3-(6-Benzoyl-5-chloro-2-oxo-2,3-dihydro-1,3-benzoxazol-3-yl)propanoic acid

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The title compound, $C_{17}H_{12}ClNO_5$, consists of a main benzothiazole group and its substituents which are one Cl atom, a benzoyl group and a propanoic acid group. There is a 0.779 (4)/0.221 (4) disorder in the terminal propanoic acid group. The crystal structure is stabilized by intermolecular O—H···O hydrogen-bond contacts.

Comment

The title compound, (I), shows analgesic activity comparable to aspirin, and results in lower anti-inflammatory activity than that of indometacin. This compound caused gastric lesions and bleeding in the stomachs of tested animals (Fereira *et al.*, 1995; Ünlü *et al.*, 2003). We report here the structure of (I), shown in Fig. 1.

(I)
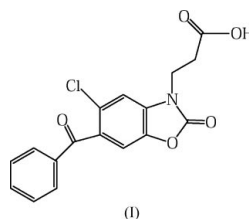
The structure of (I) exhibits disorder. Atoms C2*A*, C3*A*, C4*A*, O3*A* and O4*A* in the propanoic acid group and atom N1*A* in the benzothiazole group have a site-occupancy of 0.779 (4) *versus* 0.221 (4) for the minor component *B* of the same groups. Within experimental uncertainty, the bond lengths and angles in the disordered compound are comparable to those in related structures (Allen *et al.*, 1987; Aydın *et al.*, 2002).

In the disordered propanoic acid group, the average C—C bond length and the C—C—C bond angle are 1.506 (18) Å and 113.3 (9)°, respectively. These values are in agreement with those in 3-(6-benzoyl-2-oxo-2,3-dihydro-2-benzothiazol-
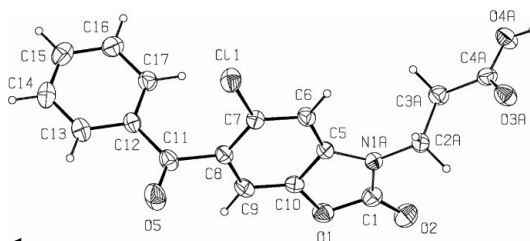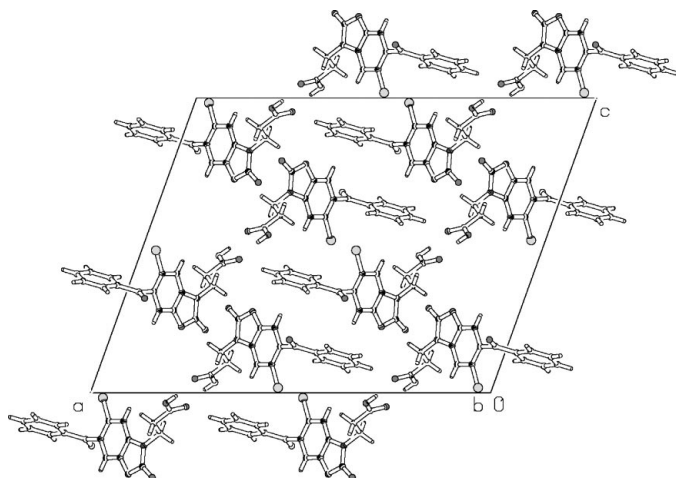

Figure 1
A view of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme. The disordered atoms of the minor component have been omitted for clarity.

Figure 2
A view of the packing of (I) along the *b* axis. The minor components of the disordered atoms have been omitted for clarity.

3-yl)propanoic acid, [1.501 (1) and 110.9 (3)°; Aydın *et al.*, 2003].

The maximum deviations from the mean plane through the bicylic benzothiazole system (O1/C1/N1*A*/N1*B*/C5–C10) are −0.105 (3), 0.38 (1), 0.015 (2) and −0.016 (2) Å for atoms N1*A*, N1*B*, C6 and C8, respectively. The dihedral angle between the least-squares planes of the benzothiazole group and the phenyl ring (C12–C17) is 72.81 (5)°, and is comparable with the angle of 74.7 (1)° in 5-chloro-6-(2-fluorobenzoyl)-1,3-benzoxazol-2(3*H*)-one (Aydın *et al.*, 2004).

The crystal structure of (I) is stabilized by intermolecular O—H···O hydrogen-bond contacts (Table 2).

Experimental

3-(6-Benzoyl-5-chloro-2-oxo-3*H*-benzoxazol-3-yl)propionitrile (0.01 mol) was added to an *N,N*-dimethylformamide–water–sulfuric acid (1:1:2) mixture (50 ml). After stirring at room temperature for 2 h, the mixture was refluxed for 4 h. The mixture, cooled to room temperature, was poured into ice–water. The resulting precipitate was filtered off by suction filtration, washed with water, dried and crystallized from ethanol–water (yield 78%, m.p. 468–469 K). IR ν_{max} cm^{-1} (KBr): 3060, 1770, 1665, 1600, 1480; ^{1}H NMR (DMSO-d_6): δ 12.40 (1H, *s*, COOH), 7.87–7.81 (4H, *m*, 2-oxobenzoxazol-H^{4,7}, benzoyl-H^{2,6}), 7.69 (3H, *m*, benzoyl-H^{3,4,5}), 4.21 (2H, *t*, N-CH$_2$), 2.89 (2H, *t*, CH$_2$CO) (Ünlü *et al.*, 2003).

