

Data collection

Enraf–Nonius CAD-4 diffractometer	$\theta_{\max} = 65^\circ$
$\omega/2\theta$ scans	$h = 0 \rightarrow 10$
Absorption correction: none	$k = 0 \rightarrow 12$
2554 measured reflections	$l = 0 \rightarrow 31$
2544 independent reflections	3 standard reflections
2189 reflections with $I > 2\sigma(I)$	every 100 reflections
R_{int} not available (see below)	frequency: 60 min
	intensity decay: <2%

Refinement

Refinement on F^2	$(\Delta/\sigma)_{\max} = 0.004$
$R[F^2 > 2\sigma(F^2)] = 0.051$	$\Delta\rho_{\max} = 0.194 \text{ e } \text{\AA}^{-3}$
$wR(F^2) = 0.178$	$\Delta\rho_{\min} = -0.231 \text{ e } \text{\AA}^{-3}$
$S = 0.913$	Extinction correction: none
2542 reflections	Scattering factors from
307 parameters	<i>International Tables for</i>
H atoms riding	<i>Crystallography</i> (Vol. C)
$w = 1/[\sigma^2(F_o^2) + (0.0708P)^2]$	
where $P = (F_o^2 + 2F_c^2)/3$	

Table 1. Selected torsion angles ($^\circ$)

C1—O1—C0'—N1	178.8 (5)
O1—C0'—N1—C1A	172.7 (4)
C0'—N1—C1A—C1'	108.8 (5)
N1—C1A—C1'—N2	167.8 (4)
C1A—C1'—N2—C2A	173.5 (4)
C1'—N2—C2A—C2'	-106.4 (5)
N2—C2A—C2B—C2D1	-170.6 (4)
N2—C2A—C2B—C2D2	-47.6 (6)
N2—C2A—C2'—N3	115.3 (4)
C2A—C2'—N3—C3A	-169.6 (4)
C2'—N3—C3A—C3'	-100.0 (5)
N3—C3A—C3B—C3G1	-67.9 (5)
C3A—C3B—C3G1—C3G2	77.5 (7)
N3—C3A—C3'—O4	-29.6 (6)

Table 2. Hydrogen-bonding geometry (\AA , $^\circ$)

D—H...A	D—H	H...A	D...A	D—H...A
N4—H1N4...O0'	0.86	2.10	2.917 (6)	159
N1—H1N1...O2''	0.86	2.11	2.937 (6)	161
N2—H1N2...O1''	0.86	2.05	2.899 (5)	171
N3—H1N3...O3''	0.86	2.16	3.010 (5)	171

Symmetry codes: (i) $\frac{1}{2} + x, \frac{1}{2} - y, 2 - z$; (ii) $x - \frac{1}{2}, \frac{1}{2} - y, 2 - z$; (iii) $\frac{1}{2} + x, -\frac{1}{2} - y, 2 - z$.

The title structure was solved by direct methods and refined by full-matrix anisotropic least squares assuming all H atoms riding in calculated positions with fixed isotropic U 's. The data collection was not continued beyond $\theta_{\max} = 65^\circ$ due to the large number of too-weak reflections, and also because of the sudden failure in the encoders of the goniometer device. R_{int} was not available since the data collection and processing were carried out by a fees-for-service organization which sent only hkl , F_o and $\sigma(F_o)$, and deleted the files before *Acta Crystallographica Section C*'s requirements regarding R_{int} became known. Since we used the *TWIN* option, the Flack parameter was suppressed.

Data collection: *SDP* (Frenz, 1978). Cell refinement: *SDP*. Data reduction: *SDP*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ZORTEP* (Zsolnai, 1997). Software used to prepare material for publication: *SHELXL93* and *PARST* (Nardelli, 1983, 1995).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1355). Services for accessing these data are described at the back of the journal.

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4-(Dimethylaminomethylene)-2-(2-nitrophenyl)oxazol-5(4H)-one

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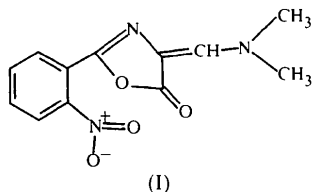
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Abstract

The crystal structure of the title compound, $\text{C}_{12}\text{H}_{11}\text{N}_3\text{O}_4$, has been determined as part of a study of the luminescent activity of oxazolin-5-ones [Singh & Singh (1994). *Indian J. Chem.* **33B**, 232–235]. The dihedral angle between the 2-oxazoline (4,5-dihydrooxazole) and phenyl rings is $12.48(8)^\circ$. A conjugation effect is observed in the dimethylaminomethylene moiety.

Comment

The numbering scheme of the title compound, (I), is shown in Fig. 1. Bond distances and angles for the



oxazoline ring and the dimethylaminomethylene moiety are within expected ranges (Leban *et al.*, 1991; Ahmet *et al.*, 1994). The bond distances of the nitro group [N—O 1.212 (2) and 1.220 (2) Å] are within expected values [N—O 1.217 (11) Å; Allen *et al.*, 1987].

