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Draft Indian Standard

METHODS OF TEST FOR ENVIRONMENTAL STRESS-CRACK RESISTANCE OF BLOW-MOULDED POLYETHYLENE CONTAINERS

(*First Revision* of IS 8747)

(ICS 83.080)

Plastics packaging Sectional Committee, PCD 21

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FOREWORD

(Formal clause will be added later**)**

This Indian Standard was originally published in 1977. In this revision, editorial mistakes have been rectified.

With the growth of new technologies conventional containers such as metal and glass, are being replaced fast by blow-moulded polyethylene containers for various industrial applications. Estimation of shelf-life of plastics containers is one of the important considerations in their performance evaluation. Results of the container resistance to environmental stress cracking can be used for estimating their shelf-life.

Environmental stress cracking of blow-moulded containers is governed by many factors. It is summation of the influence of container design, resin, blow-moulding conditions and post treatment.

In this standard, two methods have been given for the evaluation of stress-crack resistance of the blow-moulded containers. Method I gives the suitability of the actual container for the storage of the product to be packed into it and is to be carried out by the processor, whereas Method II is useful for determining the suitability of resin on the stress-crack resistance of the container. Method II pertains to the raw material and should be carried out by the raw material manufacturer. A certificate from the raw material manufacturer stating that the raw materials, namely HDPE granules and the resin, fulfil the requirements of the method will be acceptable.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’.

1 SCOPE

1.1 This standard prescribes the methods of test for environmental stress-crack resistance under specified conditions of stress and in the presence of environments, such as soaps, wetting agents, oils or detergents.

2 TERMINOLOGY

2.1 For the purpose of this standard, the following definition shall apply.

2.1.1 *Failure* **—** During this test, the formation of any imperfection, such as a crack, which results in a loss of pressurizing gas or stress-cracking agent.

A container has failed when:

a) it has lost pressure through any aperture other than heat-seal areas; or in procedure, when there is a detectable flow of supply air into the bottle;
b) when there is a crack completely through the container wall which is visible to an observer with normal eyesight; and
c) there is evidence of the contained liquid on the outside of the container through any aperture other than one at the heat-sealed area, or the contained liquid volume has been reduced.

3 TEST METHODS

3.1 Two test methods are prescribed in the standard to determine the effect of each of the factors influencing the stress-crack resistance.

3.2 Method I — Consists of exposing any filled blow-moulded container to the action of a potential stress-cracking agent within the container at an elevated temperature. The time of failure is observed. This method is useful for determining the effect of container design on stress-crack resistance.

3.2 Method II — Consists of exposing a partly filled and sealed blow moulded standard container to the action of poly-oxyethylated nonyl-phenol (PONP), a stress-cracking agent, within the container, as well as to the action of this agent as an external environment, at an elevated temperature. The time of failure is observed. This method is useful for determining the effect of resin on the stress-crack resistance of the container.

4 APPARATUS

4.1 For Methods I and II

4.1.1 *Circulating Air-Oven* — Capable of maintaining a temperature of 60 ± 1 °C and an air-flow rate of 8.5 to 17 m^3/ min.

CAUTION — A high-temperature safety switch is highly recommended on this oven. Some test liquids can cause extreme pressure build-up upon heating. Under these conditions bottles can rupture with explosive

force. This condition can cause injury to the operator as well as damage to the oven. The override cut-off switch should be set to turn off the oven heat if the test temperature is exceeded by as much as 10 ºC.

4.1.2 *Balance* — Accurate to within ± 1.0 g (for weighing containers and contents) or a volumetric filling apparatus accurate to ±1 ml.

4.1.3 *Heat-Seal Laminate* — For sealing the containers.

4.1.4 *Heat-Sealing Unit*

4.1.5 *Torque Meter*

4.1.6 *Glass Beakers* — large enough to hold the contents of one test container.

4.2 Polyethylene Bags for Method I Only

Approximately 0.038 mm thick, large enough to enclose completely a test container. The bag should fit loosely around the container and be long enough so that the bag opening can be closed above the container closure.

5 REAGENTS

5.1 For Method I

5.1.1 Any environmental stress-cracking agent.

5.2 For Method II

5.2.1 *Poly-Oxyethylated Nonyl-Phenol* — A stress-cracking agent.

NOTE — Poly-oxyethylated nonyl-phenol is hygroscopic and the undiluted agent should be kept tightly stoppered.

5.2.2 *Poly-Oxyethylated Nonyl-Phenol Solution or Teepol Grade B — 300* — Prepare a 10 percent solution, by volume, of the stress-cracking agent in distilled water in sufficient volume to fill a minimum of fifteen 500 ml containers to one-third of overflow capacity (170 ml).

