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sp-9-(*meta*-Methylphenyl)-9-fluorenol

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The title compound, $C_{20}H_{16}O$, (I), whose 9-aryl group is freely rotating in solution, crystallizes exclusively as its *sp* rotamer, which exhibits intermolecular hydrogen bonding involving $O—H\cdots\pi$(fluorene) but not involving $O—H\cdots OH$. The molecules pack as centrosymmetric dimers incorporating two $O—H\cdots\pi$(fluorene) hydrogen bonds. Although (I) melted sharply without decomposition, its melt failed to recrystallize on cooling.

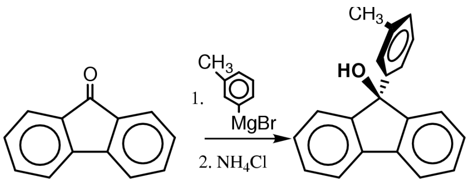
Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.004 Å
R factor = 0.045
wR factor = 0.142
Data-to-parameter ratio = 13.6

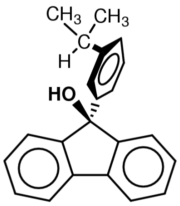
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Our current results show that crystalline 9-(*meta*-methylphenyl)-9-fluorenol, (I), exhibits properties very similar to those of 9-(*meta*-*tert*-butylphenyl)-9-fluorenol, (II), which we recently reported (Meyers *et al.*, 2003). Both crystallize as their *sp* rotamers exclusively, although NMR clearly shows that their aryl moieties rotate freely in solution; both display intermolecular $O—H\cdots\pi$(fluorene) hydrogen bonding, but no $O—H\cdots OH$ hydrogen bonding. Also, both compounds melt sharply without decomposition, although their melts fail to recrystallize on cooling over an extended time.



(I)



(II)

The structure of (I), with its atom numbering, is shown in Fig. 1. The angle between the least-squares fluorene and subsituted phenyl planes is 87.38 (10)°. No unusual inter- or intramolecular geometric parameters were noted. Fig. 2 shows the $O—H\cdots\pi$(fluorene) hydrogen-bonding scheme and Table 1 lists the hydrogen-bond geometry. Two molecules are related by an inversion center and dimerize as a result of the hydrogen-bond interactions. The two crystallographically equivalent hydrogen bonds linking the dimerized molecules are drawn to C1 since it is in closest proximity to H1, which is readily apparent in the molecular packing (Fig. 3).

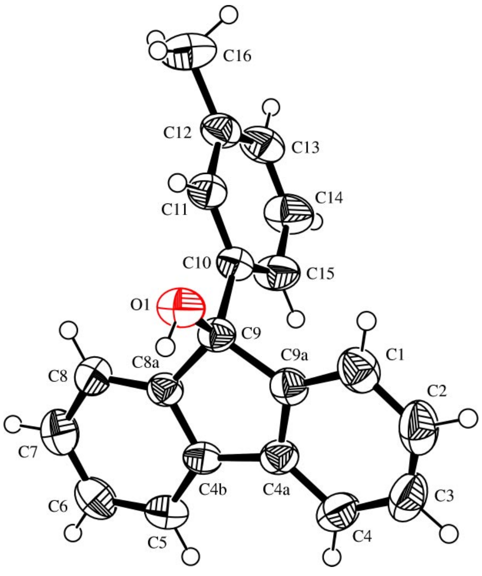


Figure 1
The molecular structure and atom-numbering scheme for (I), with displacement ellipsoids at the 50% probability level.

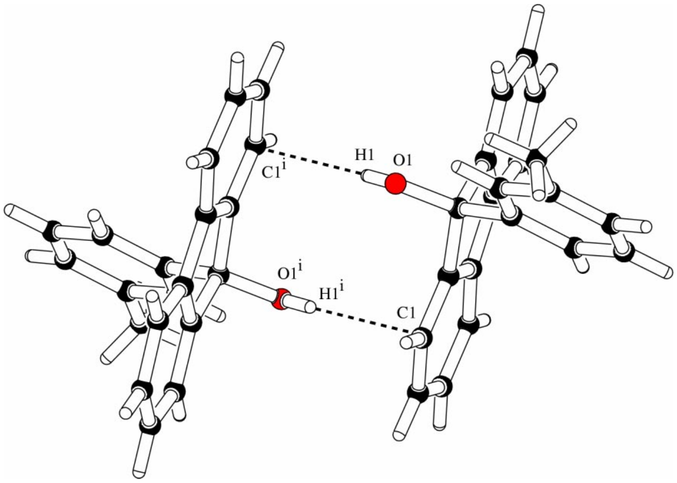


Figure 2
The O—H···π(fluorene) hydrogen-bonding scheme in (I). [Symmetry code: (i) −x, −y, −z.]

