Designation: D3720 – 90 (Reapproved 2019)

Standard Test Method for Ratio of Anatase to Rutile in Titanium Dioxide Pigments by X-Ray Diffraction[1]

This standard is issued under the fixed designation D3720; 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 determination of the ratio of anatase to rutile in titanium dioxide pigments. The method is also applicable to pigment mixtures and pigmented coatings containing titanium dioxide.

1.2 The values stated in SI units are to be regarded as the standard. The values given 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]

D215 Practice for the Chemical Analysis of White Linseed Oil Paints (Withdrawn 2005)[3]

D2371 Test Method for Pigment Content of Solvent-Reducible Paints

D2698 Test Method for Determination of the Pigment Content of Solvent-Reducible Paints by High-Speed Centrifuging

D3925 Practice for Sampling Liquid Paints and Related Pigmented Coatings

3. Summary of Test Method

3.1 The X-ray diffraction pattern obtained from a material is characteristic of that material. The intensity of a diffraction peak entirely due to one component of a mixture is dependent upon the amount of that substance in the mixture. To a minor extent the peak intensity of the component is also dependent on the mass absorption coefficient of other materials present. Since the test method utilizes the ratio of the intensities of diffraction peaks of two chemically similar materials, it is expected that the effects of other constituents will be the same for both materials.

3.2 The intensity of the diffraction maxima for anatase and rutile is measured by X-ray diffractometry. The intensity of the anatase peak is converted to anatase content relative to rutile and the rutile content is determined by difference.

3.3 The X-ray diffraction measurement is made on single pigments, pigment mixtures, films of pigmented coatings, and films prepared from liquid coatings, if interfering materials are not present. When interfering materials are present, the pigment is separated from the redissolved (or ignited film, or from the liquid coating and treated to isolate the titanium dioxide.

4. Significance and Use

4.1 This test method is used by titanium dioxide pigment manufacturers and users for process control and product acceptance.

5. Interferences

5.1 Calcium sulfate interferes, but its effect is eliminated by chemical removal (see Practice D215). It is desirable to assure by analysis that any residual $CaSO_4$ is considerably less than the level of anatase being sought. The insoluble residue after removal of calcium sulfate should be ignited above 700°C. Chrome yellow and the valentinite form of antimony trioxide also interfere if not removed. High amounts of iron render analysis difficult due to increased background (see Note 1). Additives, such as antimony and zinc, and impurities, such as niobium and zirconium, are generally present in solid solution and thus would not have interfering diffraction peaks. Surface treatments such as silica and alumina do not interfere. Extreme

[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 D01.31 on Pigment Specifications.

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