Designation: C 978 – 02

Standard Test Method for Photoelastic Determination of Residual Stress in a Transparent Glass Matrix Using a Polarizing Microscope and Optical Retardation Compensation Procedures[1]

This standard is issued under the fixed designation C 978; 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 residual stresses in a transparent glass matrix by means of a polarizing microscope using null or retardation compensation procedures.

1.2 Such residual stress determinations are of importance in evaluating the nature and degree of residual stresses present in glass matrixes due to cord, or the degree of fit, or suitability of a particular combination of glass matrix and enamel, or applied color label (ACL).

1.3 The retardation compensation method of optically determining and evaluating enamel or ACL residual stress systems offers distinct advantages over methods requiring physical property measurements or ware performance tests due to its simplicity, reproducibility, and precision.

1.4 *Limitations*—This test method is based on the stress-optical retardation compensation principle, and is therefore applicable only to transparent glass substrates, and not to opaque glass systems.

1.5 Due to the possibility of additional residual stresses produced by ion exchange between glasses of different compositions, some uncertainty may be introduced in the value of the stress optical coefficient in the point of interest due to a lack of accurate knowledge of chemical composition in the areas of interest.

1.6 This test method is quantitatively applicable to and valid only for those applications where such significant ion exchange is not a factor, and stress optical coefficients are known or determinable.

1.7 The extent of the ion exchange process, and hence the magnitudes of the residual stresses produced due to ion exchange will depend on the exchange process parameters. The residual stress determinations made on systems in which ion exchange has occurred should be interpreted with those dependencies in mind.

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

1.9 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*

C 162 Terminology of Glass and Glass Products[2]

C 770 Test Method for Measurement of Glass Stress-Optical Coefficient[2]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[2]

F 218 Test Method for Analyzing Stress in Glass[2]

3. Terminology

3.1 *Definitions*—For additional definitions of terms used in this test method, refer to Terminology C 162.

3.1.1 *cord*—an attenuated glassy inclusion possessing optical and other properties differing from those of the surrounding glass. **C 162**

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *residual stress*—permanent stress that is resident in a glassy matrix. Such residual stress may result either from heat treatment above the strain point of the glass, or from differences in thermal expansion between the glass matrix and a cord, applied enamel, or ACL decoration.

3.2.1.1 *Discussion*—The residual stress may be modified either by heat treatment above the strain point, remelting and homogenizing the glass melt, or by removal of a fired-on ceramic or glass decoration. Residual stress caused by ion exchange may only be relieved by either reexchanging the glass to its original state, removing the exchanged glass from the matrix, or by remelting the exchanged glass and homogenizing the resulting glass melt.

3.2.2 *applied color label (ACL)*—vitrifiable glass color decoration or enamel applied to and fused on a glass surface.

3.2.3 *polarizer*—an optical assembly that transmits light

[1] This test method is under the jurisdiction of ASTM Committee C14 on Glass and Glass Products and is the direct responsibility of Subcommittee C14.10 on Glass Decoration.

Current edition approved Apr. 10, 2002. Published May 2002. Originally published as C 978 - 87. Last previous edition C 978 - 87 (1996).

[2] *Annual Book of ASTM Standards*, Vol 15.02.

vibrating in a single planar direction, typically positioned between a light source and the specimen being evaluated.

3.2.4 *retardation compensator*—an optical device, variants of which are used to quantify the optical retardation produced in transparent birefringent materials, typically positioned between the specimen being evaluated and the analyzer.

3.2.5 *analyzer*—a polarizing element, typically positioned between the specimen being evaluated and the viewer.

4. Summary of Test Method

4.1 This test method provides for the quantitative determination of residual stresses in transparent glass matrixes by means of photoelastic retardation compensation procedures. Compensation is achieved by producing a retardation null or extinction in the specimen using either rotating (11.2), birefringent quartz wedge (11.3), or tilting (11.4) optical retardation compensators.

5. Significance and Use

5.1 The quality and performance of an article of glassware may be affected not only by the presence of residual stresses due to heat treatment above the strain point in the ware, but also by additional residual stresses caused by differences in thermal expansion between the glass substrate, and either cord, fired-on vitreous enamel, or ACL decoration.

5.2 The effects of those additional residual cord, enamel, or ACL stresses and the resulting performance of such items may be evaluated by performance test procedures. Such evaluations of enamel or ACL stresses may also be accomplished through the determination of appropriate physical properties of the decoration and matrix glass, or by analytical methods.

5.3 This test method offers a direct and convenient means of determining the magnitudes and spatial distributions of residual stress systems in glass substrates. The test method is simple, convenient, and quantitatively accurate.

5.4 This test method is useful in evaluating the degree of compatibility between the coefficient of thermal expansion of an enamel or ACL applied to a glass substrate.

6. Apparatus

6.1 *Microscope*, monocular or binocular polarizing, having a rotating, and preferably graduated, sample stage. Binocular microscope heads frequently contain a second, separate polarizing element intended to minimize internal reflections. If such a binocular microscope is used, care should be taken to ensure that the antireflection polarizing element is removed from the field of view. An eyepiece containing mutually perpendicular or otherwise easily referenced crosshairs should be provided. For retardation determinations using rotating compensation methods, the polarizing microscope must be equipped with a rotatable analyzer element, having a scale graduated in degrees of rotation, capable of being read to at least 1°, and a quarter-wave plate, properly indexed.

6.2 *White Light Source* should be provided, together with strain-free objective lenses yielding overall magnifications ranging typically from 25 to 100×.

