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5-(2-Chlorobenzoyl)-1,3-benzoxazol-2(3*H*)-one

Şamil Işık,[a] Yavuz Köysal,[a]* Metin Yavuz,[a] Meriç Köksal[b] and Hakkı Erdoğan[c]

[a]Department of Physics, Faculty of Arts and Sciences, Ondokuz Mayıs University, Kurupelit, 55139 Samsun, Turkey, [b]Faculty of Pharmacy, Yeditepe University, 34755 Istanbul, Turkey, and [c]Department of Pharmaceutical Chemistry, Faculty of Pharmacy, Hacettepe University, 06100 Ankara, Turkey

Correspondence e-mail: yavuzk@omu.edu.tr

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.042
wR factor = 0.125
Data-to-parameter ratio = 19.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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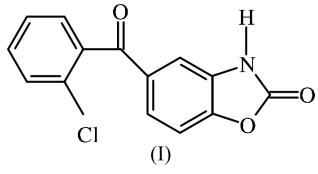
The title compound, $C_{14}H_{18}ClNO_3$, contains a planar dihydrobenzoxazolone ring system and a planar chlorobenzoyl group; the dihedral angle between the two planes is 86.84 (4)°. In the crystal structure, there are intermolecular C—H···O and N—H···O hydrogen bonds, together with π–π stacking interactions.

Comment

Acylated benzoxazolone derivatives have been extensively synthesized and evaluated for their potential medical use, in view of their analgesic, antipyretic, anticonvulsant, hypnotic and antimicrobial activities (Sam & Valentine, 1969; Aichaoui, Lesieur & Henichart, 1992; Aichaoui, Lesieur, Lespagnol *et al.*, 1992; Liacha *et al.*, 1999; Uçar *et al.*, 1998). Although both the ring N and O atoms in benzoxazolone are electron-donating, it has been claimed that the acylation product cannot be easily predicted (Aichaoui *et al.*, 1991). Many investigations on benzoxazolin-2-one have shown that direct acylation always gives only one product, *viz*. that with the acyl group at the 6-position (Aichaoui *et al.*, 1992; Yous *et al.*, 1994).



(I)

The site of acylation in 6-acylbenzoxazolin-2(3*H*)-ones has been confirmed by X-ray single-crystal diffraction (Mairesse *et al.*, 1984) and ^{1}H NMR spectroscopy (Aichaoui *et al.*, 1991; Yous *et al.*, 1994). However, there are few examples of alternative synthetic routes to 5-acylbenzoxazol-2(3*H*)-ones; only a few sets of ^{1}H NMR data are available for these compounds

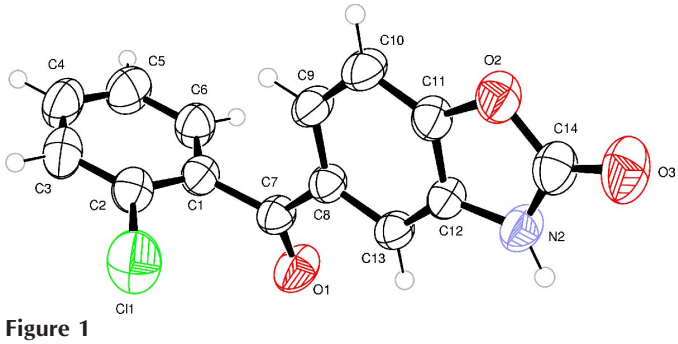


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level.

(Aichaoui *et al.*, 1991; Lesieur *et al.*, 1990). It was our aim to synthesize 5-(2-chlorobenzoyl)-1,3-benzoxazol-2(3*H*)-one, (I), by an alternative route and the results are presented here.

The 1,3-benzoxazol-2(3*H*)-one ring system is essentially planar, as was found in a related structure (Köysal *et al.*, 2004). The maximum deviation from the plane of the nine-membered ring system is 0.014 (3) Å for atom C13. The 1,3-benzoxazol-2(3*H*)-one and substituent benzene groups are almost perpendicular to each other, with a dihedral angle of 86.84 (4)°.

In the crystal structure, there are intermolecular C—H···O and N—H···O hydrogen bonds (Table 2), which link the molecules into discrete pairs parallel to the *ac* plane. There are also π–π stacking interactions between parallel 1,3-benzoxazol-2(3*H*)-one ring systems. The closest perpendicular separation is 3.412 Å between the ring system at (x, y, z) and that at ($-x$, $2-y$, $-z$). A short Cl···Cl contact of 3.41 (1) Å exists between the Cl atom at (x,y,z) and that at ($1-x$, $1-y$, $1-z$).

