INTERNATIONAL SOCIETY of BEVERAGE TECHNOLOGISTS

Test Method for Evaluating the Relative Stress Crack Resistance of Poly(ethylene terephthalate) Carbonated Soft Drink Bottles

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ISBT Document: *PTC-Stress Crack Test Methods 031207*

This test method is intended only for the qualification of bottles as regards new or modified base, preform and bottle designs, new or modified resins and resin additives, and any other change that may impact the stress crack resistance of the bottle in the market. The test is sensitive to procedural variations, and must be conducted under carefully controlled laboratory conditions exactly as specified in this method.

It is not recommended as a tool for routine quality control or quality assurance.

Complete English-language documents may be obtained online from the ISBT at www.bevtech.org.

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INTRODUCTION

This document has been prepared to provide guidelines to manufacturers of PET container, Closures and Filling industry personnel to identify key characteristics for maintaining the quality of both preforms and bottles. Individual needs, however, may dictate the application of other requirements negotiated between Consumer Product companies and suppliers of preform/containers.

The International Society of Beverage Technologists does not warrant the efficacy, accuracy or completeness of these guidelines.

Test Method for Evaluating the Relative Stress Crack Resistance of Poly(ethylene terephthalate) Carbonated Soft Drink Bottles Using Sodium Hydroxide

1. Scope
   1.1. This test method describes a procedure for determining the stress crack resistance of blow-molded polyethylene terephthalate (PET) carbonated soft drink bottles by measuring the propensity for base cracking upon exposure to an aqueous sodium hydroxide solution.
   1.2. This test method is designed for use on bottle bases with a "footed" design.
   1.3. This test method is intended to be used as a comparative test; i.e., one which compares the performance of various bottle characteristics with that of an appropriate standard.
   1.4. *This test method is intended only for the qualification of bottles as regards new or modified base, preform and bottle designs, new or modified resins and resin additives, and any other change that may impact the stress crack resistance of the bottle in the market. The test is sensitive to procedural variations, and must be conducted under carefully controlled laboratory conditions exactly as specified in this method. It is not recommended as a tool for routine quality control or quality assurance*.

2. Summary of Test Method
   2.1. This test method involves an evaluation of the resistance of PET bottle bases to a sodium hydroxide solution as the stress crack initiator. The bases of test bottles are exposed to a standard aqueous sodium hydroxide solution under a controlled environment. When the test bottles and the standard bottles are simultaneously tested the failure rate of the test bottles can be compared. Judgments can then be made as to the relative stress crack resistance of the test bottles.

3. Significance
   3.1. The hydroxide ion is a major catalyst for stress cracking of PET bottles. A bottle base that shows more resistance to sodium hydroxide attack should be more resistant to stress crack initiators during its lifetime.

4. Apparatus
   4.1. Individual testing stations.
   4.2. Stop watch or timer.
   4.3. Compressed air regulated to 531 +/- 4 kilopascals (77 +/-0.5 psi).
   4.4. Distilled water.
   4.5. Bottle closures.
   4.6. Titration apparatus (to determine alkalinity of NaOH solution).
   4.7. pH paper.
   4.8. 0.200% by weight of aqueous sodium hydroxide (NaOH) solution.
   4.9. Solution can be prepared as described in Appendix 1 or purchased as a commercial solution of 0.05N NaOH.

5. Safety Concerns. The following list outlines known hazardous conditions. However, the user is responsible for conducting a comprehensive job safety analysis to address any required safety or health issues before using this test.

5.1. NaOH is a strong alkali, which is corrosive to skin and eyes. Solid NaOH is also hygroscopic. Dilution of solid NaOH with water is an exothermic reaction. Always add NaOH slowly to water. Do not add water to NaOH. The pH of the 0.2% solution is about 12.7.
5.2. Total body protection is recommended when handling the solution and the pressurized bottles.
5.3. The possibility for bottle explosion is always present. Proper precautions should be taken to prevent injury from such occurrences.
5.4. NaOH solutions must be disposed of according to MSDS guidelines and relevant regulatory requirements.

6. Test Specimens and Conditioning
6.1. The standard sample quantity per test is 30 bottles.
6.2. Bottles should be no older than two weeks. Prior to testing, bottles are conditioned as follows:
6.2.1. Age at 50°C +/- 1°C (122°F +/- 2°F) and 50 percent relative humidity for 24 hours.
6.2.2. After aging, store at 22°C +/- 1°C (72°F +/- 2°F) for 16 hours minimum.

