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Optical and Crystallographical Properties of 1-(ρ -anisoyl)-4-methoxynaphthalene

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With 2 figures in the text

Abstract

Crystals of 1-(ρ -anisoyl)-4-methoxynaphthalene are monoclinic holohedral.

$$\beta = 94^\circ 56' \quad a/b = 1.398$$

The Crystals are colourless, biaxial with average $n = 1.650$. Parallel extinction with negative elongation. $2V = +86^\circ 30'$; $\Delta = 0.045$. These crystal-optical data are an indispensable means of identifying the compound.

Introduction

1-(ρ -Anisoyl-4-)methoxynaphthalene $C_{19}H_{16}O_3$ was prepared by ELKASHEF and SAID-AHMED²⁾ by the reaction between anisoyl chloride and α -methoxynaphthalene in the presence of anhydrous aluminium chloride in carbon disulphide medium.

For its identification only two tests are available, namely the melting point determination and the red colour it yields with concentrated sulphuric acid. However, these two tests can not be considered to be specific for the identification of the compound, as many other compounds will yield the same results. On the other hand, the crystal-optical properties give rise to several characteristics by means of which the compound can be identified at once with certainty.

In the present investigation the authors give the directly observed optical properties of this crystalline compound and its standard values.

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²⁾ M. Sc. Thesis, Cairo University, 1959.

Behaviour of the crystals on the heating stage

Before starting in the preparation of crystals for goniometric and optical measurements, it was necessary to establish that the compound does not exist in more than one crystallographical modification at different temperatures, for if this were so, each one would have to be crystallized out separately within the temperature range of its stability. A few crystals of the substance under investigation were heated on the heating stage and no change of phase was observed till the crystals melt at 108° C.

Crystallographical Properties

Measurements on the theodolite goniometer were carried out on five crystals grown from a hot ethyl alcohol solution. Their average length was four millimeters, their width 1.5 millimeters and their thickness about two millimeters.

The following table gives the observed forms, their corresponding average φ and ρ values and the character of their signals.

Table 1.

Miller face symbols	φ	ρ	Type of signal
(001)	90° 00'	4° 56' ($\pm 1'$)	Very sharp
(100)	90° 00'	90° 00'	Very sharp
(110)	35° 41' ($\pm 3'$)	90° 00'	Sharp

The calculated crystallographical constants (using the above φ and ρ values) are found to be:

$$\beta = 94^\circ 56'$$

$$a/b = 1.398$$

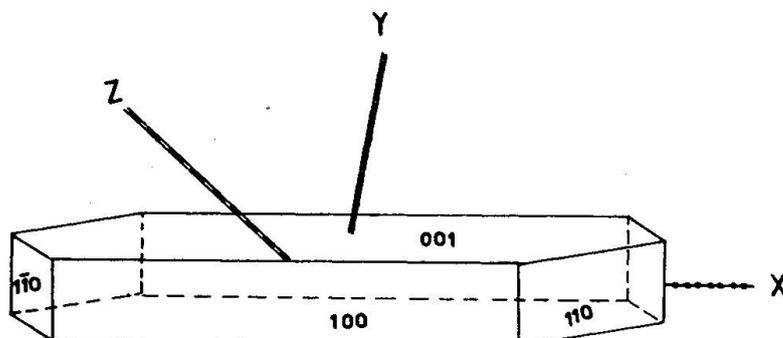


Fig. 1. An idealized crystal of 1-(ρ -Anisoyl)-4-methoxynaphthalene. Z, X and Y are optical directions.

c/b cannot be calculated because of the absence of domal or pyramidal faces.

From the previous crystallographical data, it appears that crystals of 1-(*p*-anisoyl)-4-methoxynaphthalene belong to the holosymmetrical class of the monoclinic system.

An idealized crystal drawn from the stereographic projection using RITTMANN's³⁾ nomogram for crystal drawing is shown in Fig. 1.

Optical Properties

The following optical properties were determined on flat-lying microcrystals grown on a glass carrier. Fig. 2 is a photomicrograph of these crystals.

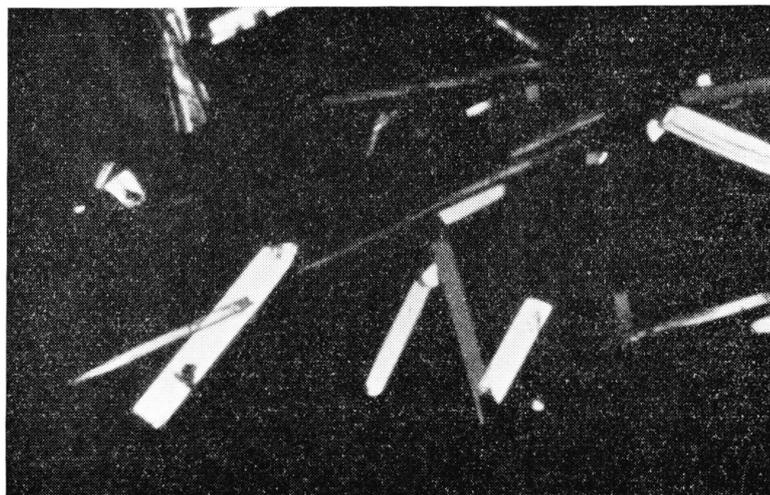


Fig. 2. Photomicrograph of crystals of 1-(*p*-Anisoyl)-4-methoxynaphthalene. Crossed Nicols, 94 \times .

Observations in polarized light

The crystals are colourless. Two types of faces are distinct: the first (100) is rectangular, while the second (001) is six-sided. Profile angles measured on (001), i. e. the angles between the edges [(010)/(110)], [(110)/(1 $\bar{1}$ 0)], and [(1 $\bar{1}$ 0)/(010)] are 126 $^\circ$; 108 $^\circ$ and 126 $^\circ$ respectively.

The indices of refraction could not be determined with sufficient accuracy, as the crystals are soluble in the immersion media. However,

³⁾ Schweiz. Min. Petrogr. Mitt., IX, p. 190, 1929.

the average index of refraction n was found to be slightly lower than that of α -monobromonaphthalene (1.658) and may be estimated to be about 1.650.

Observations between crossed nicols

The crystals are anisotropic. Both (100) and (001) show straight extinction and negative elongation, indicating that the crystals are prismatic parallel to the b -axis.

The relative birefringences Δ_0 measured on both (100) and (001) have been found to be 0.031 and 0.038 respectively.

Observations in the conoscope

On (100) the interference figure is monosymmetric S_{13} (according to the classification of the interference figures by the senior writer) with a positive character. The quotient of the extreme retardations (Q value) = 0.53 while $1/Q'$, which is the quotient of the intermediate retardation and the maximum one = 0.59 (using a numerical aperture of 0.85).

Having an average index of refraction of 1.650, the optic axial angle $2V$ has been found to be $+87^\circ$, while the maximum birefringence Δ was found to be 0.045.

On (001), the interference figure is monosymmetric, S_{13} , with a positive character.

Q value = 0.63, while $1/Q' = 0.76$, $2V = +86^\circ$ and $\Delta = 0.044$.

From the above conoscopical observations, it is evident that the average optic axial angle is $+86^\circ 30'$ while the average maximum birefringence $\Delta = 0.045$. The optical orientation is shown in Fig. 1.

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February 1960.