Experimental

As direct acylation of 1,3-benzoxazol-2(3*H*)-one gives the 6-acyl derivative, the synthetic procedure for 5-acyl derivatives was started from 2-aminophenol, followed by acylation and then cyclization of the acylated aminophenol (Aichaoui *et al.*, 1991). To protect the amino group of the starting material, 2-aminophenol, acetic anhydride (0.14 mol) was added dropwise to a 60 ml suspension of 2-aminophenol (0.09 mol) in distilled water, with stirring. Friedel–Crafts acylation of 2-acetylaminophenol (0.05 mol) with 2-chlorobenzoyl chloride (0.07 mol) yielded 2-(acetylamino)-4-(2-chlorobenzoyl)phenol (Lesieur *et al.*, 1990). After deprotection of the acetyl group in an alkaline medium by heating in concentrated NaOH, the resulting 2-amino-4-(2-chlorobenzoyl)phenol (0.1 mol) and urea (0.12 mol) were mixed and heated at 413 K for 1.5 h, and at 443 K for a further 2.5 h. The final product was then cooled, water (30 ml) was added and the mixture stirred for 1 h. The resulting precipitate was filtered, washed with water and crystallized from ethanol. Analysis calculated: C 61.44, H 2.95, N 5.12%; found: C 62.03, H 2.42, N 5.27%. The title compound was obtained as white prisms (yield 28%; m.p: 482–483 K). IR data (KBr, cm^{-1}): 3200 (N—H), 3123, 3003 (C—H), 1787 (C=O, lactam), 1668 (C=O, ketone), 1628, 1621, 1592 (C=C), 1274 (C—O—C).

Crystal data

$C_{14}H_8ClNO_3$
M_r = 273.66
Monoclinic, $P2_1/c$
a = 10.4550 (9) Å
b = 7.8954 (4) Å
c = 14.7960 (12) Å
β = 93.724 (7)°
V = 1218.78 (16) Å^3
Z = 4
D_x = 1.491 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 16 248 reflections
θ = 2.0–29.5°
μ = 0.32 mm^{-1}
T = 293 (2) K
Prism, white
0.80 × 0.54 × 0.23 mm

Data collection

Stoe *IPDS*-2 diffractometer
ω scans
Absorption correction: by integration (*X-RED*32; Stoe & Cie, 2002)
T_{min} = 0.846, T_{max} = 0.953
21273 measured reflections
3386 independent reflections
2456 reflections with $I > 2\sigma(I)$
R_{int} = 0.072
θ_{max} = 29.6°
$h = -14 \rightarrow 14$
$k = -10 \rightarrow 10$
$l = -20 \rightarrow 19$

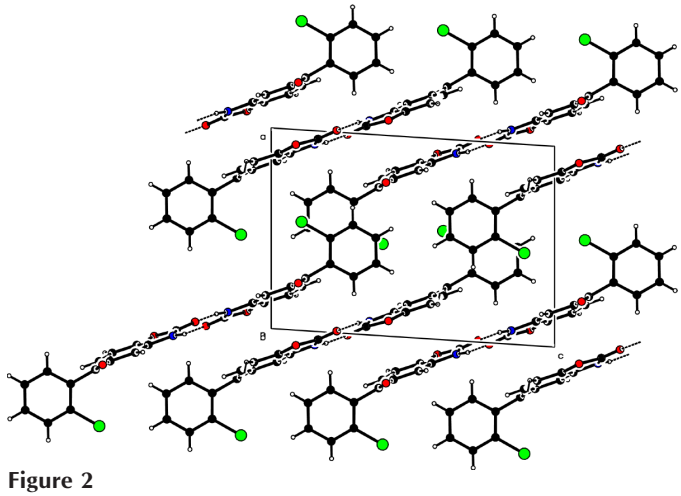


Figure 2
The crystal structure of (I), projected along the b axis. Dashed lines indicate hydrogen bonds.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.125
S = 1.06
3386 reflections
173 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.065P)^2 + 0.0757P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.016 (3)

Table 1
Selected bond lengths (Å).

Cl1—C2	1.7290 (16)	C7—O1	1.2119 (16)
N2—C14	1.3420 (19)	C11—O2	1.3797 (15)
N2—C12	1.3859 (16)	O3—C14	1.2061 (17)
C12—C11	1.3826 (17)	C14—O2	1.3699 (18)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O3^{i}	0.86	1.90	2.7498 (16)	170
C10—H10···O1^{ii}	0.93	2.44	3.2948 (17)	153

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

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 Å, N—H = 0.86 Å and U_{iso}(H) = 1.2U_{eq}(parent atom).

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED*32 (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*III (Burnett & Johnson, 1996); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PARST* (Nardelli, 1995).

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