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N-(2′,7′-Di-*tert*-butyl-9,9′-spirobifluoren-2-yl)aniline

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.003 Å
R factor = 0.045
wR factor = 0.103
Data-to-parameter ratio = 14.2

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

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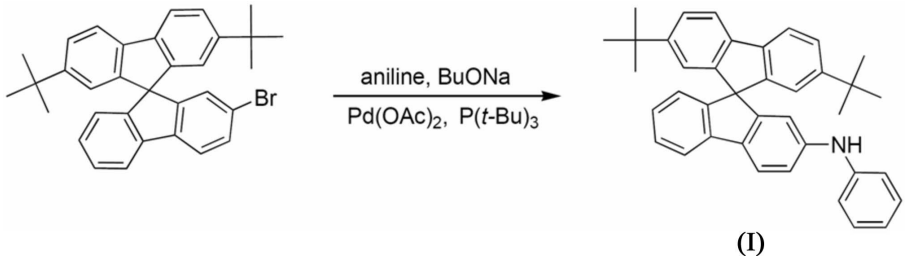
The title compound, $C_{39}H_{37}N$, is a precursor for the production of hole-transporting and/or emitting materials. The widespread interest in high T_g materials for optical light-emitting device (OLED) applications led us to design the syntheses of the desired compounds using the 9,9′-spirobifluorene unit as a building block.

Comment

The title compound, (I), has been shown to be an excellent precursor for the production of hole-transporting and/or emitting materials (Shen *et al.*, 2005). Intramolecular bond rotation and vibration in the spiro-linked unit are not possible and an increase in T_g is anticipated.



(I)

The compound is soluble in organic solvents and was considerably purified by column chromatography. The molecular structure, including the definition of rings for mean plane calculations, is shown in Fig. 1. The dihedral angles between the fluorene (*P*1 and *P*2) and phenyl (*P*3) rings are 85.89 (8) and 45.09 (7)°, and that between the two fluorene rings is 86.61 (8)°.

Experimental

The title compound was synthesized by the following procedure. 2-Bromo-2′,7′-di-*tert*-butyl-9,9′-spirobifluorene (2.53 g, 5.0 mmol), aniline (0.61 g, 6.5 mmol), $Pd(OAc)_2$ (23 mg, 0.1 mmol), $P(^tBu)_3$ (20 mg, 0.1 mmol), sodium *tert*-butoxide (0.72 g, 7.5 mmol) and toluene (50 ml) were charged in a two-necked flask kept under nitrogen. The mixture was heated to reflux for 12 h. After cooling, it was quenched with 5 ml of water. The solvent was removed under vacuum and the residue was extracted with dichloromethane/water (2:1). The organic layer was dried over $MgSO_4$ and filtered. Evaporation of the solvent left a brown residue that was chromatographed through silica gel using a dichloromethane/hexane (1:4) mixture as eluent. The compound was obtained as a white solid in 85% yield. FAB MS: *m*/*e* 519 (M^+). Analysis calculated for $C_{39}H_{37}N$: C 90.13, H 7.18, N 2.70%; found: C 89.66, H 7.31, N 2.76%.

Crystal data

$C_{39}H_{37}N$
M_r = 519.70
Monoclinic, $C2/c$
a = 43.971 (3) Å
b = 11.0785 (7) Å
c = 27.6079 (16) Å
β = 120.042 (3)°
V = 11641.9 (12) Å^3
Z = 16
D_x = 1.186 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5849 reflections
θ = 2.3–23.2°
μ = 0.07 mm^{-1}
T = 100.0 (1) K
Prism, colourless
0.16 × 0.14 × 0.08 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.919, T_{max} = 0.994
79866 measured reflections
10284 independent reflections
6304 reflections with $I > 2\sigma(I)$
R_{int} = 0.105
θ_{max} = 25.0°
$h = -52 \rightarrow 52$
$k = -13 \rightarrow 13$
$l = -32 \rightarrow 32$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.103
S = 0.89
10284 reflections
722 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0424P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.00049 (3)

H atoms were positioned geometrically and treated as riding atoms, with C—H = 0.93–0.96 Å, N—H = 0.86 Å, and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

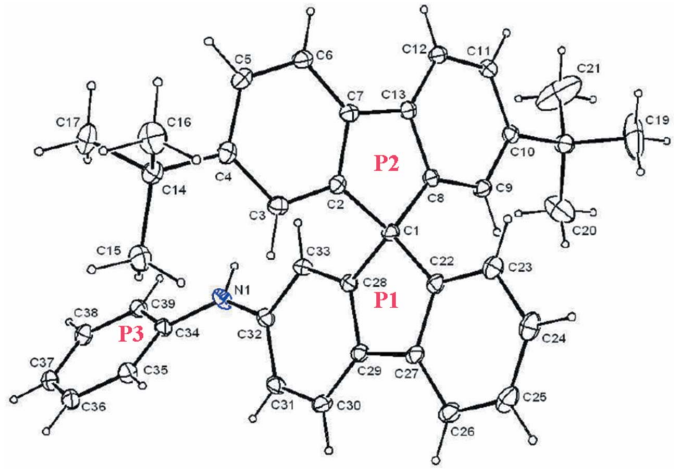


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

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