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Crystal and molecular structure of 4 (4' N, N-dimethylamino) benzylidene-2-phenyloxazolin-5-one

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Abstract The crystal structure of the title compound, $C_{18}H_{16}N_2O_2$ has been determined by X-ray diffraction. The benzylidene ring A, the oxazolin-5-one ring system B and the attached phenyl ring C of the title compound (II) are almost planar having interplanar angles between A and B and that between B and C are 4.10 ° and 3.03 ° respectively. The N,N-dimethyl group is nearly coplanar with the benzylidene ring plane. There is no intermolecular hydrogen bond and the molecule is stabilised by the normal van der Waals interaction in the crystalline assembly.

Keywords Crystal structure, $C_{18}H_{16}N_2O_2$, N,N-dimethyl, van der Waals' interaction

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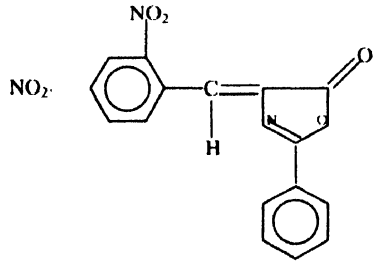
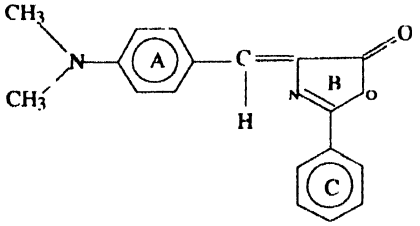
1. Introduction

Oxazoline compounds which form acyl-enzyme complexes with papain, are of medicinal importance in biology. Although the first alkyloxazoline was reported by Gabriel [1], the crystal structures of this class of compounds are limited because few of the known oxazoline are solid at room temperature. The crystal structure of 4-(4-2-dinitro) benzylidene-2-phenyloxazolin-5-one (I) has been reported in literature [2]. Crystallographic study of the analogous 4-(4-N,N- dimethylamino) benzylidene-2-phenyloxazoline-5-one (II) has been reported in this paper.



I.4 (4,2-dinitro)benzylidene-2-phenyloxazolin-5-one



II. 4 (4/ N,N-dimethylamino) benzylidene-2-phenyloxazoline-5-one

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2. Experimental details

0.05 mole of hipparic acid was dissolved in 100 ml. of dimethylformaide saturated with sulfurtrioxide. To this solution 0.05 mole of (4 N,N-dimethyl) benzyldehyde was added at room temperature and stirred in a magnetic stirrer for 30 minutes. The red precipitate obtained was recovered and recrystallised from 95% of ethanol. Preliminary X-ray study revealed monoclinic crystal system. The systematic absences established the space group as $P2_1/c$. The unit cell parameters were refined by least squares method on the basis of 25 independent high angle reflections. The data were corrected for Lorentz and polarisation effects, but no absorption correction was made.

3. Structure solution and refinement

The structure was solved by direct methods using the program SHELXS 97 [3] and refined on F^2 using the program SHELXL97 [4]. Full matrix least-squares refinement with anisotropic temperature factors to non-hydrogen atoms led to the R value

Table 1. Crystal data for the title compound

Chemical formula	$C_{18}H_{16}N_2O_2$
Molecular weight	292.33
Crystal system	monoclinic
Space group	$P2_1/c$
Lattice parameters	a = 12.177(4) Å
	b = 3.966(1) Å
	c = 30.944(8) Å
	b = 101.170(10)°
Volume	1466.1(7) Å^3
Z	4
Measured density, Dm	1.320 Mg m^{-3}
Calculated density Dx	1.324 Mg m^{-3}
Temperature	293 K
Crystal size	0.45 × 0.18 × 0.14 mm^3
Crystal colour	Pink
Radiation	CuKα
Wavelength	1.5418 Å
Absorption coefficient (mu)	0.70 mm^{-1}
0 (Theta) max.	68°
Index ranges	$0 < h \leq 14$, $0 \leq k < 4$, $36 \leq l \leq 36$
No of reflections measured	2325
No. of unique reflections	2289
No. of observed reflections	1996 [$I > 2\sigma(I)$]
R_{int}	0.078
F (000)	616
Goodness of fit	1.134
Final R	0.0787
R_w	0.2073

Table 2. Fractional atomic coordinates for non hydrogen atoms and equivalent isotropic displacement parameters (Å^2)

