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(6*E*)-*N*-[(4*Z*)-2,5-Dimethyl-4-(*p*-tolylimino)cyclohexa-2,5-dienylidene]-4-methylaniline

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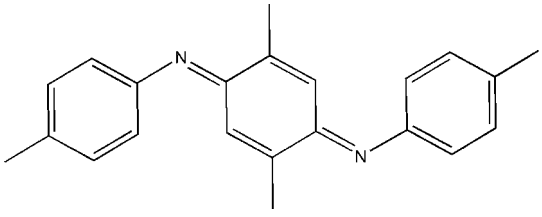
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; R factor = 0.072; wR factor = 0.233; data-to-parameter ratio = 17.8.

The title compound, $C_{22}H_{22}N_2$, was prepared by the reaction of 4-aminotoluene with sodium carbonate, sodium hydroxide and potassium permanganate. The molecule is disposed about a crystallographic inversion centre with one half-molecule comprising the asymmetric unit. The dihedral angle between the terminal and central benzene rings is 88.05 (1)°. The crystal packing is stabilized by van der Waals forces.

Related literature

For related literature, see: Boyer *et al.* (2000); Hadek (1968); Hadek *et al.* (1969)



Experimental

Crystal data

$C_{22}H_{22}N_2$
M_r = 314.42
Trigonal, $R\overline{3}$
a = 21.173 (8) Å
c = 10.476 (2) Å
V = 4067 (2) Å^3
Z = 9
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 293 (2) K
0.21 × 0.18 × 0.15 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: none
6148 measured reflections
1956 independent reflections
793 reflections with $I > 2\sigma(I)$
R_{int} = 0.075
3 standard reflections
every 100 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.072
$wR(F^2)$ = 0.233
S = 1.02
1956 reflections
110 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1990); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2328).

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supporting information

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(6*E*)-*N*-[(4*Z*)-2,5-Dimethyl-4-(*p*-tolylimino)cyclohexa-2,5-dienylidene]-4-methylaniline

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S1. Comment

It is now well established that conformational characteristics of the polyaniline polymer play a crucial role for its physical properties, including transport characteristics (Boyer *et al.*, 2000). Detailed analysis of the crystal structures of polyaniline oligomers containing alternating benzoid and quinoid rings with amine and/or imine groups can help in the understanding of the spectroscopic behaviour of the compounds and possibles mechanism for their electrical conductivity (Hadek,1968; Hadek *et al.*, 1969). Here we report the crystal structure of the title compound, (I).

The structure of (I) consists of discrete molecules disposed about a crystallographic inversion centre with half the molecule comprising the asymmetric unit (Fig. 1). The atoms (N1, C1 - C7) are planar with the greatest deviation from planarity for N1 of 0.042 (1) Å). The bond lengths and angles are usual for this type of compound (Boyer *et al.*, 2000). The mean planes p1(C2 - C7) and p2(C8 - C10,C8a - C10*a*) make a dihedral angle of 88.06 (1)°. The dihedral angle formed by ring (N1,C1 - C7)and ring (N1,C5 - C11,N1a,C5a) is 1.52 (1)°. The crystal packing (Fig.2) is stabilized by van der Waals forces.

S2. Experimental

P-aminotoluene (2.14 g, 0.02 mol) was dissolved in water (100 ml), then sodium carbonate (0.53 g, 0.005 mol), sodium hydroxide (0.80 g, 0.02 mol) and potassium permanganate (1.58 g, 0.01 mol) was added with stirring. The mixture was allowed to react at room temperature for 12 h to give a precipitate which wasfiltered and recrystallized from acetone to afford the title compound (0.956 g, yield 89.5%). Single crystals suitable for X-ray measurements were obtained by recrystallization from acetone at room temperature.

S3. Refinement

H atoms were fixed geometrically and allowed to ride on their parent atoms, with C—H distances of 0.93–0.96 Å and with U_{iso}=1.2–1.5 U_{eq} of the parent atoms.

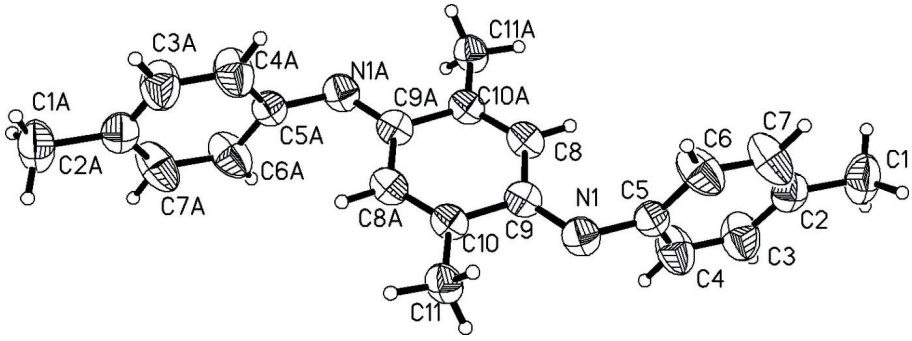

Figure 1

The molecular structure and atom-labeling scheme for (I), with displacement ellipsoids drawn at the 30% probability level. ′A′ atoms were generated by symmetry (-*x* + 1/3, -*y* + 2/3, -*z* - 1/3).