6.3 *Iris Diaphragm*, enabling collimation of the light beam transmitted through the specimen being evaluated.

6.4 *Compensator*, fixed full-wave retardation, commonly referred to as a sensitive tint plate, full-wave plate, or gypsum plate, having a fixed retardation value centered on 565-nm wavelength.

6.5 *Compensator*, [3] appropriate variable retardation, used to null or compensate, and thereby determine, the magnitude of the stress-optical retardation effect produced by the residual stress induced in the glass substrate. Variable compensators may be used.

6.5.1 *Wedge*, graduated birefringent, of continuously varying thickness, typically made of crystalline quartz, calibrated to yield retardation values directly and covering a range of four to six orders of retardation, or approximately from 2200 to 3300-nm total retardation.

6.5.2 *Tilting Compensator*, [4] typically capable of allowing determination of five orders of retardation.

6.5.3 *Rotating Compensator*, [5] typically allowing a determination of retardation of one order or one wavelength in magnitude to be determined. A monochromatizing filter is usually provided by the rotating compensator manufacturer. Care should be taken to use the appropriate matching filter for the particular rotating compensator being used.

6.6 *Data Conversion Tables*—The latter two tilting and rotating variable compensator types provide raw data in the form of angles of rotation, from which retardation data may be obtained through the use of conversion tables provided by the manufacturer, specific to the particular rotating compensator being used.

6.7 *Glass Immersion Dish*, strain-free, flat bottomed, of sufficient diameter to conveniently fit on the microscope stage. The immersion dish should not, in and of itself, add any significant optical retardation to the field of view. The dish should be of sufficient depth to enable the specimen section being evaluated to be completely immersed in an index of refraction matching immersion fluid.

6.8 *Suitable Immersion Fluid*, having an index of refraction matching that of the glass substrate being evaluated, generally to within ±0.01 units in refractive index as mentioned in Test Method F 218.

6.9 *Sample Holder*, to orient and maintain the planes of stress at the point of interest (POI), parallel to the optical column of the microscope, if the geometry of the specimen section is such that the planes of stress to be examined do not initially parallel the optical axis of the microscope.

6.10 *Means of Preparing the Section Containing the POI to be Analyzed*, such as an abrasive or diamond-impregnated cutoff wheel, or a hot wire bottle-cutting apparatus. Care should be taken to ensure that the section is not heated during cutting so as to affect the residual stress distribution in the specimen section.

6.11 *Means of Physically Measuring the Optical Path Length*, paralleling the stress planes through the thickness of the section containing the POI to within 0.03 mm (0.001 in.).

[3] Compensators of Senarmont, Berek, and Friedel type and graduated quartz compensators have been found suitable for this purpose.

[4] A compensator of the Berek type has been found satisfactory for this purpose.

[5] Compensators of the Friedel or Senarmont type have been found satisfactory for this purpose.

7. Sampling

7.1 The test specimens may be sections cut from appropriate locations containing areas of interest to be evaluated in production sampled articles of commerce, fired decorated or enameled ware, or laboratory specimens especially prepared for evaluation.

8. Test Specimens[6]

8.1 Ensure that the test specimen is appropriately annealed, in that retardation due to inappropriate annealing could affect the retardation due to the stress systems being evaluated at the POI.

NOTE 1—To ensure proper annealing, determine the stress-optical retardation in a comparable reference area of the test specimen away from the POI, free of ACL and other residual stress sources. Proper annealing should result in minimal retardation due to annealing stress in the selected reference area.

8.2 Cut a section, of generally not less than 2.0 mm (0.08 in.) and not more than 30.0 mm (1.18 in.) in optical path length, from the portion of the ware containing the POI. The section may then consist of a bar, a ring, or other appropriately shaped section.

8.2.1 In the case of ring section specimens, especially those used for cord, vitreous enamel, or ACL stress evaluations, open the ring section with a vertical saw cut to form a narrow kerf, relieving whatever architectural stresses may be present in the section.

8.2.2 Care should be taken to ensure that both cut section surfaces are parallel to each other, and are perpendicular to the optical path length of the section paralleling the planes of residual stress in the POI being evaluated.

8.3 If the sections being cut contain high magnitudes of retardation at the POI, the cut section thickness may be decreased proportionately from the thickness values listed in 8.2 to decrease the magnitude of retardation to be measured at the POI.

9. Preparation of Apparatus

9.1 Ensure that the microscope optical system is properly aligned and the objectives to be used in the examination are properly centered. The objectives should be relatively low powered, 2.5 to 10× being used during the initial examination procedure. The microscope eyepiece should contain a pair of mutually perpendicular or otherwise easily referenced crosshairs.

9.2 Orient the eyepiece such that one or both of the eyepiece crosshairs parallel the 45° diagonal positions in the field of view. The crosshairs will be used to orient the sections for which retardation determinations are to be made.

9.3 The microscope polarizing element should be oriented in the optical column at 0° or in an East-West (E-W) alignment, while the analyzer should be set in the field of view at 90° or a North-South (N-S) alignment, perpendicular to the polarizer. The microscope field of view should be at maximum darkness or extinction at this point if the polarizing elements are properly oriented, that is, mutually perpendicular to one another with no compensator installed.

9.4 If the field of view should not be at maximum darkness or extinction, the less-than-dark or brightened field indicates that the polarizing elements are not mutually perpendicular. The East-West alignment of the polarizer should be checked and then the analyzer should be rotated to a mutually perpendicular alignment with the polarizer, a position where the field of view is at its darkest, extinction position.

