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Methyl 1-*n*-butyl-2-(3,4-dichlorophenyl)-1*H*-benzimidazole-5-carboxylate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.043
wR factor = 0.117
Data-to-parameter ratio = 18.2

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

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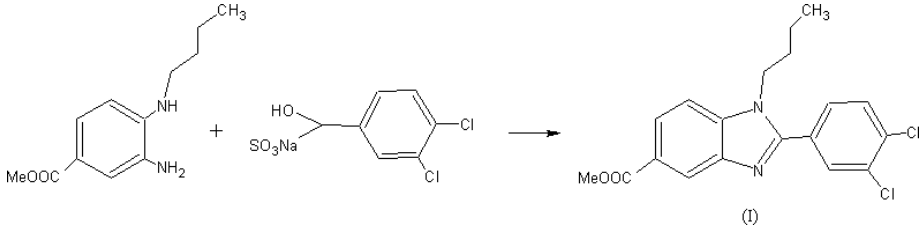
A new benzimidazole compound, methyl 1-*n*-butyl-2-(3,4-dichlorophenyl)-1*H*-benzimidazole-5-carboxylate, $C_{19}H_{18}Cl_2N_2O_2$, has been synthesized by the condensation of methyl 3-amino-4-(*n*-butylamino)benzoate with an $Na_2S_2O_5$ adduct of 3,4-dichlorobenzaldehyde. The molecule is twisted with a C—C—C—N torsion angle of −39.7 (3)° between the phenyl and benzimidazole groups. In the crystal structure, symmetry-related molecules are linked by C—H···O interactions, forming a chain.

Comment

The benzimidazole ring system is of interest because of its diverse biological activities, including antifungal (Göker *et al.*, 2002), antibacterial (Weidner-Wells *et al.*, 2001), antiparasitic (Navarrete-Vazquez *et al.*, 2001), anticancer (Badawey & Kappe, 1999), anti-allergic (Nakano *et al.*, 2000), anti-ulcer (Göker & Düver, 1990) and antihypertensive (Matsumori, 2003). New drugs carrying a benzimidazole moiety, such as omeprazole (Göker & DüVer, 1990), candesartane (Matsumori, 2003) and mizolastine (Dubertret *et al.*, 1999), have been used clinically, and considerable effort has been invested recently to discover new potent agents (Mekapati & Hansch, 2001). From our laboratory, the synthesis and crystal structure analyses of several benzimidazoles have already been reported (Göker *et al.*, 1995, 1999; Özbey *et al.*, 1998; Kendi *et al.*, 1999). The versatility of this ring system has prompted us to synthesize new analogs, including the title compound, (I).



(I)

The molecular structure of (I) is shown in Fig. 1 and selected bond distances and angles are given in Table 1. The dihedral angle between the plane of the ring defined by atoms N1/C7/N2/C8/C9 and the C1–C6 phenyl ring is 36.68 (7)°, with a C3—C4—C7—N1 torsion angle of −39.7 (3)°. The molecule shows small deviations from planarity, the largest being 0.014 (2) Å for atom C8 in the benzmidazole ring system and 0.015 (4) Å for atom C1 in the C1–C6 phenyl ring. The C18═O1 bond length is 1.198 (2) Å and the C19—O2—C18—C11 torsion angle is 178.78 (17)°. In the molecule, the C—Cl bond lengths are very similar, Cl—Cl1 being 1.725 (2) Å and C6—Cl2 being 1.729 (2) Å.

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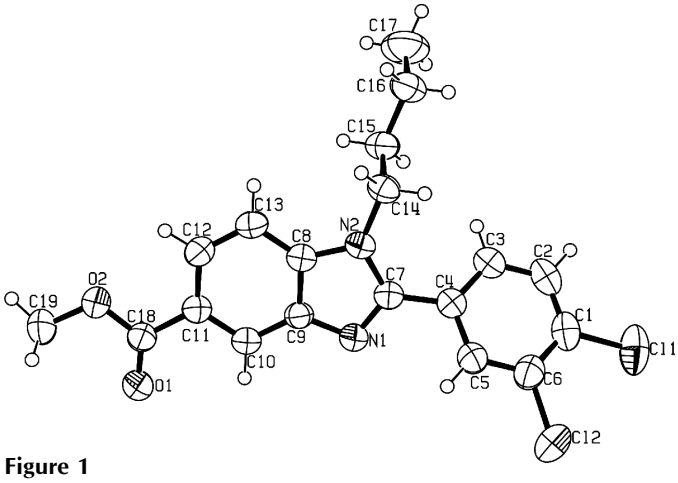

Figure 1
An *ORTEP*-3 (Farrugia, 1997) view of (I), with the atomic numbering scheme and 50% probability displacement ellipsoids.

