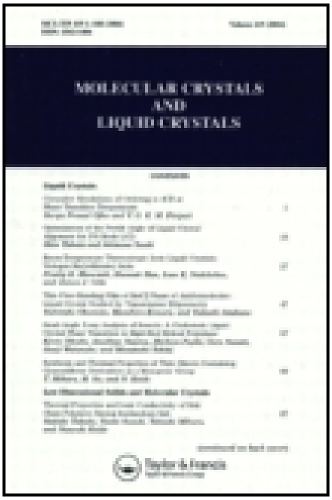

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Crystal Structure of a Chalcone Derivative

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The crystal and molecular structure of 1-(4-methoxyphenyl)-3-(phenyl)-2-propen-1-one derivative is determined by X-ray diffraction method. The compound, $C_{16}H_{14}O_2$, crystallises in the orthorhombic space group *Pbca* with $a = 10.921(2)$ Å, $b = 30.583(1)$ Å, $c = 7.535(3)$ Å, $V = 2516.7(9)$ Å^3, $Z = 4$, $D_{calc} = 1.242$ Mg/m^3, $\mu = 0.327$ mm^{-1}, $F_{000} = 504$, $\lambda(\text{Mo}K\alpha) = 0.71069$ Å and the structure was refined to $R = 0.044$.

Keywords: Crystal growth; Chalcone; NLO; Structure analysis

INTRODUCTION

The most commonly used second-order non-linear optical (NLO) crystals in the bulk form tend to be inorganic crystals such as ADP, ADP-isomorphs, KDP, ADA, etc. and the corresponding deuterated versions, the ABO_3 type of ferroelectrics such as $LiIO_3$, $LiNbO_3$, $KNbO_3$, etc. and the borates such as β-BaB_2O_4, LiB_3O_5, etc. Although the III-V and II-VI compounds such as GaAs, InSb, GaP, etc. generally have large dielectric coefficients, there is no birefringence that can be used to compensate for material dispersion because their structures are cubic. Therefore, they cannot be phase-matched in the bulk and are useful only in waveguide forms. Organic crystals hold promise because of the large variety of

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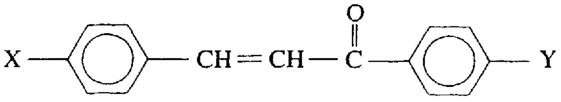
such materials and the potentiality to synthesize molecules according to some design principles.As a result, there have been extensive efforts to develop such materials for applications in nonlinear optics[1,2]. The organic NLO material contains one or more delocalized bonds, typically a ring structure like benzene. When substituted with donor and acceptor at the para position, they show large induced dipole moment under the influence of electromagnetic fields. Organic NLO materials are inherently poor in mechanical hardness, have low melting point and poor chemical inertness. Among many organic compounds reported for their second harmonic generation, chalcone derivatives are important materials for their excellent blue light transmittance and good crystallizability[3].

EXPERIMENTAL

Synthesis

Substituted chalcone derivatives can be synthesized by Claisen-Schmidt condensation reaction method[4]. This is the reaction of substituted acetophenone with substituted benzaldehyde in the presence of aqueous alkali. The basic molecular structure of substituted chalcone is given below.



A chalcone derivative with X = H and Y = OCH_3 was synthesized by a solution of benzaldehyde (0.01 mol) and appropriate p-methoxyacetophenone (0.01 mol) in ethanol (20 ml) and treating with an aqueous solution of potassium hydroxide (10 ml, 5%). The mixture was stirred well and kept aside for 24 hours. The resulting solid mass was collected by filtration, dried and recrystallised three to four times from ethanol.

Solubility

The compound is sparingly soluble in ethanol, methanol and highly soluble in acetone, benzene, N,N-dimethylformamide, xylene and 1,4-dioxane. The solubility is calculated using the formula

$$\text{Solubility (wt \%)} = \frac{\text{wt(solute)}}{\text{wt(solute + solvent)}} \times 100$$

Analysis

The UV/visible spectra is shown in figure 1. It is transparent in the entire visible region of the spectra. The IR spectra of the sample confirms the existence of funcional groups in the material and is represented in figure 2. The sample is also found to be chemically stable and non-hygroscopic.