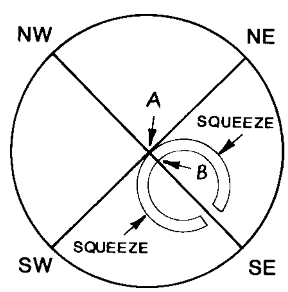
9.5 On insertion of a fixed, sensitive tint plate or a full-wave retardation plate in the microscope accessory slot, which plate is aligned at 45° between properly crossed polarizing elements, the darkened extinction field of view should then become reddish-purple or magenta in color.

10. Calibration and Standardization

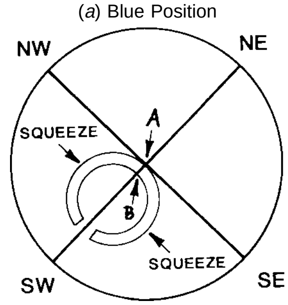
10.1 For microscopes and compensators that are not factory-standardized to determine the optical sign of stresses, the sense of the stresses being evaluated, that is, their tensile or compressive nature, must be established for the particular microscope being used with either a sensitive tint plate or full-wave fixed retardation compensator installed in the microscope column accessory slot between crossed polarizers. This may be accomplished, for instance, by positioning a well-annealed split ring section, containing a saw cut or kerf, in the field of view as shown in Fig. 1. A bar section, or other calibration section, may be similarly bent producing an identical effect.

NOTE 2—The calibration section used should have stress-optical retardation characteristics similar to the section being evaluated.

10.2 Orient the outer original surface of the section, directly opposite the kerf, to lie parallel to the diagonal Northeast-Southwest (NE-SW) direction in the field of view as seen in Fig. 1(*a*).



(*a*) Blue Position



(*b*) Orange Position

FIG. 1 Split Ring Section Used in Establishing Stress Sense and Proper Specimen Orientation

[6] "Polariscopic Examination of Glass Container Sections," *Journal of the American Ceramic Society*, Vol 27, No. 3, March, 1944.

10.3 Gently squeeze the ring section across a diameter paralleling the NE-SW diagonal to produce a tensile stress on the original outside section surface at the region of interest (POI) at Point *A*. A simultaneous compressive stress will be generated on the inside section surface near the POI at Point *B*, directly opposite Point *A* on the tensile surface.

10.4 Note and record the specimen POI orientation relative to the two diagonal positions, and the retardation color produced on the outside tensile surface of the section at Point *A* with the sensitive tint plate installed in the microscope.

10.5 Rotate the section 90° clockwise, such that the POI on the outer original section surface at Point *A*, opposite the saw kerf, is now oriented parallel to the Northwest-Southeast (NW-SE) diagonal in the field of view as seen in Fig. 1(*b*).

10.6 Gently squeeze the section in a direction paralleling the NW-SE diagonal and again note and record the POI orientation and the retardation color produced on the outside surface of the section due to the tensile stress at Point *A*.

10.7 The blue position is defined as that specimen POI orientation parallel to which a planar *tensile* stress of sufficient magnitude will be revealed by a bluish retardation color, between crossed polarizers with a sensitive tint plate or full-wave compensator installed. A *compressive* stress, of sufficient and equal magnitude, will be revealed by an orangy retardation color in the same blue specimen position.

10.8 When the specimen section POI is then rotated 90° from the blue position to the position where its outside surface parallels the diagonal position opposite the blue position, that same *tensile* stress will appear as an orangy retardation color, hence the name, orange position. The corresponding *compressive* stress, of sufficient and equal magnitude, will now appear as a bluish retardation color in the orange position.

10.9 Retardation readings should be referenced to the particular position, that is, blue or orange position, in which the retardation readings were made.

10.10 Typical specimen section POI orientations, relative to the particular compensator slow-wave direction necessary to provide proper blue- and orange-position locations for the variable compensators described in this test method, are shown in Table 1.

10.11 The particular diagonal specimen POI orientation corresponding to the blue position in a specific quadrant within the field of view may vary for different microscopes, depending on the particular orientation of the polarizing elements and compensators being used. Therefore the specimen-section positioning procedures outlined in 10.2 through 10.8 should be periodically checked and reaffirmed.

TABLE 1 Retardation Color Equivalents With and Without Sensitive Tint Plate (Observed Color in Flint Soda-Lime-Silica Glass Only)[A]

NOTE 1—Letters *a* through *o* indicate the most distinctive colors for various ranges. When using the tint plate in the orange position, if the color appears to fall between *c* and *e*, reorient the POI to the blue position, and verify that the retardation color at the POI is indeed *d*.

NOTE 2—The retardation colors indicated in the table are referenced only to transparent colorless flint soda-lime-silica glasses.

With 565-nm Sensitive Tint Plate

Blue Position	Orange Position	Equivalent Retardation, nm
violet-red	violet-red	0
violet-blue	(a) red	(a) 20
(b) dark blue	red-orange	(b) 35
blue	(c) orange	(c) 75
(d) blue-green	orange-yellow	(d) 120
deep green	(e) gold-yellow	(e) 150
(f) green	yellow	(f) 180
pale green	(g) pale yellow	(g) 220
(h) yellowish green	yellow-white	(h) 255
greenish yellow	(i) white	(i) 290
pale yellow	gray-white	330

Without 565-nm Sensitive Tint Plate

Orange position	Equivalent Retardation, nm
black	0
gray (various shades)	up to 255
gray-yellow	290
(j) dirty yellow	(j) 330
(k) dirty brown	(k) 380
(l) brown-orange	(l) 440
(m) brown-red	(m) 480
(n) violet-red	(n) 565
(o) blue-green	(o) 675

[A] "Polariscopic Examination of Glass Container Sections," *Journal of the American Ceramic Society*, Vol 27, Number 3, March 1944.

