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# Device and method of optically orienting biaxial crystals for sample preparation

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An optical instrument we refer to as the "biaxial orientation device" has been developed for finding the optical plane, acute bisectrix, and obtuse bisectrix in biaxial crystals by means of optically aligning conoscopically formed melatopes and measuring the angular coordinates of the melatopes, where the angular values allow for determination of the optical plane containing the optical axes using a vector algebra approach. After determination of the optical plane, the instrument allows for the sample to be aligned in the acute bisectrix or obtuse bisectrix orientations and to be transferred to a simple mechanical component for subsequent grinding and polishing, while preserving the orientation of the polished faces relative to the optical plane, acute bisectrix, and obtuse bisectrix during the grinding and polishing process. Biaxial crystalline material samples prepared in the manner are suitable for accurate spectroscopic absorption measurements in the acute bisectrix and obtuse bisectrix directions as well as perpendicular to the optical plane. © 2014 AIP Publishing LLC. [http://dx.doi.org/10.1063/1.4894555]

## I. INTRODUCTION

### A. Challenges in orientating biaxial crystals for optical measurement

Optical absorption spectra of biaxial crystals are, by convention, usually measured along the fast, slow, and perpendicular to the optical plane directions corresponding to the directions in which the $\alpha$, $\beta$, and $\gamma$ refractive indices would be measured. In monoclinic crystals, two of these principal optical directions do not align with the crystal axes and in triclinic crystals none of them do. Thus, it is usually necessary to use optical methods rather than X-ray methods to align such crystals for spectroscopic study. Well established methods exist for crystals less than a millimeter in size using spindle stages and the methods of optical crystallography. These methods are generally not practical for larger crystals. In practice, the objective is to align a crystal such that when the spectrum is taken, the incident light is traveling down either the Bxa, the Bxo, or the optic normal directions (Fig. 1) so that linearly polarized light can be aligned with two of the three principal optical directions that coincide with the Bxa, Bxo, and optic normal directions.

Small crystals can be aligned in these orientations using the multi-axis stage accessory on petrographic microscopes. However after orientation, transferring the sample in the aligned condition for grinding and polishing of the input and output optical faces needed for spectroscopy is problematic. X-ray diffraction systems are able to align and mechanically preserve the orientation of orthorhombic biaxial crystals, and can locate the b-axis of monoclinic crystals, but these systems are expensive and much slower than the apparatus developed in this work. X-ray systems are able to orient biaxial crystals that are not transparent to the visible light spectrum. While the biaxial orientation device described herein is operated in the visible light spectrum, modifications to work in the UV (ultraviolet), NIR (near-infrared), and IR (infrared) regions are theoretically possible.

When viewing the biaxial crystal in convergent (spherical waves) polarized light, the fast and slow light paths of the crystal will diffract the incident rays at angles related to the refractive indices of the fast and slow axes. When viewed through crossed polarizers, the different ray paths created by the fast and slow indices appear as dark bands where the phase addition of the fast and slow rays add to form linearly polarized light that is rotated by 90°. These dark bands are referred to as isogyres, and their curvature is greatest near the circular figure formed around the centers of each of the crystalline axes of the crystal. The point of emergence of an optic axis, referred to as a melatope, is the point at the center of the bulls-eye pattern of nearly circular thin dark bands (isochromes) in Figure 2. There are two melatopes on each side of the crystal, one for each of the optic axes. Once the melatopes are found, the optic plane has been found. Finding the melatopes optically can be challenging if, for example, the sample is oriented in the apparatus in such a way that one or both of the axes are not able to be viewed optically.

In practice, biaxial crystal samples often have no reference on the external surfaces that provide an indication for locating the optic plane. Further, natural crystals are often found with fractured edges that are not ideal for optical analysis. To accommodate fractured edges and any possible random geometric external surface shape, a method of launching light into and out of the crystal sample while minimizing light ray angular errors must be utilized, such as immersion in an index matching liquid.

