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Optical Crystallography of Silver Sulfadiazine

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KEYWORDS

Silver sulfadiazine, spindle stage, optical crystallography, pinacoid, Excalibr, polarized light microscopy (PLM), Raman microspectroscopy, energy dispersive spectroscopy

ABSTRACT

Silver sulfadiazine was encountered during casework, but its optical properties could not be found in a literature search. A standard was obtained and its optical properties determined by the spindle stage method. The standard crystals were determined to be monoclinic prisms with strong inclined dispersion, and the following optical properties at several wavelengths. For λ=485 nm: (+)2V=59.1°, n_x=1.762, n_y=1.766, n_z=1.777, n_z-n_x=0.015; for λ=589 nm: (+)2V=76.6°, n_x=1.736, n_y=1.739, n_z=1.751, n_z-n_x=0.015; for λ=650 nm: (+)2V=77.2°, n_x=1.726; n_y=1.731; n_z=1.738; n_z-n_x=0.012.

INTRODUCTION

An item of clothing submitted for analysis was observed to have a white stain present. Examination of the stain material by polarized light microscopy revealed the presence of small, high index crystals that were seen to exhibit dramatic dispersed extinction (Figure 1). These crystals were characterized optically using the polarized light microscope, but additional analyses by Raman microspectroscopy and energy dispersive spectroscopy were required in order to identify the material as silver sulfadiazine. Silver sulfadiazine is a compound with antimicrobial properties, and is commonly administered as a topical cream to prevent skin infections, especially for burn victims. Its common use and its presence on skin make it likely to be encountered during routine casework as stains on clothing.

After the material was identified, literature searches for its optical properties were conducted. No published reports of its optical properties were encountered, making it impossible to use the optical properties measured using the polarized light microscope as identifying features. In order to make the optical properties of silver sulfadiazine available to the scientific community, and to confirm that the optical properties of the crystals on the submitted evidence are consistent with silver sulfadiazine, a standard sample was obtained and its optical properties determined using the spindle stage method.

METHODS

A single crystal of silver sulfadiazine was isolated and mounted on the tip of a tungsten needle using red nail polish (Figure 2). The needle with the crystal was then inserted into a Bloss detent spindle stage for optical characterization (Figure 3). Extinction data were obtained on the crystal using a Zeiss AxioImager. A1m polarized light microscope, and photomicrographs were taken using a Zeiss AxioCam MRc5 camera. Chroma Technology Corporation monochromatic filters were used with wavelengths of 485 nm, 589 nm and 650 nm while obtaining extinction data and mea-

