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Growth and Characterization of 8-Hydroxy Quinoline Nitrobenzoate

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Abstract

In this work we report the newly formed crystal structure of 8-Hydroxy quinoline nitro benzoate. The systematic investigation has been carried out on the growth and characterizations with a view of using this organic material in semiconductor devices apart from its various biological applications. Single crystals of 8-Hydroxy Quinoline Nitro Benzoate (8-HQNB) were grown successfully by slow evaporation solution growth technique. The new formation of the crystal with molecular formula $C_{16}H_{14}N_2O_6$ is confirmed by single crystal X-ray diffraction analysis. The crystallographic data has been deposited in Cambridge Crystallographic Data Centre [CCDC NO. 1005192]. Fourier Transform Infra Red (FTIR) spectroscopic analysis confirms the functional groups of the newly confirmed crystal. The UV-Vis-NIR studies reveal that there is no remarkable absorption in the visible region which proves its suitability for optical applications.

Keywords

Crystal Growth, Organic, FTIR

1. Introduction

The organic materials have been well known for their wide applications in superconductors [1], semiconductors [2], nonlinear optical devices [3] and photonic devices [4] [5]. 8-Hydroxy quinoline is a derivative of the heterocycle quinoline by placement of an OH group on carbon number 8. It is a monoprotic bidentate chelating agent. It exhibits antiseptic, disinfectant and pesticide properties [6]. It once was of interest as an anti-cancer drug and its solution in alcohol is used in liquid bandages [7]. Growth, structural, thermal and optical properties of 8-hydroxy quinoline by slow evaporation solution growth technique were already reported [8] [9]. In the present investigation an attempt has been

made to grow 8-hydroxy quinoline nitro benzoate single crystals. And the newly formed crystal structure has been reported. The newly formed crystal has been confirmed by the single crystal X-ray diffraction analysis and the data has been deposited in Cambridge crystallographic data centre (CCDC).

2. Materials and Methods

Analar grade samples of 8-hydroxy quinoline, 2-nitro benzoic acid acid and acetone were obtained from Sigma Aldrich for the growth of single crystals.

The scheme of preparation was done in the following way. 8-hydroxy quinoline and 2-nitro benzoic acid were taken into 1:1 molar ratio and was dissolved in acetone. They are stirred well for about 20 minutes using magnetic stirrer in order to achieve homogeneous mixing of the solvent. The saturated solution was filtered using Whatmann filter paper and transferred into crystallizing vessel and kept undisturbed for crystallization. After a period of one week, good quality crystals were harvested from the solution and are shown in Figure 1.

3. Results and Discussion

Single crystal X-ray diffraction analysis proves the newly formed crystal with molecular formula $C_{16}H_{14}N_2O_6$ and lattice parameters a = 7.693(5) Å, b = 23.203(5) Å and c = 8.654(5) Å. It crystallizes in monoclinic crystal system under centrosymmetric $P2_1/n$ space group. FT-IR spectrum was recorded to confirm the presence of functional groups. The optical transmittance spectrum for the newly formed crystal shows its wide transparency in the visible region.

3.1. Single Crystal X-Ray Diffraction Analysis

The grown crystals were subjected to single crystal X-ray diffraction analysis using Enraf Nonius-CAD 4 diffractometer. Crystallographic data of HQNB has been deposited with the Cambridge Crystallographic Data Centre [CCDC No. 1005192]. The crystallographic data are presented in Table 1.

The crystal structure of the grown sample is shown in Figure 2.

