

4'-{[2-(But-2-enyl)-4-chloro-5-formyl-1*H*-imidazol-1-yl]methyl}biphenyl-2-carbonitrile

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Key indicators

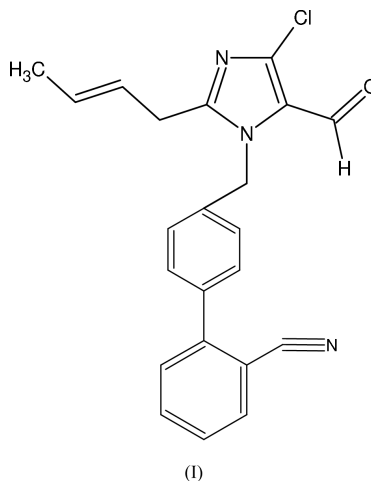
Single-crystal X-ray study
T = 293 K
Mean $\sigma(\text{C}—\text{C})$ = 0.004 Å
R factor = 0.064
wR factor = 0.184
Data-to-parameter ratio = 17.0

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

In the title structure, $\text{C}_{22}\text{H}_{18}\text{ClN}_3\text{O}$, the dihedral angle between the benzene rings of the biphenyl system is $41.6(1)^\circ$; they are approximately perpendicular to the planar imidazole ring. The crystal structure is stabilized by $\text{C}—\text{H}\cdots\text{Cl}$, $\text{C}—\text{H}\cdots\text{O}$ and $\text{C}—\text{H}\cdots\text{N}$ hydrogen bonds and $\text{C}—\text{H}\cdots\pi$ interactions.

Comment

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. Imidazole-4-acetic acid is a catabolite of histamine and is present in the brain (Prell *et al.*, 1996), and an imidazole succinic acid complex is an active non-steroidal aromatase inhibitor (Schiavo *et al.*, 1988). The title compound, (I), serves as a key intermediate for the preparation of the antihypertensive drug losartan potassium (Griffiths *et al.*, 1999).



As expected, the cyanide group is linear, with angle $\text{C14}—\text{C19}—\text{N20}$ equal to $176.9(3)^\circ$. Angles $\text{C9}—\text{C10}—\text{C11}$ of $117.6(2)^\circ$ and $\text{C14}—\text{C13}—\text{C18}$ of $117.1(2)^\circ$ are contracted, due to the steric hindrance of the biphenyl system. The but-2-enyl chain is in an extended conformation, as noted from the torsion angles $\text{N1}—\text{C2}—\text{C21}—\text{C22}$ [$156.2(3)^\circ$], $\text{C2}—\text{C21}—\text{C22}—\text{C23}$ [$-165.5(5)^\circ$] and $\text{C21}—\text{C22}—\text{C23}—\text{C24}$ [$178.0(5)^\circ$]. The dihedral angle between the benzene rings of the biphenyl system is $41.6(1)^\circ$; rings $\text{C7}—\text{C12}$ and $\text{C13}—\text{C18}$ make angles of $73.3(1)^\circ$ and $85.9(1)^\circ$, respectively, with the plane of the imidazole ring.

The crystal structure of (I) is stabilized by $\text{C}—\text{H}\cdots\text{Cl}$, $\text{C}—\text{H}\cdots\text{O}$ and $\text{C}—\text{H}\cdots\text{N}$ hydrogen bonds and $\text{C}—\text{H}\cdots\pi$ interactions (Table 1 and Fig. 2). The two interactions $\text{C}—\text{H}\cdots\text{Cl}$ and the $\text{C}—\text{H}\cdots\text{N}$ involving atom N3 of the imidazole result

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in the formation of a two-dimensional network in the *bc* plane. A C—H... π interaction exists between C11 and benzene ring C13–C18 at $(-x, -y, 1 - z)$, the distance between C11 and the centroid of the ring being 3.983 Å.

Experimental

To a suspension of sodium methoxide (0.03 mol, 1.62 g) in dimethylformamide (DMF, 25 ml) was added a solution of 2-(but-2-enyl)-4-chloro-5-formylimidazole (0.03 mol, 5.655 g) in DMF. The mixture was stirred at 298 K for 30 min, and to this was added dropwise a solution of 4-bromomethyl-2'-cyanobiphenyl (0.025 mol, 6.80 g) in DMF (25 ml). The mixture was stirred at room temperature for 24 h and evaporated to a residue under vacuum. The residue was dissolved in ethyl acetate (70 ml), washed with brine (20 ml), then water (50 ml), dried using Na₂SO₄ and evaporated to yield a crude product; this was purified by column chromatography using a mixture (7:2) of *n*-hexane and ethyl acetate as eluant to give the title product, which was recrystallized from CCl₄.