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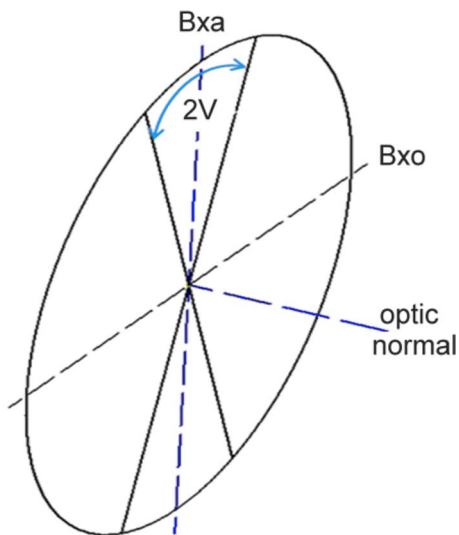a)Electronic mail: Tim.Thomas@gia.edu. URL: http://www.gia.edu.

FIG. 1. Diagram illustrating the optical plane containing the acute bisectrix (Bxa), obtuse bisectrix (Bxo), the 2 V angle between the optical axes of the biaxial crystal, and the direction normal to the plane.

Indexing matching liquids are only an approximate match for the refractive index range of the crystal. As such, the in plane 2V angle of the crystal is approximate, with the error related to the mismatch of the index of the index matching liquid and the range of refractive indices of the crystal, as well as the particular shape of the crystal faces. Similarly, out of plane positions of the melatopes can be caused by refractive index matching liquid mismatching, and external surface geometries of the crystal.

The optical system used to create the convergent polarized light must have an optical axis that is stable and aligned relative to the mounted sample. Since the goal is to attach the sample to a mechanical component (a transfer dop) after the crystal is oriented, the mechanism for attaching the dop must have mechanics that are aligned to the optical axis of the instrument. These mechanics must allow for x, y, and z placement of the transfer dop so that the transfer dop can be bonded to an appropriate portion of the crystal sample using a photo-

initiated adhesive, and hold the transfer dop stable during the curing process of the adhesive.

During observation of the melatopes, the crystal is viewed through an immersion cell containing the index matching liquid. Once the crystal is aligned, the immersion cell must be removed prior to attachment of the transfer dop. The immersion liquid must be cleaned from the crystal sample without disturbing its alignment so that the photo-initiated adhesive will bond correctly.

## B. Basis for the biaxial orientation device, the conoscope, and the uniaxial orientation device

The Western Electric Conoscope[1–3] instrument illustrated the concept of a conoscope with convergent polarized light. The instrument contained a light source, spherical lens elements for collecting and collimating the light, a polarizer, focusing lenses to form the convergent ray pattern into the sample, an immersion cell for containing the sample, collection optics to collimate the output of the sample, and a cross polarizer analyzer to create the isogyre pattern viewed by the user. Using this concept as the basis of his instrument, Emmett (Crystal Chemistry, Brush Prairie, Washington) built a similar instrument, but substituted plastic Fresnel lenses for the spherical lens elements used in the Western Electric instrument, and inexpensive film polarizers. Emmett's instrument was specifically for aligning uniaxial crystals. Emmett shared his uniaxial instrument design with the Gemological Institute of America (GIA), where Thomas collaborating with Emmett added laser alignment capability to the optical components, a reference fiducial for aligning the isogyre pattern of uniaxial crystals, and precision mechanics for attaching the grinding and polishing dop while preserving the c-axis alignment of the sample. The resulting uniaxial instrument, "Corundum c-Axis Device for Sample Preparation,"[4] can be viewed on GIA's website.

