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Plastics/rubber — Polymer dispersions and synthetic rubber latices — Freeze-thaw cycle stability test

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*Plastiques/caoutchouc — Dispersions de polymères et latex de caoutchouc
synthétique — Essai de stabilité à des alternances de gel et de dégel*

ISO 1147:1995

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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.

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.

International Standard ISO 1147 was prepared by Technical Committee ISO/TC 61, *Plastics*, in collaboration with ISO/TC 35, *Paints and Varnishes*, ISO/TC 45, *Rubber and rubber products*, and CEN/TC 193, *Adhesives*.

This third edition cancels and replaces the second edition (ISO 1147:1988), which has been technically revised.

ISO 1147:1995

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Plastics/rubber — Polymer dispersions and synthetic rubber latices — Freeze-thaw cycle stability test

1 Scope

This International Standard specifies a procedure for the evaluation of the freeze-thaw cycle stability of polymer dispersions and synthetic rubber latices. It does not apply to natural rubber latex.

The test has no significance if the sample does not freeze under the test conditions.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 2555:1989, *Plastics — Resins in the liquid state or as emulsions or dispersions — Determination of apparent viscosity by the Brookfield Test Method*.

ISO 3219:1993, *Plastics — Polymers/resins in the liquid state or as emulsions or dispersions — Determination of viscosity using a rotational viscometer with defined shear rate*.

ISO 12000:—¹⁾, *Polymer dispersions and rubber latices (natural and synthetic) — Definitions and review of test methods*.

3 Principle

The viscosity of a sample of the polymer dispersion under test is measured. The sample is placed in a test chamber for 16 h at a temperature of -5°C , -10°C or -20°C , then held for 8 h at 23°C . Finally, the appearance of the polymer dispersion is checked. If coagulation has occurred, the test is considered to be completed.

In cases of doubt, the viscosity is remeasured. If it has changed by more than the manufacturer's specification limit, then the test is considered to be completed. If it has not changed by more than the manufacturer's specification limit, the cycle is repeated, i.e. the sample is stored at the selected temperature for another 16 h and then held at 23°C for 8 h, up to a maximum number of three cycles, the viscosity of the dispersion being remeasured after each cycle.

NOTE 1 The freeze temperature selected will depend on the chemical nature of the polymer dispersion and the temperature to which it is exposed in service. If required, preliminary tests may be carried out to determine the appropriate test temperature.

4 Apparatus

4.1 Cylindrical container, with a large opening fitted with a stopper, and with the following dimensions:

— height: 100 mm

— inside diameter: 40 mm

— wall thickness: 2 mm

1) To be published.

NOTE 2 It is important not to deviate from these dimensions as the results are sensitive to the size of the container.

At freeze temperatures of $-5\text{ }^{\circ}\text{C}$ or $-10\text{ }^{\circ}\text{C}$, the container may be made of polyethylene; at lower freeze temperatures a stainless-steel container shall be used.

4.2 Test chamber, comprising a suitable cabinet, room or enclosure large enough to contain the samples to be tested, allowing at least 25 mm of air space between the sides of adjacent containers and capable of being maintained at temperatures of $-5\text{ }^{\circ}\text{C} \pm 0,5\text{ }^{\circ}\text{C}$, $-10\text{ }^{\circ}\text{C} \pm 0,5\text{ }^{\circ}\text{C}$ and $-20\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$.

4.3 Thermometer or thermocouple, capable of measuring the temperature inside the test chamber to the nearest $0,5\text{ }^{\circ}\text{C}$.

4.4 Viscometer, as specified in ISO 3219 or ISO 2555, as appropriate, to determine the viscosity of the polymer dispersion.

5 Procedure

NOTE 3 By agreement between the interested parties, a property other than viscosity may be used for the test.

5.1 Put $100\text{ g} \pm 1\text{ g}$ of test sample into the cylindrical container (4.1).

Measure the viscosity of the sample at $23\text{ }^{\circ}\text{C}$ in accordance with ISO 3219 or ISO 2555, and note the result.

5.2 Stopper the container and place it for 16 h in the test chamber (4.2) set at the selected freeze temperature kept constant to within $\pm 0,5\text{ }^{\circ}\text{C}$.

5.3 Remove the container from the test chamber and let it thaw at $23\text{ }^{\circ}\text{C}$ for 8 h.

5.4 Check the condition of the dispersion by inserting a glass rod. If there is complete coagulation or the formation of clots of coagulum that cannot be dispersed by stirring, regard the test as completed and record the appearance of the dispersion.

If this check is not conclusive, measure the viscosity of the sample at $23\text{ }^{\circ}\text{C}$ as described in 5.1. If the viscosity has changed from its initial value by more than the manufacturer's specification limit, regard the test as completed. If not, subject the dispersion to a further complete freeze-thaw cycle.

5.5 Homogenize the sample if necessary, e.g. if the sample shows a water layer on the surface or has thickened.

5.6 If necessary, continue the test until three freeze-thaw cycles have been completed, remeasuring the viscosity of the sample at $23\text{ }^{\circ}\text{C}$ after each cycle.

5.7 Note the freeze temperature used, the appearance of the sample after each cycle, the number of cycles completed without formation of visible, indispersable coagulum or clots (or without a change in viscosity of more than the manufacturer's specification limit), the initial viscosity and the final viscosity.

5.8 In the special case of dispersions in which the polymer tends to crystallize [for example poly(vinylidene chloride)], an additional check may be carried out by verifying whether the aptitude for film formation is still good after the freeze-thaw cycles.

6 Expression of results

The freeze-thaw stability of the polymer dispersion at a selected freeze temperature is expressed as the number of cycles endured without the formation of visible and indispersable coagulum or clots or without a change in viscosity exceeding the manufacturer's specification limit.

NOTE 4 Failure to meet either of these requirements does not necessarily mean that the polymer dispersion will be unsuitable for a particular application.

7 Test report

The test report shall include the following particulars:

- a reference to this International Standard;
- all details necessary for complete identification of the product tested;
- the freeze temperature used;
- the freeze-thaw stability at this freeze temperature, expressed as the number of cycles endured without coagulation;
- the method used to measure the viscosity;
- the initial and final viscosities of the polymer dispersion;
- details of any modification to the procedure described and details of any circumstances which may have affected the results;
- if the aptitude for film formation was verified, the method employed and the result obtained;
- the date of the test.