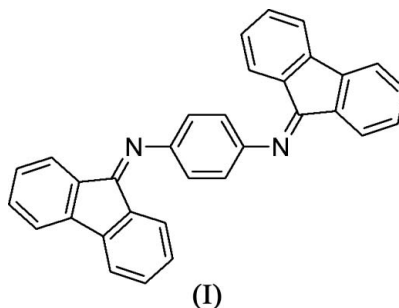


***N,N'*-Bis(9*H*-fluoren-9-ylidene)benzene-1,4-diamine**

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Key indicatorsSingle-crystal X-ray study
 $T = 298$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.038
 wR factor = 0.102
Data-to-parameter ratio = 14.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.Molecules of the title compound, $\text{C}_{32}\text{H}_{20}\text{N}_2$, synthesized by the *p*-toluenesulfonic acid-assisted Schiff base reaction between 9-fluorenone and *p*-phenylenediamine, are located on centers of inversion.Received 22 March 2006
Accepted 24 March 2006**Comment**Diimines derived from *p*-phenylenediamine and 9-fluorenone derivatives have been synthesized to study methodologies of imine synthesis (Reddeslien, 1910; Taylor & Fletcher, 1956), the biological effects of these compounds (Abou-Gharbia *et al.*, 1978), and their uses as potential dyes (Kubo *et al.*, 1973).

The title compound, (I) (Fig. 1), was synthesized to study the spatial relationship between the two fluoren-9-ylidene units with respect to the central aromatic ring (derived from *p*-phenylenediamine). A similar compound, *N,N'*-bis(9*H*-fluoren-9-ylidene)benzene-1,2-diamine, was found to be a wholly aromatic chiral diimine that owes its chirality to the *trans* orientation of the two fluoren-9-ylidene units with respect to the central aromatic ring (Glagovich *et al.*, 2005); the steric bulk of the two fluoren-9-ylidene units precludes the two groups from adopting a *syn* geometry. It was expected that the remoteness of the two fluoren-9-ylidene units in the title compound would prevent steric interaction with each other, and therefore not require a *trans* orientation of the two units. Regardless, the title compound would be achiral. The crystal structure of (I) reveals that a *trans* orientation of the groups attached to the central ring is still preferred, at least in the solid state.

Compound (I) crystallizes with the molecule lying on an inversion center. To avoid unfavorable steric interactions between H atoms on atoms C3 and C16, the fluoren-9-ylidene substituent ring system makes a dihedral angle of $65.09(4)^\circ$ with respect to the central aromatic ring. The C1–N1–C14 angle of $121.03(9)^\circ$ and the N1–C1 bond distance of $1.2830(12)$ Å are in close agreement with the four published structures of similar molecules (Glagovich *et al.*, 2004*a,b,c*, 2005).

Experimental

In a 100 ml round-bottomed flask equipped with a Hickman still and a reflux condenser were combined *p*-phenylenediamine (0.273 g, 2.5 mmol), 9-fluorenone (0.75 g, 4.2 mmol), *p*-toluenesulfonic acid (0.0004 g, 2.3 μmol) and toluene (20 ml). The resulting mixture was heated with refluxing for 72 h. After this time, the orange solution was concentrated under reduced pressure, producing an orange solid. The solid was purified by flash chromatography (SiO₂, 80% hexanes/10% ethyl acetate/10% CH₂Cl₂) which yielded 0.54 g of *N,N'*-bis(9*H*-fluoren-9-ylidene)benzene-1,4-diamine as a bright-orange solid (30%).

Crystal data

C₃₂H₂₀N₂ *D*_x = 1.300 Mg m⁻³
*M*_r = 432.50 Mo *K*α radiation
 Monoclinic, *P*2₁/*c* Cell parameters from 5041 reflections
a = 5.1360 (3) Å *θ* = 3.9–29.8°
b = 14.6772 (10) Å *μ* = 0.08 mm⁻¹
c = 15.0469 (9) Å *T* = 298 (2) K
β = 103.154 (5)° Plate, orange
V = 1104.51 (12) Å³ 0.55 × 0.30 × 0.20 mm
Z = 2

Data collection

Oxford Diffraction Excalibur 8876 measured reflections
 diffractometer with Sapphire3 2844 independent reflections
 detector 2225 reflections with *I* > 2σ(*I*)
ω scans *R*_{int} = 0.014
 Absorption correction: multi-scan *θ*_{max} = 29.8°
 (*CrysAlis RED*; Oxford *h* = -6 → 6
 Diffraction, 2005) *k* = -19 → 20
*T*_{min} = 0.991, *T*_{max} = 1.007 *l* = -20 → 19

Refinement

Refinement on *F*² *w* = 1/[σ²(*F*_o²) + (0.0548*P*)²
R[*F*² > 2σ(*F*²)] = 0.038 + 0.0927*P*
wR(*F*²) = 0.102 where *P* = (*F*_o² + 2*F*_c²)/3
S = 1.07 (Δ/*σ*)_{max} = 0.010
 2844 reflections Δ*ρ*_{max} = 0.18 e Å⁻³
 194 parameters Δ*ρ*_{min} = -0.15 e Å⁻³
 All H-atom parameters refined

Table 1

Selected geometric parameters (Å, °).

| | | | |
|---------------|-------------|--------|-------------|
| C1–N1 | 1.2830 (12) | C14–N1 | 1.4179 (12) |
| C1–N1–C14 | 121.03 (9) | | |
| C15–C14–N1–C1 | 122.90 (11) | | |

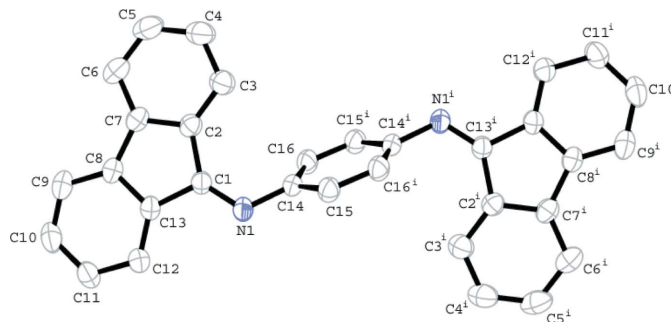


Figure 1

A view of (I). Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity. [Symmetry code: (i) -*x*, 1 - *y*, 1 - *z*.]

H atoms were found in difference maps and were refined isotropically [*C*–*H* = 0.954 (15)–1.015 (15) Å].

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2005); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2005); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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