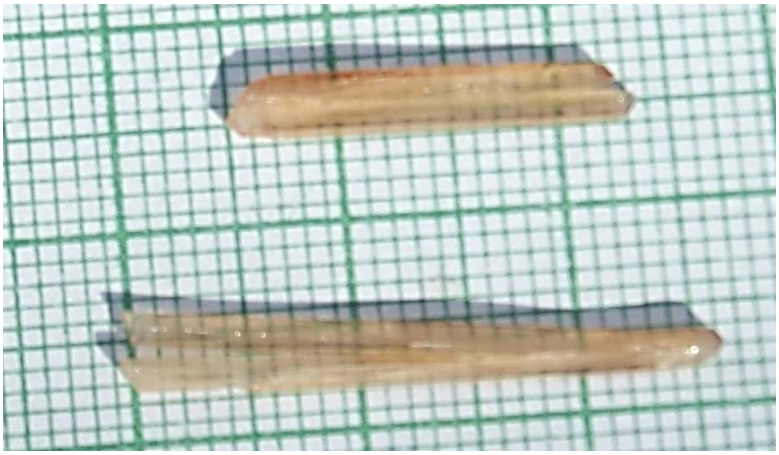


Figure 1. Photograph of 8-HQNB single crystals.

Table 1. Crystal data.

Crystal data	
Empirical formula	$C_{16}H_{14}N_2O_6$
Formula weight	330.29
Temperature	293(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, $P2_1/n$
Unit cell dimensions	a = 7.693(5) Å alpha = 90.000(5)˚
	b = 23.203(5) Å beta = 106.067(5)˚
	c = 8.654(5) Å gamma = 90.000(5)˚
Volume	1484.4(13) $Å^3$
Z	4
Calculated density	1.478 Mg/m^3

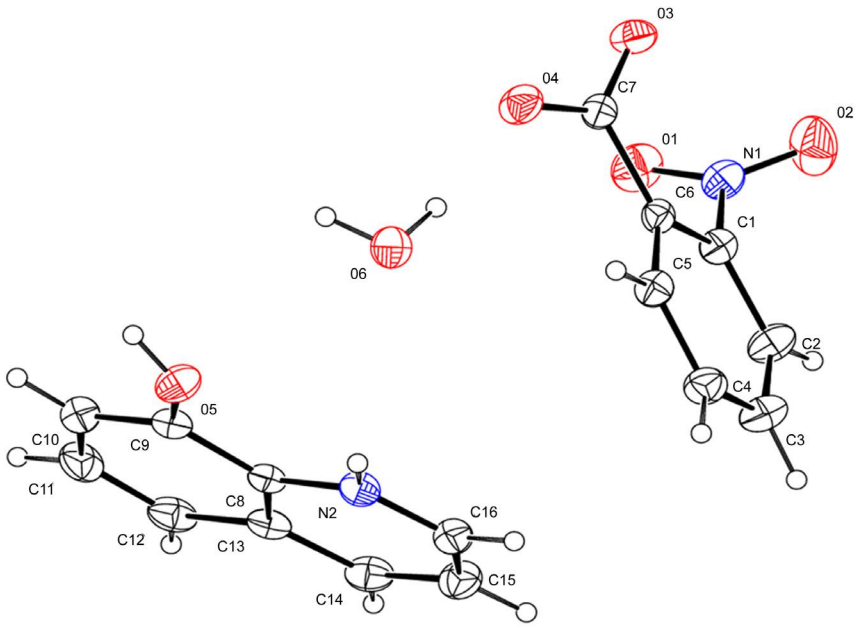


Figure 2. Crystal structure of 8-HQNB.

3.2. FT-IR Spectral Analysis

FT-IR spectrum was recorded to confirm the presence of functional groups using Bruker: RFS 27 spectrometer. The recorded spectrum of 8-HQNB crystal is depicted in **Figure 3**.

The O–H stretching vibration appears at 3385 cm^{-1} and the aromatic C–H stretching appears at 3076 cm^{-1}. The absorption band at 2889 cm^{-1} is assigned due to symmetric C–H stretching vibration of $-CH_2$ group. The carbonyl stretching vibrations band at 1716 cm^{-1} as well as anti symmetric and symmetric stretching vibration bands at 1534 cm^{-1} and 1375 cm^{-1} of COO^- group are obtained. The peak observed at 1601 cm^{-1} is assigned to C=N ring stretching vibration.