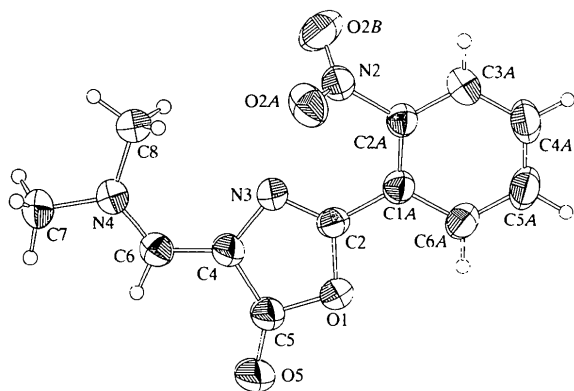


Fig. 1. Displacement ellipsoid plot of the title compound (H atoms omitted). Ellipsoids have been plotted at the 50% probability level.

Atoms C1A, C2, N3, C4 and C6 are coplanar. The deviation from 120° observed for the C6—C4—N3 bond angle [128.9 (2)°] of the oxazoline ring might be a consequence of repulsion between the lone pair of electrons on the N atom and the H atom of C8 (Centore *et al.*, 1996). A conjugation effect is observed in the dimethylaminomethylene moiety. The N4—C6 bond length of the aminomethylene moiety in the present structure is 1.314 (2) Å.

The dihedral angle between the phenyl and oxazoline rings is 12.48 (8)°. The nitro group is twisted from the plane of the phenyl ring. The angle between the plane of the C—NO₂ group and the phenyl ring is 108.61 (7)°. The phenyl and oxazole rings adopt planar conformations, as evidenced by the torsion angles (Table 1).

Short intermolecular contacts are observed in (I): O1···O5(−*x*+2, −*y*+2, −*z*+1) 3.329 (3) and O2A···N2(−*x*+1, −*y*+1, −*z*) 3.186 (2) Å.

Experimental

N-(3,4-Dinitrobenzoyl)glycine (0.01 mol) was dissolved in 10 ml of dimethylformamide. The reaction mixture was cooled to 273 K and phosphorus oxytrichloride (2.8 ml) was added dropwise with stirring. The reaction mixture was allowed to reach room temperature and was then poured onto crushed ice, filtered and dried. Recrystallization was performed from methanol (yield 85%; m.p. 423 K).

Crystal data

C₁₂H₁₁N₃O₄
M_r = 261.24
 Triclinic
P $\bar{1}$
a = 8.049 (2) Å
b = 12.756 (2) Å
c = 6.209 (2) Å
 α = 98.82 (2)°
 β = 100.94 (3)°
 γ = 93.99 (2)°
V = 615.3 (3) Å³
Z = 2
D_x = 1.410 Mg m^{−3}
D_m not measured

Mo *K*α radiation

λ = 0.7017 Å

Cell parameters from 25 reflections

θ = 2–25°

μ = 0.108 mm^{−1}

T = 293 (2) K

Rectangular

0.20 × 0.15 × 0.10 mm

Lustrous light orange

Data collection

Rigaku AFC-7S diffractometer

ω -2 θ scans

Absorption correction:

ψ scan (North *et al.*, 1968)

T_{min} = 0.979, *T_{max}* = 0.989

2469 measured reflections

2170 independent reflections

1628 reflections with

I > 2 σ (*I*)

R_{int} = 0.035

θ_{\max} = 24.66°

h = −9 → 9

k = −15 → 15

l = 0 → 7

3 standard reflections

every 100 reflections

intensity decay: negligible

Refinement

Refinement on *F*²

R [*F*² > 2 σ (*F*²)] = 0.041

wR(*F*²) = 0.134

S = 1.060

2170 reflections

178 parameters

H atoms constrained

w = 1/[$\sigma^2(F_o^2)$ + (0.0751*P*)² + 0.0784*P*]

where *P* = (*F_o*² + 2*F_c*²)/3
 (Δ/σ)_{max} = 0.002

$\Delta\rho_{\max}$ = 0.437 e Å^{−3}

$\Delta\rho_{\min}$ = −0.464 e Å^{−3}

Extinction correction:

SHELXL93 (Sheldrick, 1993)

Extinction coefficient:

0.050 (10)

Scattering factors from

International Tables for Crystallography (Vol. C)

Table 1. Selected geometric parameters (Å, °)