## II. SETUP

In this section, the optical components, optical alignment, and use of the biaxial orientation device are described. All
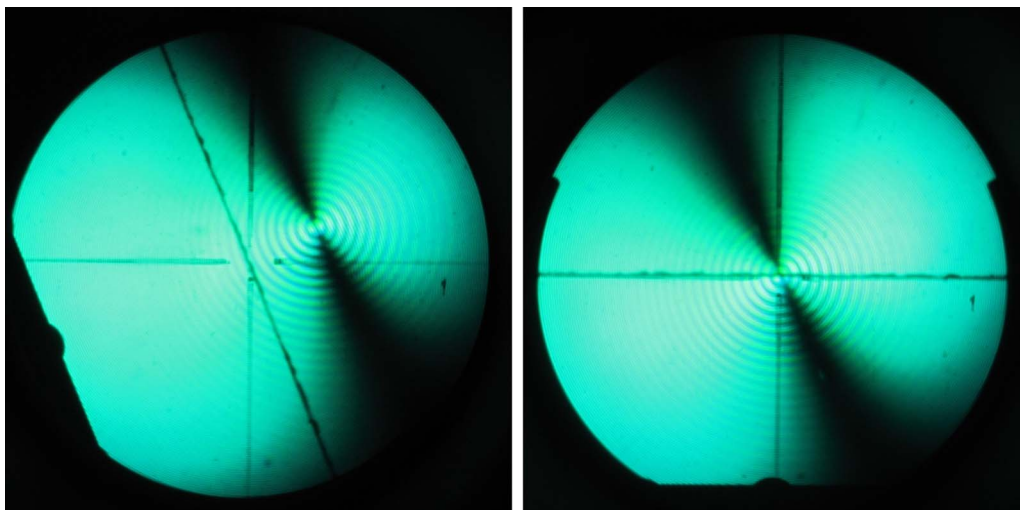


FIG. 2. Melatope images produced on the instrument. Topaz in immersion, misaligned left, aligned right.

FIG. 3. The biaxial orientation device.

components are listed and the details of the calculations for determining the orientation of the crystal sample are shown.

## A. Optical components of the biaxial orientation instrument

Fig. 3 is an image of the instrument and Fig. 4 illustrates schematically the optical components of the biaxial orientation device. The light source is an Edmund Optics MI-150 tungsten lamp fiber optic illuminator. The light output of the illuminator is delivered to the instrument using an Edmund Optics 39-366 0.125 in. aperture fiber optic, 48 in. in length. The output of the fiber passes through a Thorlabs FW1A filter wheel that contains one each of Edmund Optics 46-156, 46-158, and 46-160 transmission filters that are centered on wavelengths 450, 550, and 650 nm, respectively. The filters are useful for increasing the isogyre image contrast in colored samples. The output face of the fiber optic is collimated by the first Fresnel lens, an Edmund Optic 43-025 lens with a focal length of 2 in.. The Edmund Optics 45-668 polarizer film is placed after the first Fresnel lens to polarize the light. The polarized light now enters the second Fresnel lens, another Edmond Optics 43-025, where the convergent polarized rays are now formed. The convergent polarized rays enter a glass immersion cell such as the Hellma 704.002 40 mm cell. The immersion cell is filled with index matching liquid chosen to be appropriate for the crystal sample and may include,

for example, methylene iodide, benzyl benzoate, or benzaldehyde, with refractive indices of 1.741, 1.568, and 1.544, respectively. The Hellma immersion cell is mounted on a Melles Griot 04MFS001 rack and pinion stage to allow the cell to be easily raised or lowered. The biaxial sample is bonded to a small diameter dop and inserted into the spindle of the instrument. The spindle allows for manual rotation of the sample and has gradations in degrees, with fiducial marks every 2°. The axis of the spindle is mounted in a yoke that allows for the elevation angle of the sample to be manually adjusted. The elevation scale range is 110° with fiducials every 2°, allowing elevation adjustment of the sample from −40° to +70° away from perpendicular alignment relative to the instrument base.