$$\left(U_{eq} = (1/3)\sum_i\sum_j U_{ij}a^*a_j^*a_ia_j\right)$$

	x	y	z	U_{eq}
O1	0.2152(2)	0.7461(7)	0.12868(8)	0.0555(3)
O2	0.2052(2)	0.5231(9)	0.06047(9)	0.0723(4)
C3	0.7799(3)	1.1018(9)	0.07764(8)	0.0451(4)
N4	0.3902(2)	0.9565(8)	0.14226(9)	0.0482(4)
C5	0.5540(3)	0.9142(8)	0.07425(7)	0.0416(4)
N6	0.8890(2)	1.1973(9)	0.07951(6)	0.0576(4)
C7	0.6102(3)	0.8396(5)	0.04014(7)	0.0489(4)
C8	0.7241(3)	1.1841(9)	0.11209(9)	0.0469(4)
C9	0.7192(3)	0.9369(8)	0.04093(8)	0.0487(4)
C10	0.4409(3)	0.7959(9)	0.07120(9)	0.0486(4)
C11	0.2767(3)	1.0075(9)	0.19961(8)	0.0488(4)
C12	0.6142(3)	1.0846(9)	0.11036(9)	0.0452(4)
C13	0.3715(3)	0.8203(9)	0.10010(7)	0.0461(4)
C14	0.2592(3)	0.6695(8)	0.09119(9)	0.0539(4)
C15	0.2999(3)	0.9096(8)	0.15709(8)	0.0476(4)
C16	0.3594(3)	1.1631(9)	0.23009(9)	0.0588(4)
C17	0.3406(4)	1.2517(8)	0.27074(9)	0.0676(4)
C18	0.1543(4)	1.0355(9)	0.25182(8)	0.0679(4)
C19	0.1737(3)	0.9426(8)	0.21104(9)	0.0580(4)
C20	0.9528(4)	1.3575(9)	0.1182(2)	0.0661(5)
C21	0.2377(4)	1.1862(8)	0.28157(7)	0.0674(4)
C22	0.9452(4)	1.1234(7)	0.0437(2)	0.0673(6)

of 0.0882. The final R value was 0.0787 with the inclusion of the hydrogen atoms from the difference Fourier maps with isotropic thermal parameters. The peak heights in the final difference Fourier map were in the range of 0.28 to –0.33 eÅ^{-3}. The atomic scattering factors used were taken from the International Table for X-ray Crystallography Vol. IV [5]. The molecular geometry was calculated using the program SHELXL 97 [4]. The crystal data and the fractional coordinates and equivalent isotropic thermal parameters for all non-hydrogen atoms are shown in Table 1 and Table 2 respectively.

4. Result and discussion

The ORTEP [6] view of the molecule with atom numbering scheme and the packing of the molecule are shown in Figure 1 and Figure 2 respectively. The molecule consists of an essentially planner oxazolin-5-one moiety linked to phenyl and benzylidene rings *via* C-C and CH groups. The torsion angles (Table 3) an

that the molecule is almost planar; the maximum deviation of an atom (C3) is 0.026(4) Å. The planarity of the molecule is presumed

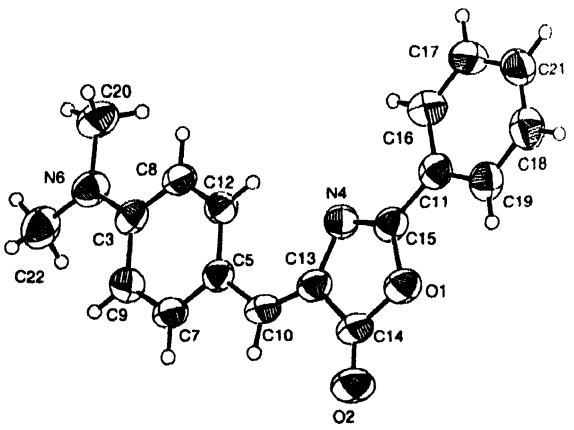
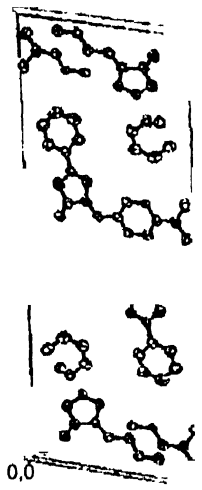


Figure 1. ORTEP diagram of the molecule with atom numbering scheme the H-atoms are shown by circles.

Table 3. Selected bond distances (Å), angles (°) and the torsion angles (^{0}) and their esd's in parenthesis

N4 – C13 = 1.389(4)	C22 – N6 – C20 = 117.9(4)	C22 – N6 – C3 – C8 = – 177.9(4)
N4 – C15 = 1.284(4)	C22 – N6 – C3 = 120.8(3)	C20 – N6 – C3 – C9 = 177.8(3)
N6 – C3 = 1.372(4)	C20 – N6 – C3 = 121.2(3)	C13 – C10 – C5 – C7 = 176.6(5)
C14 – O2 = 1.196(3)	O1 – C15 – C11 = 116.8(3)	C5 – C10 – C13 – C14 = – 177.7(4)
N6 – C20 = 1.442(5)	N4 – C15 – C11 = 128.0(3)	C16 – C11 – C15 – O1 = 178.0(3)
N6 – C22 = 1.441(6)	N4 – C15 – O1 = 115.2(2)	C19 – C11 – C15 – N4 = 179.3(4)

o be due to conjugation effect. The dihedral angle between the oxazolin-5-one moiety (N4,C13,C14,O1,C15,O2) and the attached henyl ring (C11,C16,C17,C21,C18,C19) is 3.0(2)^0, while that etween the oxazolin-5-one and the benzylidene (C5,C7,C9,C3,C8,C12) parts is 4.1(2)^0. The bond lengths and bond ngles (Table 3) of the title compound are comparable to those bserved in related oxazolin-5-one compounds [7-10]. The crystal tructure is stabilised by van der Waals' contacts.



igure 2. Packing of the molecule viewed along the b-axis, the H-atoms ere omitted for clarity

Acknowledgment

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