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Plastics — Polymer dispersions — Freeze-thaw cycle stability test

Plastiques — Dispersions de polymères — Essai de stabilité à des alternances de gel et de dégel

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Reference number ISO 1147: 1988 (E)

## **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 approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 1147 was prepared by Technical Committee ISO/TC 61, Plastics.

This second edition cancels and replaces the first edition (ISO 1147: 1975), of which it constitutes a minor revision.

# Plastics — Polymer dispersions — Freeze-thaw cycle stability test

#### 1 Scope

This International Standard specifies a procedure for the evaluation of the freeze-thaw cycle stability of aqueous dispersions of polymers and copolymers.

The freezing temperature is normally -10 °C but in special cases a lower temperature can be used.

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

The procedure is suitable for all aqueous polymer and copolymer dispersions.

#### 2 Principle

Placing of a sample of the dispersion being tested in a refrigerator at -10 °C for 16 h, then holding it at room temperature (about 20 °C) for 8 h.

Checking of the condition of the dispersion; if there is no coagulum, repetition of the freeze-thaw cycle until it appears, up to a maximum of five cycles.

Freeze-thaw cycle stability is indicated by the number of cycles endured.

### 3 Apparatus

- **3.1 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

It may be made of polyethylene.

- **3.2** Refrigerator, the temperature of which can be controlled at -10 °C  $\pm$  0,5 °C.
- 3.3 Laboratory balance, accurate to the nearest 0,5 g.

#### 4 Procedure

**4.1** Put 100 g  $\pm$  1 g of test sample into the cylindrical container (3.1). Stopper the container and place it in the refrigerator (3.2), set at -10 °C  $\pm$  0,5 °C, for 16 h.

- **4.2** Remove the container from the refrigerator and let it thaw at room temperature (about 20 °C) for 8 h.
- **4.3** Check the condition of the dispersion by insertion of a glass rod. If there is complete coagulation or clots of coagulum that cannot be dispersed by stirring, the test is regarded as completed.

If this test is not conclusive, expose the dispersion to a further complete freeze-thaw cycle.

- **4.4** If necessary, continue the test until five freeze-thaw cycles are completed.
- **4.5** Note the number of cycles completed without coagulation or clotting.
- **4.6** In the special case of dispersions in which the polymer tends to crystallize (for example vinylidene chloride), the test may be completed subsequently by checking whether the aptitude for film formation is still good after the freeze-thaw cycles.
- **4.7** In special cases, the test may be repeated with similar test conditions but at different freezing temperatures.

#### 5 Expression of results

The freeze-thaw cycle stability of the dispersion is expressed as the number of freeze-thaw cycles endured without coagulation.

Under the specifications for the test, the maximum stability is 5 (see 4.4).

NOTE - The results may not be applicable to larger-volume vessels.

#### 6 Test report

The test report shall include the following particulars:

- a) a reference to this International Standard;
- b) complete identification of the product under test;
- c) freeze-thaw stability at -t °C expressed as the number of cycles endured without coagulation;
- d) the freezing temperature  $(-t \, {}^{\circ}C)$ ;
- e) if the aptitude for film formation has been verified, indication of the result and the method employed.