While the sample is rotated and tilted in the index matching liquid, the polarized light passing through the sample is collected by the last Fresnel lens, another Edmund Optics 43-025, located approximately a focal length away from the sample. The last Fresnel lens has two black lines inscribed on its surface to form the alignment crosshair viewed by the user. The mechanical center of the Fresnel lens is easily found using a conventional microscope to aid in creating the crosshair lines on the surface of the Fresnel lens. The transfer dop is held by a fabricated aluminum vee-block attached to a Thorlabs KB1×1 magnetic kinematic base. This allows for the removal and installation of the transfer dop with micrometer level precision. The magnetic kinematic base is positioned using Siskiyou Design Z-X 1620 precision crossed roller bearing stages mounted with fabricated aluminum components. The Z-X 1620 stage assembly is permitted to slide toward or away from the sample while attached to a Deltron BSGS16W-1-180 stainless steel recirculating ball slide with 180 mm of travel. A pinhole lens of diameter 0.75 mm is positioned on the optical axis of the instrument and used for viewing the isogyre figure. The mount of the pinhole lens and the mount of the fiber optic serve as the reference bores for performing the optical alignment of the instrument. When all optical components are removed, the permanently mounted Thorlabs S2011 635 nm laser is adjusted using a pinhole centered in the bore of the fiber optic mount, and the bore of the pinhole lens, until the laser is centered on both locations. This forms the reference beam for aligning the optical components of the instrument. The immersion cell is placed on the rack and pinion stage and the stage mounts are adjusted to retroreflect the laser from the immersion cell faces back to the laser, and thereby establish the walls of the immersion to be perpendicular to the laser. A special dop tool is prepared with a polished, reflective end that has been carefully polished to have the reflective surface perpendicular to the axis of the dop. The dop tool is inserted into the instrument using the kinematic magnetic base and vee-block. The alignment of the dop tool axis can be adjusted using the fabricated aluminum mounts supporting the magnetic base such that the laser is reflected from the dop tool. The mounts are adjusted so that the laser is retroreflected from the dop tool back to the laser thereby establishing the dop mechanical axis to be parallel to the instrument optical axis. The last Fresnel lens, Fresnel lens 3, and analyzer polarizer are mounted together on a removable mount to allow them to be removed during the attachment of the transfer dop to the sample. The mount of Fresnel lens 3
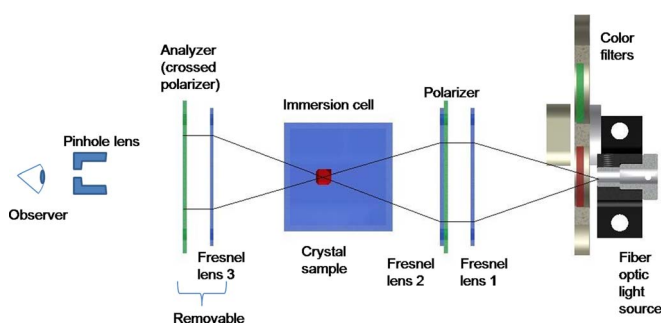


FIG. 4. Optical components of the biaxial orientation device.

is adjusted to center the transmitted laser beam on the pinhole target mounted in the pinhole lens bore, thereby aligning Fresnel lens 3. Next, Fresnel lenses 1 and 2 are installed. They are adjusted iteratively until the laser output is centered on the pinhole target mounted in the pinhole lens bore. At this point, the instrument lenses are aligned and the laser is switched off. The fiber optic is inserted into the instrument and the illuminator is powered on and the 550 nm filter on the filter wheel is selected. A PTFE (polytetrafluoroethylene) rod machined with a flat parallel to the axis of the rod is inserted into the spindle. The PTFE flat is positioned to be perpendicular to the instrument beam and the filled immersion cell is raised to bring the PTFE rod into the solution. While viewing the flat on the PTFE rod, the mount containing Fresnel lenses 1 and 2 slides on a rail that has been machined to be parallel to the optical axis of the instrument, until the smallest focused spot on the PTFE rod surface is observed. The PTFE rod is removed, and the instrument is now aligned and ready for use.

## B. Use of the biaxial orientation instrument

A biaxial crystal sample of interest is first attached to a temporary dop with a small contact area, simply to hold the sample during alignment. All the dops have a flat machined into the non-sample end of the dop such that the flat is parallel to the mechanical axis of the dop while the depth of the flat is controlled precisely. The dop holding the sample, Fig. 5, is inserted into the instrument spindle and a thumbscrew in the spindle is gently tightened against the flat on the dop which aligns the dop flat to 0° on the spindle, as a result of the thumbscrew threads having been machined for that purpose. The immersion cell containing the appropriate index matching liquid is raised with the rack and pinion stage to immerse the sample. The fiber optic illuminator is set to an appropriate illumination level and the user peers through the pinhole lens. While looking through the pinhole lens, the user rotates the spindle and changes the axis of the spindle in elevation until the pattern of a melatope is found. The spindle angle and elevation angle are recorded when the melatope is centered on the crosshair. The user again rotates and tilts the spindle until the second melatope is found. The spindle rotation and elevation angles are again recorded. The spindle rotation and elevation angles are entered into the Javascript

