

5.9 *Hot Plate*, capable of operating to provide 120 to 130°C to boiling solution, or heating mantle for round-bottom flasks operating in the same temperature range.

5.10 *Drying Oven*, capable of controlling at 100°C Type 1B oven in accordance with Specification E145.

6. Reagents

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.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.

6.2 *1,2-Dichlorobenzene*.

6.3 *Methanol*.

6.4 *Osmium Tetroxide (0.004 M Solution in Toluene 1 kg/m³ (0.1 g/100 cm³))*—Protect the solution from light. Decomposition is indicated by formation of a black precipitate. *The vapors are highly toxic*. Therefore, use due precaution in reagent preparation, use, and disposal. One cubic centimetre of this stock solution is needed for each analysis. In view of the toxicity of the reagent, the amount of solution prepared should not greatly exceed the number of determinations to be made over a reasonable period of time, depending on the stability of the solution and the work load.

6.5 *Sulfuric Acid (density 1.84 Mg/m³)*—(H₂SO₄).

6.6 *Tertiary Butyl Hydroperoxide (2-Methyl-2-Hydroperoxypropane)*, approximately 70 % purity assay is typical.

6.7 *Toluene*.

7. Use of Osmium Tetroxide

7.1 *Precautions:*

7.1.1 Store osmium tetroxide ampules (maximum total of 3 g) inside capped pipe or its equivalent.

7.1.2 Persons working with osmium tetroxide or its solutions should wear rubber gloves, a rubber apron, and a face shield or goggles.

7.1.3 Keep solutions of osmium tetroxide (maximum of 0.5 g) in unbreakable containers and store and use in the hood. The volume of air mechanically exhausted should be such that the hood-face velocities are within acceptable limits (a recommended minimum of (0.5 m/s) 100 ft/min).

7.1.4 Mark all containers of osmium tetroxide, whether concentrated or diluted with “DANGER” labels.

7.1.5 Transfer all osmium tetroxide or its solutions to be disposed of, to polyethylene bottles and incinerate. Filtrates are

collected in a dump-can set aside for chlorinated solvents, and the contents are also incinerated. Do not accumulate waste solutions.

7.2 *Hazards:*

7.2.1 Osmium tetroxide is a yellow crystalline mass at room temperature. It can be hazardous in either its crystalline or vapor form. On contact with the skin or eyes, it produces irritation and if not removed immediately, may cause dermatitis and ulceration of the skin and intense conjunctivitis and corneal ulceration to the eye. Inhalation of osmium tetroxide may cause capillary bronchitis and dermatitis. Even small amounts, if inhaled over a considerable period, cause headache, insomnia, pharyngeal and laryngeal distress, and digestive disturbance. (**Warning**—Analysts must thoroughly familiarize themselves with a current Material safety data Sheet (MSDS) for osmium tetroxide.)

7.2.2 The vapor has a pronounced acrid, chlorine-like odor, which should be taken as a warning of the toxic concentration in the atmosphere, and personnel should immediately remove to an area of fresh air. The minimum perceptible concentration is 0.02 g/m³ of air. The threshold safety limit is 0.002 mg/m³ of air. Therefore, it cannot be detected by odor at the threshold safety limit. The melting point of the crystals is 56°C and the boiling point at 100 kPa is 130°C. However, it begins to sublime and distill well below the boiling point.

8. Sampling

8.1 If the sample is latex, prepare a dried film in accordance with Specification D1076.

8.2 If the sample is an oil-extended rubber, conduct an ETA extraction in accordance with Test Methods D1416.

8.3 In all cases, use a random unhomogenized sample.

9. Procedure

9.1 For a sample containing less than 30 % block styrene, cut approximately 0.5 g of rubber weighed to the nearest 0.1 mg into small pieces. Add the pieces to a 250-cm³ Erlenmeyer flask (or round-bottom flask), to which 35 cm³ of 1,2-dichlorobenzene has been previously added. For rubbers containing more than 30 % block styrene, use a 0.3-g specimen.

9.2 Fit an air condenser to the flask and heat it and its contents to 120 ± 5°C, until the rubber dissolves.

9.3 Remove the flask from the hot plate or heating mantle and cool it just enough to stop refluxing. Separate the air condenser from the flask, add 10 cm³ of the tertiary butyl hydroperoxide solution (see 6.6) and 1 cm³ of the osmium tetroxide solution (see 6.4). Replace the air condenser and continue heating the resulting solution at 100 to 120°C for 12 to 15 min. (Polystyrene is itself slowly degraded in this process, therefore digestion time should be constant and not lengthy.)

9.4 Remove the flask from the hot plate or heating mantle. When it has cooled so that it can be held without discomfort, pour the solution slowly and with constant stirring into a 600-cm³ beaker containing 350 cm³ of methanol to which 5

⁴ *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 *Annual 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.