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N,*N*-Dibenzyl-4-(4,4-diphenylbuta-1,3-dienyl)-3-methylaniline

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
R factor = 0.054
wR factor = 0.131
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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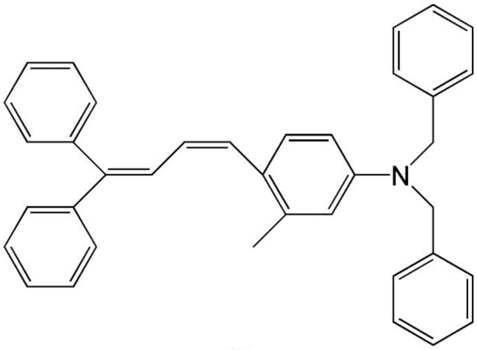
The title compound, $C_{37}H_{33}N$, was synthesized by the Wittig–Horner reaction of 4-(*N*,*N*-dibenzylamino)-2-methylbenzaldehyde and the phosphonate carbanion, derived from 1,1-diphenyl-3-chloropropylene and triethyl phosphite by the Arbuzov reaction. The butadiene fragment has a planar *cisoid* conformation, while the two benzyl fragments are almost parallel to each other, forming a dihedral angle of 9.8 (2)°.

Comment

Hole transporting materials (HTMs) play an important role in the fabrication of organic photo-conductors (OPCs) (Wu *et al.*, 2005) and organic light-emitting diodes (OLEDs) (Li *et al.*, 2005; Satoh *et al.*, 2003). Substances containing the butadiene structure have been widely investigated, because of their easy preparation by the Wittig reaction and favorable hole-transport properties when used as HTMs (Enokida & Hirohashi, 1991). We report here the synthesis and crystal structure of the title compound, (I), synthesized by the Wittig-Horner reaction of 2-methyl-4-(*N*,*N*-dibenzyl)aminobenzaldehyde and the phosphonate carbanion, derived from 1,1-diphenyl-3-chloropropylene and triethyl phosphite by the Arbuzov reaction.



(I)

Fig. 1 shows the molecular structure of (I). The butadiene fragment C13=C14–C15=C16 is planar, with a maximum deviation of 0.003 (7) Å for atom C15, and has a *cisoid* conformation. Both the C1/C13/C7 plane and the plane of the C17–C22 benzene ring show substantial deviations from the butadiene plane, forming dihedral angles with the latter of 9.3 (2)° and 130.3 (2)°, respectively. The dihedral angles formed by the plane of the butadiene fragment with the planes of the C1–C6 and C7–C12 rings are 69.6 (2)° and 39.2 (2)°, respectively. The two benzyl fragments (C24–C30) and (C31–C37) are almost parallel to each other, forming a dihedral angle of 9.8 (2)°. The C1–C13 [1.487 (3) Å] and C7–C13

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[1.480 (3) Å] bond distances are a little longer than C16–C17 [1.467 (3) Å].

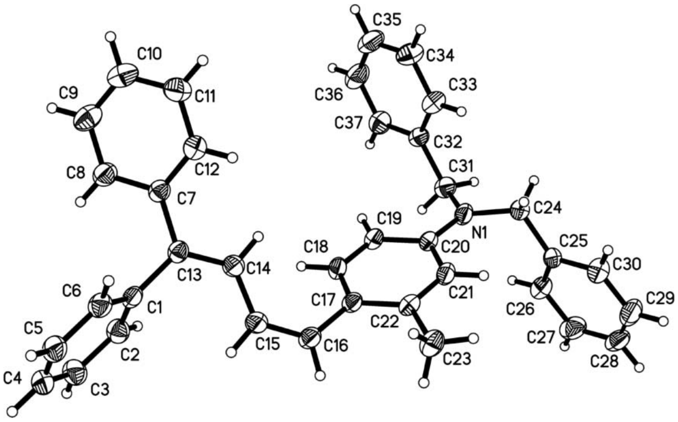

Figure 1
The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small circles of arbitrary radii.

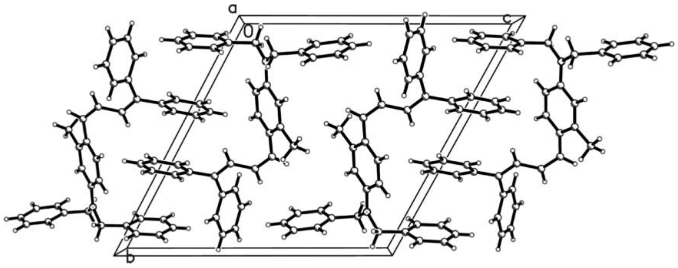

Figure 2
Packing diagram of (I).

Experimental

A mixture of 1,1-diphenyl-3-chloropropylene (11.4 g, 0.05 mol) and triethyl phosphit (18.0 ml, 0.05 mol) was refluxed in 50 ml of xylene for 10 h and the xylene was then removed *in vacuo*. After cooling, 2-methyl-4-(*N*,*N*-dibenzyl)aminobenzaldehyde (12.6 g, 0.04 mol) and 100 ml of dimethylformamide were added to the flask containing the residue. Thereafter, 4.5 g (0.04 mol) of potassium *tert*-butoxide was added in small portions. The resulting mixture was stirred for 5 h and then poured into methanol. The resulting precipitate was separated from the liquid by filtration, purified by silica gel column chromatography (eluent: toluene/ethyl acetate = 2:1), recrystallized from hexane, and dried to obtain yellow crystals (yield: 41.2%, m.p. 420 K).

Crystal data

$C_{37}H_{33}N$
$M_r = 491.64$
Triclinic, $P\bar{1}$
$a = 6.1629\ (16)$ Å
$b = 15.935\ (4)$ Å
$c = 16.323\ (4)$ Å
$\alpha = 117.824\ (4)^\circ$
$\beta = 90.749\ (5)^\circ$
$\gamma = 99.054\ (5)^\circ$
$V = 1393.2\ (6)$ Å^3

$Z = 2$
$D_x = 1.172$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1753 reflections
$\theta = 2.5–26.3^\circ$
$\mu = 0.07$ mm^{-1}
$T = 294\ (2)$ K
Plate, yellow
$0.40 \times 0.16 \times 0.08$ mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
$T_{\min} = 0.974$, $T_{\max} = 0.995$
7091 measured reflections

4873 independent reflections
2678 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.027$
$\theta_{\max} = 25.0^\circ$
$h = -7 \rightarrow 5$
$k = -18 \rightarrow 18$
$l = -15 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.131$
$S = 1.02$
4873 reflections
344 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0464P)^2 + 0.1576P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.14$ e Å^{-3}
$\Delta\rho_{\min} = -0.18$ e Å^{-3}

H atoms were positioned geometrically and refined in the riding-model approximation, with C–H = 0.93–0.98 Å and $U_{\rm iso}(H) = 1.2U_{\rm eq}(C)$ for aromatic and methine H atoms [$U_{\rm iso}(H) = 1.5U_{\rm eq}(C)$ for CH_3].

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); 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, 1997); software used to prepare material for publication: *SHELXTL*.

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