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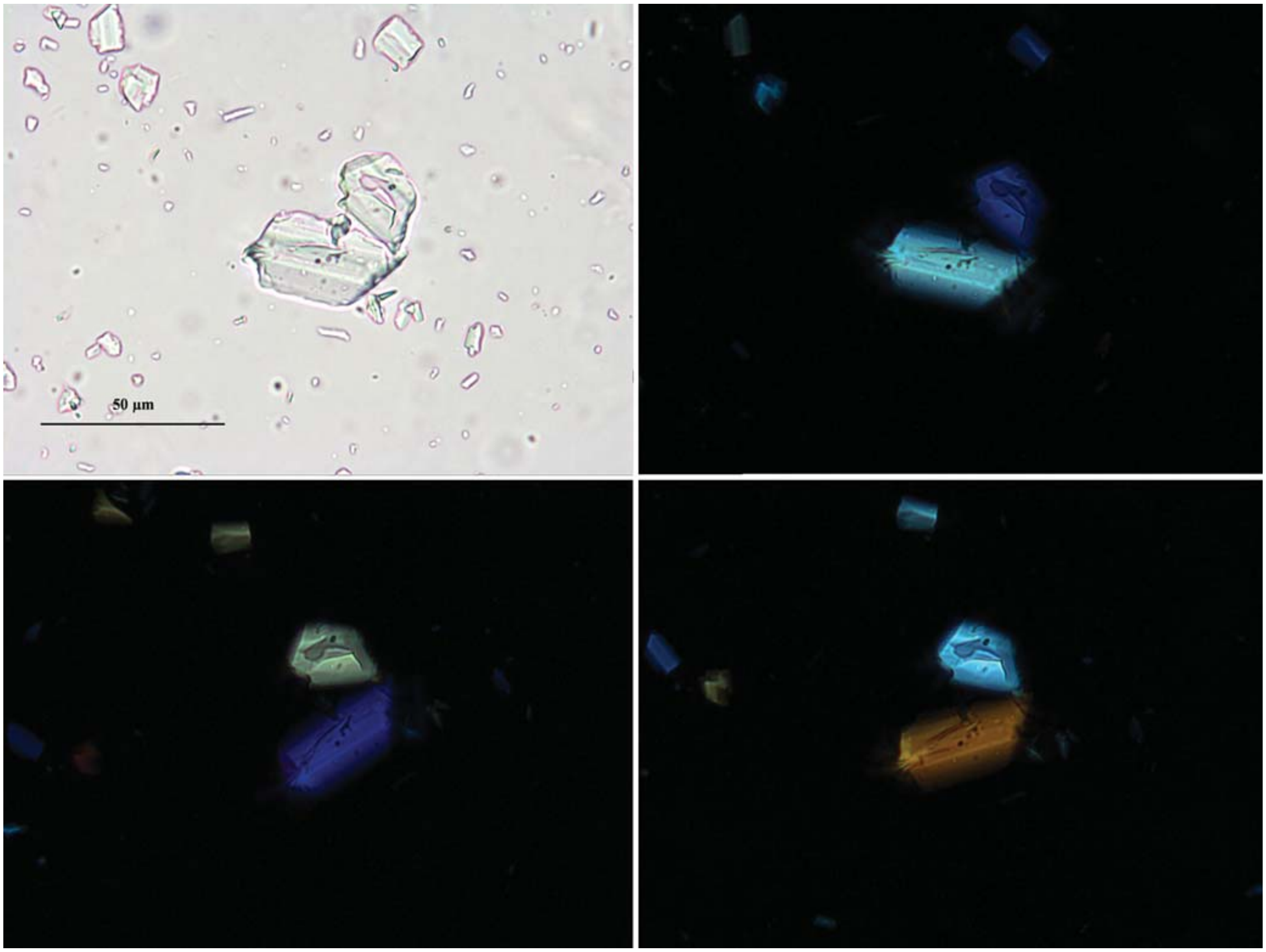

Figure 1. Silver sulfadiazine crystals shown in plane polarized light (top left) and again in crossed polars in three orientations (top right and bottom) illustrating its dramatic dispersed extinction. The mounting medium is 1.660.

suring refractive indices (Figure 4). Extinction data were obtained for the same crystal using all three wavelengths and the extinction data were entered into the Excalibr software program, which may be downloaded for free online (1). The output from Excalibr was used to locate the acute bisectrix, the obtuse bisectrix and the optic normal for refractive index measurement of the mounted crystal.

For information regarding the theory and use of the spindle stage, the reader is referred to the references (2, 3). The refractive indices were measured using the Becke line immersion method and Cargille refractive index liquids. All three refractive indices were measured in 589 nm light. For 485 nm and 650 nm light, two refractive indices were measured and the third was calculated using Equation 1 for a positive optic sign crystal and the 2V value provided by Excalibr for that wavelength.

Equation 1:

$$\cos^2 V\gamma = \frac{\alpha^2 (\gamma^2 - \beta^2)}{\beta^2 (\gamma^2 - \alpha^2)}$$

Once extinction data were obtained for all three wavelengths, the dispersion of the indicatrix was determined by Excalibr. Interfacial angles and extinction angles were measured using a graduated rotating stage on a polarized light microscope. For additional information on the measurement of refractive indices, in-

