

***N*-9*H*-Fluoren-9-ylidene-3,4-dimethylaniline**Neil M. Glagovich,<sup>a</sup> Elizabeth M. Reed,<sup>a</sup> Guy Crundwell,<sup>a\*</sup> James B. Updegraff III,<sup>b</sup> Matthias Zeller<sup>b</sup> and Allen D. Hunter<sup>b</sup><sup>a</sup>Department of Chemistry, Central Connecticut State University, New Britain, CT 06053, USA, and <sup>b</sup>Department of Chemistry, Youngstown State University, One University Plaza, Youngstown, Ohio 44555-3663, USACorrespondence e-mail:  
crundwellg@mail.ccsu.edu**Key indicators**Single-crystal X-ray study  
*T* = 100 K  
Mean  $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$   
*R* factor = 0.054  
*wR* factor = 0.147  
Data-to-parameter ratio = 18.5For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.The title compound, C<sub>21</sub>H<sub>17</sub>N, was synthesized by the *p*-toluenesulfonic acid-assisted Schiff base reaction between 9-fluorenone and 3,4-dimethylaniline. The crystal structure has been determined at 100 K.

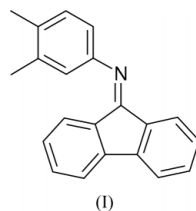
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**Comment**

Our interest in imines results from the potential use of chiral amines as auxiliaries in the separation of racemic mixtures of aldehydes and ketones. Chiral amines can form Schiff bases with racemic aldehydes and ketones to form diastereomeric mixtures of imines. These diastereomers can potentially be resolved by either flash chromatography or differential recrystallization.

To test the ease with which imines can be formed, a series of imines were made by reacting various aromatic amines with 9-fluorenone. A previous study has shown that an aromatic amine with an electron-donating group forms the imine with 9-fluorenone readily and in good yield (Glagovich *et al.*, 2004). The title compound, (I), was made in order to test what effects disubstitution of the amines will have on imine formation.The title compound crystallized with molecules in general positions. The planes defined by the dimethylphenyl and the fluorenylidene moieties are nearly at right angles to each other [89.61 (3)°]. An *ORTEP*-3 (Farrugia, 1997) diagram of (I) is shown in Fig. 1.**Experimental**In a 100 ml round-bottomed flask equipped with a Hickman still and reflux condenser, 2.5 g (14 mmol) 9-fluorenone, 2.04 g (16.8 mmol) 3,4-dimethylaniline, 0.014 g (74 mmol) *p*-toluenesulfonic acid, and 50 ml toluene were combined. The resulting mixture was heated under reflux for 24 h. After this time, the orange solution was concentrated under reduced pressure to produce an orange solid. The solid was purified by flash chromatography (SiO<sub>2</sub>, 80% hexanes–ethyl acetate) which produced 3.36 g of (I) as an orange solid (87%). *R<sub>F</sub>* 0.59 (SiO<sub>2</sub>, 80% hexanes–ethyl acetate); m.p. 355.8 K; IR (CHCl<sub>3</sub>) 3040, 3061, 3017, 1649, 1450, 1376, 1304, 790, 730, 650 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  7.92 (*d*, 1H, *J* = 7.4 Hz), 7.60 (*d*, 2H, *J* = 7.4 Hz), 7.46 (*t*, 1H, *J* = 7.4 Hz), 7.35 (*d*, 1H, *J* = 7.8 Hz), 7.32 (*d*, 1H, *J* = 7.5 Hz), 7.16 (*d*, 1H, *J* = 7.8 Hz), 6.95 (*t*, 1H, *J* = 7.5 Hz), 6.81 (*s*, 1H),

6.73 (*t*, 1H, *J* = 7.8 Hz), 2.33 (*s*, 3H), 2.29 (*s*, 3H); <sup>13</sup>C NMR (300 MHz, CDCl<sub>3</sub>): δ 162.65, 149.42, 143.73, 141.75, 137.62, 137.48, 132.05, 131.64, 131.55, 131.26, 130.30, 128.33, 127.59, 127.09, 123.21, 120.27, 120.09, 119.50, 115.50, 19.85, 19.29; UV/Vis (CH<sub>2</sub>Cl<sub>2</sub>; λ<sub>max</sub>, log ε) 404 nm, 2.685; MS calculated for C<sub>21</sub>H<sub>17</sub>N: M<sup>+</sup>: 283, measured: 283. Analysis calculated for C<sub>21</sub>H<sub>17</sub>N: C 89.01, H 6.05, N 4.94%; found: C 89.26, H 6.18, N 4.81.