Biscectrix Calculator. The Bisectrix Calculator immediately displays the rotation angle of the spindle and the elevation angle of the spindle such that the acute bisectrix or the obtuse bisectrix will be centered on the optical axis of the instrument. The Bisectrix Calculator displays an indicator if the solution found is the bisectrix or obtuse solution, and shows a graphical representation of the melatopes and optical plane location. The mounting surface for the kinematic magnetic dop support is supported on the rotation axis of a Thorlabs RSP2C rotation stage. The rotation stage has a 2 in. diameter glass window installed that is bisected by a diamond scribe line. The window inside the rotation stage is aligned so that the diamond scribe line is at 0° on the rotation stage in a previous operation. The diamond scribe line on the window is parallel to the mounting surface the magnetic kinematic support. The Bisectrix Calculator produces the angle that the rotation stage is then adjusted to such that the diamond scribe line on the window is now parallel to the optical plane of the biaxial crystal. Fresnel lens 3 and the analyzer polarizer are removed. The immersion cell is lowered and protected with a cover glass. A small, light-weight vessel is placed on top of the immersion cell to collect waste solvent that is dripped carefully on the sample to clean the immersion liquid from the sample surface. Photo-initiated adhesive is applied to the end of a transfer dop and the dop is placed in the vee-block and attached to the magnetic kinematic base. The Siskiyou Z-X 1620 stages are moved forward on the Deltron slide until the transfer dop is within a few millimeters of the sample, Fig. 6.

The axes of the Z-X 1620 are adjusted to center the transfer dop on the sample. The Siskiyou Z-X 1620 stages are then moved forward on the Deltron slide until the transfer dop slightly contacts the sample. A 405 nm laser pen pointer is then used to activate the photo-initiated adhesive and attach the transfer dop to the sample. After sufficient curing of the adhesive, the thumbscrews securing both dops can be loosened and both dops can be removed from the instrument. The dop initially held in the spindle is no longer needed, and can be removed by heating or by holding the sample firmly and breaking the bond of the initial dop with finger pressure.

At this point, the sample has the acute bisectrix or obtuse bisectrix aligned with the mechanical axis of the dop, and the optical plane is parallel to the flat machined in the opposite end of the dop. The user must now determine which directions
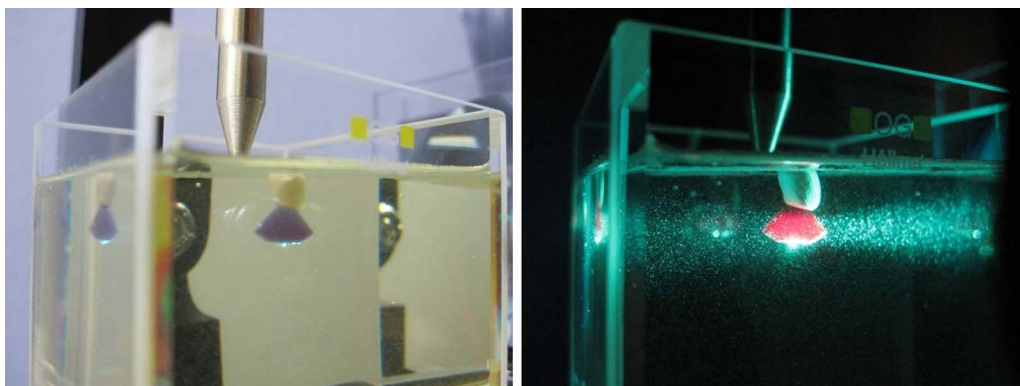


