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4′-(2-Butyl-4-chloro-5-formylimidazol-1-ylmethyl)-biphenyl-2-carbonitrile

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Key indicators

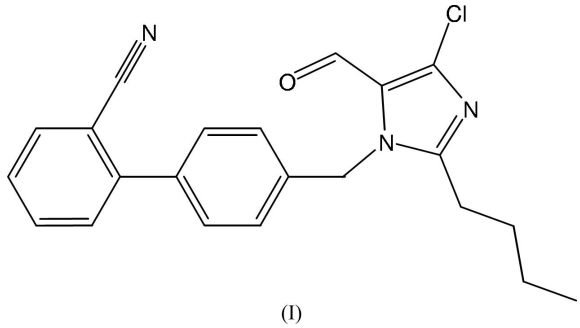
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
R factor = 0.042
wR factor = 0.112
Data-to-parameter ratio = 18.2

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

The title compound, $C_{22}H_{20}ClN_3O$, (I), is used as an intermediate for the synthesis of the antihypertensive drug losartan. Bond lengths and angles are unexceptional. The crystal packing is stabilized by one C—H···O and one C—H···N contact. It is noteworthy that (I) is isomorphous with a closely related compound which differs in having a but-2-enyl chain instead of a butyl chain on the imidazole ring.

Comment

Imidazole derivatives have applications in pharmaceuticals, agrochemicals, dyestuffs and high-temperature polymer products (Nagaraj *et al.*, 2005).



(I)

Imidazole is a fundamental building block of many proteins and other biological systems. It also acts as a ligand that will bind readily to a metal ion in aqueous systems. A survey of the literature reveals that the crystal structure of a closely related compound, namely 4′-{[2-(but-2-enyl)-4-chloro-5-formyl-imidazol-1-yl]methyl}biphenyl-2-carbonitrile, (II), has been reported recently (Malathy Sony *et al.*, 2005). The only difference between the two structures is that the imidazole

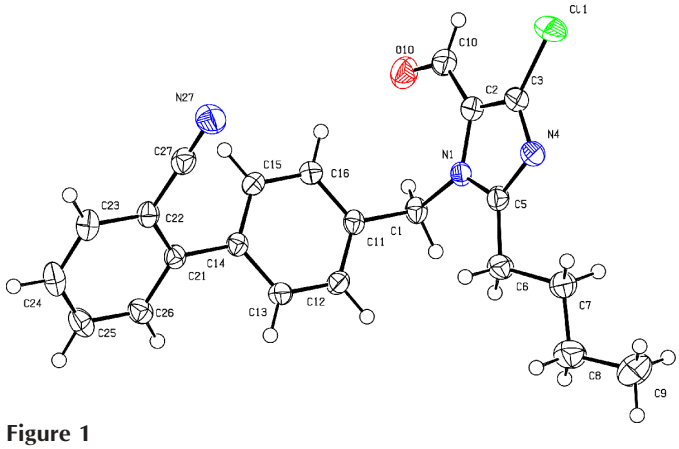


Figure 1
A perspective view of (I) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

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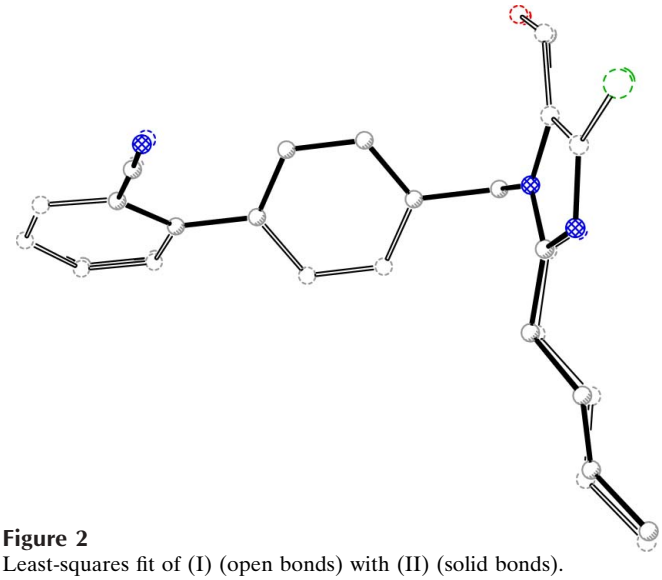

Figure 2
Least-squares fit of (I) (open bonds) with (II) (solid bonds).

ring in the title compound, (I), carries a butyl chain, whereas (II) carries a but-2-enyl chain.

The title compound is used as a key intermediate for the synthesis of the antihypertensive drug losartan (Griffiths *et al.*, 1999). In a continuation of our work on derivatives of imidazole (Yathirajan *et al.*, 2005), and in order to establish the conformation of the title compound, the crystal structure determination was carried out.

