



Designation: C978 – 04 (Reapproved 2019)

Standard Test Method for Photoelastic Determination of Residual Stress in a Transparent Glass Matrix Using a Polarizing Microscope and Optical Retardation Compensation Procedures¹

This standard is issued under the fixed designation C978; 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

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.

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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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.10 *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

C162 Terminology of Glass and Glass Products

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

F218 Test Method for Measuring Optical Retardation and Analyzing Stress in Glass

3. Terminology

3.1 *Definitions:*

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

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

3.2 *Definitions of Terms Specific to This Standard:*

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

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.

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

3.2.4 *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.

3.2.4.1 *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.

3.2.5 *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.

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*, 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*, typically capable of allowing determination of five orders of retardation.

6.5.3 *Rotating Compensator*, 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 F218.

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.).

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 Specimens³

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).

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.

³“Polariscopic Examination of Glass Container Sections,” *Journal of the American Ceramic Society*, Vol 27, No. 3, March, 1944.

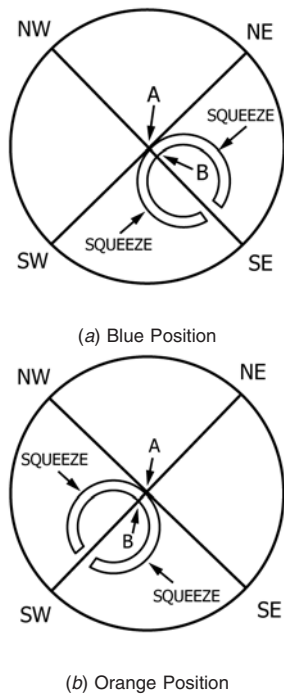


FIG. 1 Split Ring Section Used in Establishing Stress Sense and Proper Specimen Orientation

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.

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.

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 i 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.

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

11.1.2 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

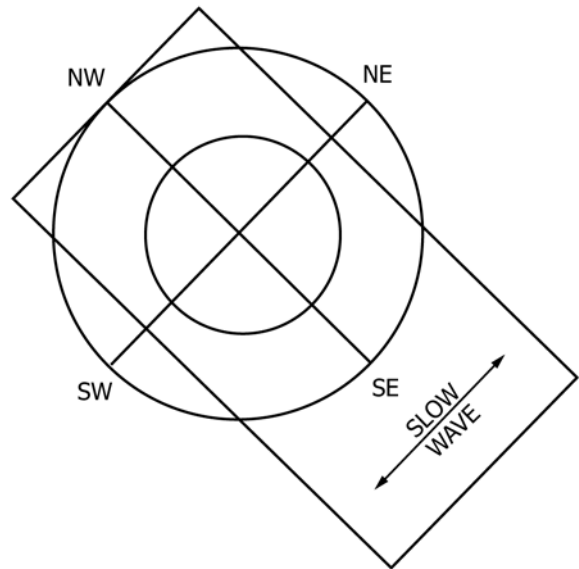


FIG. 2 Orientation of the Fixed Retardation Compensator Slow-Wave Vibration Direction

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.

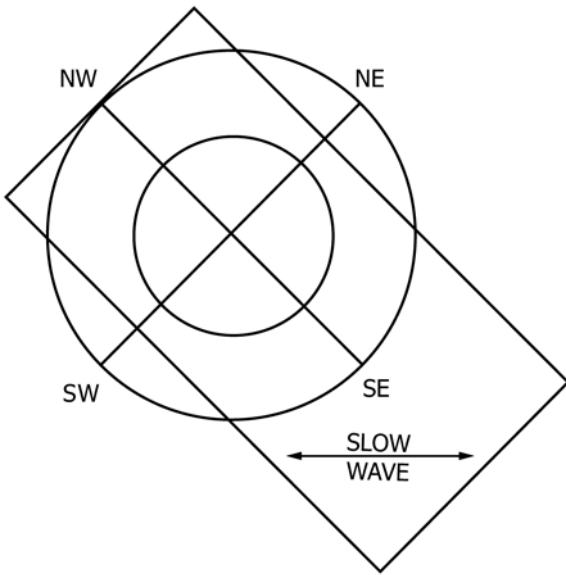


FIG. 3 Orientation of the Rotating Compensator Slow-Wave Vibration Direction

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.

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

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 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 11.2.6 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:

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

where:

- λ = wavelength, and
- a* = angle of rotation.

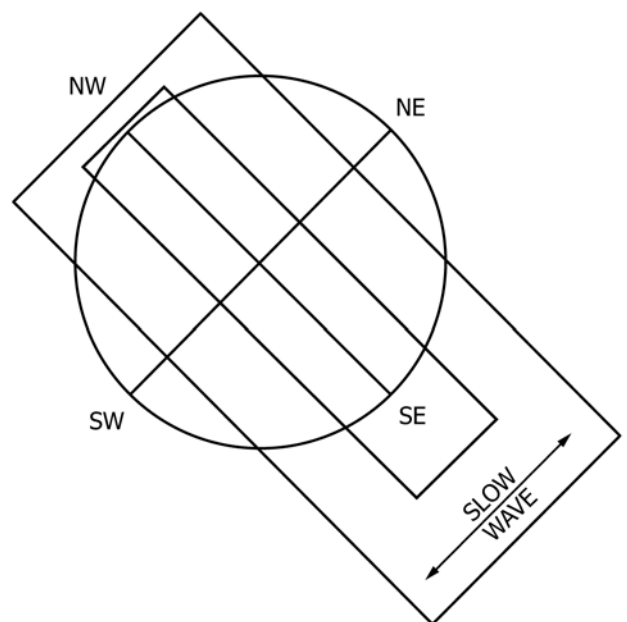


FIG. 4 Orientation of the Birefringent Wedge-Compensator Slow-Wave Vibration Direction