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2-(1*H*-1,2,3-Benzotriazol-1-ylmethyl)-1-(4-chlorobenzoyl)ethyl nicotinate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.043
wR factor = 0.089
Data-to-parameter ratio = 14.4

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

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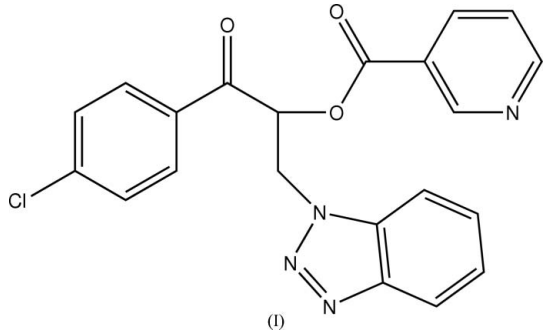
In the title compound, $C_{21}H_{15}ClN_4O_3$, molecules are linked into chains along the *b* axis by C—H···O hydrogen bonds. The chains are further connected by intermolecular C—H···O hydrogen bonds, forming two-dimensional layers parallel to the *ab* plane.

Comment

We have recently reported the structure of 2-(1*H*-1,2,3-benzotriazol-1-ylmethyl)-1-benzoylethyl 4-chlorobenzoate, (II) (Wan *et al.*, 2006). In our ongoing studies of benzotriazole compounds, the title compound, (I), was obtained.



(I)

The bond lengths and angles in (I) show normal values (Allen *et al.*, 1987) and are comparable to those found in (II). The benzotriazole system is essentially planar, with a dihedral angle of 1.0 (2)° between the triazole (*A*, atoms N1–N3/C10/C15) and benzene rings (*B*, atoms C10–C15). The dihedral angles between the mean plane of the benzotriazole system and rings *C* (atoms C1–C6) and *D* (atoms N4/C17–C21) are 6.9 (1) and 71.1 (1)°, respectively. The dihedral angle between rings *C* and *D* is 74.4 (1)°.

There exists an intramolecular C21—H21*A*···O2 hydrogen bond, forming a five-membered ring. In the crystal structure, molecules of (I) are linked into chains along the *b* axis by C—H···O hydrogen bonds. Further C—H···O interactions connect the chains into two-dimensional layers parallel to the *ab* plane (Fig. 2 and Table 2 for geometry values and symmetry code).

Experimental

Bromine (3.2 g, 0.02 mol) was added dropwise to a solution of 3-(benzotriazol-1-yl)-1-phenylpropan-1-one (5.0 g, 0.02 mol) and sodium acetate (1.6 g, 0.02 mol) in acetic acid (50 ml). The reaction was allowed to proceed for 13 h. Water (50 ml) and chloroform (20 ml) were then added. The organic layer was washed successively with a saturated sodium bicarbonate solution and brine, dried over

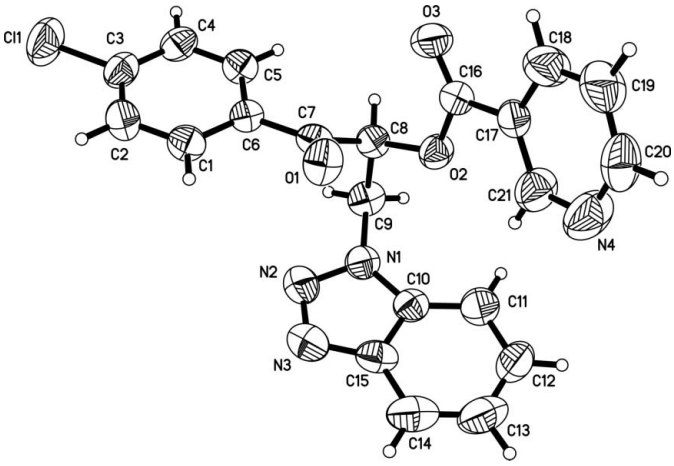


Figure 1
The structure of the compound (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

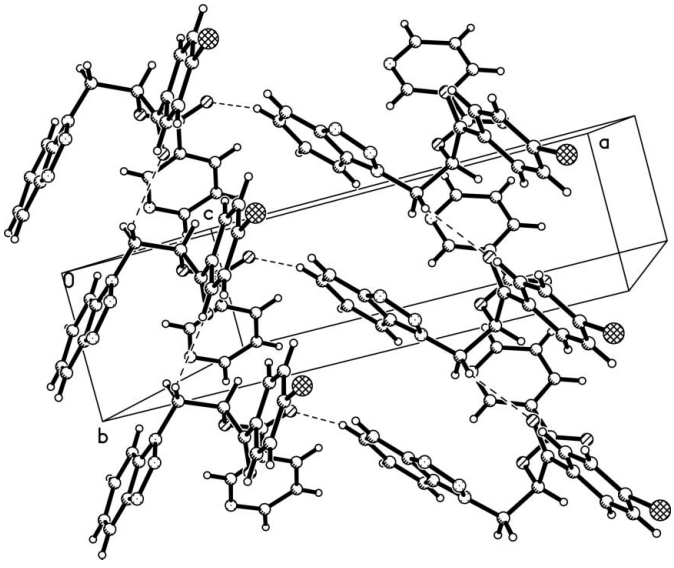


Figure 2
The two-dimensional layers of (I). Hydrogen bonds are indicated by dashed lines.

anhydrous magnesium sulfate and the chloroform solution filtered. After cooling with ice–water, an acetone solution (10 ml) of nicotinic acid (3.1 g, 0.02 mol) and triethylamine (2.8 ml) were added. The mixture was stirred at room temperature for about 2 h, then filtered, concentrated and purified by flash column chromatography (silica gel, petroleum ether–ethyl acetate, 3:1 *v*/*v*) to afford the title compound. Single crystals were obtained by slow evaporation of a petroleum ether–ethyl acetate (1:1 *v*/*v*) solution at room temperature over a period of one week.

Crystal data

$C_{21}H_{15}ClN_4O_3$
M_r = 406.82
Orthorhombic, $Pca2_1$
a = 18.820 (2) Å
b = 5.3367 (6) Å
c = 19.027 (2) Å
V = 1911.0 (4) Å^3
Z = 4
D_x = 1.414 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 293 (2) K
Column, colourless
0.40 × 0.19 × 0.10 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.913, T_{max} = 0.977
10277 measured reflections
3763 independent reflections
2880 reflections with $I > 2\sigma(I)$
R_{int} = 0.036
θ_{max} = 26.1°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.089
S = 1.01
3763 reflections
262 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.036P)^2 + 0.0701P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}
Absolute structure: Flack (1983), 1811 Friedel pairs
Flack parameter: 0.01 (7)

Table 1
Selected bond lengths (Å).

Cl1—C3	1.732 (3)	O3—C16	1.194 (3)
O1—C7	1.212 (3)	C7—C8	1.520 (4)
O2—C16	1.356 (3)	C8—C9	1.525 (4)
O2—C8	1.437 (3)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9B···O1^{i}	0.97	2.57	3.058 (3)	111
C13—H13A···O3^{ii}	0.93	2.49	3.351 (4)	155
C21—H21A···O2	0.93	2.39	2.739 (4)	102

Symmetry codes: (i) $x, y-1, z$; (ii) $x-\frac{1}{2}, -y+3, z$.

All H atoms were located in difference Fourier maps and constrained to ride on their parent atoms, with C—H = 0.93–0.98 Å and U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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