Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4′-[2-Butyl-4-chloro-5-(hydroxyiminomethyl)-1*H*-imidazol-3-ylmethyl]biphenyl-2-carbonitrile

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Received 4 February 2005
Accepted 14 February 2005
Online 19 February 2005

Key indicators

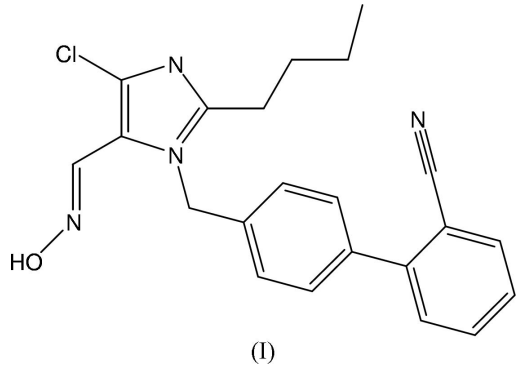
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.043
wR factor = 0.105
Data-to-parameter ratio = 13.8

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

The title compound, $C_{22}H_{21}ClN_4O$, is used as an intermediate for the synthesis of biologically active compounds. Geometric parameters are in the usual ranges. The packing is stabilized by O—H···N hydrogen bonding.

Comment

Imidazole derivatives have applications as pharmaceuticals, agrochemicals, dyes and high-temperature polymer products (Rasmussen, 1999; Ambalavanan *et al.*, 2003). The title compound, (I), is used as an intermediate for the synthesis of biologically active novel heterocycles (Rai *et al.*, 1992). In order to determine the conformation of (I), the crystal structure determination was carried out.



(I)

A perspective view is shown in Fig. 1. Bond lengths and angles can be regarded as normal (Cambridge Structural Database, Version 1.7; *MOGUL* Version 1.0; Allen, 2002).

The hydroxyiminomethyl residue is almost coplanar with the planar (r.m.s. deviation = 0.002 Å) imidazole ring (Table 1). The dihedral angle between the two benzene rings is

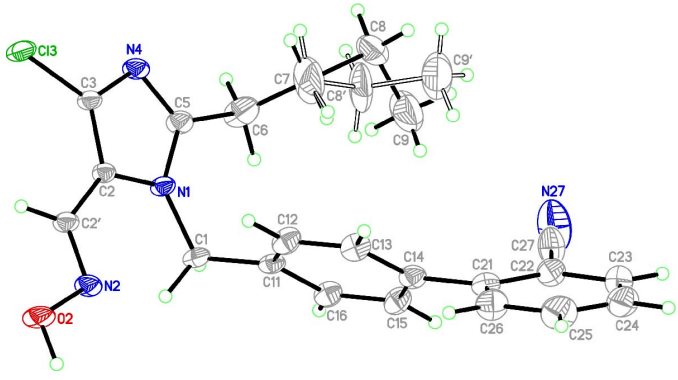


Figure 1
Perspective view of the title compound, with the atom numbering; displacement ellipsoids are drawn at the 50% probability level. The minor occupancy disordered atoms are shown with open bonds.

38.93 (7)°. It is interesting to note that the butyl group and the biphenyl residue are located on the same side of the imidazole ring. There is an intermolecular hydrogen bond between the hydroxyl group and the unsubstituted imidazole N atom (Table 2).

