INTERNATIONAL STANDARD

ISO 2028

Fourth edition
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Synthetic rubber latex — Preparation of dry polymer

Latex de caoutchouc synthétique — Préparation du polymère sec

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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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 2028 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 2028:1989), which has been technically revised.

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 that may 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 that are used in the manufacture of synthetic rubber latices, no one method is suitable for all latices. The methods given in this International Standard are suitable for a wide variety of latices, but their applicability should 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.

It should be noted that the dry polymer as isolated may contain residual organic acids or their aluminium salts which may affect the properties of the polymer. This should be taken into account when performing analytical tests.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 123:—[1)], *Rubber latex — Sampling*.

ISO 124:1997, *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 °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.

1) To be published. (Revision of ISO 123:1985)

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: An aqueous solution of a polyamine polyelectrolyte, prepared by dissolving 2,5 g of polyamine polyelectrolyte in 1 dm^3 of hot, 50 °C water and then diluting the resultant solution to 10 dm^3.

NOTE 1 Suitable polyamine polyelectrolytes include “Prodefloc CRC 300C” (CAS No. 42751-79-1) available from Industrie Chimiche Caffara SpA, Casella postale 327, 25100 Brescia, Italy, and “EPI DNA” available from AcqueNYMCO SpA, 20032 Cormano MI, Via dei Giovi 6, Italy, Fax 66301278.

NOTE 2 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: A solution of aluminium sulfate in dilute sulfuric acid. Prepare dilute sulfuric acid by slowly and cautiously adding 82 cm^3 of concentrated sulfuric acid to 1 dm^3 of water and cooling. Separately, prepare a solution of aluminium sulfate by adding 200 cm^3 of 500 g/dm^3 aluminium sulfate solution to 200 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 dm^3 with water.

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

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

Prepare an aqueous emulsion of antioxidant containing 25 g of antioxidant in 100 cm^3 of emulsion.

4.4 Aluminium sulfate solution, prepared by dissolving 4 g of aluminium sulfate in water and diluting the resultant solution to 100 cm^3.

4.5 Ethanol, minimum purity 99 %.

5 Apparatus

Standard laboratory equipment plus the following:

5.1 Stainless-steel beaker, 5 dm^3 capacity.

5.2 Graduated cylinders, 10 cm^3, 500 cm^3 and 1 000 cm^3 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: An aluminium or enamel-lined pan with a suitable surface area (approximately 500 cm^2) on which to spread out the crumb for drying. It is desirable that the pan should be relatively light in weight.

5.8 Ventilated oven, capable of being maintained at 70 °C ± 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 cm^3 of the antioxidant emulsion (4.3). Stir well.

6.2.2 Transfer 2 $dm^3 \pm 50$ cm^3 of coagulant A (4.1) to a 5 dm^3 stainless-steel beaker (5.1) and heat to 65 °C ± 5 °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 6.2.1). 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 (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 °C ± 5 °C. Allow to soak for a further 120 s ± 15 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 weighings 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 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 6.3.1) in a 1 dm^3 beaker and add 500 cm^3 of water. Stir well. Proceed as described in 6.2.2, 6.2.3 and 6.2.4.

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 cm^3 of the antioxidant emulsion (4.3). Stir well.

6.4.2 Transfer 2 dm^3 of the diluted aluminium sulfate solution (4.4) to a 5 dm^3 stainless-steel beaker (5.1) and raise the temperature to 65 °C ± 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 cm^3 of the antioxidant emulsion (4.3). Stir until thoroughly mixed.

6.5.2 Transfer 2 dm^3 of ethanol (4.5) into a 5 dm^3 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;

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. 6.2, 6.3, 6.4 or 6.5) 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.