Designation: F218 – 13 (Reapproved 2020)

Standard Test Method for Measuring Optical Retardation and Analyzing Stress in Glass[1]

This standard is issued under the fixed designation F218; 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 analysis of stress in glass by means of a polarimeter based on the principles developed by Jessop and Friedel (**1, 2**).[2] Stress is evaluated as a function of optical retardation, that is expressed as the angle of rotation of an analyzing polarizer that causes extinction in the glass.

1.2 There is no known ISO equivalent to this standard.

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:*[3]

C162 Terminology of Glass and Glass Products

C770 Test Method for Measurement of Glass Stress—Optical Coefficient

C978 Test Method for Photoelastic Determination of Residual Stress in a Transparent Glass Matrix Using a Polarizing Microscope and Optical Retardation Compensation Procedures

C1426 Practices for Verification and Calibration of Polarimeters

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this test method, refer to Terminology C162.

4. Significance and Use

4.1 The performance of glass products may be affected by presence of residual stresses due to process, differential thermal expansion between fused components, and by inclusions. This test method provides means of quantitative evaluation of stresses.

5. Calibration and Standardization

5.1 Whenever calibration of the polarimeter is required by product specification, Practices C1426 for verification and calibration should be used.

6. Polarimeter

6.1 The polarimeter shall consist of an arrangement similar to that shown in Fig. 1. A description of each component follows:

6.1.1 *Source of Light*—Either a white light or a monochromatic source such as sodium light (λ 589 nm) or a white light covered with a narrow-band interferential filter B (see Fig. 1) transmitting the desired monochromatic wavelength.

NOTE 1—The white light should provide a source of illumination with solar temperature of at least that of Illuminant A.

6.1.2 *Filter*—The filter should be placed between the light source and the polarizer, or between the analyzer and the viewer (see Fig. 1).

6.1.3 *Diffuser*—A piece of opal glass or a ground glass of photographic quality.

6.1.4 *Polarizer*—A polarizing element housed in a rotatable mount capable of being locked in a fixed position shown in Fig. 2 and Fig. 4.

6.1.5 *Immersion Cell*—Rectangular glass jar with strain-free, retardation-free viewing sides filled with a liquid having

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[1] This test method is under the jurisdiction of ASTM Committee C14 on Glass and Glass Products and is the direct responsibility of Subcommittee C14.04 on Physical and Mechanical Properties.

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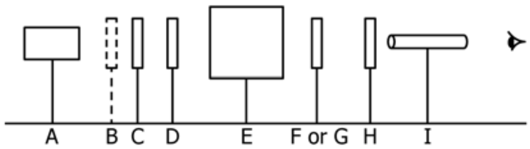
[2] The boldface numbers in parentheses refer to the list of references at the end of this test method.

[3] 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.



A—Light source (white, sodium vapor, or mercury vapor arc)
B—Filter (used only with mercury arc light) (used with white light)
C—Diffuser
D—Polarizer
E—Immersion cell
F—Full-wave plate (used only with white light)
G—Quarter-wave plate
H—Analyzer
I—Telescope

FIG. 1 Polarimeter

the same index of refraction as the glass specimen to be measured. It may be surmounted with a suitable device for holding and rotating the specimen, such that it does not stress the specimen.

NOTE 2—Suitable index liquids may be purchased or mixed as required. Dibutyl phthalate (refractive index 1.489), and tricresyl phosphate (index 1.555) may be mixed to produce any desired refractive index between the two limits, the refractive index being a linear function of the proportion of one liquid to the other. Other liquids that may be used are:

Liquid	Refractive Index
Cinnamic Aldehyde	1.62
Oil of Cassia	1.61
Monochlorobenzene	1.525
Carbon Tetrachloride	1.463
Dipentene (Eastman)	1.473

NOTE 3—Cases may arise where the refraction liquid may contaminate the specimen. When the sample is viewed through faces that are essentially parallel, elimination of the liquid will cause only a minor error. However, when viewing through faces of the sample that are not parallel, the use of liquid of same refraction index is essential.

6.1.6 *Full-wave (Sensitive Tint) Plate,* having a retardation of 565 ± 5 nm, which produces, with white light, a violet-red color. It should be housed in a rotatable mount capable of being locked in a fixed position shown in Fig. 2.

6.1.7 *Quarter-wave Plate,* having a retardation equivalent to one quarter of the wavelength of monochromatic light being used, or 141 ± 5 nm when white light is used. It should be housed in a rotatable mount capable of being locked in a fixed position shown in Fig. 2.