Experimental

A mixture of 4′-(2-butyl-4-chloro-5-formyl-1*H*-imidazol-3-ylmethyl)biphenyl-2-carbonitrile (3.77 g, 10 mmol), hydroxylamine hydrochloride (0.69 g, 10 mmol) and sodium acetate (0.82 g, 10 mmol) in ethanol (25 ml) was warmed at 323 K for 30 min and then cooled; the product was recrystallized from a mixture of dichloromethane and methanol (yield, 90%; m.p. 480 K). IR (KBr, ν cm^{-1}): 3463 (br), 3257 (*w*), 2919 (*s*), 2827 (*w*), 2217 (*s*), 1668 (*m*), 1508 (*s*), 1379 (*m*), 1213 (*m*). ^{1}H NMR ($CDCl_3$, p.p.m.): 0.9 (*t*, 3H, CH_3–), 1.4 (*m*, 2H, CH_2–), 2.1 (*m*, 2H, CH_2–), 2.7 (*t*, 2H, CH_2–), 4.65 (*s*, 2H, C_{H2}–), 5.95 (*s*, 1H, CH–), 6.75 (*s*, 1H, OH–), 7.1–7.4 (*m*, 5H, ArH–), 7.6–7.8 (*m*, 3H, ArH–); ^{13}C NMR ($CDCl_3$, p.p.m.): 13.2 (*q*, CH_3–), 21.3 (*t*, CH_2–), 24.0 (*t*, CH_2–), 33.2 (*t*, CH_2–), 41.2 (*t*, CH_2–), 115.2 (*s*, ArC—CN–), 119.0 (*s*, CN–), 123.3 (*s*, C—Cl–), 126.4 (*s*, ImC–), 127.9 (*d*, ArC–), 128.9 (*d*, ArC–), 130.2 (*d*, ArC–), 132.4 (*d*, ArC–), 132.9 (*d*, ArC–), 133.9 (*d*, ArC–), 134.7 (*s*, ArC–), 138.2 (*s*, ArC–), 141.0 (*s*, ArC–), 150.8 (*s*, ImC–), 152.8 (*d*, CH–). Analysis calculated for $C_{22}H_{21}ClN_4O$. C 67.26, H 5.39, N 14.26%; found: C 67.29, H 5.41, N 14.22%.

Crystal data

$C_{22}H_{21}ClN_4O$
M_r = 392.88
Triclinic, $P\overline{1}$
a = 7.6974 (6) Å
b = 7.8682 (6) Å
c = 19.1907 (16) Å
α = 79.823 (6)°
β = 84.248 (7)°
γ = 64.220 (6)°
V = 1029.77 (14) Å^3
Z = 2
D_x = 1.267 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 29 921 reflections
θ = 3.6–25.7°
μ = 0.21 mm^{-1}
T = 173 (2) K
Block, colourless
0.36 × 0.33 × 0.32 mm

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.927, T_{max} = 0.941
16 926 measured reflections
3815 independent reflections
3517 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 25.6°
$h = -9 \rightarrow 9$
$k = -9 \rightarrow 9$
$l = -23 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.105
S = 1.12
3815 reflections
276 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0421P)^2 + 0.7181P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C5	1.367 (2)	C3—Cl3	1.7228 (17)
N1—C2	1.395 (2)	N4—C5	1.332 (2)
C2—C3	1.374 (2)	C2′—N2	1.280 (2)
C2—C2′	1.451 (2)	N2—O2	1.4121 (18)
C3—N4	1.365 (2)		
N2—C2′—C2	125.07 (16)	C2′—N2—O2	109.53 (14)
C3—C2—C2′—N2	−166.97 (18)	C2—C2′—N2—O2	178.05 (16)

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···N4^{i}	0.92 (3)	1.88 (3)	2.7776 (19)	163 (3)

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

H atoms, except those attached to the disordered atoms, were located in a difference map. All CH H atoms were geometrically positioned and refined with fixed individual displacement parameters [set at 1.2 times U_{eq} of the parent atom (1.5 for methyl groups)] using a riding model, with C—H distances ranging from 0.95 to 0.99 Å. The hydroxyl H atom was refined freely. The two terminal atoms of the butyl chain are disordered over two sites with occupancy factors of 0.567 (11) and 0.433 (11). The bond between C7 and C8′ was restrained to 1.540 (8) Å.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

One of the authors (HSY) sincerely thanks K. M. Lokanatha Rai for spectral studies and RSN is grateful to the University of Mysore for permission to carry out the research work.

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