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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Determining Unreacted Monomer Content of Latexes Using Gas-Liquid Chromatography¹

This standard is issued under the fixed designation D 4747; 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.

^{ε1} NOTE—Unit of measurement statement added to the Scope Section editorially in May 1997.

1. Scope

1.1 This test method covers the determination of free monomer content of acrylic latexes. Monomers that have been successfully determined by this procedure include *n*-butyl methacrylate, *n*-butyl acrylate, styrene, and methyl methacrylate. The determination of other monomers has not been evaluated, but this test method is believed to be applicable. The established working range of this test method is from 100 to 1000 $\mu\text{g/g}$, but there is no reason to believe it will not work outside of this range, provided that appropriate dilutions and adjustments in specimen size are made.

1.2 The volatile composition of acrylic latexes is expected to change with time and environmental factors. This time dependence of the determination may be seen as an artificially large deviation of results, making the method mostly applicable for in-house quality control, where sampling and analysis conditions can be better controlled.

1.3 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *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.* See Section 7 for specific hazard statements.

2. Referenced Documents

2.1 ASTM Standards:

D 3980 Practice for Interlaboratory Testing of Paint and Related Materials²

E 260 Practice for Packed Column Gas Chromatography³

3. Summary of Test Method

3.1 A suitable aliquot of the latex is internally standardized with isobutyl acrylate, diluted with water, and then injected

into a gas chromatographic column containing a packing material coated with a stationary phase that separates the internal standard and monomers in question from each other and from other volatile compounds.

4. Significance and Use

4.1 Excessive amounts of unreacted monomer may cause concerns relating to toxicity and odor. This test method is designed to measure the unreacted monomer content of latexes. The results may be used to monitor the extent of polymerization during manufacture, as well as to establish maximum unreacted monomer content for regulatory purposes.

5. Apparatus

5.1 *Gas Chromatograph*, any gas-liquid chromatographic instrument having a flame ionization detector and linear temperature programming. An injection port using replacable glass liners to facilitate periodic removal of accumulated residues is recommended.

5.2 *Column*, 2 by 2-mm inside diameter glass or 6 ft by 1/8-in. outside diameter steel tubing, packed with 10 % by weight of a 2-nitroterephthalic acid derivative of a synthetic polyester wax on 100/120 mesh acid washed, silane treated diatomaceous earth.⁴ A column of equivalent or superior performance may also be used.

5.3 *Recorder*—A recording potentiometer with a full-scale deflection of 10 mV, a full-scale response time of 2 s or less, and a maximum noise level of $\pm 0.03\%$ of full scale (see Practice E 260).

5.4 *Liquid Charging Devices*, microsyringe, 10- μL capacity or an automatic liquid sampling device.

5.5 *Dropper Pipets*, glass, disposable.

5.6 *Vials*, approximately 7-mL capacity, with caps. Open top screw cap vials fitted with polytetrafluoroethylene/silicone septa are preferred.

5.7 *Autosampler Vials*, 2-mL capacity (optional).

5.8 *Analytical Balance*, accurate to 0.1 mg.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

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² *Annual Book of ASTM Standards*, Vol 06.01.

³ *Annual Book of ASTM Standards*, Vol 14.02.

⁴ Columns prepared from the stationary phases and supports have been found suitable for this purpose and are available from scientific supply houses.