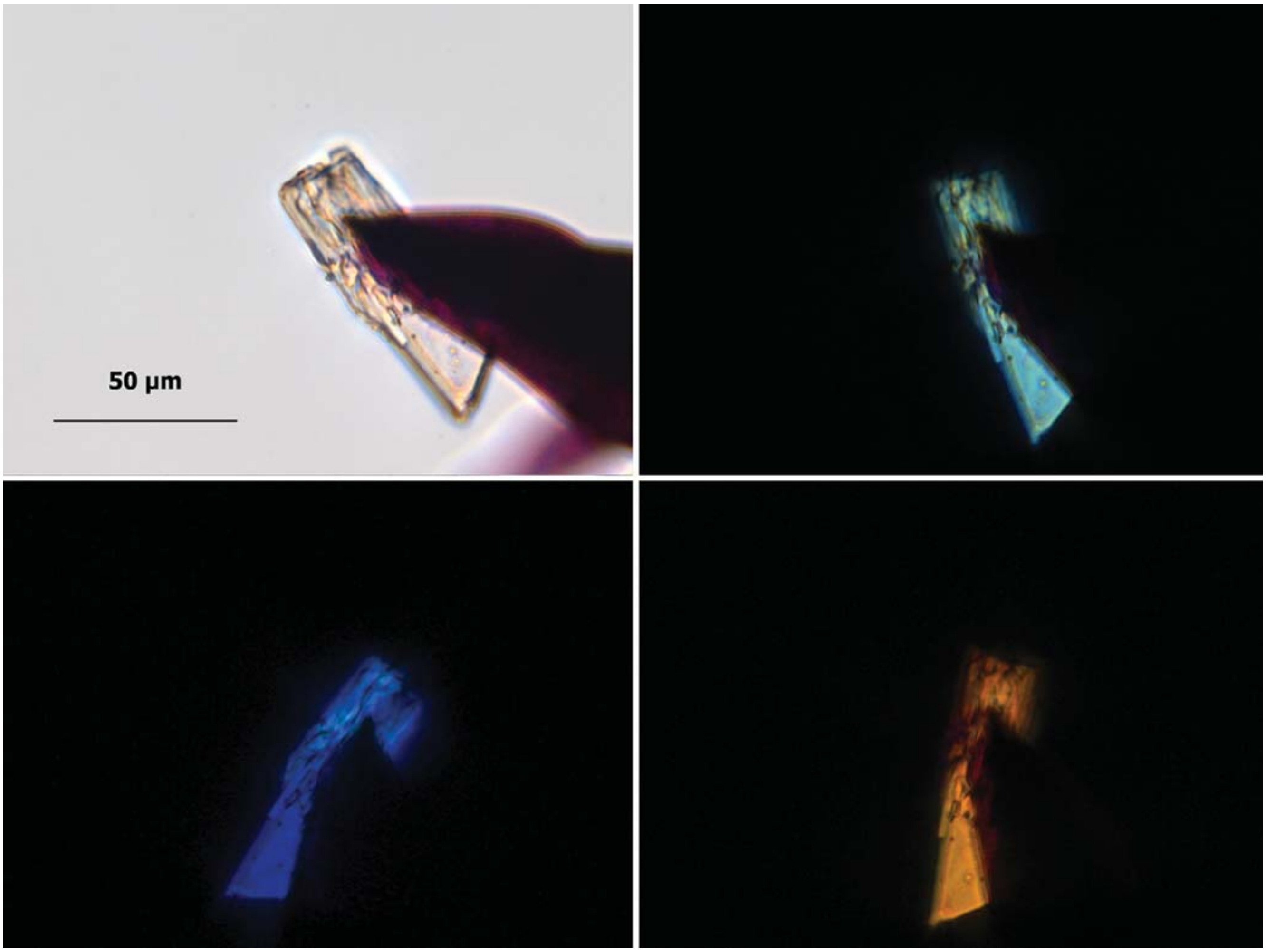

Figure 2. A silver sulfadiazine crystal mounted on the tip of a tungsten needle with red nail polish in plane polarized light (top left) and again in crossed polars in three orientations (top right and bottom).

terfacial angles and extinction angles in crystals, the reader is referred to the references (4, 5).

RESULTS

The extinction data obtained using the spindle stage for the three wavelengths are shown in Table 1*. The optic axial angles were calculated to be 59.1 (± 3.0°) for 485 nm light, 76.6 (± 3.3°) for 589 nm light and 77.2 (± 2.2°) for 650 nm light. Locations of optical directions in the crystal as indicated by Excalibr are shown for all three wavelengths in Tables 2-4.

