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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Determination by X-ray Fluorescence Spectroscopy of Titanium Dioxide Content in Paint¹

This standard is issued under the fixed designation D 4764; 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—Keywords were added editorially in April 1993.

1. Scope

1.1 This test method covers the determination of titanium dioxide content in liquid paint. This test method is applicable to both water-reducible and solvent-reducible paints.

1.2 *This standard does not purport to address all of the safety problems, 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.* Specific hazards statements are given in Section 7.

2. Referenced Documents

2.1 ASTM Standards:

D 476 Specification for Titanium Dioxide Pigments²

D 1193 Specifications for Reagent Water³

D 1394 Test Methods for Chemical Analysis of White Titanium Pigments²

D 2369 Test Method for Volatile Content of Coatings⁴

D 3925 Practice for Sampling Liquid Paints and Related Pigmented Coatings⁴

D 3980 Practice for Interlaboratory Testing of Paint and Related Materials⁴

E 180 Practice for Determining the Precision Data of ASTM Methods for Analysis and Testing of Industrial Chemicals⁵

3. Summary of Test Method

3.1 Paint containing a known amount of titanium dioxide is used as the reference standard. A solution of tetraethylammonium bromide in 2-ethoxyethanol is added to the standard paint and to each paint being analyzed. Drawdown films of the standard and test paint are made on thin plastic sheets. The intensities of key X-ray fluorescence peaks of titanium and bromine are measured. The titanium dioxide content is deter-

mined by comparing the ratio of the intensities of titanium and bromine peaks in the test paint and the standard. Differences in the nonvolatile content of the paints are taken into account in the calculation of results.

4. Significance and Use

4.1 Titanium dioxide pigments are components with high refractive index that significantly influence the opacity, color, durability, and other properties of coatings. This test method for determining titanium dioxide content is quicker and easier to use than Test Methods D 1394, a wet chemical analysis method for pigments. It is conveniently applicable to single samples and to large numbers of samples. Only a single relatively stable reagent used to prepare standards and paints under test need be stored. Drawdown specimens used as standards, once prepared, can be stored indefinitely and used repeatedly.

5. Apparatus

5.1 *X-ray Fluorescence Spectrometer*, suitable for measurement of the baseline corrected intensity of the $K\alpha$ lines of titanium and of bromine. For spectrometers equipped with a tungsten or chromium target and a lithium fluoride (200) analyzing crystal, these lines are observed at 2θ angles of 86.09° and 29.97° , respectively.

NOTE 1—Follow the recommendations of the manufacturer of the instrument used. Use operating conditions that ensure that the count rate for bromine and titanium are within the linear response range of the detector. Although the base operating conditions and count rate may be different for different instruments, these differences will not affect the outcome of the analysis.

5.2 Paint Shaker.

5.3 *Film Applicator*, to produce a 3-mil (75- μm) wet film thickness.

5.4 *Plastic Sheet*,⁶ with no interfering X-ray fluorescence peaks and not attacked by paint solvent.

5.5 *Perforated Suction Plate*, or other flat surface.

5.6 *Vials and caps*, 20 mL.

¹ This test method is under the jurisdiction of ASTM Committee D01 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 06.03.

³ *Annual Book of ASTM Standards*, Vol 11.01.

⁴ *Annual Book of ASTM Standards*, Vol 06.01.

⁵ *Annual Book of ASTM Standards*, Vol 15.05.

⁶ Leneta P-121-10N dull black plastic panels 6½ by 17 in. by 10 mils (165 by 432 by 0.25 mm) in size, available from Leneta Co., 15 Whitney Rd., Mahwah, NJ 07430, have been found suitable for this purpose.