Designation: C225 – 85 (Reapproved 2022)

Standard Test Methods for Resistance of Glass Containers to Chemical Attack[1]

This standard is issued under the fixed designation C225; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 These test methods cover the evaluation of the resistance of glass containers to chemical attack. Three test methods are presented, as follows:

1.1.1 *Test Method B-A* covers autoclave tests at 121 °C on bottles partially filled with dilute acid as the attacking medium.

1.1.2 *Test Method B-W* covers autoclave tests at 121 °C on bottles partially filled with distilled water as the attacking medium.

1.1.3 *Test Method P-W* covers autoclave tests at 121 °C on powdered samples with pure water as the attacking medium.

1.2 The values stated in SI units are to be regarded as the standard. The values in parentheses are for information only.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

A569/A569M Specification for Steel, Carbon (0.15 Maximum, Percent), Hot-Rolled Sheet and Strip Commercial [3]

D1125 Test Methods for Electrical Conductivity and Resistivity of Water

D1193 Specification for Reagent Water

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Significance and Use

3.1 The solubility of glass in contact with food, beverages, or pharmaceutical products is an important consideration for the safe packaging and storage of such materials. Autoclave conditions are specified since sterilization is often employed for the packaging of the product. It also represents one of the most extreme conditions, particularly of temperature, that containers will ordinarily experience. Any of the three test methods described may be used to establish specifications for conformity to standard values, either as specified by a customer, an agency, or "The United States Pharmacopeia:"

3.1.1 *Test Method B-A* is intended particularly for testing glass containers primarily destined for containment of products with a pH under 5.

3.1.2 *Test Method B-W* is intended particularly for testing glass containers to be used for products with a pH of 5.0 or over.

3.1.3 *Test Method P-W* is a hydrolytic autoclave test primarily intended for evaluating samples from untreated glass containers. It is often useful for testing the resistance of containers of too small capacity to permit measurements of solubility on the unbroken article by the B-W test method. Yielding the water resistance of the bulk glass, it can also be used in conjunction with the B-W test method to distinguish whether the internal surface of a container has been treated to improve its durability.

3.2 All three test methods are suitable for specification acceptance.

4. Purity of Reagents

4.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such

[1] These test methods are under the jurisdiction of ASTM Committee C14 on Glass and Glass Products and are the direct responsibility of Subcommittee C14.02 on Chemical Properties and Analysis.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Withdrawn. The last approved version of this historical standard is referenced on www.astm.org.

specifications are available.[4] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

4.2 Unless otherwise indicated, references to water shall be understood to mean distilled water or other water meeting the requirements for one of the types of reagent water covered by Specification D1193.

TEST METHOD B-A—RESISTANCE OF BOTTLES TO ATTACK BY DILUTE ACID

5. Apparatus

5.1 *Autoclave or Steam Sterilizer,* capable of withstanding a pressure of 165 kPa (24 psi) and, preferably, equipped with a constant-pressure regulator or other means for maintaining the temperature at 121 °C ± 0.5 °C (250 °F ± 0.9 °F). This temperature shall be checked by means of a suitably calibrated instrument. The autoclave shall be capable of accommodating at least six and preferably twelve of the largest containers to be tested. It shall be equipped with a rack for supporting the samples, a thermometer, a pressure gauge, and a vent cock.

6. Reagents and Materials

6.1 *Acetone,* USP grade.

6.2 *Methyl Red Indicator Solution*—Dissolve 24 mg of the sodium salt of methyl red in 100 mL of water. If necessary, neutralize the indicator solution with 0.020*N* sodium hydroxide (NaOH) solution so that the titer of five drops of the indicator solution in 100 mL of the special distilled water does not exceed 0.02 mL of 0.020*N* NaOH solution. In titrations using the methyl red indicator solution, the end point shall be taken at a pH of 5.6.

6.3 *Phenolphthalein Indicator Solution*—Dissolve 0.5 g of phenolphthalein in 60 mL of ethyl alcohol (95 %) and dilute with water to 100 mL.