The dispersion data output by Excalibr are shown in Table 5, and stereographic projections of the optical indicatrix for all three wavelengths are shown in Figure 5. The spindle stage data indicate that there is strong dispersion of the two optic axes, the acute bisectrix and obtuse bisectrix, but no dispersion of the optic normal. This type of dispersion is termed "inclined" dispersion, and is typically seen in the monoclinic crystal system (although it is also possible in triclinic crystals).

The Excalibr data in Tables 2-4 were used to locate and measure the refractive indices for each wavelength. The measured and calculated refractive indices are shown in Table 6 along with optic axial angles and calculated birefringence values. In order to determine the optic orientation of the crystal, the Excalibr data were used to locate the axes of the indicatrix and observe the crystal symmetry in relation to these directions. The crystal appears to contain only one plane of symmetry perpendicular to its optic normal (Figure 6).

* See Tables 1-6 on pp 17-18.

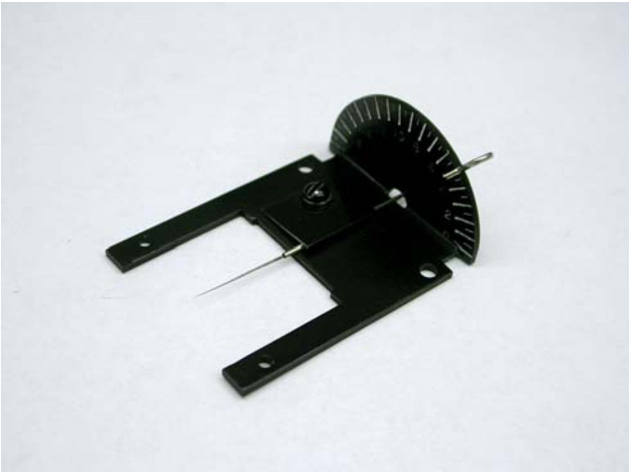

Figure 3. A Bloss detent spindle stage with an inserted tungsten needle.

Extinction angles were measured relative to the remaining crystal faces using 589 nm light. The optic orientation is illustrated in Figure 7, with the 100, 010 and 001 pinacoids as the forms generally developed. Occasionally the 110 pinacoid forms as well. The dispersion data suggest that the crystal is monoclinic, as does the symmetry observed in the external crystal form. This corresponds well with published X-ray diffraction data, which indicates that silver sulfadiazine crystallizes in the monoclinic space group $P2_1/c$ (6).

DISCUSSION AND CONCLUSIONS

Silver sulfadiazine was encountered on an item of evidence and required identification. No published optical properties could be located to assist in its identification. This compound is a common topical antimicrobial agent, and is likely to be encountered during casework by other investigators as stains on clothing and other items that come in frequent contact with the skin.

The optical properties of the crystals are very distinctive, the most characteristic feature being strong inclined dispersion resulting in dramatic dispersed extinction on the 010 face of the crystal. The optical properties were determined on a crystal of standard silver sulfadiazine using the spindle stage method and are reported above as a reference for future investigators.

