### BS ISO 2028:2015



## **BSI Standards Publication**

# Synthetic rubber latex — Preparation of dry polymer



BS ISO 2028:2015 BRITISH STANDARD

#### National foreword

This British Standard is the UK implementation of ISO 2028:2015. It supersedes BS ISO 2028:1999 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/50, Rubber - Raw, natural and synthetic, including latex and carbon black.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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## INTERNATIONAL STANDARD

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## Synthetic rubber latex — Preparation of dry polymer

Latex de caoutchouc synthétique — Préparation du polymère sec



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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 <a href="www.iso.org/directives">www.iso.org/directives</a>).

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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 fifth edition cancels and replaces the fourth edition (ISO 2028:1999), which has been technically revised with the following modifications.

- Clause 2 "Normative references" has been updated.
- In 4.1, the note giving information on which company may provide polyamine polyelectrolytes, has been deleted.

### Synthetic rubber latex — Preparation of dry polymer

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 four methods for the isolation of dry polymer from synthetic polymer latices containing anionic surfactants, for the purpose of subsequent testing. Tests to be performed include the determination of the Mooney viscosity, the bound-styrene or acrylonitrile content in the polymer chain or the mill shrinkage.

Because of the wide variety of surfactants and stabilizers used in the manufacture of synthetic rubber latices, there is not one method suitable for all latices. The methods given in this International Standard are suitable for a wide variety of latices, but their applicability is to be confirmed for individual types. Latices containing non-ionic stabilizers, in particular, may be resistant to coagulation.

These methods are not necessarily suitable, without modification, for latices containing polymers having high tack.

The dry polymer as isolated may contain residual organic acids or their aluminium salts which may affect the properties of the polymer and which are to be taken into account when performing analytical tests.

#### 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

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

#### 3 Principle

The latex is coagulated in the presence of an antioxidant, using different procedures depending on the type of latex. The resultant crumb is washed with water and dried in a ventilated oven at  $70\,^{\circ}\text{C}$  to constant mass.

NOTE Freezing is also a suitable method for the isolation of some latices; sufficient details of the method are not available for inclusion in this edition of ISO 2028.

#### 4 Reagents

During the preparation of the reagent solutions and the isolation of the polymer, use only distilled water or water of equivalent purity.

#### 4.1 Coagulant A

Coagulant A is an aqueous solution of a polyamine polyelectrolyte, prepared by dissolving 2,5 g of polyamine polyelectrolyte in 1 dm<sup>3</sup> of hot, 50 °C water and then diluting the resultant solution to 10 dm<sup>3</sup>.

NOTE Any residues of polyamine electrolyte left in the polymer following isolation are likely to interfere in the determination of bound acrylonitrile.

#### 4.2 Coagulant B

Coagulant B is a solution of aluminium sulfate in dilute sulfuric acid. Prepare dilute sulfuric acid by slowly and cautiously adding  $82~\rm cm^3$  of concentrated sulfuric acid to  $1~\rm dm^3$  of water and cooling. Separately, prepare a solution of aluminium sulfate by adding  $200~\rm cm^3$  of  $500~\rm g/dm^3$  aluminium sulfate solution to  $200~\rm cm^3$  of water and straining through a piece of gauze. Add the diluted aluminium sulfate solution to the diluted sulfuric acid and adjust the volume to  $1.5~\rm dm^3$  with water.

**4.3 Antioxidant**, for example trinonyl phenyl phosphite (TNPP).

Prepare an aqueous emulsion of antioxidant containing 25 g of antioxidant in 100 cm<sup>3</sup> of emulsion.

The antioxidant chosen should preferably be one known to be suitable for use with the polymer to be isolated.

- **4.4 Aluminium sulfate solution**, prepared by dissolving 4 g of aluminium sulfate in water and diluting the resultant solution to 100 cm<sup>3</sup>.
- **4.5 Ethanol**, minimum purity 99 %.

#### 5 Apparatus

Standard laboratory equipment plus the following.

- **5.1** Stainless-steel beaker, 5 dm<sup>3</sup> capacity.
- **5.2 Graduated cylinders**, 10 cm<sup>3</sup>, 500 cm<sup>3</sup> and 1 000 cm<sup>3</sup> capacity.
- **5.3 Electric stirrer**, with two propeller-type blades of approximately 35 mm diameter, mounted 50 mm apart at right angles to the shaft.
- 5.4 Magnetic stirrer.
- **5.5 Suitable device for injecting steam**, fed from a low-pressure source.

NOTE Steam is the most efficient method of heating liquids rapidly.

- **5.6 Gauze filter**, e.g. cheese cloth.
- **5.7 Drying pan,** aluminium or enamel-lined pan with a suitable surface area (approximately 500 cm<sup>2</sup>) on which to spread out the crumb for drying. The pan should be relatively light in weight.
- **5.8 Ventilated oven**, capable of being maintained at 70 °C  $\pm$  5 °C.
- **5.9 Balance**, capable of being read to 0.1 g.

#### 6 Procedure

#### 6.1 General

Use a test sample of the latex prepared by one of the methods described in ISO 123.

If the solids content of the latex is not known, determine it in accordance with ISO 124.