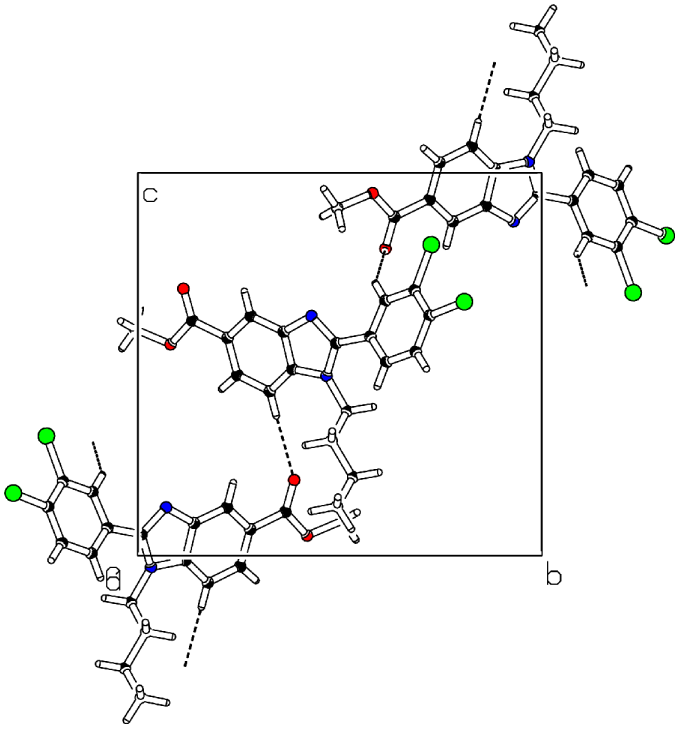

Figure 2
An *ORTEP*-3 (Farrugia, 1997) packing diagram of (I), viewed along the *a* axis. The C—H···O hydrogen bonds are shown as dashed lines.

In the crystal structure, symmetry-related molecules are connected by C—H···O hydrogen bonds, forming a polymer chain (see Table 2 and Fig. 2).

Experimental

To a suspension of methyl 3-amino-4-(*n*-butylamino)benzoate (0.22 g, 1 mmol) in dimethylformamide (1 ml), a sodium metabisulfite adduct of 3,4-dichlorobenzaldehyde (0.347 g, 1.25 mmol) was added and heated at 403 K for 4 h. The reaction mixture was cooled then poured into water. The solid product obtained was collected by filtration and washed with water. It was then chromatographed with EtOAc–*n*-hexane (1:3) (yield 0.2 g, 53%). Pale-green crystals of (I) were obtained (m.p. 353 K). IR (CO): 1706 cm^{-1}; ^{1}H NMR (DMSO-d_6): δ 0.67 (*t*, 3H, CH_2CH_3), 1.03–1.09 (*m*, 2H, CH_2CH_3), 1.54–1.58 (*m*, 2H, CH_2CH_2), 3.79 (*s*, 3H, OCH_3), 4.26 (*t*, 2H, N-CH_2, J = 7.2 Hz), 7.70–7.8 (*m*, 3H, H-5,6,7), 7.84–7.87 (*dd*, 1H, H-6, J_o = 8.6, J_m = 1.4 Hz), 7.99 (*d*, 1H, H-2, J_m = 1.8 Hz), 8.20 (*d*, 1H, H-4, J_m = 1.2 Hz); MS (ES+): 377 (M + 1) (100%).

Crystal data

$C_{19}H_{18}Cl_2N_2O_2$	D_x = 1.364 Mg m^{-3}
M_r = 377.25	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 15013 reflections
a = 9.3359 (6) Å	θ = 1.5–29.0°
b = 14.4051 (8) Å	μ = 0.37 mm^{-1}
c = 16.3707 (11) Å	T = 293 (2) K
β = 123.459 (4)°	Prismatic, pale green
V = 1836.8 (2) $Å^3$	0.50 × 0.30 × 0.10 mm
Z = 4	

Data collection

Stoe IPDS-2 two-circle goniometer diffractometer	2727 reflections with $I > 2\sigma(I)$
	R_{int} = 0.088
ω scans	θ_{max} = 27.5°
Absorption correction: none	$h = -12 \rightarrow 12$
30007 measured reflections	$k = -18 \rightarrow 18$
4133 independent reflections	$l = -21 \rightarrow 21$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0573P)^2 + 0.0182P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.043	
$wR(F^2)$ = 0.117	
S = 1.04	$(\Delta/\sigma)_{max}$ = 0.001
4133 reflections	$\Delta\rho_{max}$ = 0.29 e $Å^{-3}$
227 parameters	$\Delta\rho_{min}$ = −0.29 e $Å^{-3}$
H-atom parameters constrained	Extinction correction: *SHELXL*97
	Extinction coefficient: 0.0164 (18)

Table 1
Selected geometric parameters (Å, °).

Cl1—C1	1.725 (2)	O1—C18	1.198 (2)
Cl2—C6	1.729 (2)		
C3—C4—C7—N1	−39.7 (3)	C19—O2—C18—C11	178.78 (17)

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···O1^{i}	0.93	2.53	3.336 (3)	146
C13—H13···O1^{ii}	0.93	2.41	3.340 (2)	176

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

H atoms were included in calculated positions and treated as riding atoms; C—H = 0.93–0.97 Å and U_{iso}(H) = 1.2 or 1.5U_{eq}(C).

Data collection: *X-AREA* (Stoe & Cie, 1996); cell refinement: *X-AREA*; data reduction: *X-RED*32 (Stoe & Cie, 1996); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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