N,N'-Bis(1-naphthyl)-N,N'-diphenyl-1,1'-biphenyl-4,4'-diamine

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The title complex, $C_{44}H_{32}N_2$, is a useful charge-carrier transport material in organic electroluminescence devices. There are two half-molecules in the asymmetric unit; each molecule possesses a centre of symmetry at the mid-point of the central C—C bond.

Comment

Organic electroluminescence has come to the critical point of large-scale marketing for flat-panel displays since the first report of an efficient layered device by Kodak (Tang & Vanslyke, 1987). The materials used for charge-carrier transport layers are carefully chosen so that holes and electrons are preferentially conducted to obtain stable efficient devices (VanSlyke *et al.*, 1996). As one of the most important components in common electroluminescent (EL) structures, amorphous amines, *e.g.* N,N'-bis(1-naphthyl)-N,N'-diphenyl-1,1'-biphenyl-4,4'-diamine (α-NPD) having a glass transition temperature of 368 K, are the most successful hole-transporting materials (Naito & Miura, 1993; Thelakkat & Schmidt, 1998). However, its crystal structure has not been reported up to now. We have synthesized α-NPD by palladium-catalysed amination and have analysed its crystal structure.
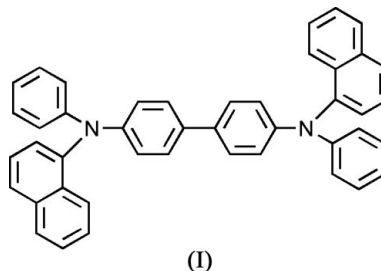
(I)

There are two half-molecules in the asymmetric unit; each molecule possesses a centre of symmetry at the mid-point of the central C—C bond (Fig. 1). The amine atoms adopt a trigonal geometry with an average C—N—C angle value near 120° and C—N bond distances [1.4102 (16)–1.4291 (17)Å, average 1.418Å] within the normal range (Kennedy *et al.*, 2002).

Experimental

Pd(OAc)$_2$ (0.002 g) and P(^{t}Bu)$_3$ (0.1 ml, 10% in hexane) were added to a 20 ml *o*-xylene solution of 4,4'-diiodobiphenyl (2 mmol), N-phenyl-1-naphthylamine (4 mmol) and NaO^{t}Bu (4.8 mmol) and the mixture was refluxed in an N$_2$ atmosphere for 3–5 h at 393 K. It was then cooled to room temperature, water (20 ml) was added, and the organic layer was extracted, concentrated to dryness under reduced
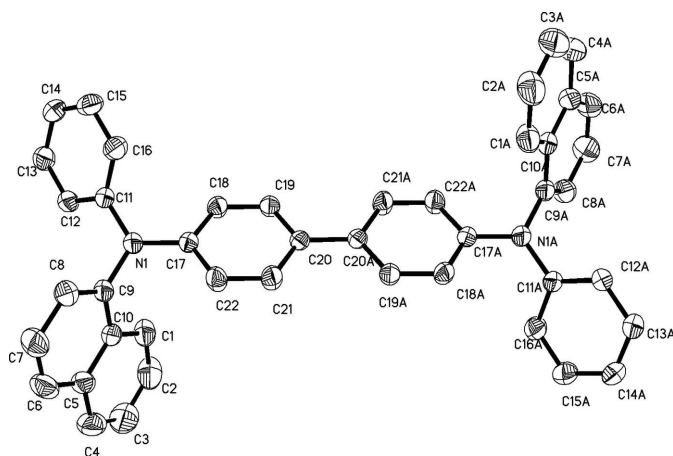
Figure 1
Perspective view of the first half-molecule in the asymmetric unit of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and all H atoms have been omitted for clarity. [Symmetry code: (A) $-x, 2-y, 1-z$.]
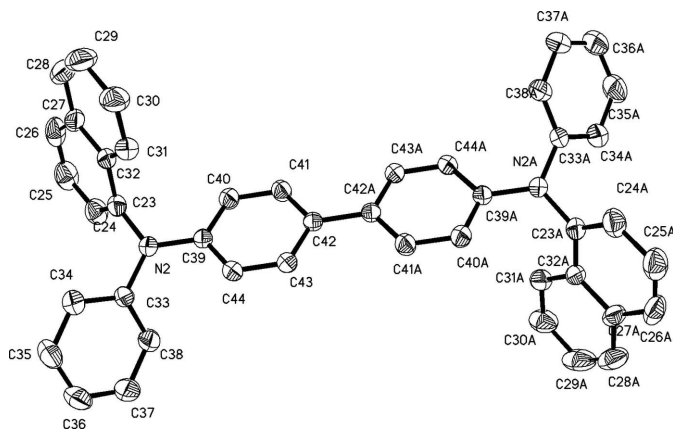

