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Optical Characterization of Sodium Lauryl Sulfate[1]

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KEYWORDS

Becke line, conoscopy, optical crystallography, polarized light microscopy, refractive index, sodium lauryl sulfate, surfactant

ABSTRACT

Sodium lauryl sulfate (SLS) is a common anionic surfactant found in household cleaning and hygiene products. Its optical properties are not available in the standard optical crystallography literature. Because SLS is widely used, it would be beneficial to have its optical properties published and accessible to microscopists. A standard of SLS was obtained, and its optical crystallographic properties were determined using polarized light microscopy (PLM). Sodium lauryl sulfate is biaxial positive (+) with 2V = 15°. The refractive indices measured (in sodium D light) are $\alpha = 1.463$, $\beta = 1.464$, $\gamma = 1.525$ (calculated), B = 0.062. Crystals commonly occur as thin plates with a negative sign of elongation.

INTRODUCTION

Some well-formed crystals of SLS were recently encountered in the casework of a colleague and identified using instrumental analysis. A secondary confirmation using PLM was not possible due to the fact that the optical properties of SLS could not be found in the optical crystallography literature. A standard of SLS was obtained from the Aldrich Chemical Company, Inc., (catalog number 86,201-0; lot number 12522JG) and characterized with PLM.

Sodium lauryl sulfate — also known as sodium dodecyl sulfate, lauryl sulfate sodium salt, dodecyl sodium sulfate, dodecyl sulfate sodium salt ($CH_3(CH_2)_{11}OSO_3Na$) and CAS registry 151-21-3 — is prepared by the sulfonation of lauryl alcohol followed by neutralization with sodium carbonate (1). Crystals of anhydrous SLS are monoclinic, in the $P2_1/c$ space group, with a = 38.915Å, b = 4.709Å, c = 8.198Å, and $\beta = 93.29°$ (2). Crystals are typically colorless with platy habit, dominated by the {100} form (2). Sodium lauryl sulfate is water soluble (1g/10mL H_2O) (1) and has a melting point between 204 °C and 207 °C (3). It is commonly used in household cleaning and hygiene products, and is also used in the textile industry as a wetting agent and detergent (1). Several hydrates of SLS have been described (2), so care should be taken when identifying crystals precipitated from aqueous solutions.

MATERIALS AND METHODS

The SLS crystals analyzed in this study occur as very thin plates dominated by the {100} form (Figure 1). The thin, platy SLS crystals tend to stack in multiple layers, making the determination of precise extinction positions challenging and rendering the crystals unsuitable for spindle stage work (Figure 2). Due to the difficulty in determining the optical properties of SLS using a spindle stage, the refractive indices were measured on crystals in grain mounts using the Becke

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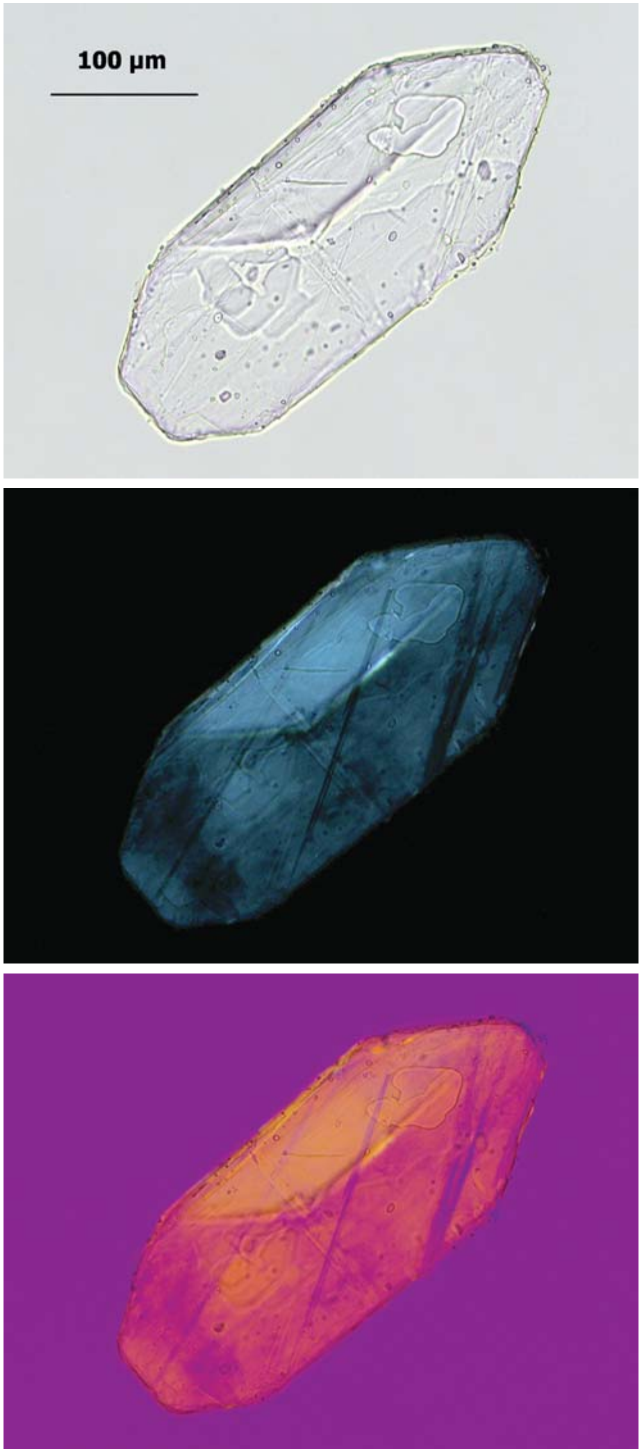