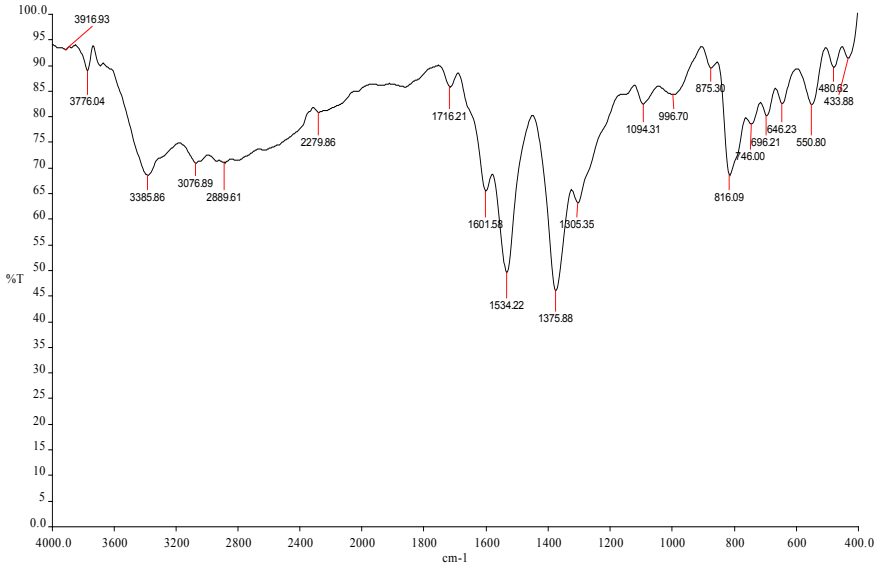


Figure3. F-T-IR spectrum of 8-HQNB.

The NO_2 stretching vibration of 2-nitrobenzoic acid moiety in the complex salt appears at 1534 cm^{-1}. The band observed at 875 cm^{-1} is assigned to out of plane bending of C–O deformation. The C-C in plane and out plane bending vibration was observed at 816 cm^{-1}. Also the vibrations around 746 - 550 cm^{-1} are assigned to in-plane bending vibration of quinoline. The observed frequencies with their assignments are summarized in **Table 2**.

3.3. UV-Vis-NIR Studies

The optical transmittance spectrum for the grown crystal was recorded using a Perkin-Elmer Lambda 35 Spectrophotometer in the wavelength range from 200 - 1200 nm. The recorded spectrum is shown in **Figure 4(a)**.

The grown crystal has a wider transparency range in the visible and NIR region. From the figure it is observed that the crystal has UV cut-off wavelength at 453 nm. A band observed at 225 nm is attributed to the excitation of aromatic ring (π to π^*). High transmittance of grown crystal in the visible region facilitates it to be a potential crystal for optoelectronics applications. The higher optical transmission in solution grown 8-HQNB crystal may be due to lesser defects and absence of inclusions, which in turn reduced scattering in HQNB crystals and increases the output intensity. The optical absorption coefficient (α) was calculated from the transmittance using the given formula

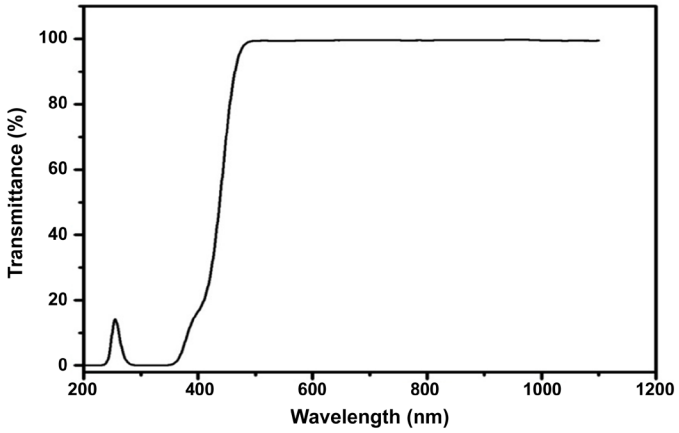
$$\alpha = \frac{2.3036 \log\left(1/T\right)}{d} \tag{4.1}$$

where T is the measured crystal transmittance and d is the thickness of the sample. Assuming parabolic trends, the relation between α and E_g is given by