#### 6.2 Styrene-butadiene (SBR) latices (of less than 55 % solids content)

- **6.2.1** If it is not known whether or not the latex contains an antioxidant, transfer a portion of the test sample containing approximately 100 g of dry polymer to a clean, dry beaker and add  $10 \text{ cm}^3$  of the antioxidant emulsion (4.3). Stir well.
- **6.2.2** Transfer  $2 \text{ dm}^3 \pm 50 \text{ cm}^3$  of coagulant A (4.1) to a  $5 \text{ dm}^3$  stainless-steel beaker (5.1) and heat to  $65 \text{ °C} \pm 5 \text{ °C}$  by injecting steam (see 5.5).

Introduce the electric stirrer and stir at approximately 105 rad/s (1 000 r/min) while adding, in a slow stream, a quantity of latex containing approximately 100 g of dry polymer with antioxidant (see <u>6.2.1</u>).

Continue stirring for 1 min to ensure uniform mixing, then slowly add 15 cm $^3$  of coagulant B (4.2) dropwise. If complete precipitation is not achieved with the formation of fine crumbs and a clear serum, add a further 1 cm $^3$  of coagulant B.

**6.2.3** Strain through a clean gauze ( $\underline{5.6}$ ) and wash the residue of precipitated rubber particles with water at 50 °C ± 5 °C for 120 s ± 15 s.

Allow the gauze to drain, then wrap it around the precipitated rubber and transfer it to a beaker containing water at 40  $^{\circ}$ C ± 5  $^{\circ}$ C.

Allow to soak for a further  $120 \text{ s} \pm 15 \text{ s}$  while holding the gauze under the water and squeezing gently with the fingers.

Remove the gauze containing the crumbs from the water and squeeze to eliminate as much water as is practical.

**6.2.4** Transfer the damp polymer crumbs to a suitable pan (5.7) lined with a double layer of clean gauze and ensure that they are well spread out over the whole surface area.

Dry in a ventilated oven (5.8) at 70 °C ± 5 °C for 5 h. Using a balance (5.9), weigh the container and dried crumbs. Record the mass to 0.1 g.

Return the container to the oven for a further 1 h and re-weigh. Continue to dry in the oven for 1 h periods until the loss in mass between successive weightings is less than 0,5 g.

#### 6.3 Carboxylated (X-SBR and X-NBR) latices

- **6.3.1** If it is not known whether or not the latex contains an antioxidant, transfer a portion of the test sample containing approximately 100 g of dry polymer to a clean, dry beaker and add  $10 \text{ cm}^3$  of the antioxidant emulsion (4.3). Stir well.
- **6.3.2** Take a portion of latex containing approximately 100 g of dry polymer with antioxidant (see <u>6.3.1</u>) in a 1 dm<sup>3</sup> beaker and add  $500 \text{ cm}^3$  of water. Stir well. Proceed as described in <u>6.2.2</u>, <u>6.2.3</u> and <u>6.2.4</u>.

#### 6.4 Nitrile (NBR) latices

- **6.4.1** If it is not known whether or not the latex contains an antioxidant, transfer a portion of the test sample containing approximately 100 g of dry polymer to a clean, dry beaker and add  $10 \text{ cm}^3$  of the antioxidant emulsion (4.3). Stir well.
- **6.4.2** Transfer 2 dm<sup>3</sup> of the diluted aluminium sulfate solution (4.4) to a 5 dm<sup>3</sup> stainless-steel beaker (5.1) and raise the temperature to 65 °C  $\pm$  5 °C by passing in steam (see 5.5).

Remove the steam injector and introduce the electric stirrer (5.3). Stir at approximately 105 rad/s (1 000 r/min) while adding, in a slow stream, a portion of the latex containing approximately 100 g of dry polymer together with antioxidant (see 6.4.1).

Continue stirring for a further 5 min, then proceed as described in 6.2.3 and 6.2.4.

#### 6.5 Concentrated latices (more than 55 % solids content)

- **6.5.1** If it is not known whether or not the latex contains an antioxidant, transfer a 200 g portion of the test sample to a clean, dry beaker and add  $10 \text{ cm}^3$  of the antioxidant emulsion (4.3). Stir until thoroughly mixed.
- **6.5.2** Transfer 2 dm<sup>3</sup> of ethanol (4.5) into a 5 dm<sup>3</sup> stainless-steel beaker equipped with a magnetic stirrer (5.4). While stirring slowly, add, in a steady stream, 200 g  $\pm$  5 g of the latex containing antioxidant (see 6.5.1).

Continue stirring until the latex has completely precipitated and the serum is clear. Continue as described in 6.2.3 and 6.2.4.

#### 7 Report

The report shall include the following information:

- a) a reference to this International Standard, i.e. ISO 2028;
- b) all details necessary for complete identification of the test sample;
- c) whether or not an antioxidant was added, and if so its identity;
- d) which method (i.e. <u>6.2</u>, <u>6.3</u>, <u>6.4</u> or <u>6.5</u>) was used;
- e) any unusual features noted during the test;
- f) full details of any operation not included in this International Standard or in any of the International Standards to which reference is made, together with any operation regarded as optional;
- g) the date of the test.





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