NOTE — Due to the viscosity of the stress-cracking agent, it has been found to be helpful to prepare the solution at an elevated temperature. A temperature of 50 °C has been found suitable.

5.2.3 *Dye Indicator Solution* — Add 0.1 percent by mass of a wetting agent to distilled water. Dissolve 0.00l percent by mass of gentian violet in water.

NOTE — Since only about 0.1 ml of this solution is added to each bottle, only a small volume is needed.

6 SAFETY PRECAUTIONS

6.1 Proper precautions should be taken to prevent overheating of the containers during testing since some products which may be tested by Method I may cause an extreme pressure build-up in the container and could cause the container to rupture explosively. Proper safety measures against overheating are described in **4.1.l.**

6.2 Sometimes container will fail due to a small pinhole. Since the container is under pressure during the test, liquid may be forced out of the opening, spraying the inside of the over and the operator, if an inspection is being made. Precautions to prevent this from happening are describing in **9.1.4.**

6.3 Care should be taken in handling the stress cracking agent since there are some possibilities of its causing dermatitis.

7 TEST SPECIMEN

7.1 For Method I — A minimum of 15 blow-moulded containers, representative of the lot to be tested, and each fitted with a screw closure affording a leak-proof seal shall be selected.

7.2 For Method II — A standard blow-moulded container shall be used for this test. It is a 500 ml cylindrical bottle weighing approximately 20 g, as shown in Fig. 1. A minimum of 15 containers shall be selected as in **7.1**. The minimum wall thickness of the container shall be not less than 0.30 mm. The pinch-off area of the container shall not extend into the chime radius.

8 CONDITIONING

8.1 Conditioning

The test specimens shall be conditioned at 27 ± 2 °C and 65 ± 5 percent relative humidity for 24 h.

8.2 Test Conditions

Conduct tests in the atmosphere of the standard conditions specified in **8.1.**

9 PROCEDURE

9.1 For Method I

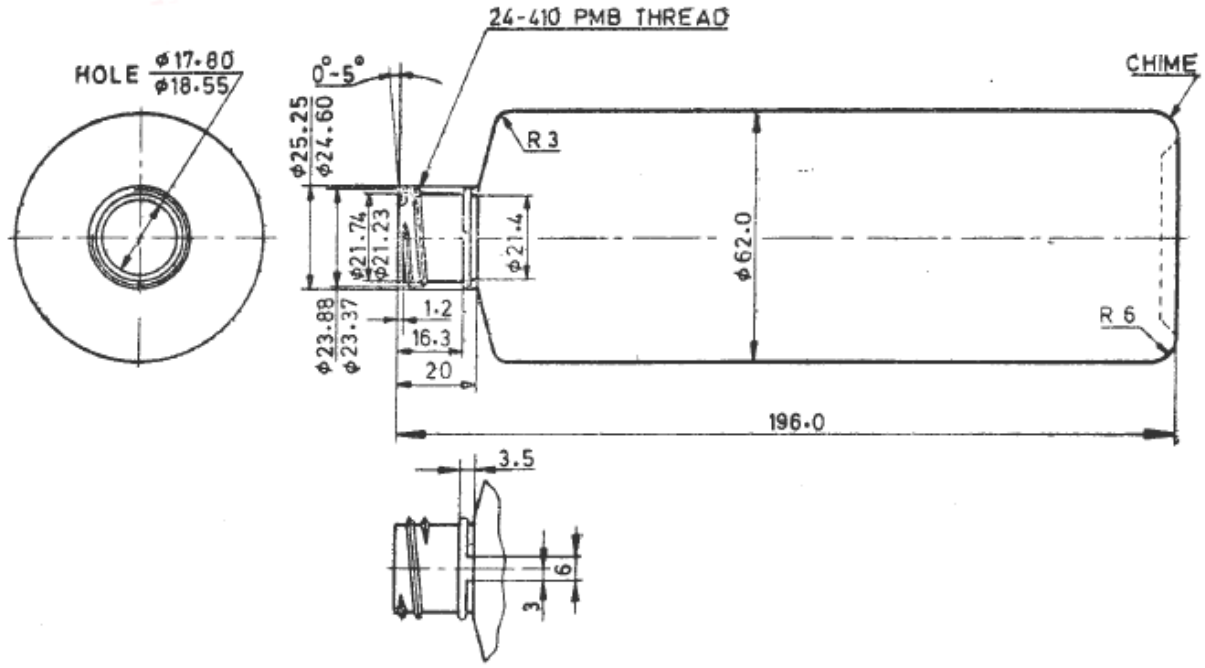
9.1.1 Fill a minimum of 15 containers to nominal capacity with test liquid that has not been used previously.