Figure 2
Perspective view of the second half-molecule in the asymmetric unit of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and all H atoms have been omitted for clarity. [Symmetry code: (A) $1-x, 1-y, -z$.]

pressure, dissolved in a minimal amount of tetrahydrofuran and recrystallized from ethanol. The product was further purified by sublimation [m.p. 552–556 K, T_g = 368 K (determined by differential scanning calorimetry)].

Crystal data

$C_{44}H_{32}N_2$	$V = 1598.4\ (2)$ Å³
$M_r = 588.72$	$Z = 2$
Triclinic, $P\bar{1}$	$D_x = 1.223$ Mg m⁻³
$a = 10.3089\ (9)$ Å	Mo $K\alpha$ radiation
$b = 11.3382\ (10)$ Å	$\mu = 0.07$ mm⁻¹
$c = 14.5056\ (13)$ Å	$T = 293\ (2)$ K
$\alpha = 82.378\ (10)°$	Plate, yellow
$\beta = 77.671\ (2)°$	$0.50 \times 0.30 \times 0.08$ mm
$\gamma = 75.565\ (10)°$	

Data collection

Bruker SMART CCD area-detector diffractometer	9404 measured reflections
φ and ω scans	6574 independent reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	4494 reflections with $I > 2\sigma(I)$
$T_{min} = 0.754$, $T_{max} = 1.000$	$R_{int} = 0.011$
	$\theta_{max} = 27.8°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0633P)^2 + 0.0935P]$
$R[F^2 > 2\sigma(F^2)] = 0.041$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.124$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.06$	$\Delta\rho_{max} = 0.19$ e Å⁻³
6574 reflections	$\Delta\rho_{min} = -0.16$ e Å⁻³
416 parameters	Extinction correction: *SHELXL97*
H-atom parameters constrained	Extinction coefficient: 0.0078 (17)

Table 1
Selected geometric parameters (Å, °).

C9—N1	1.4291 (17)	C23—N2	1.4220 (17)
C11—N1	1.4217 (16)	C33—N2	1.4156 (17)
C17—N1	1.4102 (16)	C39—N2	1.4122 (15)
C17—N1—C11	120.98 (10)	C39—N2—C33	121.31 (10)
C17—N1—C9	119.88 (11)	C39—N2—C23	120.59 (10)
C11—N1—C9	119.10 (10)	C33—N2—C23	117.93 (10)

All H atoms were positioned geomertrically and treated as riding [C—H = 0.93Å and $U_{iso}(H) = 1.2U_{eq}(C)$].

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*

References

Bruker (1998). *SAINT* (Version 4.00), *SMART* (Version 5.0) and *SHELXTL* (Version 5.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Kennedy, A. R., Smith, W. E., Tackley, D. R., David, W. I. F., Shankland, K., Brown, B. & Teat, S. J. (2002). *J. Mater. Chem.* **12**, 168–172.
Naito, K. & Miura, A. (1993). *J. Phys. Chem.* **97**, 6240–6248.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Tang, C. W. & Vanslyke, S. A. (1987). *Appl. Phys. Lett.* **51**, 913–915.
Thelakkat, M. & Schmidt, H. W. (1998). *Adv. Mater.* **10**, 219–223.
VanSlyke, S. A., Chen, C. H. & Tang, C. W. (1996). *Appl. Phys. Lett.* **69**, 2160–2162.