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rac-(*tert*-Butylamino)(diisopropylamino)-(9-fluorenyl)borane

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In the crystal structure, the title compound, $C_{23}H_{33}BN_2$, exhibits approximate C_s symmetry, with B—N bond lengths of 1.414 (2) and 1.419 (2) Å, consistent with B═N double bonds. This is also reflected by the trigonal planes around the B and butylamino N atoms, which are virtually coplanar.

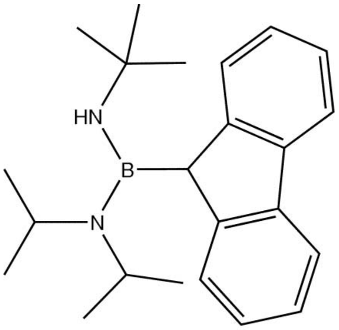
Key indicators

Single-crystal X-ray study
T = 193 K
Mean σ(C–C) = 0.002 Å
R factor = 0.054
wR factor = 0.123
Data-to-parameter ratio = 17.4

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

Comment

Ligand precursors related to the title compound *rac*-(*tert*-butylamino)(diisopropylamino)(9-fluorenyl)borane, (I), have been previously employed for the synthesis of boron-bridged constrained geometry complexes. These complexes exhibit activity for the polymerization of ethene and higher α-olefins when activated with MAO (methylaluminoxane) (Braunschweig *et al.*, 2000, 2004). In comparative studies, a strong influence of the ligand substitution patterns in corresponding complexes has been demonstrated on both catalyst activity and polymerization characteristics (Stevens 1994), justifying the synthesis of a series of boron-bridged analogues.



(I)

The structure of (I) (Fig. 1) in the solid state largely resembles that of its corresponding *N*-phenyl-substituted counterpart (η^1-$C_{13}H_9$)B(N^{i}Pr$_2$)N(H)Ph (Braunschweig *et al.*, 2003). Compound (I) adopts approximate C_s symmetry in the crystal structure. Again, both the B1 and N2 centres adopt trigonal planar geometries with the central atoms lying only 0.002 (B1) and 0.001 Å (N2) out of the planes of their respective substituents. These two trigonal planes are virtually coplanar with torsion angles of 5.5 (2) (C18—N2—B1—N1) and 5.3 (2)° (C21—N2—B1—C9). The B1—N1 and B2—N2 bond lengths of 1.414 (2) and 1.419 (2) Å, respectively, are equivalent within experimental error, indicating equal amounts of π-contribution from the two amino groups. The N2-bound isopropyl group *syn* to the fluorenyl moiety is oriented such that the methine proton is directed towards the middle of the five-membered ring, with H—π distances to the centres of the C2═C10 and C8═C3 aromatic bonds of 2.561

and 2.558 Å, respectively. No short intermolecular contacts are observed.

Experimental

The title compound was prepared by a modified literature procedure (Braunschweig *et al.*, 2003) by sequential reaction of $^{i}Pr_2NBCl_2$ (4.55 g, 25.0 mmol) with lithium fluorenide (4.30 g, 25.0 mmol) and lithium *tert*-butylamide (1.98 g, 25.0 mmol) in a hexane–toluene mixture. Two subsequent recrystallization steps from hexane at ambient temperature and from toluene at ambient temperature yielded the pure target compound (I) (1.18 g, 13.6%). Crystals of (I) suitable for single-crystal X-ray diffraction were selected directly from the sample as prepared.

Crystal data

$C_{23}H_{33}BN_2$
$M_r = 348.32$
Monoclinic, $P2_1/n$
$a = 9.2771$ (5) Å
$b = 16.0667$ (9) Å
$c = 14.5453$ (8) Å
$\beta = 97.0340$ (10)°
$V = 2151.7$ (2) Å^3
$Z = 4$

$D_x = 1.075$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 9516 reflections
$\theta = 2.5$–25.4°
$\mu = 0.06$ mm^{-1}
$T = 193$ (2) K
Block, colourless
0.35 × 0.25 × 0.21 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
$T_{\min} = 0.954$, $T_{\max} = 0.987$
35746 measured reflections

4269 independent reflections
3774 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.042$
$\theta_{\max} = 26.1°$
$h = -11 \rightarrow 11$
$k = -19 \rightarrow 19$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.123$
$S = 1.12$
4269 reflections
246 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0434P)^2 + 0.798P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.26$ e Å^{-3}
$\Delta\rho_{\min} = -0.20$ e Å^{-3}

All H atoms were visible in difference Fourier maps. H atoms bonded to N atoms were refined. H atoms bonded to C atoms were subsequently placed at idealized positions and treated as riding atoms, with C—H = 0.98 (CH_3), 0.95 (aromatic CH) and 1.00 Å (aliphatic CH). $U_{\rm eq}$ values for primary H atoms were fixed at 1.5 times and tertiary H atoms at 1.2 times the $U_{\rm eq}$ of the attached C atom.

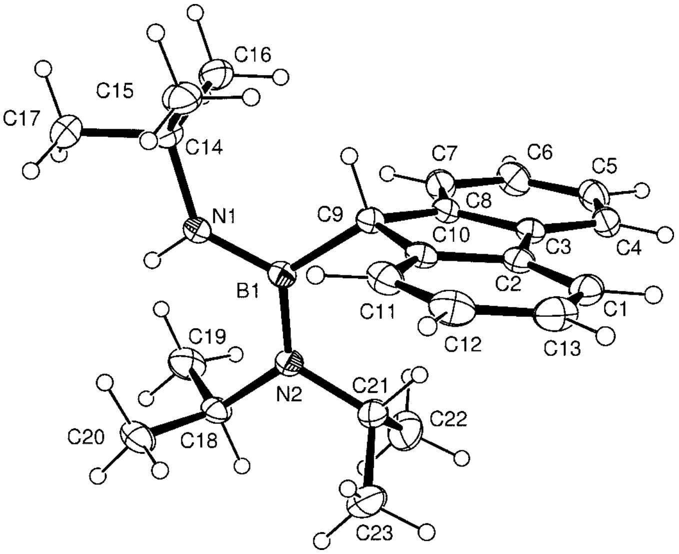


Figure 1
A view of (I), with displacement ellipsoids drawn at the 30% probability level.

Data collection: *SMART-NT* (Bruker, 2001); cell refinement: *SAINT-Plus-NT* (Bruker, 2001); data reduction: *SAINT-Plus-NT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *XP* in *SHELXTL*.

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