



## Plastics — Aqueous dispersions of polymers and copolymers — Freeze-thaw cycle stability test

~~Matières~~ **P**lastiques — Dispersions aqueuses de polymères et copolymères — Essai de stabilité à des alternances de gel et de dégel

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**Descriptors :** plastics, polymers, copolymers, dispersions, tests, physical tests, thermal cycling tests, freeze-thaw resistance.

## FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

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.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 61 has reviewed ISO Recommendation R 1147 and found it technically suitable for transformation. International Standard ISO 1147 therefore replaces ISO Recommendation R 1147-1969 to which it is technically identical.

ISO Recommendation R 1147 was approved by the Member Bodies of the following countries :

Austria	Iran	South Africa, Rep. of
Belgium	Israel	Spain
Brazil	Italy	Sweden
Czechoslovakia	Japan	Turkey
Egypt, Arab Rep. of	Korea, Rep. of	United Kingdom
France	Netherlands	U.S.A.
Germany	Poland	U.S.S.R.
Hungary	Portugal	
India	Romania	

No Member Body expressed disapproval of the Recommendation.

The Member Body of the following country disapproved the transformation of ISO/R 1147 into an International Standard :

Canada

# Plastics — Aqueous dispersions of polymers and copolymers — Freeze-thaw cycle stability test

## 1 SCOPE AND FIELD OF APPLICATION

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  $-10^{\circ}\text{C}$  but in special cases a lower temperature can be used.

The test has no significance if the sample is not frozen 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  $-10^{\circ}\text{C}$  for 16 h, then holding it at room temperature (about  $20^{\circ}\text{C}$ ) 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**, of constant diameter and fitted with a stopper, having the following dimensions :

- height : 100 mm
- inside diameter : 40 mm
- thickness : 2 mm

It may be made of "high density" polyethylene.

**3.2 Refrigerator** with temperature control for  $-10 \pm 0,5^{\circ}\text{C}$ .

**3.3 Laboratory balance**, accurate to the nearest 0,5 g.

## 4 PROCEDURE

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

**4.2** Remove the container from the refrigerator and let it thaw at room temperature (about  $20^{\circ}\text{C}$ ) 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 further complete freeze-thaw cycles.

**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 by the number of freeze-thaw cycles endured without coagulation.

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

## 6 TEST REPORT

The test report shall include the following particulars :

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