Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4′-Bromomethylbiphenyl-2-carbonitrile

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.045
wR factor = 0.088
Data-to-parameter ratio = 15.8

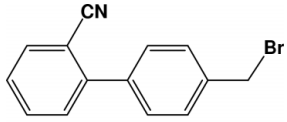
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 4 November 2003
Accepted 5 November 2003
Online 15 November 2003

In the title compound, $C_{14}H_{10}BrN$, the dihedral angle between the two aromatic rings is 48.34 (14)°. Weak C—H···Br and C—H···N interactions are the principal intermolecular forces.

Comment

The title compound, (I), is well known as a key intermediate in the production of Losartan (Duncia *et al.*, 1991), which is very useful in the treatment of hypertension by inhibiting angiotensin II (Campbell *et al.*, 1995). We are interested in how the molecular conformation of (I) (Fig. 1) affects the reactivity of the Br atom and its influence on the conversion of the cyano group to a tetrazole.



(I)

In (I), all the bond lengths and angles are normal. The two aromatic rings are non-coplanar; the dihedral angle is 48.34 (14)°. Weak C—H···Br and C—H···N interactions are the principal intermolecular forces, mediating the formation of the three-dimensional framework (Fig. 2 and Table 1).

Experimental

A solution of bromine (16 g, 0.1 mol) in 1,2-dichloroethane was added dropwise to a mixture of 4′-methyl-2-cyanobiphenyl (19.3 g, 0.1 mol) and azodiisobutyronitrile (0.8 g, 0.005 mol) in 1,2-dichloroethane (150 ml) at 343 (3) K with stirring for 2 h. The reaction mixture was then heated continuously at the same temperature, with monitoring by thin-layer chromatography, until the raw materials disappeared. The mixture was then treated with water (80 ml) three

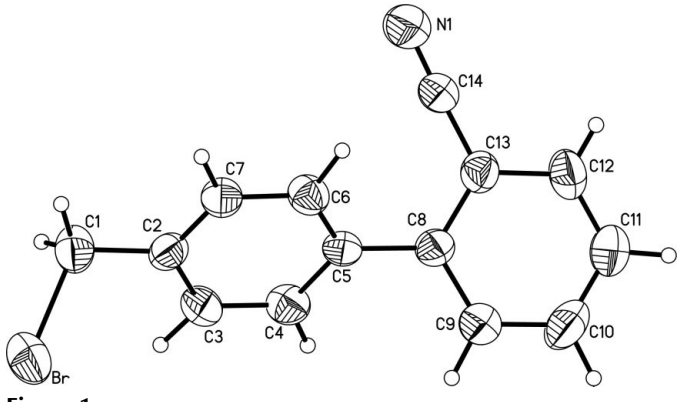


Figure 1
The molecular structure of (I), shown with 50% probability displacement ellipsoids.

times, dried and concentrated to dryness *in vacuo* to obtain crude (I). Crystallization from 1,2-dichloroethane–hexane yielded the pure solid (25 g, 92%) with a purity of 99.3% (HPLC). Colorless single crystals of (I) suitable for diffraction analysis were obtained from a tetrahydrofuran solution after two weeks at 293 (3) K.

Crystal data

$C_{14}H_{10}BrN$
M_r = 272.14
Orthorhombic, *Fdd*2
a = 47.219 (12) Å
b = 24.229 (6) Å
c = 4.1085 (10) Å
V = 4700 (2) Å^3
Z = 16
D_x = 1.538 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1496 reflections
θ = 2.4–20.6°
μ = 3.47 mm^{-1}
T = 293 (2) K
Prism, colorless
0.3 × 0.2 × 0.2 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.43, T_{max} = 0.50
6379 measured reflections
2284 independent reflections
1610 reflections with $I > 2\sigma(I)$
R_{int} = 0.075
θ_{max} = 26.0°
$h = -58 \rightarrow 52$
$k = -29 \rightarrow 24$
$l = -5 \rightarrow 4$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.088
S = 1.05
2284 reflections
145 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.02P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}
Absolute structure: Flack (1983), 965 Friedel pairs
Flack parameter = 0.014 (15)

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1B···Br^{i}	0.93	2.82	3.699 (6)	158
C1—H1A···N1^{ii}	1.08	2.61	3.634 (8)	159
C9—H9···Br^{iii}	0.96	3.05	3.750 (6)	131

Symmetry codes: (i) $x, y, z-1$; (ii) $\frac{3}{4}-x, \frac{1}{4}+y, \frac{3}{4}+z$; (iii) $\frac{1}{2}-x, 1-y, \frac{1}{2}+z$.

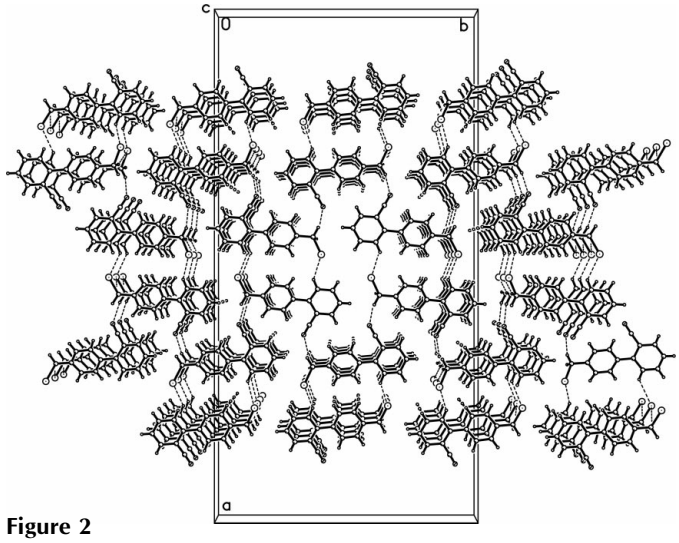


Figure 2
A packing diagram of (I), viewed down the c axis. Intermolecular C—H···N and C—H···Br interactions are shown as dashed lines.

All the H atoms were located in a difference-density maps and refined isotropically as riding. The C—H bond lengths are in the range 0.90–1.08 Å and $U_{iso} = 1.2U_{eq}$(C).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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