6.4 *Sodium Hydroxide Solution, Standard* (0.020*N*)—Dissolve 100 g of NaOH in 100 mL of water in a 150 mL test tube. Avoid wetting the top of the test tube. Stopper the tube loosely with a stopper covered with tinfoil and allow to stand in a vertical position until the supernatant liquid is clear. Withdraw some of the clear solution in a measuring pipet and deliver 1.3 mL into a paraffin-lined bottle containing 1 L of carbon dioxide (CO_2)-free water. Stopper the bottle with a two-hole stopper carrying a glass siphon tube (for delivering the solution to a buret) and a soda-lime or soda-asbestos guard tube. Standardize the 0.020*N* NaOH solution against the National Institute of Standards and Technology Standard Sample No. 84h of acid potassium phthalate. Transfer 0.2000 g of the phthalate to a 250 mL Erlenmeyer flask and dissolve in about 75 mL of CO_2-free water. Add five drops of phenolphthalein indicator solution and titrate with the NaOH solution to the first persistent pink color. Adjust the standard NaOH solution to 0.020*N* strength.

6.4.1 Calculate the normality *N* of the NaOH solution as follows:

$$N = 0.9798/\text{mL of NaOH} \quad (1)$$

6.5 *High-Purity Water*—This water shall be free of heavy metals, particularly copper, as shown by a dithizone test and have a conductivity (consult Test Methods D1125) not exceeding 0.15 µS/cm.

6.5.1 The source water shall be distilled, then passed through a deionizer cartridge packed with a mixed bed of nuclear-grade resin, then through a cellulose ester membrane having openings not exceeding 0.45 µm. Pass the purified water through an in-line conductivity cell to verify its purity. After flushing discharge lines, suitable water should be dispensed directly into the test vessels.

NOTE 1—Copper tubing should not be used in the discharge lines. TFE-fluorocarbon or pure tin are suitable.

NOTE 2—Reference should be made to Specification D1193. Type I reagent water as defined therein complies with the present 6.5. In the interest of practicality and demonstrated sufficiency, 6.5 allows the following deviations from Type I reagent water specifications.

(1) Source water is unspecified whereas Type I specifies source water having a maximum conductivity of 20 µS/cm at 25 °C.

(2) The final step is filtration through a membrane having openings not exceeding 0.45 µm. Type I directs filtration through a 0.2 µm membrane.

(3) The conductivity immediately before dispensing is required not to exceed 0.15 µS/cm at 25 °C whereas Type I is limited to 0.06 µS/cm at 25 °C.

The distillation step is essential to minimize or avoid cultivation of microorganisms in the ion-exchange bed and consequent clogging of the membrane filter. When preceded by distillation, the ion-exchange bed should have a long life, but as the conductivity begins to rise toward the limit it should be replaced by a new bed.

Distillation from phosphoric acid with a conductivity of the product between 0.5 µS/cm and 1.0 µS/cm was specified as water for extraction in Test Methods C225. Water prepared as described herein gave results averaging about 8 % higher than water prepared by distillation from phosphoric acid when Test Method B-W was applied to soda-lime and borosilicate glass bottles in seven laboratories. The trend to slightly greater extraction may be associated with the higher average purity of this water. The limit on conductivity of 0.15 µS/cm for water prepared by this means was set because water of less conductivity is readily obtained and when 0.15 µS/cm is exceeded, the conductivity rises rapidly on further use of the system.

6.6 *Sulfuric Acid, Standard* (0.020*N*) containing approximately 0.58 mL of concentrated sulfuric acid (H_2SO_4, sp gr 1.84) in 1 L of solution. Prepare 0.1*N* H_2SO_4 containing 3.0 mL of concentrated sulfuric acid (H_2SO_4, sp gr 1.84)/L. Dilute 200 mL of the 0.1*N* H_2SO_4 to 1 L and standardize against 0.020*N* NaOH solution, using methyl red indicator solution. Finally, adjust the standard H_2SO_4 to 0.020*N* strength.

6.7 *Sulfuric Acid, Standard* (0.0005*N*)—Mix 1 volume of 0.1*N* H_2SO_4 with 199 volumes of water. Adjust the strength to be 0.0005 ± 0.000025*N*.

6.8 *Sulfuric Acid, Standard* (0.0002*N*)—Mix 1 volume of 0.1*N* H_2SO_4 with 499 volumes of water. Adjust the strength to be 0.0002 ± 0.00001*N*.

[4] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.