Figure 1. A typical thin, platy crystal of sodium lauryl sulfate is shown in plane polarized light (top), between crossed polars (middle) and in crossed polars with a 530 nm compensator (bottom). The mounting medium is 1.540 liquid.

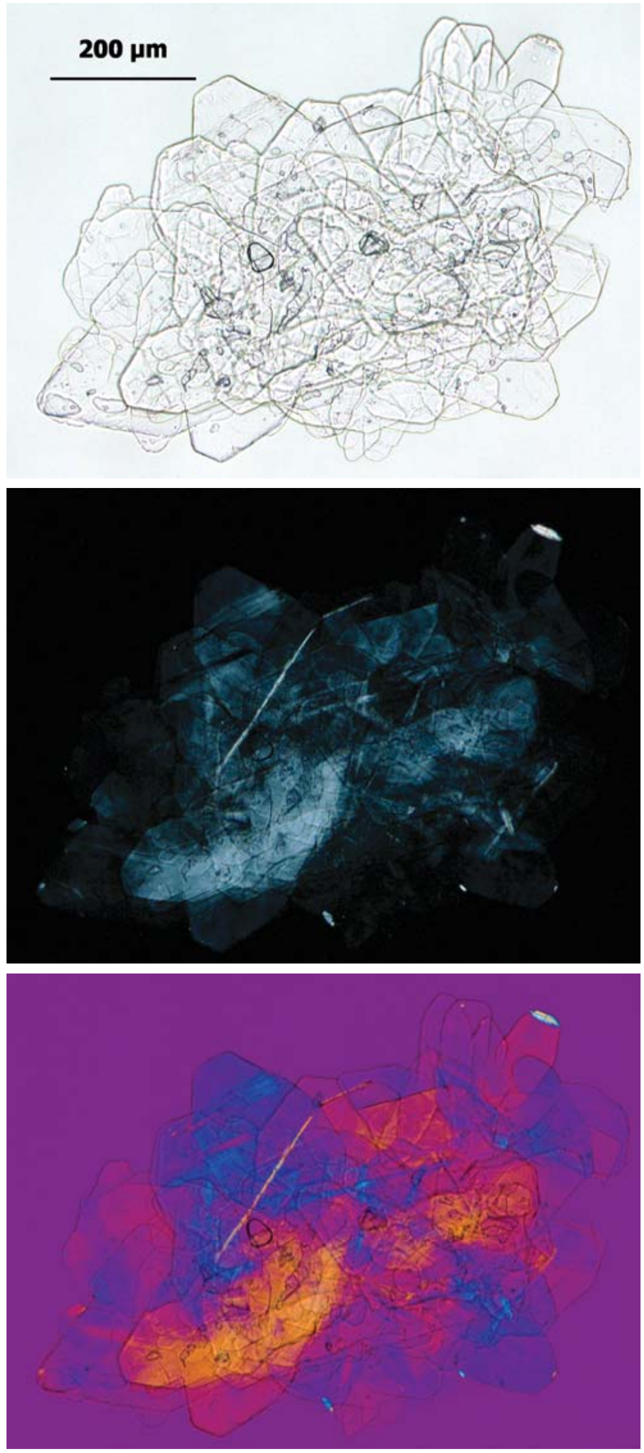

Figure 2. Stacked layers of sodium lauryl sulfate crystals are shown in plane polarized light (top), between crossed polars (middle) and in crossed polars with a 530 nm compensator (bottom). The mounting medium is 1.540 liquid.