6.1.8 *Analyzer*—Identical to the polarizer. It should be housed in a rotatable mount capable of being rotated 360°, and a graduated dial indicating the angular rotation α of the analyzer from its standard position. The polarizer must be lockable in position shown in Fig. 2.

6.1.9 *Telescope,* short-focus, having a suitable magnifying power over the usable focusing range.

7. Setup of Polarimeter

7.1 The standard setup of the polarimeter is illustrated in Fig. 2. Two reference directions must be identified:

7.1.1 Vertical direction (*V*) (in polarimeters transmitting the light in horizontal direction) or NS, that is usually a symmetry axis of an instrument using a vertical light path, and polarizers are in a horizontal plane.

7.1.2 Horizontal (*H*), or EW (perpendicular to the vertical or NS) (see Fig. 4).

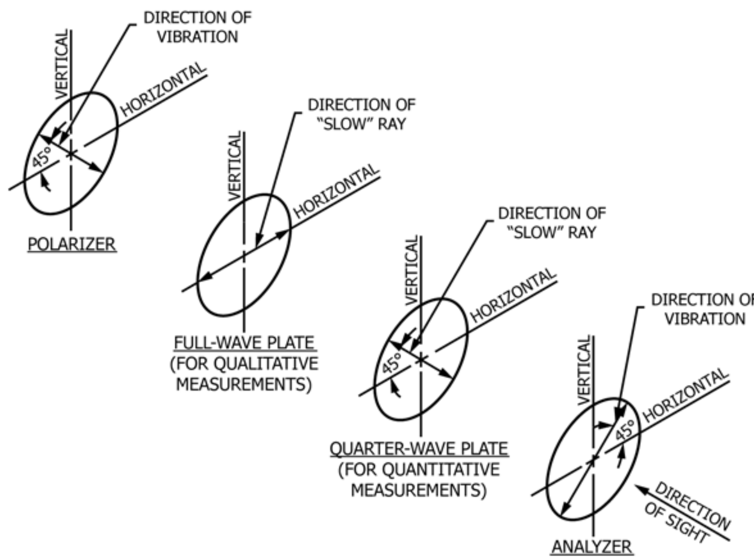
7.2 As usually employed, the polarimeter measures retardations in a sample that is placed in the polarimeter and rotated until the measured stresses S_x and S_y are oriented along V and H (vertical or a horizontal) direction. This is accomplished by setting the vibration direction of the polarizer at an angle of 45° to the vertical and clockwise to the horizontal (as shown in Fig. 2 and Fig. 4). The vibration direction of the analyzer must be "crossed" with respect to that of the polarizer; that is, the two directions must be at right angles to each other. In this relationship a minimum amount of light will pass through the combination. To check the 45° angle at which the directions of the polarizer and analyzer must be set, use may be made of a rectangular-shaped Glan-Thompson or Nicol prism. The prism is set so that its vibration direction is 45° to the vertical and horizontal. The polarizer is then rotated until extinction occurs between it and the prism. The position of the analyzer is then determined in the same way, but by first rotating the Glan-Thompson or Nicol prism through 90°; or the analyzer may be rotated to extinction with respect to the polarizer after the latter has been set in position with the prism.

7.3 When a quarter-wave plate is used, its "slow" ray direction must be set 45° clockwise from the horizontal in a northwest-southeast direction (see Fig. 2). Adjusted in this position, maximum extinction occurs when direction of axes of all three elements (polarizer, analyzer and quarter-wave plate) are in agreement with Fig. 2.

7.4 When the full-wave plate is used with the quarter-wave plate, its "slow" ray direction must be placed in a horizontal position (see Fig. 2). Adjusted in this position, a violet-red background color is seen when the three elements (polarizer, full-wave plate, and analyzer) are placed in series.

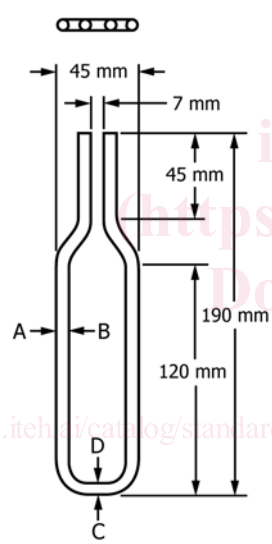
7.5 Subsections 7.3 and 7.4 describe orientations of the quarter- and full-wave plates in the standard positions that have been generally adopted. However, the direction of the " slow" rays may be rotated 90° without changing the functions of the apparatus. This does, however, cause the analyzer rotations (in the case of the quarter-wave plate) and the colors (in the case of the full-wave plate) to have opposite meanings. Tables 1 and 2 define these meanings in whatever is being measured or observed with the "slow" ray directions in either the standard or the alternate positions.