11. Procedure

11.1 *Specimen Orientation*—Rotate the graduated microscope stage containing the specimen section so that the POI in the specimen section to be evaluated is in a N-S orientation.

11.1.1 The specimen section POI containing the residual stress system to be analyzed should be uniformly dark, with the fixed compensator removed from the field, as should the background field of view exterior to the specimen. The specimen POI is said to be in the EXTINCTION position in this orientation.

11.1.2 The POI should exhibit complete extinction on being rotated to successive 90° positions relative to the initial N-S POI orientation.

11.1.3 Rotate the microscope stage bearing the specimen section POI exactly 45° clockwise using either the graduated rotating stage scale or the eyepiece crosshairs as references from the N-S orientation achieved in 11.1, to put the specimen section surface containing the POI to be analyzed in a DIAGONAL (NE-SW) orientation.

11.1.4 The POI should exhibit its maximum brightness or highest order retardation color in this position.

11.1.5 Rotate the specimen section 90° counterclockwise to the opposite diagonal position, that is, paralleling the DIAGONAL (NW-SE) orientation.

11.1.6 Observe and note the orientation and the retardation color seen in the specimen POI, both with and without the sensitive tint plate installed, in both orientations.

11.1.7 The complementary retardation colors observed in the POI when oriented in opposing diagonal positions, both with and without the tint plate installed, may be used in conjunction with Table 1 to qualitatively determine retardation values corresponding to various retardation colors observed in the POI in colorless or flint soda-lime-silica glass only.

11.1.8 Table 1 may be used to obtain an initial estimate of the magnitude of retardation present at the POI. This estimation procedure also serves as a verification of the respective quantitative retardation determination procedures detailed in 11.1.9 through 11.1.13.

11.1.9 Note the orientation of the slow-wave reference direction indicated on the body of the fixed sensitive-tint or full-wave compensator.

11.1.10 Insert the fixed compensator into the accessory slot of the microscope optical column. The slow-wave direction of the fixed compensator should parallel the NE-SW diagonal position as shown in Fig. 2.

11.1.11 Observe and note the change in the retardation color exhibited at the POI in the specimen positioned under the intersection of the eyepiece crosshairs upon complete insertion of the compensator.

11.1.12 If the retardation color at the POI increases in order on insertion of the fixed retardation compensator, that is, different values of retardation color corresponding to increasing retardation values are seen, as depicted in a Michel-Levy retardation color chart, the specimen POI is oriented in the ADDITIVE position.

11.1.13 However, if the retardation color at the POI decreases in order on insertion of the fixed compensator, the specimen POI is said to be in the SUBTRACTIVE position.

11.1.14 To make a valid retardation determination, extinction, or retardation compensation, must be produced in the POI in the specimen section under examination. Since tensile or compressive stresses will produce positive or negative retardations, respectively, in most common glass systems, the sense of the residual stress systems must be established using the procedures outlined in 10.2 through 10.8, and the POI properly oriented to the slow-wave direction of the particular variable compensator being used. The retardation produced in the POI must then be exactly compensated or nulled by the particular variable retardation compensator being used.

11.2 *Rotating Compensator Retardation Determination*:

11.2.1 With the specimen section containing the POI removed from the field of view, insert a monochromatizing filter, appropriate to the particular rotating compensator being used, into the microscope optical path below the microscope condensing lens assembly. This filter monochromatizes white light for the rotating compensator retardation measurement procedure, and is closely matched to a specific rotating compensator. Rotating compensators may be constructed to require either a 546 or a 589-nm monochromatizing filter. Ensure that the correct filter is being used with the appropriate rotating compensator. The transmitted wavelength must be known, and is used in the computation of the measured retardation.

11.2.2 Insert the fixed subparallel retardation compensator fully into the microscope accessory slot with the double-headed arrow indicating the slow-wave direction marked on the compensator body in an East-West (E-W) orientation, as shown in Fig. 3. Adjust the subparallel compensator such that the field is at maximum extinction. Secure the compensator in the maximum EXTINCTION position.

NOTE 3—The microscope polarizer should have been previously aligned to a 0° or E-W orientation, and the analyzer should have been set to a 90° N-S orientation producing extinction or a darkened field of view, as detailed in Section 9 on Preparation of Apparatus.

11.2.3 Position the specimen POI containing the tensile stress to be analyzed under the microscope crosshairs, and orient the specimen section to a diagonal (NE-SW) alignment as shown in Fig. 1(*a*), using the specimen orientation procedure detailed in 10.2 through 10.8.

11.2.4 If the specimen contains residual strain located at the POI, the POI will exhibit a retardation effect in the form of a brightened field at the POI, rather than appearing completely dark or extinct. Maintain the specimen POI in its (NE-SW) diagonal position.