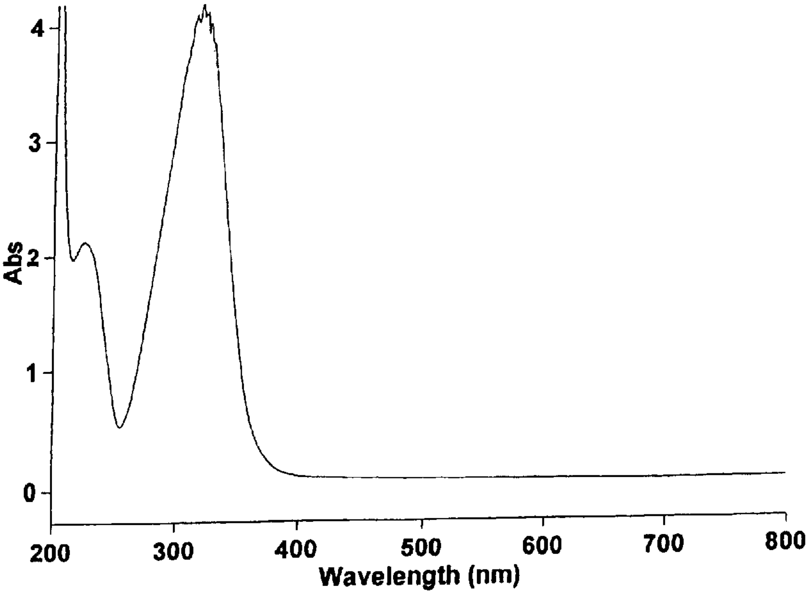


FIGURE 1 UV / Visible spectra

Crystal growth

The crystals were grown by slow evaporation technique[5]. Acetone and benzene were found to be the best solvents for the growth of crystals. A crystal of size 30 × 29 × 4 mm^3 was grown using benzene as solvent in 19 days. Another crystal of the same sample of size 35 × 25 × 4 mm^3 was obtained in 25 days using acetone as solvent and the same is shown in figure 3. Large parts of the crystals were found to be transparent.

Density measurement

The density was determined by flotation technique using small crystals of the sample. The measured density is 1.242(5) gm/cc.

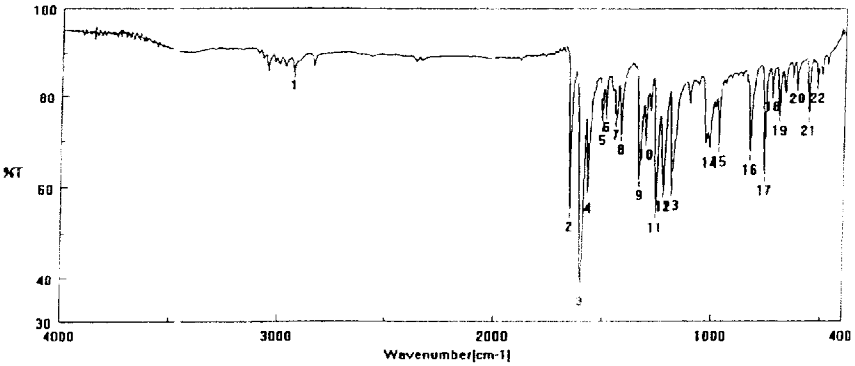


FIGURE 2 IR spectra

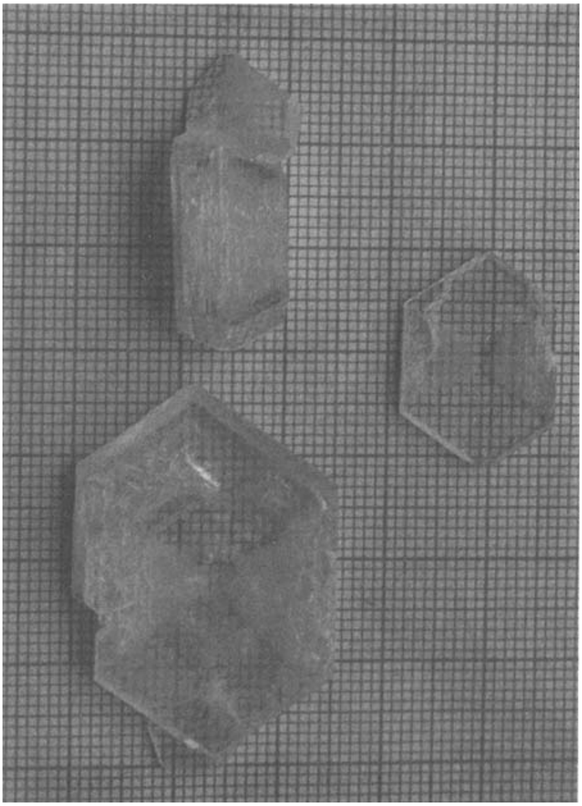


FIGURE 3 Crystal grown in acetone (See Color Plate II at the back of this issue)

TABLE I Atomic coordinates and equivalent thermal parameters of the non-hydrogen atoms