O1—C2	1.372 (2)	N4—C8	1.458 (2)
O1—C5	1.413 (2)	N4—C7	1.467 (2)
N3—C2	1.286 (2)	C2A—C1A	1.391 (3)
N3—C4	1.398 (2)	C1A—C6A	1.397 (2)
O5—C5	1.211 (2)	C6A—C5A	1.388 (3)
C2—C1A	1.461 (2)	C5—C4	1.430 (3)
N2—O2B	1.212 (2)	C4—C6	1.382 (2)
N2—O2A	1.220 (2)	C3A—C4A	1.385 (3)
N2—C2A	1.472 (2)	C4A—C5A	1.369 (3)
N4—C6	1.314 (2)		

C2—O1—C5	105.41 (13)	C2A—C1A—C6A	116.6 (2)
C2—N3—C4	105.21 (14)	C2A—C1A—C2	123.03 (15)
N3—C2—O1	115.49 (15)	C6A—C1A—C2	120.3 (2)
N3—C2—C1A	127.5 (2)	C5A—C6A—C1A	121.0 (2)
O1—C2—C1A	116.96 (14)	O5—C5—O1	120.2 (2)
O2B—N2—O2A	124.5 (2)	O5—C5—C4	135.5 (2)
O2B—N2—C2A	117.8 (2)	O1—C5—C4	104.35 (14)
O2A—N2—C2A	117.7 (2)	C6—C4—N3	128.9 (2)
C6—N4—C8	123.16 (15)	C6—C4—C5	121.5 (2)
C6—N4—C7	120.2 (2)	N3—C4—C5	109.54 (15)
C8—N4—C7	116.6 (2)	N4—C6—C4	129.3 (2)
C3A—C2A—C1A	122.8 (2)	C2A—C3A—C4A	118.9 (2)
C3A—C2A—N2	116.2 (2)	C5A—C4A—C3A	120.0 (2)
C1A—C2A—N2	120.93 (14)	C4A—C5A—C6A	120.6 (2)
C4—N3—C2—O1	0.2 (2)		
C4—N3—C2—C1A	-179.3 (2)		
C5—O1—C2—N3	0.1 (2)		
O2B—N2—C2A—C3A	70.3 (2)		
O2A—N2—C2A—C3A	-106.4 (2)		
O2B—N2—C2A—C1A	-112.3 (2)		
O2A—N2—C2A—C1A	71.0 (2)		
N2—C2A—C1A—C2	6.0 (3)		
N3—C2—C1A—C2A	12.2 (3)		
C2—O1—C5—O5	-179.7 (2)		
C2—O1—C5—C4	-0.4 (2)		
C2—N3—C4—C6	175.9 (2)		
C2—N3—C4—C5	-0.4 (2)		
O5—C5—C4—C6	3.0 (3)		
O1—C5—C4—C6	-176.19 (15)		
O5—C5—C4—N3	179.6 (2)		
O1—C5—C4—N3	0.5 (2)		
C8—N4—C6—C4	0.2 (3)		
C7—N4—C6—C4	-177.9 (2)		
N3—C4—C6—N4	0.2 (3)		
C5—C4—C6—N4	176.2 (2)		
C1A—C2A—C3A—C4A	-3.1 (3)		
N2—C2A—C3A—C4A	174.3 (2)		
C2A—C3A—C4A—C5A	1.2 (3)		
C3A—C4A—C5A—C6A	1.6 (3)		
C1A—C6A—C5A—C4A	-2.7 (3)		

All non-H atoms were refined with anisotropic displacement parameters. All H atoms were placed in fixed positions.

Data collection: Rigaku AFC software. Cell refinement: Rigaku AFC software. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ZORTEP* (Zsolnai, 1997). Software used to prepare material for publication: *SHELXL93*. Geometric calculations: *PARST* (Nardelli, 1983).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: DE1078). Services for accessing these data are described at the back of the journal.

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Ethyl 5-Ethoxy-3-methyl-1*H*-indole-2-carboxylate

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Abstract

The title compound, C₁₄H₁₇NO₃, having an ethoxy-carbonyl group at C2, serves as an important precursor in the synthesis of fused-indole heterocycles of biological importance. The indole moiety is planar and it forms dihedral angles of 2.3 (1) and 3.9 (1)° with the ethoxycarbonyl group at C2 and the ethoxy group at C5, respectively. The centrosymmetrically related molecules are held together by hydrogen bonds across a centre of symmetry and form dimers.

Comment

Indole derivatives have important pharmacological uses because of the range of anti-allergic, central nervous system depressant and muscle relaxant properties (Harris & Uhle, 1960; Wei & Stanley, 1970; Reynolds & Carson, 1970; Ho *et al.*, 1986).

The indole ring of the title compound, (I), is planar, with a maximum deviation of 0.007 (5) Å from the best plane. The ethoxy group at C5 and the ethoxycarbonyl group at C2 are inclined with respect to the mean plane of indole at angles of 3.9 (1) and 2.3 (1)°, respectively. The methyl group at C3 is in the plane of the indole moiety, with a deviation of 0.018 (2) Å.