7.6 To assure proper orientation of the directions of the "slow" ray of the quarter-wave and full-wave plates with respect to the vibration directions of the polarizer and analyzer, use may be made of a U-shaped piece of annealed cane glass as illustrated in Fig. 3. Squeezing the legs together slightly will develop a tensile stress on the outside and a compressive stress on the inside. A flat rectangular beam in bending, containing a region where the direction and sign of stresses is known can also be used. Then, if the "slow" ray directions of the quarter-wave and full-wave plates are oriented in the standard position, the stress conditions of columns 1 through 4 of Table 1 will be noted in the vertical and horizontal sides of the U-tube. If the opposite meaning of the color definition is preferred, it will be necessary to rotate the "slow" ray directions of the full-wave plate 90° to the alternate positions. The orientation of the full-wave plate can be verified, comparing



The direction of vibration of polarizer and analyzer may be oriented 90° from indicated positions.

FIG. 2 Orientation of Polarimeter in Standard Position



NOTE 1—When the legs are squeezed together, Sides A and C become tensile and Sides B and D become compressive.

NOTE 2—Material—Cane glass of approximately 7 mm diameter, annealed after forming.

NOTE 3—When viewed in the polarimeter, immerse in a liquid having the same refractive index as the glass.

FIG. 3 Reference Specimen

the observed colors to the expected colors shown in the Table 2. The orientation of the quarter wave plate can be verified, checking that a clockwise rotation of the analyzer will decrease the light intensity, whenever a black (zero-order) line is very near the point of interest.

7.7 If a major stress component lies in any direction other than vertical or horizontal, its measurement requires that either:

7.7.1 The entire optical system be rotated so that the vibration directions of the polarizer and analyzer are set at 45° to the stress direction, or

7.7.2 That the part containing the stress direction be rotated to suit assure the orientation shown in Fig. 4.

8. Procedure

8.1 Before proceeding with measurements, evaluate the stress field by observing the sample with and without the full-wave plate (tint plate) in place. The colors observed when the tint plate is introduced provide an initial evaluation of the retardation.

8.2 *Identify Directions and Sign of Stresses:*

8.2.1 Remove the tint plate from the path of light. Rotate the sample in its plane. Observe the point of interest (POI) becoming dark (minimum transmitted light intensity) whenever the direction of stress S_x or S_y is parallel to the polarizer. From the position of extinction, rotate the sample 45°, placing one of principal stresses, S_x, in vertical orientation, at 45° to the polarization axes. In this position, maximum brightness is observed. (See Fig. 4.)

8.2.2 For a region near the POI exhibiting small retardation (<150 nm), place the tint plate in the field of view, oriented as shown in Fig. 2 and Fig. 4. The colors observed when the tint plate is introduced provide an evaluation of the retardation, and identification of the sign of stress S_x (tension [+], compression [–]). If the colors observed (see Table 2) are ..red, orange.., the stress S_x is tensile (or $S_x - S_y > 0$). If the colors observed are blue..blue green, the stress S_x is compressive (or $S_x - S_y < 0$).

8.2.2.1 A 90° rotation of the tint plate will reverse the sign convention.

8.3 In regions where the retardation is larger (>150 nm), use the analyzer rotation to identify the sign of S_x, or $S_x - S_y$. With the tint plate removed, rotate the Analyzer clockwise, and observe the sequence of changing colors.

