Designation: D 3316 – 96 (Reapproved 2000)

An American National Standard

Standard Test Method for Stability of Perchloroethylene with Copper[1]

This standard is issued under the fixed designation D 3316; 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.

1. Scope

1.1 This test method covers the evaluation of the corrosiveness to copper metal by perchloroethylene.

1.2 *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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

E 200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis[2]

3. Summary of Test Method

3.1 Clean copper strips are subjected to the action of liquid and vapor phases of boiling perchloroethylene for 72 h in the presence of light. Weight loss of copper strips and acid formation are determined at the end of the test period.

4. Significance and Use

4.1 This test method is to be used as a guide in selecting or eliminating certain grades of perchloroethylene used for dry-cleaning fabrics or degreasing metal parts.

5. Apparatus

5.1 *Flask*, 300-mL, 24/40 standard-taper joint.

5.2 *Soxhlet Extractor*, 30-mm inside diameter, 24/40 standard-taper bottom joint, 34/45 standard-taper upper joint.

5.3 *Allihn Condenser*, bulb type, 34/45 standard-taper bottom joint.

5.4 *Bottle*, wide-mouth, 8 oz.

5.5 *Funnel*, 8-mm outside diameter stem, 35-mm diameter opening.

5.6 *Heater*, variable control.

5.7 *Light Bulb*, 100 W.

5.8 *Beaker*, 400 mL.

5.9 *Analytical Balance*.

6. Reagents and Materials

6.1 *Purity of Reagents*—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 specifications are available.[3] 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.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean distilled water or water of equal purity.

6.3 *Acetone*, practical grade.

6.4 *Copper*, electrolytic foil, 0.05 mm thick. Cut three copper strips, 20 by 75 mm. Immerse the strips in concentrated HCl until the surface is bright and free of tarnish. Scribe consecutive identification numbers on the strips. Rinse thoroughly in running water and dry with a towel. Weigh the strips to the nearest 0.1 mg on an analytical balance.

6.5 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl). Handle the concentrated HCl solution as indicated in the supplier's direction.

6.6 *Sodium Hydroxide, Standard Solution* (0.01 $N \pm 0.001$)—Dissolve 0.4 ± 0.04 g of anhydrous sodium hydroxide (NaOH) in 1 L of water and standardize in accordance with Practice E 200.

6.7 *Phenolphthalein Indicator*—Dissolve 5 g of phenolphthalein in 500 mL of 95 % ethanol, dilute to 1 L with distilled water, and mix thoroughly.

7. Procedure

7.1 Clean the flask and Soxhlet extractor with soap and water. Should any residue be present, clean thoroughly with acid and rinse with water. Flush out the condenser with water. Rinse the flask, Soxhlet, and condenser with acetone and dry by means of a stream of filtered air.

7.2 Place one of the strips in the flasks, one in the Soxhlet, and the third in the condenser. Bend the condenser strip lengthwise in the form of a U and force it into the condenser so

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[1] This test method is under the jurisdiction of ASTM Committee D26 on Halogenated Organic Solvents and Fire Extinguishing Agents and is the direct responsibility of Subcommittee D26.04 on Test Methods.

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[2] *Annual Book of ASTM Standards*, Vol 15.05.

[3] *Reagent Chemicals, American Chemical Society Specifications*, 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.

that approximately half of it is inside. Measure 100 mL of the solvent into the flask and add 0.2 mL of water. Assemble the apparatus as shown in Fig. 1. Fill the wide-mouth bottle approximately half full of water (about 100 mL) and insert the funnel so that it is less than ¼ in. from the water surface. Use a slotted cork to hold the funnel and provide a means of releasing pressure. Turn on the condenser cooling water and adjust the heat source so that the Soxhlet siphon cycle is between 8 and 10 min. Place the light bulb 1 in. from the vertical center of the Soxhlet sidearm vapor tube. Continue refluxing for 72 h.

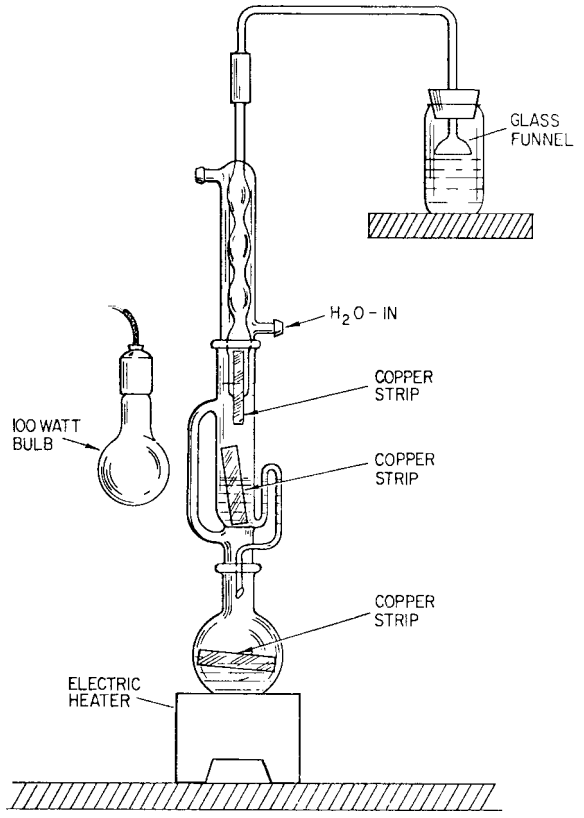

FIG. 1 Copper Stability Apparatus for Perchloroethylene

7.3 Turn off the heat source and allow the apparatus to cool for 15 min. By means of a funnel, pour the contents of the wide-mouth bottle into the top of the condenser. This will wash any acid-forming materials from the condenser and Soxhlet into the flask. Remove the flask, thoroughly shake, and transfer the contents to a 400-mL beaker. Titrate the aqueous layer using 0.01*N* NaOH solution to a phenolphthalein end point. Record the millilitres of NaOH required to reach the pink end point. Wash the copper strips individually in concentrated HCl until substantially free of all corroded matter. Rinse with running water and dry thoroughly by wiping with a towel. Weigh to the nearest 0.1 mg.

8. Report

8.1 Report any acidity as millilitres of 0.01*N* NaOH solution. Report loss in weight in milligrams, of the respective copper strips as “Flask loss,” “Soxhlet loss,” and“ Condenser loss.” This provides an indication of the quality of the perchloroethylene.

9. Precision and Bias

9.1 Results of duplicate determinations should not differ more than 20 % on acidity as millilitres of 0.01*N* NaOH solution or on weight losses of copper strips in the respective locations.

10. Keywords

10.1 acidity; copper; corrosion; dilute hydrochloric acid; dilute sodium hydroxide; indicator; perchloroethylene; weight loss