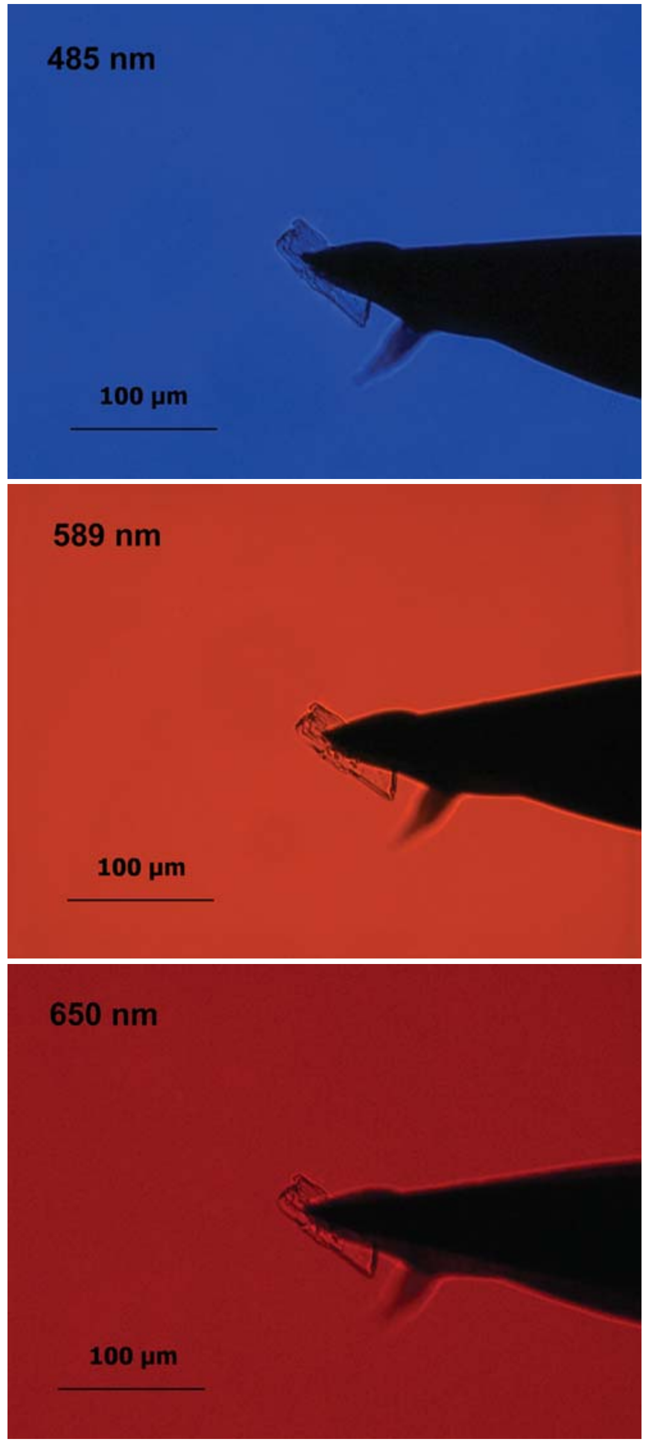

Figure 4. A silver sulfadiazine crystal mounted on the tip of a tungsten needle shown with a 485 nm filter (top), a 589 nm filter (middle), and a 650 nm filter (bottom).