8.3.1 The sequence Yellow-BlueGray-Brown-Yellow-BlueGray, or for larger retardation (approximately >300 nm)

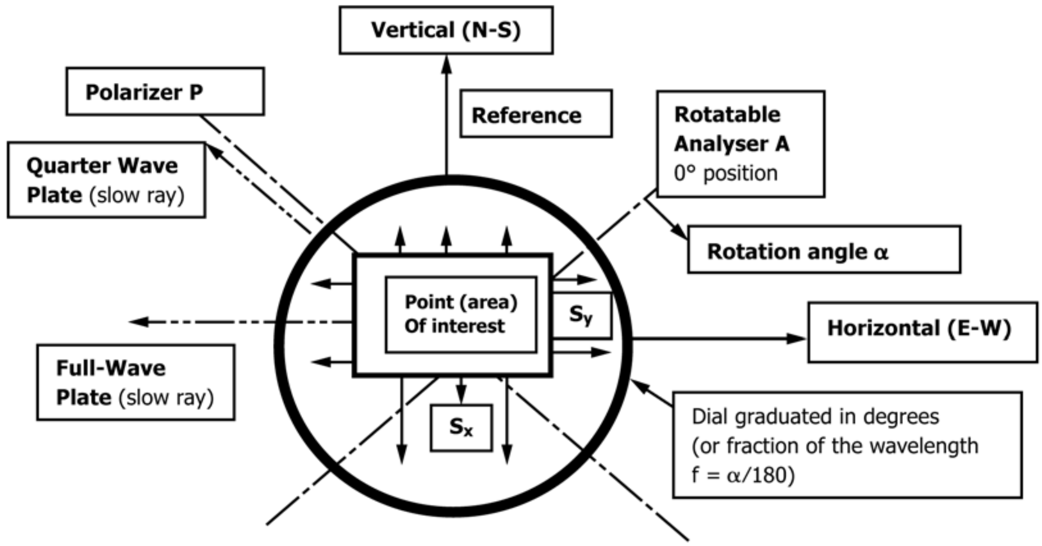


Fig. 4 Orientation of the Polarizer, Analyzer, Quarter-wave plate, Full Wave plate, and of stresses S_x, and S_y in the region of interest. Stress S_x in Vertical (NS) position

NOTE 1—Stress S_x in Vertical (NS) Position.

FIG. 4 Orientation of Polarizer, Analyzer, Quarter-wave Plate, Full-wave Plate, and of Stresses S_x and S_y in Region of Interest

TABLE 1 Orientation of "Slow" Ray Direction of Full-wave Plate with Corresponding Stresses

	Orientation of "Slow" Ray With Respect to the Horizontal is Standard			
	When Stress Component Lies in the Vertical		When Stress Component Lies in the Horizontal	
	Column 1	Column 2	Column 3	Column 4
Approximate Color Indicates	yellow tension	green compression	yellow compression	green tension

Yellow-Blue-Red-Orange-Yellow-LightYellow-Blue, indicates tensile stress ($S_x > 0$ or $S_x - S_y > 0$).

8.3.2 The reverse sequence Yellow-Brown- BlueGray-Yellow, or for larger retardation (approximately >300 nm) Yellow-Orange-Red-Blue-Yellow-Orange-Red, indicates compressive stress ($S_x < 0$ or $S_x - S_y > 0$).

8.4 *Measure Retardation:*

8.4.1 To measure the retardation at any given point, remove the tint plate, place the monochromatic filter in the field of view, and rotate the analyzer with respect to its initial position until maximum extinction (darkness) occurs at the POI. The angle α through which the analyzer must be rotated to the left or the right is a measure of the retardation at the point.

8.4.1.1 In white light, the color of the fringe moving toward the POI will keep changing. To eliminate possible errors and to increase the contrast, the monochromatic filter, *B*, must be inserted for this operation, or the monochromatic lamp must be used.

8.4.2 The rotation of the Analyzer must be clockwise. If the stress is tensile (S_x or $S_x - S_y > 0$), the measured angle α is indicated directly on the dial, in degrees. When a fractional graduation of the dial is used, the fraction $f = a/180$ is indicated on the dial.

8.4.3 If the stress is compressive (S_x or $S_x - S_y < 0$), the indicated dial angle on a 0 to 180° dial is β.

8.4.3.1 The measured angle α used to calculate the retardation and stress is given by:

$$\alpha = 180 - \beta$$

8.4.3.2 Similarly, the indicated fraction is a compliment, and the measured fraction is:

$$f = 1 - \text{indicated fraction}$$

8.4.3.3 Instruments equipped with a dual scale, 0 to 180° CW and 0 to 180° CCW, the angle α is indicated directly when the analyzer is rotated CCW.

8.4.4 When the retardation is required to be measured in a given area or section where several extinction points may exist, rotate the analyzer (CW or CCW) until the maximum extinction is achieved at each selected point. Use the procedure previously described in this section to measure retardation at those points, and the sequence of the observed colors described in 8.3 to differentiate between tensile or compressive stress.