7. Sodium Hydroxide Solution Preparation and Standardization
7.1. Prepare a 0.200 percent by weight NaOH test solution according to the procedure in Appendix 1. The solution should be equilibrated to 22°C +/- 1°C (72°F +/- 2°F)
7.2. The acceptable range of alkalinity for this solution is 2402-2602 mg/l $CaCO_3$. This should be measured before each use.
7.3. The solution must be stored in a closed container to prevent $C0_2$ absorption. The solution must be used within 7 days of preparation.

8. Procedure
8.1. Label each bottle in order of testing.
8.2. Fill each bottle with the target net contents of water (e.g., a 2 liter bottle would contain 2000ml of water). The water should be equilibrated to 22°C +/- 1°C (72°F +/- 2°F).
8.3. Pressurize each bottle with compressed air to the equivalent internal pressure of 531 +/- 4 kilopascals (77 +/ - 0.5 psi).
8.4. Mark the liquid level on each bottle 5 minutes after pressurization.
8.5. Gently place each bottle into an individual pocket of 0.200% NaOH solution at 22C +/-1C (72 +/- 2 F). The solution must cover the base up to the top of the strap.
8.6. Record the start time as the time that the bottle was placed in the 0.200% NaOH solution.
8.7. The bottles should be checked for signs of failure according to the following schedule.

0-30 minutes... Continual monitoring
30-60 minutes... Check every 2 minutes
60-90 minutes: ......Check every 5 minutes
>90 minutes: ....Check every 10 minutes
(Note: if the observer leaves the room, it should be noted on the data collection sheet.)

8.8. Record the time to failure in minutes for each bottle. Failure is defined as a burst or a slow leak. A slow leak is evidenced by a visual fill point drop.
8.9. Continue the test until either all bottles have failed or the maximum specified test time is reached. Maximum time for this test will be specified between the supplier and customer. It is recommended that the test should not be conducted for longer than 2 hours.
8.10. After each use, carefully rinse and dry each test station.

9. Report
   9.1. Report the results as the total number of bottles which failed and the total time of the test. Report these specific items:
      9.1.1. Whether or not total test time reached the maximum test time..
      9.1.2. Measured alkalinity of the NaOH solution.
      9.1.3. Room temperature, bottle temperature, and NaOH solution temperature.
      9.1.4. Preform numbers and blow cavity numbers.
      9.1.5. Time to failure in minutes for each bottle.
      9.1.6. Location of failure according to one of five categories:
         9.1.6.1. Passing through the gate.
         9.1.6.2. Amorphous region (around gate and stretch rod area).
         9.1.6.3. Oriented region (base of foot).
         9.1.6.4. Strap area.
         9.1.6.5. Stretch rod impression.
      9.1.7. Type of failure, i.e. catastrophic (burst) or slow leak.
      9.1.8. Manufacturing defects, if present.

Appendix 1: Preparation of Standard 0.200 Percent (by weight) Sodium Hydroxide Test Solution.

1. Safety and Precautionary Notes.
   1.1. Sodium hydroxide is corrosive to skin and eyes. Use appropriate personal protective equipment. See the test methods and MSDS for safety precautions.
   1.2. The reaction of water with sodium hydroxide is exothermic. Use care when preparing solutions.

2. Preferred Sodium Hydroxide Preparation Method From Solid Sodium Hydroxide.
   2.1. Precautionary Note.
      2.1.1. The sodium hydroxide concentration is critical for this test. However, sodium hydroxide absorbs moisture and carbon dioxide from the atmosphere. This can change the potency of the chemical. Therefore, be certain that the sodium hydroxide has been stored in a tightly sealed container and the beads (or powder) are free flowing.

   2.2. General Instructions
      2.2.1. Weigh the proper amount of distilled water and pour it into the mixing vessel.
      2.2.2. Weigh the proper amount of sodium hydroxide (100% active) and add slowly, with stirring, to the distilled water in the mixing vessel. Stir until the sodium hydroxide is well dissolved and the solution is homogeneous.
      2.2.3. Titrate the solution to confirm total alkalinity of 2502 ppm (mg/L) as $CaCO_3$.
      2.2.4. Transfer the prepared solution to a tightly closed container with minimal headspace.
      2.2.5. Solution may be stored for up to one week before discarding.
      2.2.6. The table below can be used to determine the proper recipe for a given batch size:

Batch Size			
	1000 gm	10, 000 gm	18945 gm (~5 gal)
Bead/Powdered Sodium Hydroxide	2.00 gm	20.0 gm	37.9 gm
Distilled Water	998 gm	9980 gm	18907gm