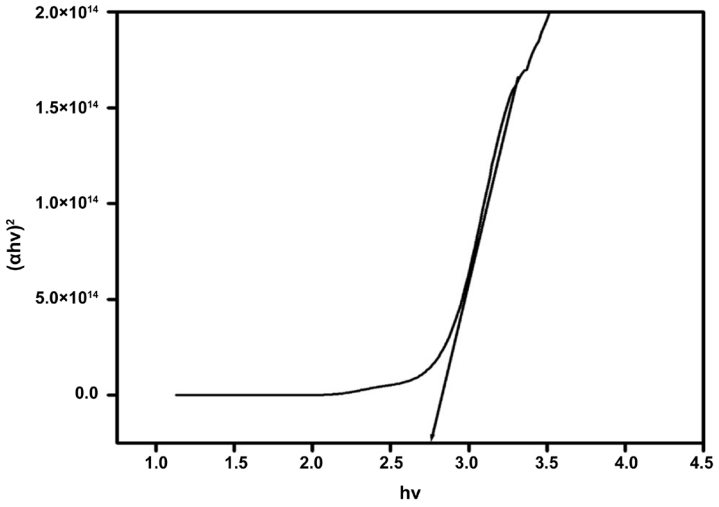
$$\alpha = \frac{A\left(h\nu - E_g\right)^n}{h\nu} \tag{4.2}$$

Table 2. FT-IR spectral data.

Wave number (cm^{-1})	Assignments
3385	O–H stretching
3076	aromatic C–H stretching
2889	C–H stretching of $–CH_2$ group
1716	carbonyl stretching
1534	anti-symmetric stretching of COO^-
1375	symmetric stretching of COO^-
1601	C=N ring stretching
1534	NO_2 stretching
875	out of plane bending of C–O deformation
816	C–C in plane and out plane bending
746 - 500	in-plane bending of quinoline



(a)



(b)

Figure 4. (a) Optical transmittance spectrum of 8-HQNB; (b) Plot of $(ahv)^2$ versus photon energy of 8-HQNB.

where A is a constant, E_g is the optical band gap of the material, υ is the frequency of the incident photon and h is the Planck's constant.

Figure 4(b) shows the plot of hυ and $(ahv)^2$. Optical band gap of the material was obtained by extrapolating the linear portion of the plots of $(ahv)^2$ and hv. The optical band gap was found to be 2.75 eV. Crystals with wide band gap expected to possess high laser damage threshold and large transmittance in the visible region.

4. Conclusion

Single crystals of 8-hydroxy quinoline nitro benzoate ($C_{16}H_{14}N_2O_6$) were grown by slow evaporation technique. The single crystal X-ray diffraction analysis shows that the grown crystal crystallizes under monoclinic crystal system with lattice parameters a = 7.693 Å, b = 23.203 Å and c = 8.654 Å. It belongs to centrosymmetric P_{21}/n space group with volume 1484.4 $Å^3$ and density 1.478 mg/m^3. The crystallographic data has been deposited in Cambridge Crystallographic Data Centre [CCDC No. 1005192]. The presence of functional groups was confirmed by FT-IR spectroscopic analysis. The UV-Vis-NIR studies reveal that there is no remarkable absorption in the visible region. The lower cut off wavelength of 8-HQNB is found to be 453 nm which proves its suitability for optical applications. The optical band gap energy of the grown crystal was calculated as 2.75 eV. Hence all the above attributes 8-hydroxy quinoline nitrobenzoate to a novel material for device applications.

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Conflicts of Interest

The author declares no conflicts of interest regarding the publication of this paper.

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