Atom	*x*	*y*	*z*	U_{eq}
O1	0.09952(17)	0.13031(5)	0.0996(2)	0.0720(6)
C10	0.0488(2)	0.26455(7)	0.1373(3)	0.0475(6)
O3	−0.07855(16)	0.32453(5)	0.1999(2)	0.0721(6)
C15	0.1590(2)	0.24800(7)	0.0744(3)	0.0524(6)
C9	0.0235(2)	0.31181(7)	0.1524(3)	0.0526(6)
C14	0.1806(2)	0.20354(7)	0.0596(3)	0.0532(6)
C13	0.0896(2)	0.17440(7)	0.1078(3)	0.0522(6)
C12	−0.0215(3)	0.19034(8)	0.1702(3)	0.0632(7)
C11	−0.0410(2)	0.23416(8)	0.1844(3)	0.0568(6)
C8	0.1183(2)	0.34386(7)	0.1060(3)	0.0558(6)
C4	0.1769(2)	0.41922(7)	0.0112(3)	0.0560(6)
C7	0.0936(2)	0.38485(8)	0.0687(3)	0.0601(7)
C3	0.1308(3)	0.45757(8)	−0.0615(3)	0.0711(8)
C16	0.2146(3)	0.11198(8)	0.0476(4)	0.0717(8)
C5	0.3043(3)	0.41465(8)	0.0194(3)	0.0639(7)
C6	0.3793(3)	0.44694(9)	−0.0471(4)	0.0800(8)
C2	0.2062(3)	0.48978(9)	−0.1260(4)	0.0830(10)
C1	0.3313(3)	0.48437(9)	−0.1192(4)	0.0834(9)

where $U_{eq} = 1/3\pi^2 \Sigma_i \Sigma_j a_i a_j \overline{a_i^* \cdot a_j^*}$

TABLE II Bond Lengths (Å)

Atoms	*Length*	*Atoms*	*Length*
O1-C13	1.354(3)	C12-C11	1.361(3)
O1-C16	1.430(3)	C8-C7	1.313(3)
C10-C15	1.389(3)	C4-C3	1.389(3)
C10-C11	1.397(3)	C4-C5	1.400(4)
C10-C9	1.476(3)	C4-C7	1.456(3)
O3-C9	1.234(3)	C3-C2	1.373(4)
C15-C14	1.385(3)	C5-C6	1.377(4)
C9-C8	1.468(3)	C6-C1	1.371(4)
C14-C13	1.384(3)	C2-C1	1.377(4)
C13-C12	1.389(3)		

NLO property

A preliminary study of second harmonic generation (SHG) measurement on powdered samples was undertaken[6]. The efficiency of the sample is less than that of urea.

Crystal structure analysis

A pale yellow rectangular crystal of dimension 0.2 × 0.2 × 0.3 mm was selected for X-ray data collection. All measurements were made an a Rigaku AFC7S diffractometer with graphite monochromated (MoKα) radiation. The data were collected at a temperature of 293K using the ω-2θ scan technique. Least squares refinement of 24 reflections in the range $28.84° \leq 2\theta \leq 37.38°$ yielded a primitive orthorhombic cell. The data were corrected for Lorentz and polarization effects. An empirical absorption correction [7] based on the azimuthal scans of three reflections was applied which resulted in the transmission factors ranging from 0.861 to 1.00. A total of 3644 intensities were collected in the 2θ range of $5 \leq 2\theta \leq 65°$ of which 2881 were unique ($R_{\mathrm{int}} = 0.0261$). The structure was solved by direct methods (SHELXS-97) [8] and the resulting fragment was expanded using difference Fouier maps. The function minimized throughout refinement was $\Sigma\, w(|F_o| - |F_c|)^2$ with $w=1/\sigma^2(F_o)$. Final refinement was carried out with anisotropic thermal parameters for all non-hydrogen atoms. Hydrogen atoms were placed at calculated positions and were not refined. The final cycle of full-matrix refinement [9] based on 3664 observed reflections ($I \geq 2\sigma(I)$) and 176 parameters converged to $R = 0.0445$ with goodness of fit = 1.086. The largest peak and deepest hole in the final difference map are 0.185 and −0.226 e.Å^{-3} respectively.

