Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5-[4′-(Bromomethyl)biphenyl-2-yl]-2-trityl-2*H*-tetrazole

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.04 Å
R factor = 0.044
wR factor = 0.102
Data-to-parameter ratio = 14.4

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

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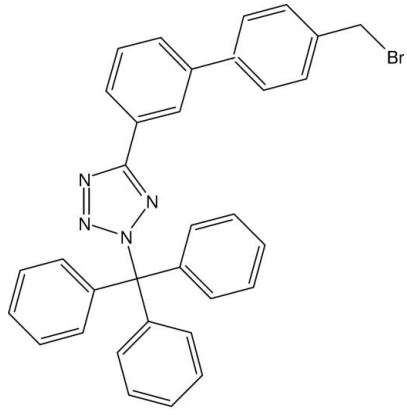
Received 14 February 2005
Accepted 18 February 2005
Online 26 February 2005

The title compound, $C_{33}H_{25}BrN_4$, belongs to the class of substituted tetrazoles. This type of compound is an important starting material for the synthesis of pharmaceutically active materials.

Comment

The title compound, (I), is a key intermediate for the synthesis of the antihypertensive drug losartan (Griffiths *et al.*, 1999) and it is also used as a starting material for the synthesis of trityl losartan (Sieron *et al.*, 2004). In view of the importance of (I), its crystal structure has been determined.



(I)

A perspective view of the title compound 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 tetrazole heterocycle and the phenyl ring attached to it is 82.11 (9)°. The dihedral angle between the benzene rings of the biphenyl group is 52.88 (8)°. The C—Br bond is rotated almost perpendicular to the benzene ring to which this group is attached (Table 1).

Experimental

Compound (I) was synthesized according to the procedure given by Aldrich *et al.* (1989). 5-(4′-Methylbiphenyl-2-yl)-1*H*-tetrazole (2.36 g, 10 mmol), trityl chloride (2.78 g, 10 mmol) and triethylamine (2 ml) were stirred at room temperature in dichloromethane (25 ml) for 2 h. The product was treated with 1-bromo-2,5-pyrrolidinedione (1.78 g, 10 mmol) and benzoyl peroxide (0.1 g) in carbon tetrachloride (10 ml) to obtain the title compound. Compound (I) was recrystallized from ethyl methyl ketone (yield 80%, m.p. 411 K). IR (KBr, cm^{-1}): υ 3029 (*m*), 2921 (*m*), 1660 (*w*), 1620 (*w*), 1452 (*m*), 1215 (*w*), 893 (*w*), 802 (*s*), 722 (*s*), 705 (*s*); ^{1}H NMR ($CDCl_3$, p.p.m.): 4.40 (*s*, 2H,

CH_2), 7.15 (*d*, 2H, ArH), 7.30–7.70 (*m*, 21H, ArH); ^{13}C NMR ($CDCl_3$, p.p.m.): 38.2 (*t*, CH_2), 62.1 (*s*, C), 126.2 (*d*, ArC), 127.9 (*d*, ArC), 128.2 (*d*, ArC), 128.9 (*d*, ArC), 129.6 (*d*, ArC), 131.0 (*d*, ArC), 135.9 (*s*, ArC), 136.2 (*s*, ArC), 137.8 (*s*, ArC), 138.0 (*s*, ArC), 143.2 (*s*, ArC). Analysis calculated for $C_{33}H_{25}BrN_4$: C 71.10, H 4.52, N 10.05%; found: C 71.18, H 4.59, N 10.01%.

Crystal data

$C_{33}H_{25}BrN_4$
M_r = 557.48
Monoclinic, $P2_1/n$
a = 10.6150 (6) Å
b = 14.9140 (5) Å
c = 17.3961 (9) Å
β = 102.263 (4)°
V = 2691.2 (2) Å^3
Z = 4
D_x = 1.376 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 46712 reflections
θ = 3.6–25.6°
μ = 1.56 mm^{-1}
T = 173 (2) K
Block, colourless
0.28 × 0.26 × 0.25 mm

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.670, T_{max} = 0.687
31845 measured reflections
4939 independent reflections
4290 reflections with $I > 2\sigma(I)$
R_{int} = 0.036
θ_{max} = 25.4°
$h = -12 \rightarrow 12$
$k = -18 \rightarrow 17$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.102
S = 1.09
4939 reflections
343 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.047P)^2 + 3.0688P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.87 e Å^{-3}
Extinction correction: none

Table 1
Selected geometric parameters (Å, °).

N1—N5	1.325 (3)	C3—N4	1.356 (3)
N1—N2	1.347 (3)	N4—N5	1.328 (3)
N2—C3	1.328 (3)	C57—Br1	1.966 (3)
N5—N1—N2	113.42 (19)	N5—N4—C3	106.3 (2)
C3—N2—N1	101.90 (19)	N1—N5—N4	106.08 (19)
N2—C3—N4	112.3 (2)		
C55—C54—C57—Br1	78.8 (3)		

All H atoms were located in a difference map but were positioned geometrically and refined with fixed individual displacement parameters (set to 1.2 times U_{eq} of the parent C atom) using a riding model, with C—H = 0.95 Å for aromatic and 0.99 Å for methylene H atoms.

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).

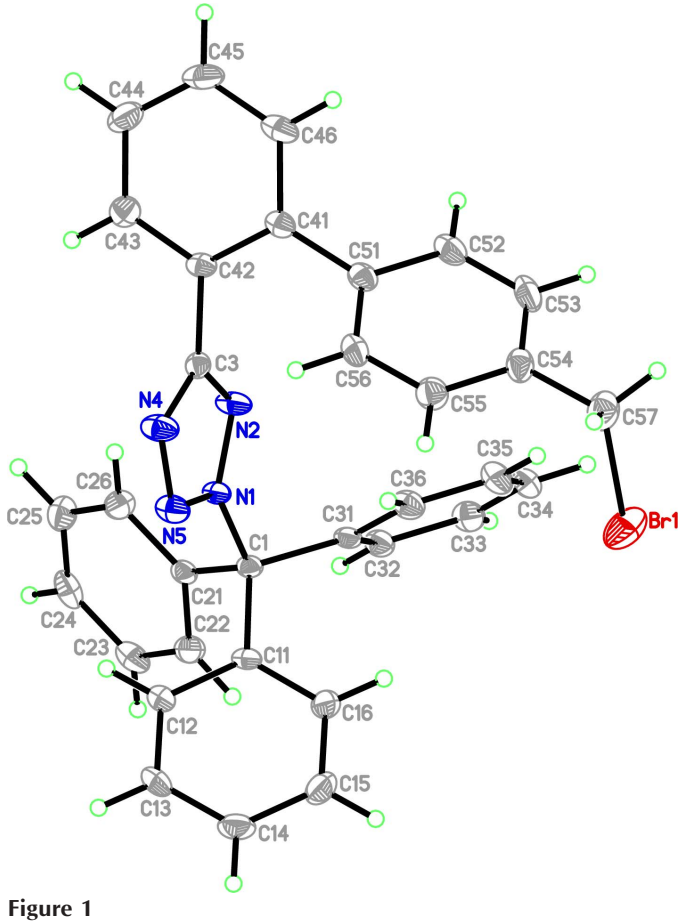


Figure 1
Perspective view of the title compound, with the atom-numbering scheme; displacement ellipsoids are drawn at the 50% probability level.

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