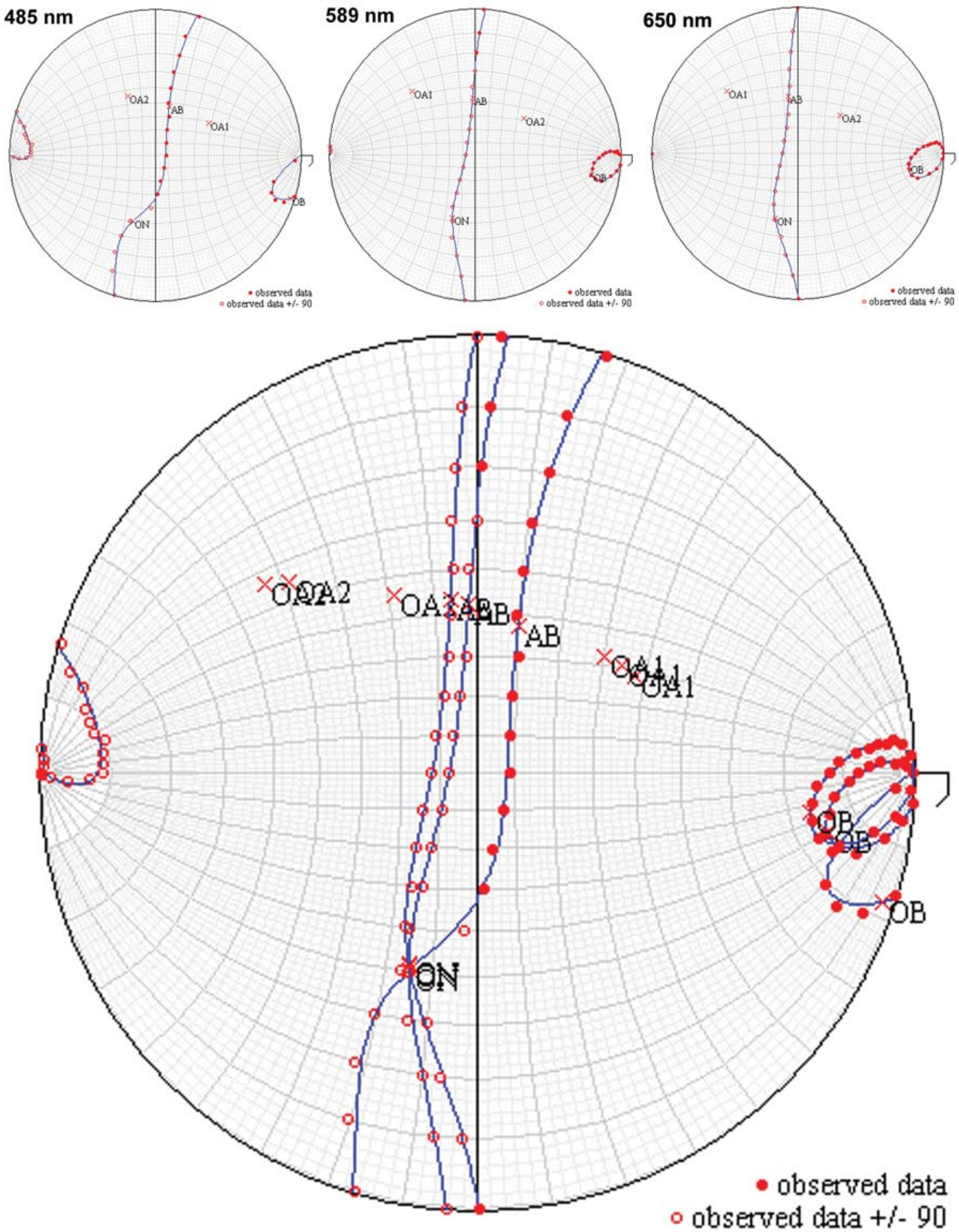

Figure 5. Dispersion data for silver sulfadiazine plotted on stereonets. The top three plots represent the individual wavelengths — 485 nm (left), 589 nm (middle), and 650nm (right)— and the bottom plot contains data from all three wavelengths plotted together.

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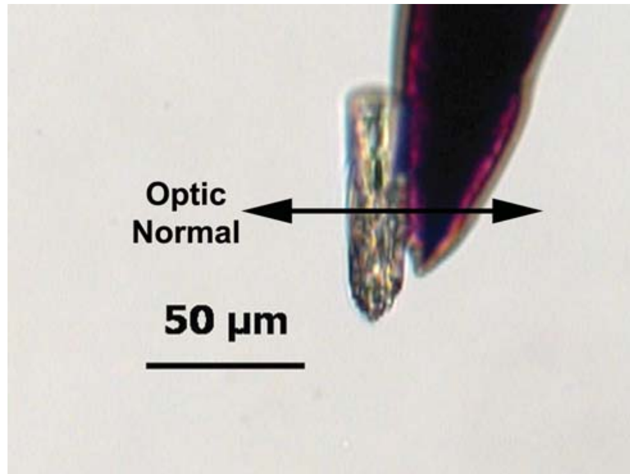

Figure 6. A silver sulfadiazine crystal mounted on the tip of a tungsten needle is shown with its optic normal oriented east-west, as indicated by the arrows. A plane of symmetry is located perpendicular to the optic normal.

