested;

c) the mean of the duplicate determinations;

d) details of any unusual features noted during the test;

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e) details of any operation not included in this International Standard or in any of the International Standards to which reference is made, together with details of any operation regarded as optional;

f) the date of the test.

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