11.2.5 Rotate the analyzer element to an increasing rotation scale value, beginning at the 90° EXTINCTION position. Observe whether a null or extinction is produced at the exact location within the POI where compensation is to be determined. Compensation will be achieved at an analyzer rotation where a zero order or dark retardation band becomes positioned at the POI under the crosshairs as the analyzer rotation

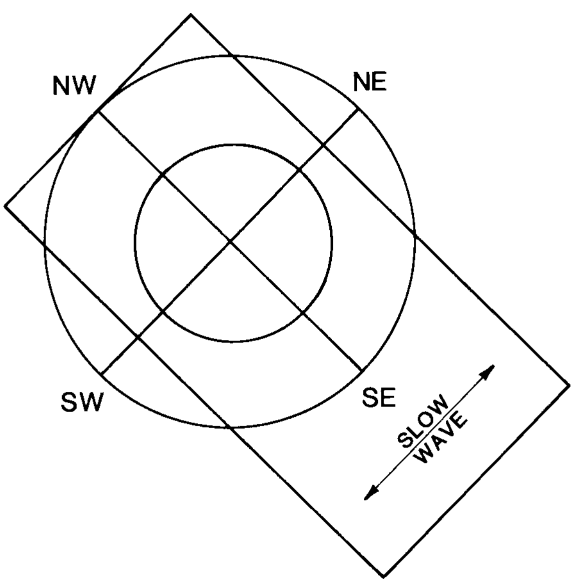


FIG. 2 Orientation of the Fixed Retardation Compensator Slow-Wave Vibration Direction

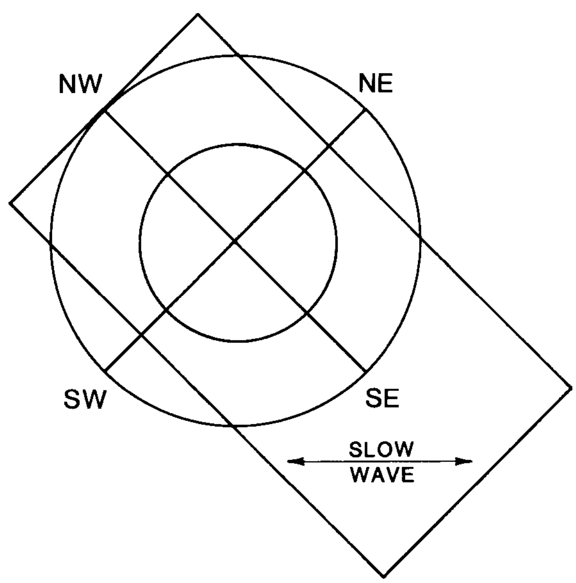


FIG. 3 Orientation of the Rotating Compensator Slow-Wave Vibration Direction

is increased from its initial 90° setting. Note and record the analyzer rotation setting corresponding to the extinction position at the POI.

11.2.6 If extinction, in the form of a darkened zero-order retardation band, does not occur at the POI in the first 90° of analyzer rotation, that is, by an indicated angle setting of 180° rotation, the specimen POI must then be rotated 90° to the alternative diagonal position, and the compensation procedure, as detailed in 11.2.5, repeated to produce compensation or extinction at the POI.

11.2.6.1 Reverse the POI positions in the procedure in 11.2.5 and 11.2.611.2.6 in the case of a compressive stress determination.

11.2.7 The microscope analyzer setting from 90 to 180° rotation, at which compensation or extinction is produced in the POI, is then used to determine an optical retardation value, *R*, as follows:

$$R = \lambda \times a / 180° \quad (1)$$

where:

λ = wavelength, and

a = angle of rotation.

11.2.8 The retardation value is then used in the calculation procedure detailed in Section 12 to obtain the value of residual stress at the POI.

11.3 *Graduated Quartz Wedge Optical-Retardation Determination*:

11.3.1 If a graduated birefringent wedge (hereinafter referred to as *the wedge*) is to be used as the variable retardation compensator, attach the wedge device to the uppermost portion of the monocular microscope column, replacing the eyepiece normally positioned in the optical column. (See Fig. 4.)

11.3.2 Remove the normal microscope analyzer, any fixed retardation compensator, and specimen sections from the microscope column.

11.3.3 The wedge device typically consists of a sliding assembly containing the wedge compensator; the device allows the wedge to be pushed through the field of view, an eyepiece containing mutually perpendicular or other easily referenced crosshairs, and an integral polarizing element that replaces the normal microscope analyzer.

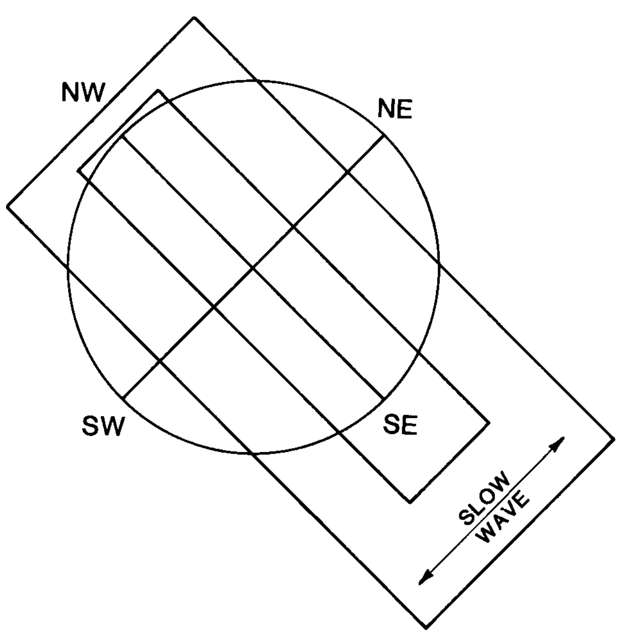


FIG. 4 Orientation of the Birefringent Wedge-Compensator Slow-Wave Vibration Direction

11.3.4 The presence of the additional integral polarizing element in the wedge assembly requires the removal of the normal microscope analyzing element from the microscope optical column.