Crystal data

C ₂₂ H ₁₈ ClN ₃ O	$D_x = 1.273 \text{ Mg m}^{-3}$
$M_r = 375.84$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 20332 reflections
$a = 9.080$ (6) Å	$\theta = 1.8\text{--}27.4^\circ$
$b = 22.782$ (15) Å	$\mu = 0.21 \text{ mm}^{-1}$
$c = 10.055$ (7) Å	$T = 293$ (2) K
$\beta = 109.476$ (10)°	Rectangular block, colourless
$V = 1961$ (2) Å ³	$0.30 \times 0.25 \times 0.22 \text{ mm}$
$Z = 4$	

Data collection

Bruker SMART CCD area-detector diffractometer	3164 reflections with $I > 2\sigma(I)$
ω scans	$R_{\text{int}} = 0.022$
Absorption correction: none	$\theta_{\text{max}} = 27.4^\circ$
20332 measured reflections	$h = -11 \rightarrow 11$
4143 independent reflections	$k = -29 \rightarrow 28$
	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0786P)^2 + 1.127P]$
$R[F^2 > 2\sigma(F^2)] = 0.064$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.184$	$(\Delta/\sigma)_{\text{max}} = 0.083$
$S = 1.05$	$\Delta\rho_{\text{max}} = 0.64 \text{ e Å}^{-3}$
4143 reflections	$\Delta\rho_{\text{min}} = -0.36 \text{ e Å}^{-3}$
244 parameters	
H-atom parameters constrained	

Table 1

Hydrogen-bonding geometry (Å, °) for (I).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
C6—H6B...O26	0.97	2.40	3.048 (3)	124
C8—H8...O26	0.93	2.71	3.463 (4)	139
C21—H21A...N20 ⁱ	0.97	2.62	3.578 (5)	170
C6—H6A...N20 ^j	0.97	2.68	3.625 (4)	165
C15—H15...O26 ⁱⁱ	0.93	2.84	3.421 (4)	122
C17—H17...N3 ⁱⁱⁱ	0.93	2.74	3.532 (4)	144
C18—H18...O26 ^{iv}	0.93	2.76	3.677 (5)	171
C24—H24A...Cl ^v	0.96	2.91	3.818 (5)	159
C11—H11...C _g ^{vi}	0.93	3.27	3.983	135

Symmetry codes: (i) $x - 1, y, z$; (ii) $1 - x, -y, -z$; (iii) $-x, y - \frac{1}{2}, -\frac{1}{2} - z$; (iv) $-x, -y, -z$; (v) $x - 1, y, z - 1$; (vi) $-x, -y, 1 - z$. C_g is the centroid of the benzene ring C13–C18.

All H atoms were positioned geometrically (C—H = 0.93–0.98 Å) and allowed to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2$ or 1.5 times $U_{\text{eq}}(\text{parent atom})$.

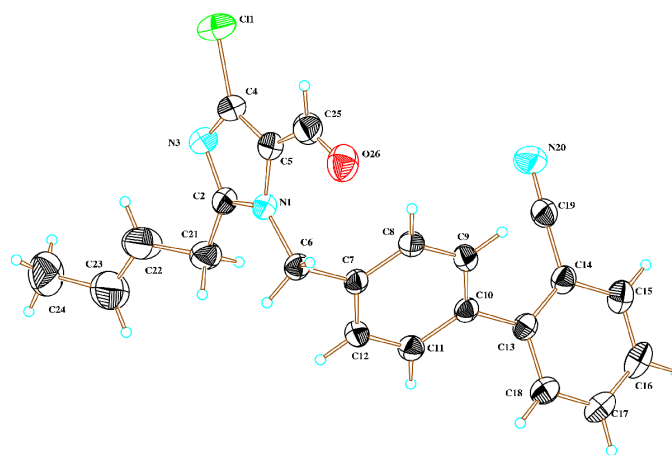


Figure 1

ZORTEP (Zsolnai, 1998) plot of the title molecule, showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

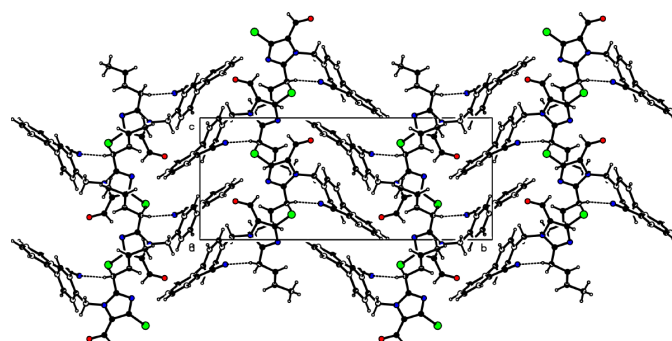


Figure 2

A packing diagram of the crystal structure, viewed down the *a* axis. Dashed lines represent hydrogen bonds.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: PLATON (Spek, 2003), ORTEP-3 (Farrugia, 1997) and ZORTEP (Zsolnai, 1998); software used to prepare material for publication: PLATON.

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