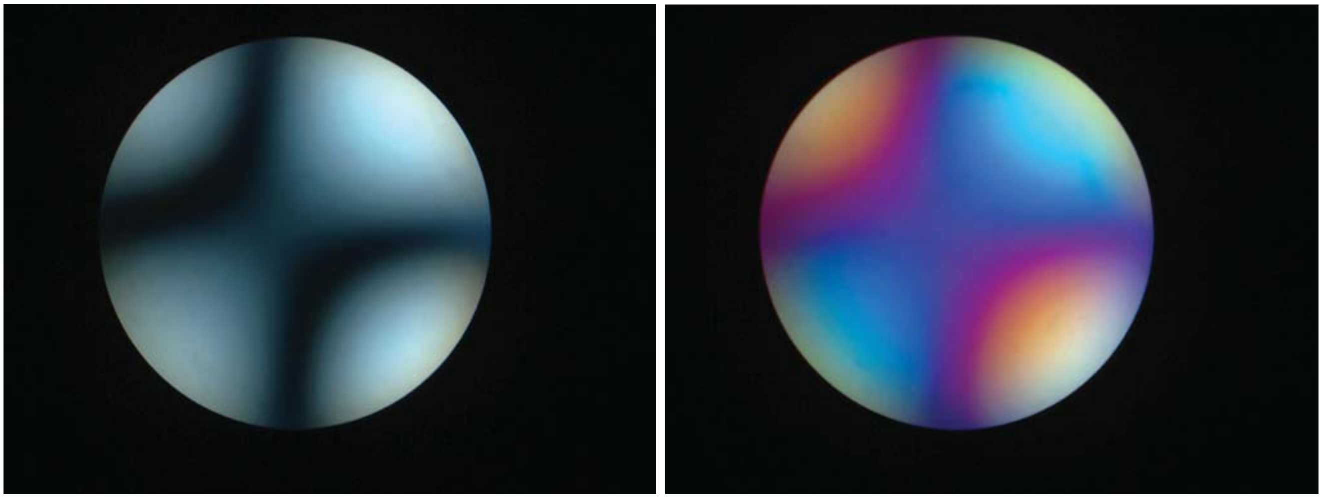

Figure 3. A centered Bxa interference figure obtained from a crystal of sodium lauryl sulfate in crossed polars (left) and again in crossed polars with a 530 nm compensator (right). The numerical aperture of the objective is 0.80.

line immersion method.

A grain mount of SLS crystals was prepared and surveyed in order to locate a crystal with well-defined edges that behaved optically as a single crystal should (complete extinction, uniform contrast along its edges). Only crystals meeting these criteria were used for the determination of optical crystallographic properties. Next, a single crystal, oriented to give a centered acute bisectrix (Bxa) interference figure, was located. The interference figure was oriented such that the two melatopes sat at a diagonal from southeast to northwest (Figure 3). The stage was then rotated 45° clockwise to align β parallel to the vibration direction of the polarizer so that its refractive index could be determined using the Becke line immersion method. The Becke line test was conducted on crystals mounted in standard Cargille refractive index liquids using sodium D light. The stage was then rotated 90° from β so that the α refractive index was parallel to the vibration direction of the polarizer.

The optic axial angle was determined using the method described by McCrone (4). The apparent optic axial angle (2E) was first determined and used to calculate the true optic axial angle (2V). To do this, a Bxa interference figure was oriented so that the two black isogyres were as far apart as possible. The distance between the center of the isogyres (d) and the diameter of the field of view (D) were measured in ocular scale divisions. The ratio of d/D together with the numerical aperture (NA) of the objective can be plotted on a table (4) to determine 2E. Alternatively, Equation 1 can be used to calculate 2E directly using d, D and NA. The 2E value, NA, and β refractive index are then used to determine 2V. Again, this can be done by means of a table (4) or by calculation using Equation 2. The 2V value together with the α and β refractive indices were then used to calculate the γ refractive index by means of Equation 3.

Equation 1:

$$\sin E = \frac{d\,(NA)}{D}$$

Equation 2:

$$\sin V = \frac{\sin E}{\beta}$$

Equation 3:

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

RESULTS

The optical properties determined for sodium lauryl sulfate using sodium D light are $\alpha = 1.463$, $\beta = 1.464$, $\gamma = 1.525$ (calculated), 2V = 15°, B = 0.062. The crystals are biaxial (+) with a negative sign of elongation. Orthographic projections with the optic orientation are illustrated in Figure 4.