Crystal data

|                                   |   |
|-----------------------------------|---|
| C <sub>21</sub> H <sub>17</sub> N | Z = 2                                     |
| M <sub>r</sub> = 283.36           | D <sub>x</sub> = 1.251 Mg m <sup>-3</sup> |
| Triclinic, P $\bar{1}$            | Mo K $\alpha$ radiation                   |
| a = 8.476 (3) Å                   | Cell parameters from 6350 reflections     |
| b = 8.870 (3) Å                   | θ = 2.3–28.4°                             |
| c = 10.376 (4) Å                  | μ = 0.07 mm <sup>-1</sup>                 |
| α = 78.886 (6)°                   | T = 100 (2) K                             |
| β = 79.375 (6)°                   | Parallelepiped, orange                    |
| γ = 89.483 (6)°                   | 0.40 × 0.34 × 0.10 mm                     |
| V = 752.1 (5) Å <sup>3</sup>      |   |

Data collection

|   |                                 |
|---|---------------------------------|
| Bruker SMART APEX diffractometer                            | 3722 independent reflections    |
| ω scans   | 3472 reflections with I > 2σ(I) |
| Absorption correction: multi-scan (SADABS; Sheldrick, 2003) | R <sub>int</sub> = 0.035        |
| T <sub>min</sub> = 0.967, T <sub>max</sub> = 0.995          | θ <sub>max</sub> = 28.5°        |
| 7401 measured reflections                                   | h = -11 → 11                    |
|   | k = -11 → 11                    |
|   | l = -13 → 13                    |

Refinement

|   |   |
|---|---|
| Refinement on F <sup>2</sup>                    | w = 1/[σ <sup>2</sup> (F <sub>o</sub> <sup>2</sup> ) + (0.0842P) <sup>2</sup> + 0.226P] |
| R[F <sup>2</sup> > 2σ(F <sup>2</sup> )] = 0.054 | where P = (F <sub>o</sub> <sup>2</sup> + 2F <sub>c</sub> <sup>2</sup> )/3               |
| wR(F <sup>2</sup> ) = 0.147                     | (Δ/σ) <sub>max</sub> < 0.001  |
| S = 1.03  | Δρ <sub>max</sub> = 0.49 e Å <sup>-3</sup>  |
| 3722 reflections                                | Δρ <sub>min</sub> = -0.28 e Å <sup>-3</sup>   |
| 201 parameters                                  |   |
| H-atom parameters constrained                   |   |

All H atoms were placed in calculated positions, with C—H distances of 0.95 and 0.98 Å (for aromatic and methyl H atoms, respectively), and were included in the refinement in the riding-model approximation, with U<sub>iso</sub>(H) = 1.2U<sub>eq</sub>(carrier atom).

Data collection: SMART (Bruker, 1997–1999); cell refinement: SMART; data reduction: SAINT-Plus (Bruker, 1997–1999); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997);

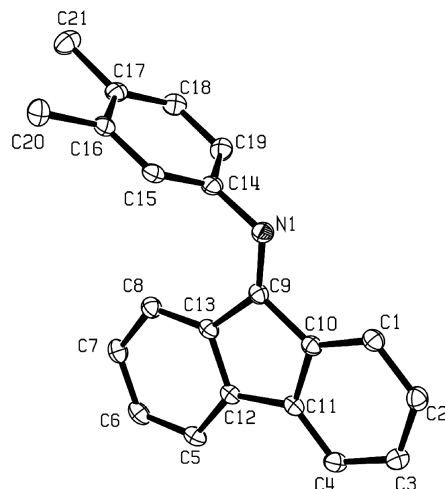


Figure 1

A view of (I) (Farrugia, 1997). Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity.

molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

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