11.3.5 The wedge assembly eyepiece lens should be focused on the crosshair and wedge retardation scale; one of the eyepiece crosshairs should be aligned parallel to the retardation scale appearing on the sliding wedge body in the field of view.

11.3.6 With the specimen section removed from the field of view, and the wedge removed from the field of view, rotate the wedge assembly such that the polarizing element in the microscope and the analyzer element in the wedge assembly are mutually perpendicular, producing a darkened extinction field. Tighten the wedge mounting attachments to firmly fix the wedge device on the microscope column in the EXTINCTION position, and prevent rotation of the assembly.

11.3.7 Slowly push the wedge compensator through the optical column, observing the progression of retardation colors passing through the field of view under the eyepiece crosshairs.

11.3.8 As the wedge is pushed through the optical column, the order of progression of retardation colors observed may initially decrease from first order retardation colors to a zero order or darkened extinction retardation band immediately under the crosshair reference line at the extinction position. Check to ensure that the extinction position corresponds to a zero retardation value on the calibrated wedge retardation scale.

11.3.9 As the wedge continues to be pushed through the optical column in the same continuing direction past the extinction point, the retardation colors will then be observed to increase in order. The progression of retardation colors produced will continue to increase in order, from black at the EXTINCTION position, to increasingly lighter shades of gray to white to yellow to red, following the sequence shown in Table 1 for increasing values of retardation.

11.3.10 The first red retardation color observed at this point in the progression of retardation colors is referred to as first-order red.

11.3.10.1 The retardation color progression continues to increase in order from red to blue to green to yellow to a second, somewhat paler than the first, red retardation color. The latter red retardation color is referred to as second-order red. The progression of retardation colors from the last mentioned yellow upward are slightly paler in appearance than the first-order series, but are slightly more intense than the third-order series which follow, and so on in the progression to higher-order colors. The retardation colors continue to fade in hue with increasing order, becoming more and more pastel, until the separate colors become relatively indistinguishable beyond the fifth order.

11.3.11 The darkened or black zero retardation line, which indicates extinction or complete retardation compensation, is observed to pass through the zero-retardation reference marking on the graduated retardation scale contained on the quartz wedge body. As the wedge is pushed through the field of view, the first-order magenta band is observed to occur at a retardation reading of 565 nm, the second-order magenta band occurs at 1130 nm, etc., as the wedge continues to be pushed through the microscope column.

11.3.12 Position the zero retardation line under the eyepiece crosshairs corresponding to a zero retardation value as noted on the wedge reference scale.

11.3.13 Insert either the sensitive tint plate or the 565-nm compensator into the microscope column accessory slot.

11.3.14 Observe and note the differences produced in the retardation bands in the field of view.

11.3.15 The black extinction band, formerly at 0°, should now be a magenta color, corresponding to a retardation of 565 nm. This results because 565 nm of retardation has been added by the sensitive tint plate to the initial retardation value of 0° at the reference point on the wedge body.

11.3.16 The former first-order magenta band, corresponding to a retardation of 565 nm should now appear as a second-order magenta band, corresponding to a retardation of 1130 nm. This latter retardation value is produced by an addition of two separate retardations, one produced by the sensitive tint plate, and the second produced by the quartz wedge.

11.3.17 The previous intermediate retardation colors are all increased by the additional 565 nm of retardation added by the sensitive tint plate, resulting in the retardation colors appearing in the field being increased by the one order of retardation produced by the compensator.

11.3.18 Remove the sensitive tint-plate compensator from the accessory slot, and reaffirm that the wedge analyzer and microscope polarizer are mutually perpendicular by the presence of a darkened field of view.

11.3.19 Now insert the specimen section containing the POI to be evaluated into the field of view.

11.3.20 Orient the specimen POI to a DIAGONAL alignment, that is, parallel to the long axis of the wedge, and perpendicular to the slow-wave direction of the wedge as detailed in 10.2 through 10.8, and shown in Fig. 1(*b*).

11.3.21 Slowly push the wedge compensator through the optical column until a darkened retardation extinction band occurs in the POI under the wedge-assembly crosshairs.

11.3.22 The darkened retardation extinction band within the POI will be observed to deflect away from the crosshair centered on the zero-retardation reference line, to either positive- or negative-retardation scale readings on the graduated wedge-reference scale. A determination must be made of the position of maximum zero-retardation band deflection in the POI which corresponds to the location of maximum positive or negative retardation.

11.3.23 The wedge must be positioned, by carefully sliding the wedge compensator back and forth in the optical column, so that the point of maximum zero retardation or extinction-band deflection in the POI is centered under the wedge-assembly crosshair.

11.3.24 Note the corresponding value of retardation, expressed in either nanometers (nm), millimicrons (mμ), or micrometres (μm) indicated on the wedge reference scale falling under the wedge-eyepiece crosshair, when that crosshair is held perpendicular to both the wedge reference scale and the POI. This reading is the retardation value produced by the wedge compensator at the POI, which nulls or compensates the retardation present in the POI. That retardation value will then be used to calculate the magnitude of the residual stress at the POI utilizing the procedure detailed in Section 12.

11.3.25 The same retardation-determination technique, detailed above, may also be repeated with the sensitive tint plate in place. The location of the point of maximum zero-order retardation-band deflection, or the extinction point in the POI, may be facilitated through the use of the sensitive tint plate. In that case, extinction will be indicated by a first-order magenta color at the POI with the sensitive tint plate in place, rather than a darkened or black retardation color, as is found with the tint plate removed.