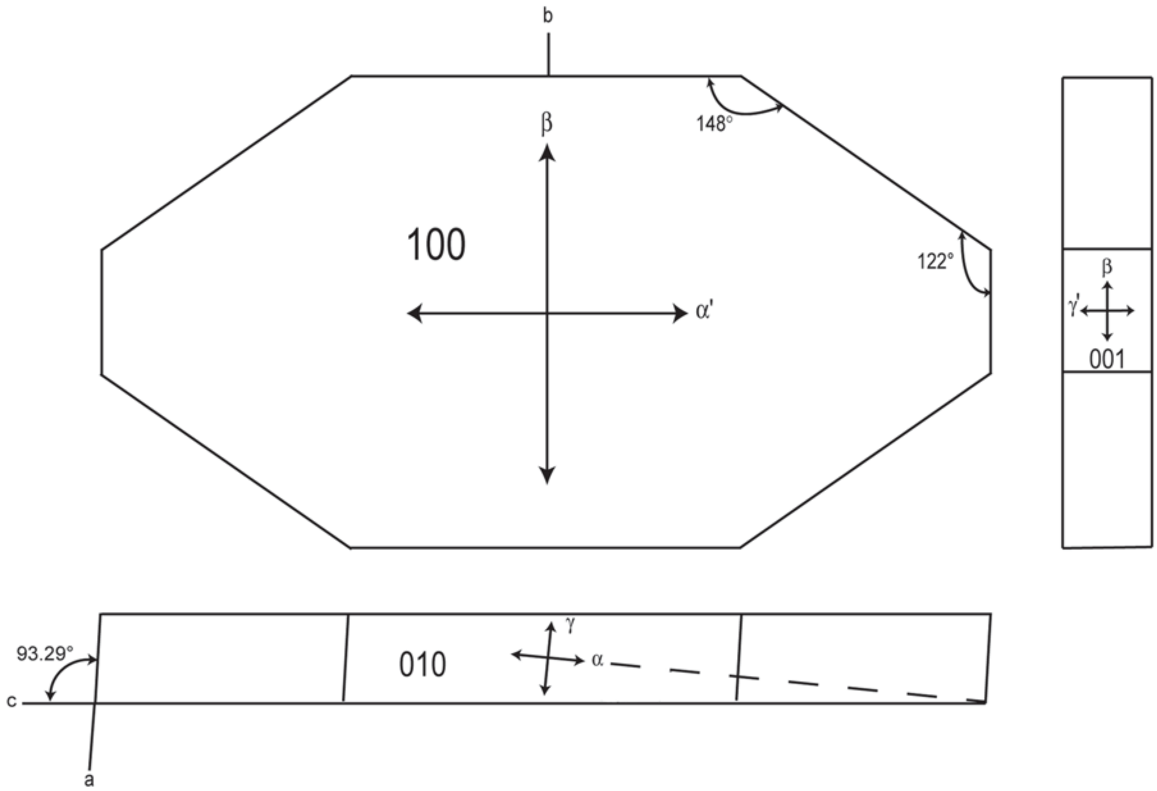

Figure 4. Orthographic projections of a typical SLS crystal with its optic orientation shown, based on measurements made with a polarized light microscope.

DISCUSSION AND CONCLUSIONS

The values of 2E (21.75°) and 2V (15°) were determined using Equations 1 and 2; 2V was rounded to the nearest whole degree. The d/D used to calculate 2E was 9/31, measured with an objective having a NA of 0.65. The γ refractive index was calculated using Equation 3. It was not possible to measure the γ refractive index directly due to the preferred orientation of the crystals in grain mount; the crystals are very thin and γ is roughly parallel to the optical axis of the microscope. Although this limited the accuracy with which the γ refractive index could be determined, it is not expected to be a limiting factor in the microscopical identification of unknown crystals, because SLS is likely to exhibit the same preferred orientation. As a result, only the α and β refractive indices, the optic axial angle and the optic sign could be determined for an unknown sample of SLS, and these properties should suffice for its identification.

It should be noted that because the 2V value is very small, a minor error in the measured value for α would result in a minor error for the calculated value of γ. The typical SLS crystal habit consists of very thin plates dominated by the {100} form that tend to stack in multiple layers with edges that often bend or curl over, similar to muscovite, talc and other phyllosilicates. This made the determination of precise extinction positions nearly impossible and rendered the crystals unsuitable for spindle stage work. The optical properties determined in grain mounts likely have greater error than those determined on suitable crystals using a spindle stage. However, grain mounts are the likely method used by analysts attempting to identify unknowns and, therefore, the data reported here should be a useful resource for microscopists hoping to identify this substance in unknown samples.

ACKNOWLEDGMENTS

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