A perspective view of (I) is shown in Fig. 1. Bond lengths and angles can be regarded as normal (Cambridge Structural Database, Version 1.7; *MOGUL*, Version 1.0.1; Allen, 2002). The dihedral angle between the two benzene rings is 41.36 (5)° [41.64 (12)° in (II)]. The imidazole ring and the central benzene ring (C11–C16) enclose an angle of 72.03 (5)° [73.28 (14)° in (II)]. The carbonyl group is almost coplanar with the imidazole ring [N1—C2—C10—O10 = 2.8 (2)°]. The butyl chain exhibits an all-*trans* conformation. The plane containing the four butyl C atoms forms an angle of 24.28 (10)° with the imidazole ring. The repulsion between the Cl atom and the formyl H atom leads to significantly different exocyclic angles at C3. On the other hand, the exocyclic angles at N1, C2 and C5 are rather similar (Table 1). The equivalent angles in (II) show the same phenomenon. There is one C—H···O and one C—H···N contact (Table 2).

A least-squares fit (r.m.s. deviation 0.052 Å) of (I) with (II), fitting all non-H atoms excluding the butyl chain (Fig. 2), shows that there are only minor differences between the two molecular conformations. The structures of (I) and (II) are isomorphous. The cell dimensions of (I) are slightly smaller than those of (II) [a = 9.080 (6) Å, b = 22.782 (15) Å, c = 10.055 (7) Å, β = 109.476 (19)° and V = 1961 (2) Å^3], but this may be due to the fact that the data collection for (II) was performed at room temperature.

Experimental

An equimolar mixture of 2-butyl-5-chloro-3*H*-imidazole-4-carboxaldehyde (1.86 g, 0.01 mol), 4′-(bromomethyl)biphenyl-2-carbonitrile (2.72 g, 0.01 mol) and anhydrous K_2CO_3 (1.66 g, 0.01 mol) was stirred in a dimethylformamide (10 ml) medium for 10 h. The mixture was quenched with water and the product formed was extracted with dichloromethane (25 ml). The solvent was then removed and the product formed was recrystallized (m.p. 372 K) from methanol (Smith *et al.*, 1994).

Crystal data

$C_{22}H_{20}ClN_3O$
M_r = 377.86
Monoclinic, $P2_1/c$
a = 8.9729 (4) Å
b = 22.6232 (9) Å
c = 9.9288 (5) Å
β = 106.427 (4)°
V = 1933.23 (15) Å^3
Z = 4
D_x = 1.298 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 35 462 reflections
θ = 2.4–27.9°
μ = 0.21 mm^{-1}
T = 173 (2) K
Block, colourless
0.32 × 0.25 × 0.22 mm

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.923, T_{max} = 0.955
31 411 measured reflections
4448 independent reflections
4037 reflections with $I > 2\sigma(I)$
R_{int} = 0.044
θ_{max} = 27.7°
$h = -11 \rightarrow 11$
$k = -29 \rightarrow 29$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.112
S = 1.05
4448 reflections
244 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.053P)^2 + 0.8177P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C3	1.7179 (14)	N4—C5	1.3401 (17)
N1—C5	1.3544 (17)	C10—O10	1.218 (2)
N1—C2	1.3969 (17)	C14—C21	1.4875 (17)
C2—C3	1.384 (2)	C22—C27	1.4409 (19)
C3—N4	1.3495 (19)	C27—N27	1.146 (2)
C5—N1—C2	107.39 (11)	N4—C3—Cl1	120.31 (11)
C5—N1—C1	125.69 (11)	C2—C3—Cl1	126.87 (11)
C2—N1—C1	126.91 (11)	C5—N4—C3	104.30 (12)
C3—C2—N1	103.43 (12)	N4—C5—N1	112.07 (12)
C3—C2—C10	128.72 (13)	N4—C5—C6	123.76 (12)
N1—C2—C10	127.83 (13)	N1—C5—C6	124.17 (12)
N4—C3—C2	112.82 (12)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1B···O10	0.99	2.41	3.0642 (18)	123
C6—H6A···N27^{i}	0.99	2.56	3.5312 (19)	166

Symmetry codes: (i) $x+1, y, z$.

All H atoms were located in a difference map but were subsequently positioned geometrically and refined with fixed individual displacement parameters (set at 1.2U_{eq} of the parent atom, or 1.5U_{eq}

for methyl groups) using a riding model (C—H = 0.95, 0.98 and 0.99 Å for aromatic, methyl and methylene H atoms, respectively).

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON* and *SHELXL97*.

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