FIG. 5. Sample mounted on dop, left, in immersion. Illumination of sample in immersion, right.
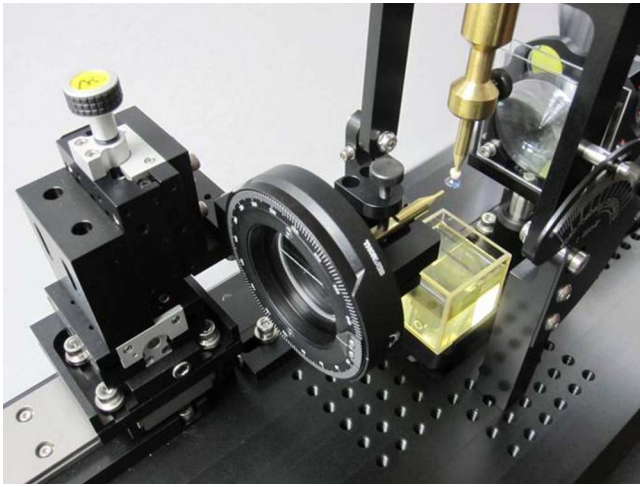
FIG. 6.  Immersion cell lowered, Fresnel lens 3 and analyzer polarizer removed, transfer dop in bonding position.

through the biaxial crystal will be measured, as the optical faces must be formed in the sample.

Fig. 7 shows a dop transfer block that is used to preserve the orientation of the optical plane. The transfer block has been machined with spring loaded fingers that when in contact with the flat portion of the dop, maintains parallelism or perpendicularity of the optical plane.

The first face of the sample is ground and polished and the dop is place into the dop transfer block to attach a new dop for forming the final face using photo-initiated adhesive. The dop used to generate the first face is removed with heat to release the adhesive.

By use of the transfer block and keeping track of the optical plane location, or acute bisectrix and obtuse bisectrix, it is possible to create optical faces accurately for the measurement of the optical properties of the biaxial crystal in the $\alpha$, $\gamma$, and $\beta$ directions. In addition, in the case of a polarized



FIG. 7.  Transfer block device used for transferring the sample to grind and polish any necessary face, while preserving the optical plane and bisectrix locations.

light spectrophotometer, it is also possible to know the E-field angle relative to the optical axes of the sample if the sample mounting hardware of the spectrophotometer is aligned for such measurements. The instrument, of course, is equally suitable for orienting uniaxial crystals.

## C. Calculation of the optical plane angle and bisectrix location

*Yaw* is the spindle rotation angle of the sample and *pitch* is the spindle axis elevation angle. Given the yaw and pitch of melatope one, $y_1$ and $p_1$, and the yaw and pitch of melatope two, $y_2$ and $p_2$, we can determine the bisectrix yaw and pitch, optical plane angle with respect to the instrument scales, and angle between the melatopes (2V).

Let

$$b_{yaw}, \quad b_p, \quad \alpha, \quad m_\alpha$$

represent the normalized bisectrix yaw angle, normalized bisectrix pitch angle, normalized optical plane angle, and angle between the melatopes, respectively. These angles are normalized with respect to the instrument scales.

Using standard vector notation we denote $\vec{v}$ as a vector, and $\hat{v}$ as a unit vector in the direction of $\vec{v}$. Similarly, $\vec{v_x}$, $\vec{v_y}$, and $\vec{v_z}$, are x, y, and z components of $\vec{v}$, while $\hat{i}$, $\hat{j}$, and $\hat{k}$ are the unit vectors of the x, y, and z axes. The relationship between spherical and Cartesian coordinates is

$$S(\theta, \varphi) = \hat{i} \sin\theta \cos\varphi + \hat{j} \sin\theta \sin\varphi + \hat{k} \cos\theta, \qquad (1)$$

and the angle between two vectors is

$$A(\vec{v_1}, \vec{v_2}) = \cos^{-1}(\vec{v_1} \cdot \vec{v_2}). \qquad (2)$$

For any given value of x and y, define *arctan2* as

$$arctan2(y, x) = \begin{cases} \tan^{-1}\left(\dfrac{y}{x}\right) & x > 0 \\[2mm] \tan^{-1}\left(\dfrac{y}{x}\right) + \pi & y \geq 0, \quad x < 0 \\[2mm] \tan^{-1}\left(\dfrac{y}{x}\right) - \pi & y < 0, \quad x < 0 \\[2mm] +\dfrac{\pi}{2} & y > 0, \quad x = 0 \\[2mm] -\dfrac{\pi}{2} & y < 0, \quad x = 0 \\[2mm] undefined & y = 0, \quad x = 0. \end{cases}$$