9.1.2 Heat-seal the containers with a suitable triple laminate polyethylene side to bottle. Apply a polyethylene or suitably lined closure with sufficient torque to ensure a double seal.

NOTE — The pressure applied during sealing should be a minimum to ensure that deformation of the container takes place. The container should not be handled in such a manner as to deform the walls during sealing. Any deformation of the container during sealing may result in a volume change which will affect the

final test pressure. An application torque of 1.7 N.m has been found sufficient for the standard container described in Fig. 1.

9.1.3 After sealing, invert the containers to coat the inside with the agent.



All dimensions in millimetres

FIG. 1 STANDARD 500 ml BLOWN CONTAINER

9.1.4 Place each container in a plastic bag and close the bag opening above the closure by folding or by means of a rubber band, string, tape etc. Do not heat-seal the bag.

CAUTION — A plastic bag is used to protect the other containers on test from the possibility of one container failing and spraying the other containers. It also protects the operator during inspection of the containers. Volatile liquids may cause safety hazards when the container is kept in an oven at 60 °C for testing.

9.1.5 Place the containers in a vertical position with the finish up, in beakers, in the oven at the test temperature of 60 ± 1 °C. Check the temperature periodically for constancy.

9.1.6 Inspect the containers for environmental stress-crack failure hourly for the first 8 h and thereafter at least once each 24 h. Remove containers that fail and record for each failure exposure time, position of failure with relation to mould number or parting line and type of failure.

9.1.6.1 It is not necessary to remove the plastic bag to inspect the containers. Failures are easily detectable with the bag in place.

9.1.6.2 During each inspection for failures, the bottles remaining on test may be moved in a random manner to new positions in the oven to eliminate any effect due to a static oven temperature profile, if one does exist.

9.1.7 Continue exposure of non-failures until all fail, or to a maximum of 360 h and record the number of containers still under test at that time.

9.2 For Method II

9.2.1 Fill a minimum of 15 containers to one-third of overflow capacity (180 ml) with the stress cracking solution described in **5.2.2**.

9.2.2 Put approximately 0.1 ml of the dye solution described in **5.2.3** in each container.

9.2.3 Heat-seal and invert the filled containers as in Method I (*see* **9.1.2** and **9.1.3**).

9.2.4 Place the containers in the oven at the test temperature of 60 ± 1 °C in a vertical position with the finish up, in beakers containing sufficient undiluted stress-cracking agent (**5.2.1**) to cover the chime area of the container. Check the temperature periodically for constancy.

9.2.5 Inspect containers for failure to a maximum exposure time of 360 h as detailed in Method I (**9.1.6** and **9.1.7**).

10 CALCULATION

10.1 Calculate the percentage of the containers that have failed at any given time by the equations in **10.1.1** or in **10.1.2** depending upon the number of containers tested and upon the frequency of inspection.

10.1.1 For 30 or more containers tested or where inspection for failures are made only once every 24 h after the first 8 h or both:

$$\text{Failures, percent} = (n/N) \times 100$$

where

n = cumulative number of containers that have failed at the given time, and
N = number of containers tested.

10.1.2 In all cases other than **10.1.1,** use $N + 1$; that is, where inspection for failures are made more frequently than regular 24 h intervals after the first 8 h, determine the percentage of the containers that have failed at any given time by the equation in **10.1.1**, except that the divisor is $N + 1$ instead of N.

10.2 F50 Failure Time

10.2.1 Plot the data on log probability graph paper with hours on the log scale and percentage failure on the probability scale. Draw the best fitting straight line for the plot. The hours indicated at the intersection of the data line with the 50 percent failure level probability line shall be reported as the F50 failure time.

10.2.2. At least one half of the containers must have failed before an F50 value can be reported.

10.3 If extremes of the distribution need to be studied, additional testing will be necessary.

11 REPORT

11.1 The report shall include the following:

a) Method used (Method I or II);
b) Complete identification and description of the containers tested, including base resin, blow-moulding conditions, colouring system, weight, any unusual material distribution, description of geometry and any other available information;
c) Description of the liquid used;
d) Number of bottles tested;
e) The time of the first observed failure Fi;
f) The estimated time at which 50 percent of the containers failed as determined from the plot described in **10.2.1** calculation (F50);
g) The observed time in hours at which 100 percent of the containers, failed (F 100);
h) If all of the containers have not failed at the end of 360 h, report F100 as 360 hours;
j) Locations and types of failures; and
k) Any unusual occurrences noted during testing.