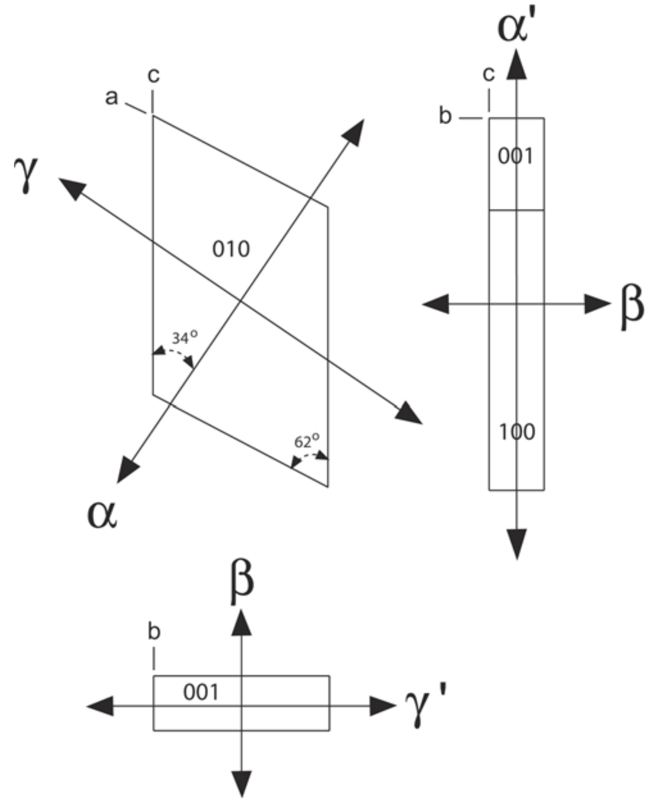

Figure 7. Orthographic projection of silver sulfadiazine based on the extinction angles and interfacial angles measured with the polarized light microscope.

Table 1. Extinction Data Measured for a Standard Silver Sulfadiazine Crystal Using the Spindle Stage

Spindle Position	485 nm	589 nm	650 nm
0	17.0	3.0	89.7
10	13.7	1.6	87.2
20	12.6	0.5	85.5
30	10.4	89.3	84.3
40	9.6	87.4	84.1
50	9.1	88.2	83.2
60	10.0	86.7	82.4
70	8.8	84.8	80.9
80	8.5	83.2	78.5
90	8.2	81.9	77.5
100	6.6	80.3	75.2
110	3.6	77.5	73.8
120	1.2	75.9	73.1
130	86.2	73.8	72.7
140	73.1	74.6	74.4
150	69.2	75.7	79.5
160	67.8	79.8	83.0
170	69.2	82.8	87.2
180	73.1	85.3	0.1

Table 2. Excalibr Data Output for 485 nm

Optic Directions	S (ese)	Es (ese)	Ms (e-w polar)
OA1	61.46 (1.87)	51.77 (1.60)	N/A
OA2	44.48 (1.55)	108.68 (1.43)	N/A
AB	52.17 (1.65)	80.12 (0.26)	99.42
OB	175.00 (2.15)	17.82 (0.74)	161.72
ON	139.56 (1.66)	104.67 (0.94)	74.87

Table 3. Excalibr Data Output for 589 nm

Optic Directions	S (ese)	Es (ese)	Ms (e-w polar)
OA1	35.19 (2.25)	128.71 (1.74)	N/A
OA2	58.37 (2.09)	55.17 (1.66)	N/A
AB	47.08 (2.17)	91.98 (0.35)	87.49
OB	129.74 (1.12)	15.16 (0.56)	164.32
ON	137.61 (2.25)	105.02 (0.57)	74.46

Table 4. Excalibr Data Output for 650 nm

Optic Directions	S (ese)	Es (ese)	Ms (e-w polar)
OA1	34.17 (1.63)	133.42 (1.19)	N/A
OA2	58.09 (1.29)	59.41 (1.08)	N/A
AB	47.16 (1.44)	96.56 (0.25)	83.15
OB	114.32 (0.91)	16.50 (0.38)	163.21
ON	138.94 (1.47)	105.07 (0.46)	74.64

Table 5. Excalibr Dispersion Data Output

	OA1	OA2	AB	OB	ON
485-589 nm	4.036 (2.340)	*****(2.211)	*****(0.692)	*****(0.441)	1.469 (2.653)
p-value	0.093	0	0	0	0.583
485-650 nm	8.199 (1.965)	*****(1.827)	*****(0.506)	*****(0.388)	1.185 (2.113)
p-value	0	0	0	0	0.578
589-650 nm	4.238 (1.987)	4.398 (2.097)	4.321 (0.227)	4.312 (0.354)	0.284 (2.606)
p-value	0.04	0.043	0	0	0.914

Table 6. Summary of Measured and Calculated Optical Properties

Wavelength	485 nm	589 nm	650 nm
Gamma (±0.002)	1.777 (calc.)	1.751	1.738
Beta (±0.002)	1.766	1.739	1.731 (calc.)
Alpha (±0.002)	1.762	1.736	1.726
2V (+)	59.1 (±3.0°)	76.6 (±3.3°)	77.2 (±2.2°)
Birefringence	0.015	0.015	0.012