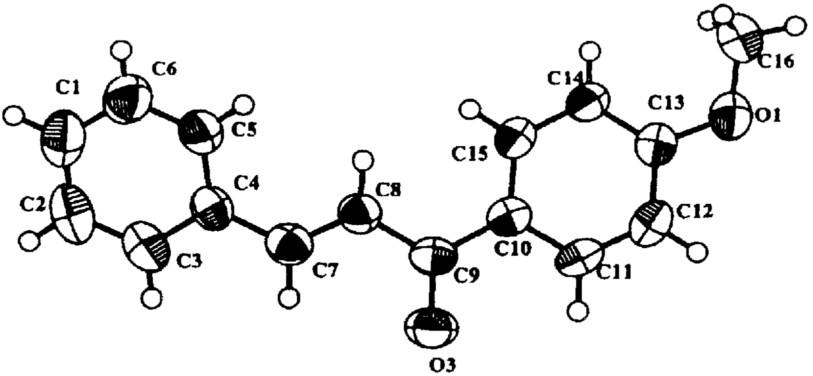


FIGURE 4 ORTEP of the molecule at 50% probability

TABLE III Bond Angles (°)

Atoms	Angle	Atoms	Angle
C13-O1-C16	118.24(19)	C11-C12-C13	120.6(2)
C15-C10-C11	116.9(2)	C12-C11-C10	121.7(2)
C15-C10-C9	123.0(2)	C7-C8-C9	122.9(2)
C11-C10-C9	120.0(2)	C3-C4-C5	117.5(2)
C14-C15-C10	122.2(2)	C3-C4-C7	120.0(2)
O3-C9-C8	119.7(2)	C5-C4-C7	1224(2)
O3-C9-C10	120.0(2)	C8-C7-C4	128.7(2)
C8-C9-C10	120.2(2)	C2-C3-C4	121.9(3)
C13-C14-C15	119.3(2)	C6-C5-C4	120.2(3)
O1-C13-C14	124.9(2)	C1-C6-C5	121.0(3)
O1-C13-C12	115.8(2)	C3-C2-C1	119.7(3)
C14-C13-C12	119.3(2)	C6-C1-C2	119.6(3)

Results and Discussion

The final positional coordinates of all the non-hydrogen atoms with equivalent isotropic thermal parameters are given in table I. The bond distances and bond angles for non-hydrogen atoms are given in tables II and III respectively. ORTEP [10] of the molecule at 50% probability is given in figure 4. Packing of the molecules along *a* axis is shown in figure 5. The weak non-linear optical property can be accounted in terms of crystal packing. The structure contains C-H...O hydrogen bonded dimers, which form a sheet parallel to *a*-axis[11].

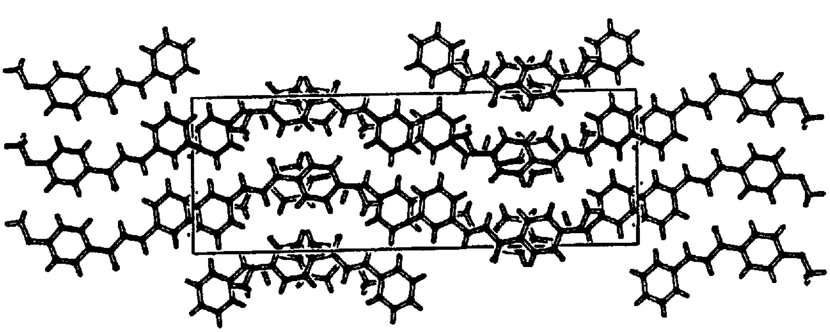


FIGURE 5 Packing of the molecules of down *a* axis

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References

[1] D.C. Wash., Nonlinear Optical Properties of Organic and Polymeric materials, D. Williams (ed.), *Am. Chem. Soc.*, (1983).
[2] D.S. Chemla and J. Zyss., Non-linear Optical Properities of Organic Molecular Crystals. **Vol. 1 and Vol. 2**, Academic Press, London, (1987).
[3] D. Fichou, T. Watanabe, T. Tanaka, S. Miyata, G. Goto and M. Nakayama., *Japan J. Appl. Phys.*, **27** (1988) L429.
[4] D.N., Dhar., The Chemistry of Chalcones and related compounds. JOHN WILEY and SONS, Newyork (1981).
[5] J.C. Brice., Crystal Growth from Solution, North Holland Publishing Company, (1973).
[6] S.K. Kurtz and T.T. Perry., *Appl. Phys.*, **39**, 3798, (1968).
[7] A.C.T. North, D.C. Philllips and F. S. Mathews., *Acta Cryst.* **A24**, 351–359 (1968).
[8] G.M. Sheldrick., SHELXS-97-Crystal Structure Solution Program, University of Göttingen, Germany (1997).
[9] G.M. Sheldrick., SHELXS-97-Crystal Structure Refinement Program, University of Göttingen, Germany (1997).
[10] L. Zsolnai., ZORTEP-Molecular Graphics Program, University of Heidelberg (1997).
[11] R. Srinivasa Gopalan., Investigation of molecular crystals using experimental charge density, JNCASR, Bangalore, India (2000).