



Designation: D 2929 – 89 (Reapproved 2000)

## Standard Test Method for Sulfur Content of Cellulosic Materials by X-ray Fluorescence<sup>1</sup>

This standard is issued under the fixed designation D 2929; 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 ( $\epsilon$ ) 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 D 871, Sections 29 to 33, and Test Methods D 817, Sections 39 to 43. 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 Note 3.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

D 817 Test Methods of Testing Cellulose Acetate Propionate and Cellulose Acetate Butyrate<sup>2</sup>

D 871 Test Methods of Testing Cellulose Acetate<sup>2</sup>

### 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  $K\alpha$  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  $K\alpha$

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  $K\alpha$  rays, as established by a standard counting period and corrected for background radiation, is then converted to percent sulfur from calibration data.<sup>3</sup>

### 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.

NOTE 1—Radiation from a chromium target tube appears to be more effective than radiation from a tungsten target tube for exciting sulfur  $K\alpha$ . This may be because a larger part of the “white” radiation of the chromium tube is at the longer wavelength region of the spectrum, or because of the use of thinner windows in the chromium tube. However, either the chromium or the tungsten target tube is suitable for sulfur analysis. If a pulse height analyzer is available as a part of the X-ray instrumentation, its use is very helpful in reducing background radiation.

<sup>1</sup> 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 D01.36 on Cellulose and Cellulose Derivatives.

Current edition approved Oct. 27, 1989. Published December 1989. Originally published as D 2929 – 70. Last previous edition D 2929 – 70(1985)<sup>1</sup>.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 06.03.

<sup>3</sup> 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.