8.5 When a maximum value is specified and the specimens are of a uniform thickness it is necessary only to set the analyzer at the angle specified and then observe whether any unclosed loop-shaped fringes are present in the stress pattern. If not, it may be concluded that the maximum retardation that is present is less than the specified maximum. If any are present, then the retardation is greater than the specified maximum. To determine the exact magnitude of the retardation, use the method outlined in 8.2 and 8.4.

8.6 When the full-wave plate (also called the "tint plate") is introduced, the polarimeter can be used to reveal a color pattern. White light must be used for this observation, and the analyzer must be set in standard position (perpendicular to the

TABLE 2 Polariscopic Colors With White Light

NOTE 1—The colors observed are affected by the color temperature of the light source, spectral transmittance of the sample and the extinction characteristics of the polarizer. For this reason, the relation between the retardation and observed color is only approximate and should not be considered quantitatively.

Color (Approximate)	Equivalent Optical Retardation (Approximate) in Degrees Rotation of Analyzer	
Blue/Green	212	
Violet	180	Colors on this side of the ″0″ line indicate:
Red	153	
Orange	128	If the slow ray of the full-wave plate is in horizontal (standard) position:
Pale Yellow	110	
Greenish Yellow	97	$S_x - S_y$ is <0. In uniaxial stress, S_x is compression or S_y is tension.
Yellowish green[A]	85	
Pale green	73	If the slow ray of the full-wave plate is in vertical position:
Green[A]	60	
Deep Green	50	$S_x - S_y$ is <0. In uniaxial stress, S_x is tension or S_y is compression.
Blue Green[A]	40	
Blue	25	
Dark Blue[A]	12	
Violet blue	7	
Violet red	0	
Red[A]	7	Colors on this side of the "0" line indicate:
Red Orange	12	
Orange[A]	25	If the slow ray of the full-wave plate is in horizontal (standard) position:
Orange Yellow	40	
Gold Yellow[A]	50	$S_x - S_y$ is >0. In uniaxial stress, S_x is tension or S_y is compression.
Yellow	60	
Pale Yellow[A]	73	If the slow ray of the full-wave plate is in vertical position:
Yellow White	85	
White[A]	97	$S_x - S_y$ is <0. In uniaxial stress, S_x is compression or S_y is tension.
Gray White	110	
Iron Gray	172	
Black	180	

[A] More distinctive color of pair.

polarizer). Table 2 shows the color distribution that may be expected together with the associated magnitude of the retardation and tension-compression indicated

8.7 When the specimen is very small, accurate evaluation of retardation with the polarimetric arrangement described becomes difficult when the magnification offered by the telescope is too low. For such specimens use a polarizing microscope containing all the basic elements of Fig. 1. Because the optic axis of the microscope is usually vertical, place the object to be observed in a strain-free glass containing the refraction liquid. A major difference may exist, however: In the polarizing microscope, the vibration directions of the polarizer and analyzer are normally crossed in north-south and east-west positions. Accordingly, the "slow" ray directions of the quarter-wave and full-wave plates are oriented 45° counterclockwise to the standard positions of Fig. 2 This simply means that the "vertical" position of the stress component is now in a northwest-southeast orientation, but it does not change the meanings of the stress directions. In essence, the polarizing microscope usually has its directions of vibration rotated 45° counterclockwise to that shown in Fig. 2.

8.7.1 When it becomes necessary to measure retardations in excess of 565 nm (180° rotation of the analyzer), use a Berek rotary compensator or quartz wedge compensator (Babinet or Babinet-Soleil), (**3-6**) capable of measuring retardations up to four or more orders (four or more times the wavelength of the light source), in place of, or in addition to the quarter-wave plate. For the use of these instruments, refer to the manufacturer's manual and to references.

9. Calculations

9.1 *Retardation:*

9.1.1 The optical retardation at the point of measurement is calculated using:

$$R = \lambda \cdot \alpha / 180 = f \lambda \qquad (1)$$

where:

R = the optical retardation, nm,
α = the measured analyzer rotation, degrees,
λ = the wavelength of monochromatic light used in the polarimeter, nm (565 nm for white light), and
f = the fractional order, $f = \alpha/180$.

9.1.2 In polariscopes equipped with a dial graduated in fractional order α/180, use the dial reading *f*, instead of α/180.

9.2 *Birefringence:*

9.2.1 The average birefringence ($n_1 - n_2$) within the thickness *t* can be calculated using Eq 2:

$$n_1 - n_2 = R/t \qquad (2)$$

9.2.2 The birefringence is dimensionless, both *R* and the thickness *t* must be expressed in the same units.

9.3 *Stresses:*