11.4 *Tilting Compensator Optical-Retardation Determination*:

11.4.1 Ensure that the tilting compensator is set at its ZERO setting prior to inserting the compensator assembly into the microscope accessory slot. (**Warning—**Failure to position the compensator dial to ZERO could result in damage to the tilting compensator body.)

11.4.2 Verify that the polarizer is set at an E-W orientation, and the analyzer is set in the mutually perpendicular 90° N-S position; remove any fixed compensators from the microscope column. The field of view should be darkened or at extinction in this orientation with the specimen section POI removed from the field of view.

11.4.3 Position the specimen containing the POI under the eyepiece crosshairs, and align the specimen POI to one of the diagonal positions, as detailed in 10.2 through 10.8, and as shown in Fig. 1(*a*) and (*b*).

11.4.4 Insert the tilting compensator into the accessory slot. Ensure that the slow wave vibration direction marked on the compensator parallels the (NE-SW) DIAGONAL position as shown in Fig. 5.

11.4.5 With the compensator set at 0° rotation, the POI should exhibit a retardation of order greater than zero, that is the POI should not be at EXTINCTION, if the POI contains residual stress.

11.4.6 Slowly rotate the compensator dial from zero to a larger angle setting in one of the two clockwise or counter-clockwise-rotation directions.

11.4.7 Observe whether a darkened extinction or zero retardation band crosses the POI from a direction paralleling the rotating compensator slow-wave or NE-SW direction, prior to the observation of a first-order red retardation band as the compensator dial setting is increased.

11.4.8 If a darkened extinction or zero retardation band is not observed at the POI prior to the observance of a first-order red retardation color, rotate the compensator dial back to its zero setting, and then rotate the compensator dial to increasing angles of tilt in the opposite direction of rotation.

11.4.9 If the first retardation color observed passing through the POI from the opposite direction of compensator tilt is red, rather than a darkened extinction zero-order retardation band,

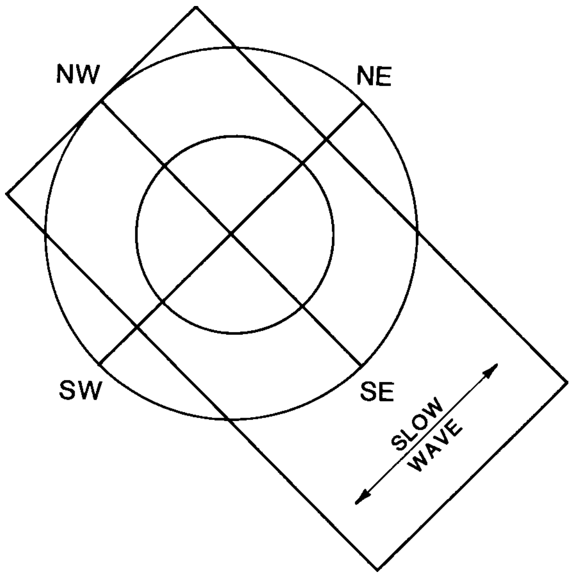


FIG. 5 Orientation of the Tilting-Compensator Slow-Wave Vibration Direction

the specimen is in the ADDITIVE diagonal position, where the separate specimen and compensator retardations add to one another. A retardation null or extinction is not possible in that ADDITIVE diagonal POI orientation. The specimen POI must be rotated 90° to the opposite SUBTRACTIVE diagonal position, where a retardation null or extinction can be achieved.

11.4.10 Rotate the specimen section 90° to a (NW-SE) diagonal alignment using the graduated specimen stage. The specimen POI should now be in the SUBTRACTIVE diagonal position, and should exhibit a retardation color of order lower than the darkened zero-order retardation band, if both residual stress is present at the POI and the analyzer dial is set at zero.

11.4.11 Rotation of the analyzer dial in one tilt direction should now move a darkened extinction or zero-order retardation band through the POI, from a direction paralleling the (NE-SW) diagonal. The analyzer dial reading should be noted once the darkened extinction band has been carefully centered on the POI.

11.4.12 The analyzer dial should then be rotated back to zero, with rotation continuing in the opposite tilt direction. A second darkened extinction or zero-order retardation band will be observed to sweep through the POI in the field of view from a direction opposite to that of the first extinction band, but paralleling the same direction of motion as the previously noted extinction band. After centering the second extinction band on the POI, the analyzer dial reading should again be noted.

11.4.13 The two analyzer-dial tilt-angle readings, corresponding to the two extinction positions produced in the POI, are used to determine an optical-retardation value in accordance with the procedure established by the manufacturer for that particular tilting compensator. That retardation value is then used in the calculation procedure detailed in Section 12 to determine the value of the residual stress at the POI.

12. Calculation

12.1 The residual stress value, corresponding to the difference in principal stresses lying parallel to the section surfaces being evaluated, may be calculated from the determined optical retardation data, the stress optical coefficient of the matrix glass in question, and the thickness of the section at the POI as follows:

$$S = \frac{R}{(K)\,(C)\,(d)} \qquad (2)$$

where:

S = calculated stress corresponding to the difference in principal residual stresses, Pa,
R = measured optical retardation value, nm,
C = stress-optical coefficient of glass,
d = section optical path length at the POI, cm, and
K = units conversion factor.