We define the vectors for melatope one and melatope two as

$$\vec{m_1} = S(p_1, y_1), \qquad (3)$$

$$\vec{m_2} = S(p_2, y_2). \qquad (4)$$

By definition, the melatopes lie in the optical plane, and the optical plane normal is

$$\vec{n}' = \vec{m_1} \times \vec{m_2}, \qquad (5)$$

and the bisectrix is

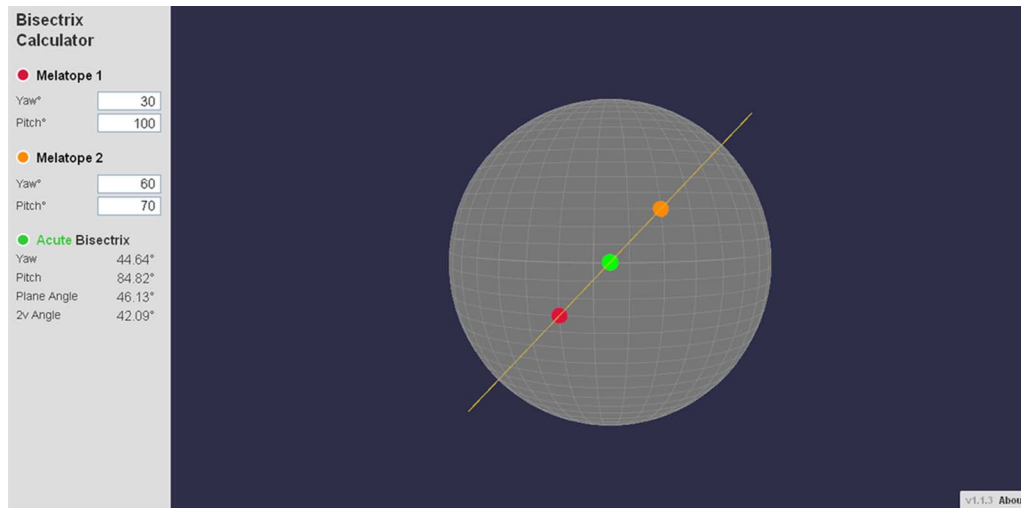$$\vec{b} = \frac{\vec{m_1} + \vec{m_2}}{2}, \qquad (6)$$

FIG. 8.   Screen display of the Javascript GUI of the biaxial orientation device.

though we wish the optical plane normal to be pointed in the $\hat{k}$ direction:

$$\vec{n} = \begin{cases} -\vec{n}', & A(\vec{n}', \hat{k}) > \frac{\pi}{2} \\ \vec{n}', & A(\vec{n}', \hat{k}) \le \frac{\pi}{2} \end{cases}. \qquad (7)$$

To position the sample so that it is centered on the bisectrix, the settings for the spindle yaw (rotation) and pitch (elevation) scale values are determined as

$$b_{yaw}{}' = arctan2(\widehat{b}_y, \widehat{b}_x), \qquad (8)$$

$$b_p = \cos^{-1}(\widehat{b}_z). \qquad (9)$$

The value of $b_{yaw}$, should not be negative, however, so

$$b_{yaw} = \begin{cases} b_{yaw}{}' + 2\pi, & b_{yaw}{}' < 0 \\ b_{yaw}{}', & otherwise \end{cases}. \qquad (10)$$

The optical plane angle is determined as follows.