3. Alternate Sodium Hydroxide Preparation Methods Using Stock Solutions of Sodium Hydroxide.
   3.1. Precautionary Notes
      3.1.1. The sodium hydroxide concentration is critical for this test. It is difficult to determine whether stock sodium hydroxide solutions have absorbed carbon dioxide and formed carbonate, or become more concentrated due to evaporation. Therefore, the preferred preparation method involves solid sodium hydroxide.
      3.1.2. If a stock solution must be used, check for any precipitate in the bottom of the container, or any white crust formation on the mouth of the container. Such items are warning signs that the potency of the solution has changed, and the stock solution should be replaced with fresh solution.
      3.1.3. If possible, titrate a known dilution of the sodium hydroxide solution, and confirm that the total alkalinity is equal to the active (phenolphthalein) alkalinity. If the two are not equal, replace the stock solution with fresh solution.

   3.2. General Instructions For Using a 50 Percent Stock Sodium Hydroxide Solution
      3.2.1. Weigh the proper amount of distilled water and pour it into the mixing vessel.
      3.2.2. Weigh the proper amount of 50% sodium hydroxide solution and add slowly, with stirring, to the mixing vessel. Stir until the solution is homogeneous.
      3.2.3. Titrate the solution to confirm total alkalinity of 2502 ppm (mg/L) as $CaCO_3$.Transfer the prepared solution to a tightly closed container with minimal headspace.
      3.2.4. Solution may be stored for up to one week before discarding.
      3.2.5. The table below can be used to determine the proper recipe for a given batch size:

Batch Size			
	1000 gm	10, 000 gm	18945 gm (~5 gal)
50% NaOH Solution	4.00 gm	40.0 gm	75.8 gm
Distilled Water	996 gm	9960 gm	18869.2 gm

General Instructions For Using a 1N Stock Sodium Hydroxide Solution

      3.2.6. Weigh the proper amount of distilled water and pour it into the mixing vessel.
      3.2.7. Weigh the proper amount of 1N sodium hydroxide solution and add slowly, with stirring, to the mixing vessel. Stir until the solution is homogeneous.
      3.2.8. Titrate the solution to confirm total alkalinity of 2502 ppm (mg/L) as $CaCO_3$.Transfer the prepared solution to a tightly closed container with minimal headspace.
      3.2.9. Solution may be stored for up to one week before discarding.
      3.2.10. The table below can be used to determine the proper recipe for a given batch size:

Batch Size			
	1000 gm	10, 000 gm	18945 gm (~5 gal)
1N NaOH Solution	50.0 gm	500 gm	947 gm
Distilled Water	950 gm	9500 gm	17998 gm

Total Alkalinity Calculations

(0.200 gm NaOH) x (1 mole NaOH) x (1 mole equiv $CaCO_3$) x (100.1 gm $CaCO_3$)	= (0.250 gm $CaCO_3$
(40.01 gm NaOH) (2 mole equiv NaOH) (1 mole $CaCO_3$) (0.250% $CaCO_3$)	= (0.250% $CaCO_3$
	= (2502 ppm (mg/L) $CaCO_3$
	= 147.2 gpg

Error Limits (4% maximum)			
Alkalinity Expression	Lower Limit	Desired Value	Upper Limit
%$CaCO_3$	0.2400	0.2500	0.260
ppm (mg/L) $CaCO_3$	2402	2502	2602
gpg	141.3	147.2	153.1

Error Limit Calculations (4%)
Percent $CaCO_3$
(0.250% $CaCO_3$) (1.04) = 0.260%
(0.250% $CaCO_3$) (0.96) = 0.240%
ppm (mg/L)
(2502 ppm $CaCO_3$) (1.04) = 2602 ppm
(2502 ppm $CaCO_3$) (0.96) = 2402 ppm
gpg
(147.2 gpg $CaCO_3$) (1.04) = 153.1 gpg
(147.2 gpg $CaCO_3$) (0.96) = 141.3 gpg

4. Alkalinity Test Kit Selection
   4.1. Alkalinity test kits should have the following capabilities:
      4.1.1. Target range of 2400 to 2600 ppm (Mg/L) as $CaCO_3$.
      4.1.2. Accuracy of 50 ppm (mg/L) as $CaCO_3$
   4.2. An example of such a test kit is as follows:

Alkalinity Test Kit with Digital Titrator
20637-00 AL-DT
10-4000 mg/L sampling range
10 mg/L smallest increment