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2,5,2″,5″-Tetramethyl-*p*-terphenyl

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Key indicators

Single-crystal X-ray study
T = 133 K
Mean σ(C–C) = 0.002 Å
R factor = 0.045
wR factor = 0.122
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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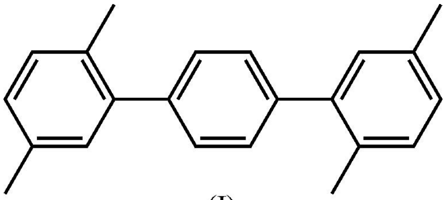
The title compound, $C_{22}H_{22}$, displays no crystallographic symmetry. The central benzene ring subtends interplanar angles of 63.00 (6) and 66.09 (6)° (in opposite senses) to the outer rings. The crystal packing involves two short C—H$\cdots\pi$ contacts.

Comment

Our interest in the crystal structures of bromomethylarene derivatives (*e.g.* Kuś & Jones, 2003) extends to the synthesis and structure of their methyl precursors. A search of the Cambridge Structural Database (Allen, 2002; Version 5.26) revealed only one hydrocarbon based on *para*-terphenyl, namely the 3,3″-dimethyl derivative (Avery *et al.*, 1998). The structure of the parent compound *para*-terphenyl is also known, but it suffers from disorder phenomena and associated phase changes, and is therefore rather imprecise (Baudour *et al.*, 1986, and references therein).



(I)

We present here the structure of the title compound, (I), $C_{22}H_{22}$ (Fig. 1), a compound first obtained from dinitrosodiacetyl-1,4-phenylenediamine and *para*-xylene by France *et al.* (1939).

The molecule of (I) displays no crystallographic symmetry. The bond lengths and angles may be regarded as normal, *e.g.* the single bond lengths between the rings of 1.490 (2) Å for C11—C21 and 1.491 (2) Å for C24—C31 [*cf.* 1.504 (3) Å in the

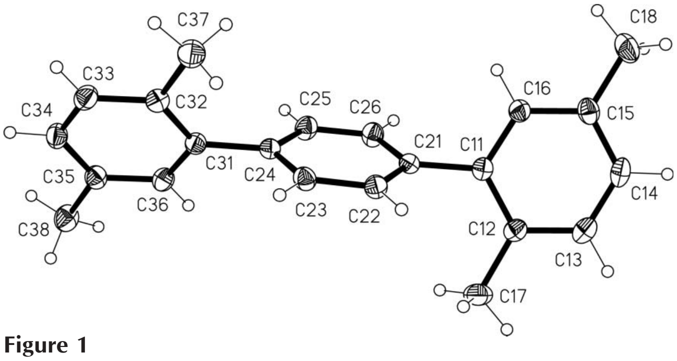


Figure 1
View of (I), showing 50% displacement ellipsoids for the non-H atoms.

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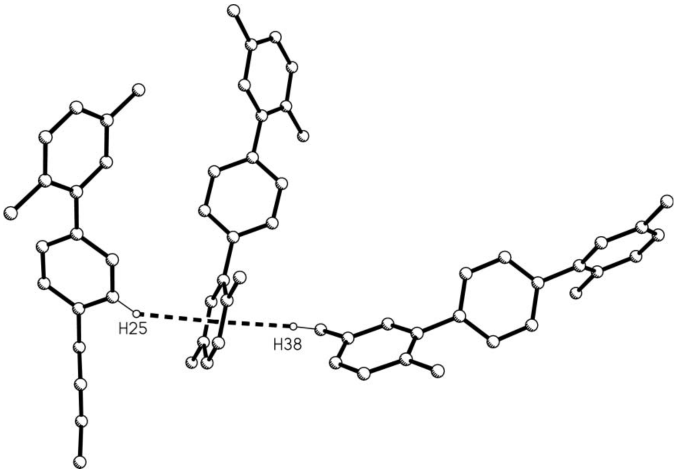

Figure 2
C—H···π contacts (dashed lines) in (I). All H atoms, except those involved in the C—H···π interactions, have been omitted for clarity.

3,3″-dimethyl derivative, which displays inversion symmetry; Avery *et al.*, 1998]. The interplanar angles to the central ring are 63.00 (6)° for ring 1 (C11) and 66.09 (6)° for ring 3 (C31) [*cf.* 35.3 (1)° in the dimethyl derivative]; the senses of rotation are opposite to each other. The centroids of the rings subtend a virtually linear angle of 179.3°.

The crystal packing in (I) involves two short C—H···π contacts to the centroid (*Cg*) of the C11–C16 ring (Fig. 2). If the C—H bond lengths are adjusted to their true, *i.e.* internuclear, values of 1.08 Å (Steiner, 1998) then the resulting geometrical parameters are C25—H25···Cg^{i} [symmetry code: (i) $x - \frac{1}{2}, \frac{3}{2} - y, 1 - z$; H···$Cg^{i}$ = 2.54 Å and C—H···Cg^{i} = 147°] and C38—H38*C*···Cg^{ii} [symmetry code: (ii) $\frac{3}{2} - x, 1 - y, z - \frac{1}{2}$; H···$Cg^{ii}$ = 2.55 Å and C—H···Cg^{ii} = 172°]. These combine to create a three-dimensional packing arrangement. There is no π–π stacking in (I).

Experimental

Compound (I) was synthesized by reaction of the Grignard reagent (*ca* 0.11 mol) derived from 2-bromo-*para*-xylene and magnesium, with 1,4-dibromobenzene (0.05 mol), using $Ni(PPh_3)_2Cl_2$ (1 mmol) as a catalyst, in boiling tetrahydrofuran [reaction time 4 h; yield: 34%; m.p. 383–385 K; literature 385–386 K (France *et al.*, 1939)]. Consistent analytical data were obtained. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.37 (*s*, 4H), 7.19 (*d*, 2H, *J* = 7.6 Hz), 7.14 (*s*, 2H), 7.10 (*d*, 2H, *J* = 7.6 Hz), 2.38 (*s*, 6H), 2.31 (*s*, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 20.30, 21.17, 128.16, 129.07, 130.55, 130.87, 132.49, 135.45, 140.66, 141.77. Single crystals were grown by slow evaporation of a methanol solution.

Crystal data

$C_{22}H_{22}$
$M_r = 286.40$
Orthorhombic, $P2_12_12_1$
$a = 6.1837$ (11) Å
$b = 15.147$ (2) Å
$c = 17.536$ (2) Å
$V = 1642.5$ (4) Å^3
$Z = 4$
$D_x = 1.158$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 7355 reflections
$\theta = 2.3$–30.5°
$\mu = 0.07$ mm^{-1}
$T = 133$ (2) K
Square prism, colourless
0.35 × 0.20 × 0.20 mm

Data collection

Bruker SMART 1000 CCD diffractometer
ω scans
Absorption correction: none
19327 measured reflections
2875 independent reflections

2476 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.041$
$\theta_{\rm max} = 30.5°$
$h = -8 \rightarrow 8$
$k = -21 \rightarrow 21$
$l = -24 \rightarrow 25$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.122$
$S = 1.05$
2875 reflections
203 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0837P)^2 + 0.0707P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.37$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.20$ e Å^{-3}

Methyl H atoms were identified in difference syntheses, idealized and then refined as rigid methyl groups (C—H = 0.98 Å and H—C—H = 109.5°) allowed to rotate but not tip. Other H atoms were included using a riding model, with C—H = 0.95 Å. The constraint $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(carrier) was applied. In the absence of significant anomalous scatterers, Friedel pairs were merged before refinement.

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: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL97*.

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