12.2 The following equations typically apply to retardation measurements and data that are obtained for soda-lime-silica glasses typical of either container, tableware, or flat-glass manufacture, in the listed units:

$$S = \frac{[1.50 \times 10^{4}] \times R}{d} \qquad (3)$$

$$S = \frac{[3.80 \times 10^{4}] \times R}{d} \qquad (4)$$

12.3 The equations in 12.2 assume that the soda-lime-silica glass section in question has a stress-optical coefficient of 2.63 Brewsters. For glass compositions whose stress-optical coefficients are different, a correction factor equal to the appropriate ratio of the respective stress-optical coefficients may be used in the above equations. Stress-optical coefficient data for representative generic glass types are listed in Table 2.

12.4 Weigh the calculated maximum residual-tensile-stress values depending on the location in the glassware specimen from which the sections containing the POI's were cut, in accordance with the following scheme:

12.4.1 Multiply tensile stress, located directly on the outside surface of the ware from which the sections were obtained, and which surface is subject to mechanical damage, by a factor of 1.

12.4.2 Multiply tensile stress, located directly on the inside surface of the ware from which the sections were obtained, and

TABLE 2 Stress-Optical Coefficients of Glasses[A]

Type of Glass	Stress-Optical Coefficients,[B] B′, nm/mm·MPa	(B, Brewsters)
Silica	701.21	(3.47)
96 % silica	742.46	(2.45–2.65)
Soda-lime	494.97–536.22	(2.45–2.65)
Lead-alkali-silicates:		
Medium lead content	515.60–556.84	(2.55–2.75)
60 % PbO	412.48	(2.05)
73 % PbO	49.50	(+0.24)
80 % PbO	206.24	(−1.05)
Borosilicate		
Low expansion	783.70	(3.9)
Borosilicate		
Low electrical loss	969.32	(4.8)
Aluminosilicate	536.22	(2.65)

[A] Shand, E. B., *Glass Engineering Handbook*, 3rd Ed., McGraw-Hill, New York, 1984, Table I-12, pp. 2–31.

[B] Conversion to metric engineering units: The stress-optical coefficient data may be converted to metric engineering units of (mµ·cm/kg) by division of each (nm/mm·MPa) value by 206.238, and multiplying each Brewster value by 209.126, respectively.

which surface is not subject to mechanical damage, by a factor of two thirds (0.667).

12.4.3 Multiply tensile stress, buried within the thickness of the section from which the sections were obtained, which are not subject to mechanical damage, and which are away from both interior and exterior surfaces, by one third (0.333).

13. Interpretation of Results

13.1 These procedures may be used to determine those residual stress values in glass substrates where the stress optical coefficient is either known or determinable.

13.2 In certain systems in which compositional changes due to ion exchange may be produced between substrates containing different alkali ion species, those compositional changes may lead to an uncertainty of the stress-optical coefficient in a POI immediately joining the two substrates. The ion exchange process itself may lead to the formation of tensile stresses in the substrate in which different sized ions are chemically exchanged. Consideration of those ion-exchange stresses must be included in any interpretation of stress systems for which residual stresses are being analyzed, and in which ion exchange is a possibility.

13.3 The presence of tin- or titanium-oxide coatings on the glass surfaces may produce an optical-retardation effect, giving the appearance of an inhomogeneity lying on the surface of a section being evaluated. Such coatings will produce a high-order retardation, lying in an extremely thin layer on the outside surface of the section. The retardation, due to such an oxide layer, will exhibit extinction on rotation of the section. If the retardation disappears after treating the section surface with a 6 volume % caustic sodium hydroxide solution at 60° C for a 1-h duration, the observed retardation is due to oxide coating, and does not represent an inhomogeneity.

14. Report

14.1 Report the following information:

14.1.1 Name of the investigator, method of retardation compensation used, and full description of the specimens evaluated, as follows:

14.1.2 Date, time, location, and manner in which the evaluated specimens were obtained.

14.1.3 Locations within each specimen from which sections containing the respective points of interest were obtained.

14.1.4 Individual retardation values determined for each specimen POI.

14.1.5 Thickness of each section at the respective point of interest.

14.1.6 Details of the calculation of residual stress, in accordance with the procedure outlined in Section 12.

14.1.7 Magnitudes of the calculated residual stresses, listing of their tensile or compressive natures, and detailed description of the appearance, orientation, and location of the stress system at each respective POI.

15. Precision and Bias

15.1 This study was performed with six laboratories using six materials with three test results per material. The minimum requirements for an ASTM study are six laboratories, four materials, and two test results per material. ASTM software, in accordance with Practice E 691, was used to compute the repeatability and reproducibility.

15.1.1 Four of the laboratories used the quartz wedge technique and two laboratories used a polarimeter.

15.2 *Precision Statement for Residual Stress*—Precision, characterized by repeatability, *(r)*, repeatability standard deviation, S_r, reproducibility, *(R)*, and reproducibility standard deviation, S_R, was determined for the materials listed in Table 3*:*

TABLE 3 Precision Calculations

NOTE 1—In order to use Practice E 691 software, negative numbers have to be eliminated. As a result of this, 90 nanometres (nm) was added to every value of retardation.

Material	Retardation AVG[A](nm)	S_r	S_R	*r*	*R*
1	165.694	4.090	28.761	11.452	80.531
2	97.122	8.594	11.409	24.062	31.945
3	213.667	4.665	18.500	13.063	51.799
4	56.028	11.018	28.926	30.851	80.992
5	214.439	6.696	25.079	18.749	70.220
6	96.833	8.179	11.357	22.903	31.801

[A] Average (AVG) was determined by round robin testing in Subcommittee C14.10.