Crystal data

$C_{17}H_{12}ClNO_5$	$D_x = 1.470$ Mg m^{-3}
$M_r = 345.73$	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 6761
$a = 26.015$ (10) Å	reflections
$b = 6.252$ (5) Å	$\theta = 1.7–28.1°$
$c = 20.412$ (4) Å	$\mu = 0.27$ mm^{-1}
$\beta = 109.749$ (16)°	$T = 293$ K
$V = 3125$ (3) Å^3	Plate, colorless
$Z = 8$	$0.60 \times 0.44 \times 0.13$ mm

Data collection

Stoe IPDS-2 diffractometer	1700 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.033$
Absorption correction: by	$\theta_{max} = 28.1°$
integration (Stoe & Cie, 2002)	$h = -32 \rightarrow 34$
$T_{min} = 0.854$, $T_{max} = 0.965$	$k = -8 \rightarrow 8$
6761 measured reflections	$l = -16 \rightarrow 26$
3791 independent reflections	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0486P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.033$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.085$	$(\Delta/\sigma)_{max} < 0.001$
$S = 0.72$	$\Delta\rho_{max} = 0.16$ e Å^{-3}
3791 reflections	$\Delta\rho_{min} = -0.17$ e Å^{-3}
243 parameters	Extinction correction: *SHELXL*97
H atoms treated by a mixture of	Extinction coefficient: 0.0037 (3)
independent and constrained	
refinement	

Table 1
Selected geometric parameters (Å, °).

Cl1—C7	1.731 (2)	O5—C11	1.206 (3)
O1—C1	1.374 (3)	N1*A*—C2*A*	1.453 (4)
O1—C10	1.379 (2)	N1*A*—C1	1.363 (3)
O2—C1	1.195 (3)	N1*A*—C5	1.381 (3)
O3*A*—C4*A*	1.220 (5)	N1*B*—C1	1.377 (10)
O3*B*—C4*B*	1.24 (2)	N1*B*—C5	1.452 (10)
O4*A*—C4*A*	1.294 (5)	N1*B*—C2*B*	1.450 (14)
O4*B*—C4*B*	1.22 (2)		
C1—O1—C10	107.35 (13)	O3*A*—C4*A*—O4*A*	123.2 (3)
C1—N1*A*—C5	109.2 (2)	O3*A*—C4*A*—C3*A*	121.9 (3)
C1—N1*A*—C2*A*	123.3 (2)	O4*B*—C4*B*—C3*B*	114.9 (11)
C2*A*—N1*A*—C5	127.2 (2)	O3*B*—C4*B*—O4*B*	128.2 (15)
C1—N1*B*—C2*B*	125.1 (8)	O3*B*—C4*B*—C3*B*	114.3 (15)
C1—N1*B*—C5	104.5 (5)	N1*A*—C5—C10	106.20 (16)
C2*B*—N1*B*—C5	128.2 (8)	N1*B*—C5—C10	104.5 (4)
O1—C1—N1*A*	107.70 (18)	N1*A*—C5—C6	132.26 (18)
O2—C1—N1*A*	129.6 (2)	N1*B*—C5—C6	131.7 (4)
O2—C1—N1*B*	127.8 (4)	Cl1—C7—C8	119.43 (13)
O1—C1—O2	122.47 (17)	Cl1—C7—C6	117.58 (12)
O1—C1—N1*B*	107.4 (5)	O1—C10—C9	128.31 (15)
N1*A*—C2*A*—C3*A*	112.2 (2)	O1—C10—C5	109.05 (14)
N1*B*—C2*B*—C3*B*	112.7 (8)	O5—C11—C12	121.53 (18)
O4*A*—C4*A*—C3*A*	114.9 (3)	O5—C11—C8	118.83 (17)

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

D—H···A	D—H	H···A	D···A	D—H···A
O4*A*—H4*A*···O3*A*^{i}	0.82	1.81	2.622 (4)	173

Symmetry code: (i) $\frac{1}{2} - x, \frac{7}{2} - y, -z$.

The minor component *B* of the disordered group was restrained to have geometry similar to that of the major component. All H atoms were placed in idealized positions (C—H = 0.97 Å for methylene groups, C—H = 0.93 Å for the phenyl group and O—H = 0.82 Å for the hydroxyl group). A riding model was used in the refinement procedure. The U_{iso} values were constrained to be 1.2 times U_{eq}(C) of the carrier atom for the phenyl and methylene groups, and 1.5 times U_{eq}(O) of the carrier atom for the hydroxyl group.

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*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 1990) and *WinGX* publication routines (Farrugia, 1999).

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