Designation: D2929 – 89 (Reapproved 2006)

Standard Test Method for Sulfur Content of Cellulosic Materials by X-Ray Fluorescence[1]

This standard is issued under the fixed designation D2929; 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 determination of sulfur content of cellulosic materials by X-ray fluorescence.

1.2 Using appropriate standards, the range of the procedure is from approximately 10 ppm to 20 % sulfur.

1.3 This test method is proposed specifically as an alternative to Test Methods D871, sections on Significance and Use, Apparatus, Reagents, Procedure and Calculation of Hydroxyl Content, and Test Methods D817, sections on Summary of Test Method, Significance and Use, Apparatus, Reagents, and Procedure of Hydroxyl Content. As applied to cellulose esters it measures the combined sulfur and sulfur in the accompanying inorganic salts.

1.4 To determine combined sulfur, the sample, when soluble, must first be reprecipitated into dilute acid to remove the noncombined sulfur compounds.

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 *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.* For a specific hazard statement, see 7.2.1.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D817 Test Methods of Testing Cellulose Acetate Propionate and Cellulose Acetate Butyrate

D871 Test Methods of Testing Cellulose Acetate

3. Summary of Test Method

3.1 The sulfur content of cellulose, cellulose modification, or cellulose derivative is determined by measuring the intensity of the secondary sulfur Kα X rays emitted on irradiation of the sample with primary X rays of higher energy from an X-ray tube with a target of tungsten or chromium. The sulfur Kα radiation is diffracted with a suitable analyzing crystal and detected with a flow-proportional counter. The entire path of the secondary radiation is purged with hydrogen or helium, or evacuated to a pressure of 0.5 mm Hg or less. The intensity of the sulfur Kα rays, as established by a standard counting period and corrected for background radiation, is then converted to percent sulfur from calibration data.[3]

4. Significance and Use

4.1 This procedure provides a method for determining sulfur content in cellulosic materials by nondestructive means. Sulfur may be in the form of sulfate esters that may contribute to thermal instability. Sulfur can also be present as salts that can cause haze in solutions.

5. Apparatus

5.1 *Wiley Mill*, equipped with 60-mesh screen.

5.2 *Sample Mold*—Chrome steel die of a size depending on the sample holder to be used.

5.3 *Laboratory Press*, capable of exerting at least 5000 psi.

5.4 *X-Ray Spectrograph*, with following equipment: tungsten or chromium target X-ray tube; hydrogen or helium purging system or vacuum system to reach 0.5 mm Hg (or less, if desired); 20-mil Soller slits; flow-proportional counter with 90 % argon-10 % methane gas mixture; and NaCl, ethylenediamine dextro tartrate (EDT), or pentaerythritol (PET) analyzing crystal.

[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 D01.36 on Cellulose and Cellulose Derivatives.

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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] This test method is compiled from four techniques, by incorporating certain features of each, as follows: "Sulfur in Cellulose Esters by X-ray Emission Spectroscopy," Eastman Kodak Co.; " Sulfur in Cellulose Acetate by X-ray Fluorescence," Tennessee Eastman Co.; "X-ray Fluorescence Analysis of Modified Cottons" by Tripp, Piccolo, Mitcham and O'Connor. *Textile Research Journal*, Vol 34, 1964, p. 773, and FMC Corp., American Viscose Div. information furnished by L. H. Phifer and W. B. Swann.