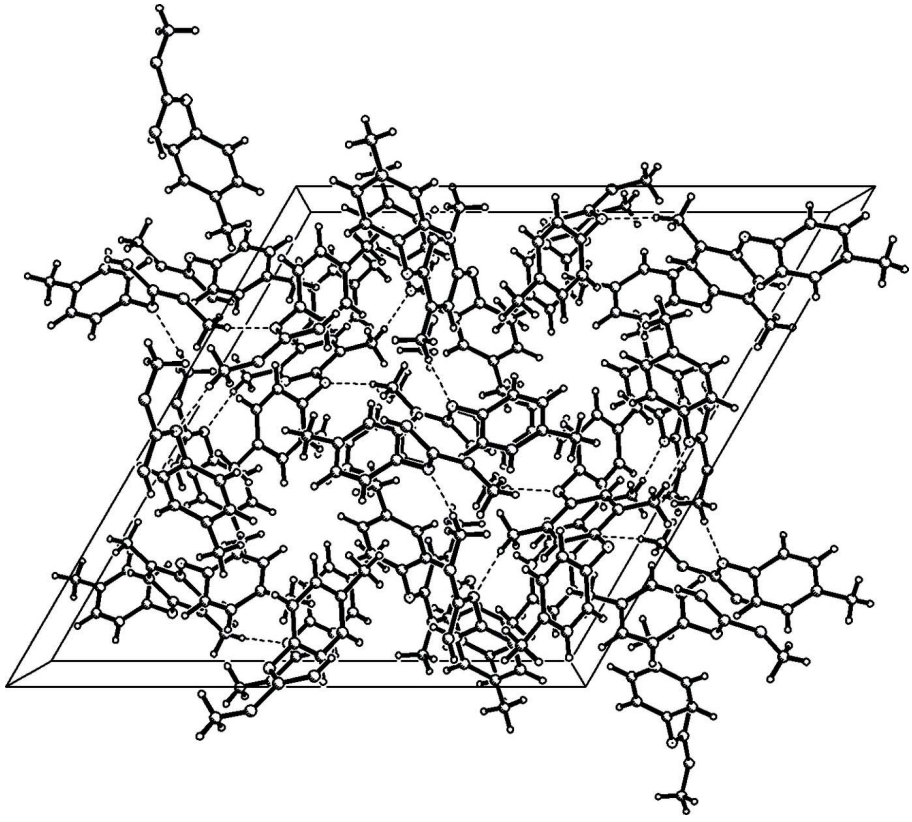

Figure 2

The crystal packing of (I),viewed down the *c* axis.

(6E)-*N*-[(4*Z*)-2,5-Dimethyl-4-(*p*-tolylimino)cyclohexa-2,5-dienylidene]-4- methylaniline

Crystal data

$C_{22}H_{22}N_2$
M_r = 314.42
Trigonal, $R\bar{3}$
Hall symbol: -R 3
a = 21.173 (8) Å
c = 10.476 (2) Å
V = 4067 (2) Å^3
Z = 9
F(000) = 1512
D_x = 1.155 Mg m^{-3}
Mo *Kα* radiation, λ = 0.71073 Å
Cell parameters from 25 reflections

θ = 4–14°
μ = 0.07 mm^{-1}
T = 293 K

Block, red
0.21 × 0.18 × 0.15 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
6148 measured reflections
1956 independent reflections
793 reflections with $I > 2\sigma(I)$

R_{int} = 0.075
θ_{max} = 27.0°, θ_{min} = 1.9°
h = −26→26
k = −26→26
l = −12→0
3 standard reflections every 100 reflections
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.072
$wR(F^2)$ = 0.233
S = 1.02
1956 reflections
110 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1023P)^2 + 1.7438P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}
Extinction correction: *SHELXL*, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0029 (9)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
N1	0.17771 (14)	0.28153 (14)	0.0673 (3)	0.0845 (9)
C1	−0.0660 (2)	0.1028 (3)	0.3942 (4)	0.1275 (17)
H1B	−0.0769	0.1330	0.4474	0.191*
H1C	−0.0529	0.0740	0.4468	0.191*
H1D	−0.1081	0.0712	0.3441	0.191*
C2	−0.00306 (19)	0.1504 (2)	0.3064 (3)	0.0882 (11)
C3	0.0232 (2)	0.1207 (2)	0.2204 (5)	0.1262 (16)
H3A	0.0010	0.0702	0.2149	0.151*
C4	0.0822 (2)	0.1636 (2)	0.1401 (5)	0.1197 (15)
H4A	0.0988	0.1413	0.0836	0.144*
C5	0.11505 (18)	0.23690 (19)	0.1441 (3)	0.0743 (9)
C6	0.0891 (2)	0.2665 (2)	0.2299 (4)	0.1182 (15)