A vector, $\widehat{b}_o$, is constructed that is orthogonal to the bisectrix:

$$\widehat{b}_o = S\left(\frac{\pi}{2}, b_{yaw}{}' + \frac{\pi}{2}\right). \qquad (11)$$

The "bisectrix normal" is defined as a vector that is orthogonal to the bisectrix and intersects $\hat{k}$. The following is a bisectrix normal:

$$\vec{b_n} = \vec{b} \times \widehat{b}_o. \qquad (12)$$

The optical plane angle is then determined as

$$\alpha' = \cos^{-1}(\vec{n} \cdot \widehat{b}_n), \qquad (13)$$

which is corrected for the rotation stage scale orientation relative to the instrument base by

$$\alpha = \begin{cases} 2\pi - \alpha', & A(\widehat{b}_o, \hat{n}) < \frac{\pi}{2} \\ \alpha', & otherwise \end{cases}. \qquad (14)$$

Finally, the angle between the melatopes (2 V) is simply

$$m_\alpha = A(\vec{m_1}, \vec{m_2}). \qquad (15)$$

With the optical plane angle determined by Eq. (13), the rotation stage on the instrument is set to that angle to establish the plane on the transfer dop to be parallel to the optical plane in the sample. To set the sample such that the bisectrix is on center with the transfer dop axis, the spindle is rotated to the angle determined by Eq. (10), and the tilt of the spindle axis is adjusted to the angle determined by Eq. (9).

A JavaScript-based application was created to make use of the instrument convenient, and to produce a graphical representation of the melatope locations, approximate 2V angle, optical plane angle, as well as displaying the values required for adjusting the instrument spindle so that either the sample acute bisectrix or obtuse bisectrix are centered on the transfer dop axis, Figure 8.

## III. PERFORMANCE

Biaxial crystals with a range of refractive indices and with a range of 2 V angles have been successfully oriented with the instrument. Tests have been conducted on solids that span refractive indices from 1.56 to 1.75, and with 2 V angles from 55º to 80º. Examples include minerals such as olivine, chrysoberyl, amblygonite, feldspars, cordierite, sapphirine, and topaz.

Sapphirine, $(Mg,Fe)_4Al_4(Al_4,Si_2)O_{20}$, with an index of refraction of about 1.71 and a 2V of about 78º serves as an example. When oriented in methylene iodide (refractive index $\sim$1.74) all four melatopes of the test sample were measured. The Bxa angles measured in the opposite hemispheres were 77.7º and 77.1º and measured Bxo angles in the two hemispheres were 103.1º and 104.5º. The close agreement of each pair of values, particularly the Bxa angles, is a measure of the precision of the orientation instrument. After alignment, the crystal was fabricated as a slab in the Bxa orientation with polished windows on the two opposite sides. When the Bxa interference figure of the polished slab was examined in a petrographic microscope, no deviation from a perfectly centered Bxa figure was observed.

Also, as a test, the same sapphirine crystal was also oriented with a liquid of lower index of refraction (benzalde-

hyde, refractive index ∼1.54). In this case, the two measured Bxa angles were 77.0º and 80.8º, and the Bxo angles were 98.9º and 103.2º. The calculated setting angles on the instrument to bring the crystal into centered Bxa orientation, in this case, differed from the settings calculated using methylene iodide by 3º in yaw and 1º in pitch.

As noted above, the accuracy of the alignment depends, in part, on the match of the index of refraction liquid to the crystal. If the crystal has a large difference between the minimum and maximum refractive indices, it will be impossible to obtain an index match in all orientations of the crystal. The accuracy also depends of the size of the 2 V angle (the angle between the two melatopes). On occasion, misorientation may occur from total internal reflections within the crystal if the surrounding liquid has a lower index of refraction or from a lens effect from the crystal due to index mismatch.

Even when such problems occur, we generally find that after cutting and polishing, the crystal is within a few degrees of providing a centered Bxa or Bxo optic figure as judged by viewing the crystal in conoscopic light in a petrographic microscope. In such cases, if higher accuracy is needed, a second round of orientation of the polished crystal in the biaxial orientation device followed by an additional grinding and pol-

ishing step will bring the crystal in alignment so good that a deviation from a centered interference figure is usually not seen in the petrographic microscope.

Overall, the orientation instrument has produced crystals that have proven excellent for optical and infrared spectroscopic measurements. It has also noticeably decreased the time required to orient biaxial crystals for UV-Vis-NIR-IR studies.

## ACKNOWLEDGMENTS

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