H6A	0.1111	0.3170	0.2358	0.142*
C7	0.0306 (3)	0.2232 (3)	0.3089 (4)	0.1189 (15)
H7A	0.0141	0.2456	0.3656	0.143*
C8	0.10079 (16)	0.28723 (16)	−0.1018 (3)	0.0740 (9)
H8A	0.0579	0.2564	−0.0583	0.089*
C9	0.16973 (16)	0.30428 (16)	−0.0440 (3)	0.0707 (9)
C10	0.23710 (16)	0.35227 (17)	−0.1164 (3)	0.0730 (9)
C11	0.30536 (15)	0.36974 (17)	−0.0619 (3)	0.0810 (10)
H11A	0.3439	0.4007	−0.1192	0.122*
H11B	0.3068	0.3257	−0.0474	0.122*
H11C	0.3115	0.3946	0.0178	0.122*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0729 (18)	0.093 (2)	0.092 (2)	0.0442 (16)	−0.0040 (15)	0.0096 (17)
C1	0.103 (3)	0.172 (4)	0.109 (3)	0.070 (3)	0.021 (3)	0.052 (3)
C2	0.082 (2)	0.112 (3)	0.081 (2)	0.055 (2)	−0.003 (2)	0.018 (2)
C3	0.105 (3)	0.081 (3)	0.179 (4)	0.036 (2)	0.031 (3)	0.017 (3)
C4	0.111 (3)	0.089 (3)	0.150 (4)	0.043 (3)	0.028 (3)	−0.012 (3)
C5	0.071 (2)	0.078 (2)	0.081 (2)	0.0428 (19)	−0.0086 (18)	0.0033 (19)
C6	0.139 (4)	0.081 (3)	0.140 (4)	0.059 (3)	0.036 (3)	0.006 (3)
C7	0.142 (4)	0.104 (3)	0.115 (3)	0.064 (3)	0.043 (3)	0.011 (3)
C8	0.0603 (19)	0.075 (2)	0.090 (2)	0.0369 (16)	0.0000 (17)	0.0017 (18)
C9	0.070 (2)	0.068 (2)	0.082 (2)	0.0400 (17)	−0.0039 (18)	−0.0034 (17)
C10	0.065 (2)	0.074 (2)	0.086 (2)	0.0388 (17)	−0.0052 (17)	−0.0016 (17)
C11	0.0528 (18)	0.093 (2)	0.097 (2)	0.0360 (17)	0.0036 (17)	0.0140 (19)

Geometric parameters (Å, º)

N1—C9	1.305 (4)	C6—C7	1.388 (5)
N1—C5	1.430 (4)	C6—H6A	0.9300
C1—C2	1.516 (5)	C7—H7A	0.9300
C1—H1B	0.9600	C8—C10^{i}	1.360 (4)
C1—H1C	0.9600	C8—C9	1.449 (4)
C1—H1D	0.9600	C8—H8A	0.9300
C2—C7	1.336 (5)	C9—C10	1.481 (4)
C2—C3	1.365 (5)	C10—C8^{i}	1.360 (4)
C3—C4	1.399 (6)	C10—C11	1.420 (4)
C3—H3A	0.9300	C11—H11A	0.9600
C4—C5	1.347 (5)	C11—H11B	0.9600
C4—H4A	0.9300	C11—H11C	0.9600
C5—C6	1.359 (5)		
C9—N1—C5	119.9 (3)	C5—C6—H6A	119.2
C2—C1—H1B	109.5	C7—C6—H6A	119.2
C2—C1—H1C	109.5	C2—C7—C6	122.4 (4)
H1B—C1—H1C	109.5	C2—C7—H7A	118.8

C2—C1—H1D	109.5	C6—C7—H7A	118.8
H1B—C1—H1D	109.5	C10^{i}—C8—C9	122.7 (3)
H1C—C1—H1D	109.5	C10^{i}—C8—H8A	118.6
C7—C2—C3	116.0 (4)	C9—C8—H8A	118.6
C7—C2—C1	122.6 (4)	N1—C9—C8	125.6 (3)
C3—C2—C1	121.3 (4)	N1—C9—C10	116.9 (3)
C2—C3—C4	122.3 (4)	C8—C9—C10	117.5 (3)
C2—C3—H3A	118.8	C8^{i}—C10—C11	121.6 (3)
C4—C3—H3A	118.8	C8^{i}—C10—C9	119.8 (3)
C5—C4—C3	120.6 (4)	C11—C10—C9	118.6 (3)
C5—C4—H4A	119.7	C10—C11—H11A	109.5
C3—C4—H4A	119.7	C10—C11—H11B	109.5
C4—C5—C6	117.2 (4)	H11A—C11—H11B	109.5
C4—C5—N1	121.2 (3)	C10—C11—H11C	109.5
C6—C5—N1	121.5 (3)	H11A—C11—H11C	109.5
C5—C6—C7	121.5 (4)	H11B—C11—H11C	109.5

Symmetry code: (i) $-x+1/3, -y+2/3, -z-1/3$.