Experimental

Compound (I) was prepared by heating a mixture of magnesium turnings (0.2872 g, 11.80 mmol), freshly distilled tetrahydrofuran (15 ml) and dibromoethane (0.30 ml, 3.48 mmol) in a flame-dried flask purged with argon, adding *m*-bromotoluene (2.0315 g, 11.89 mmol) when bubbles appeared on the surface of the magnesium turnings, stirring the mixture under reflux until all of the magnesium was consumed (*ca* 30 min), and then adding a solution of 9-fluorenone (1.0032 g, 5.573 mmol) in tetrahydrofuran (20 ml). The yellow–brown mixture was refluxed for 7 h, after which water followed by saturated aqueous ammonium chloride solution were added. The mixture was extracted with ether and the combined extracts were dried ($MgSO_4$) and concentrated *in vacuo*, leaving a tan oil which solidified on standing for several days; 1.281 g, 84.5% yield. Recrystallization from hexanes provided colorless crystals, m.p 353–354.5 K [literature (Cockerill & Lamper, 1971) m.p. 355–356 K]; the melt did not recrystallize on cooling. NMR ($CDCl_3$), ^{1}H: δ 2.29 (*s*, 3H), 2.41 (*s*, 1H), 7.02–7.06 (*m*, 1H), 7.14–7.16 (*m*, 2H), 7.22–7.25 (*m*, 3H), 7.32–7.39 (*m*, 4H), 7.65–7.69 (*m*, 2H); ^{13}C: δ 21.56, 67.15, 120.07, 122.52, 124.77, 125.94, 128.05, 128.11, 128.44, 129.05, 137.82, 139.59, 143.02, 150.49.

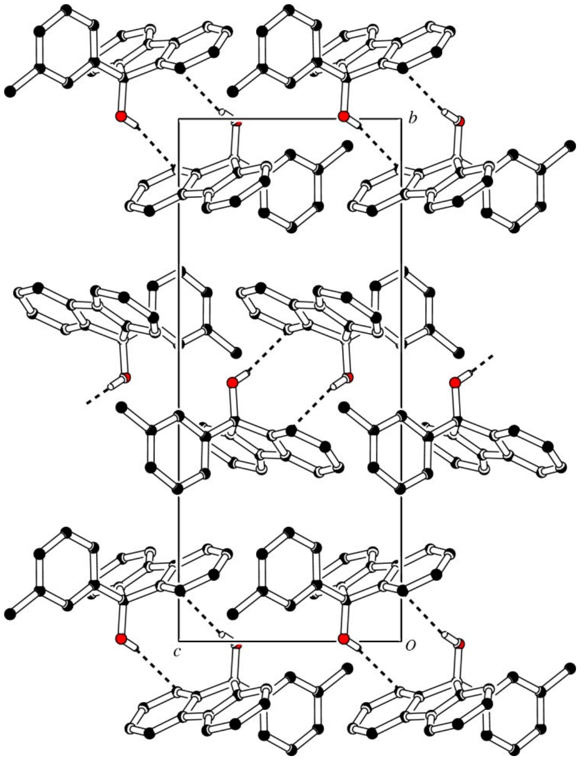


Figure 3
The molecular packing in (I), viewed down the *a** axis. Most H atoms have been omitted for clarity.

Crystal data

$C_{20}H_{16}O$
$M_r = 272.33$
Monoclinic, $P2_1/c$
$a = 9.1443\ (12)$ Å
$b = 19.4498\ (14)$ Å
$c = 8.6651\ (10)$ Å
$\beta = 106.989\ (9)^\circ$
$V = 1473.9\ (3)$ Å^3
$Z = 4$

$D_x = 1.227$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 16.0$–20.0°
$\mu = 0.07$ mm^{-1}
$T = 296$ K
Prism, colorless
0.42 × 0.34 × 0.21 mm

Data collection

Rigaku AFC-5*S* diffractometer
ω scans
Absorption correction: none
2804 measured reflections
2620 independent reflections
1500 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.027$

$\theta_{\max} = 25.1^\circ$
$h = -10 \rightarrow 10$
$k = -23 \rightarrow 0$
$l = 0 \rightarrow 10$
3 standard reflections every 100 reflections intensity decay: 0.3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.142$
$S = 1.03$
2620 reflections
192 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0675P)^2 + 0.2168P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.16$ e Å^{-3}
$\Delta\rho_{\min} = -0.23$ e Å^{-3}

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Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···Cl^{i}	0.82	2.68	3.486 (3)	167

Symmetry code: (i) $-x, -y, -z$.

The rotational orientations of the methyl and hydroxyl groups were determined by the circular Fourier refinement methods available in *SHELXL*97 (Sheldrick, 1997). All H atoms were treated as riding with an O—H distance of 0.82 Å and C—H distances in the range 0.93–0.96 Å. $U_{\rm iso}$ values for the riding H atoms equal 1.5 times (hydroxyl and methyl H atoms) or 1.2 times (all other H atoms) the $U_{\rm eq}$ of the parent atom.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1996); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *PROCESS* in *TEXSAN* (Molecular Structure Corporation, 1997); program(s) used to solve structure: *SIR*92 (Burla *et al.*, 1989); program(s) used to refine structure: *LS* in *TEXSAN* and *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2000); software used to prepare material for publication: *TEXSAN, SHELXL*97 and *PLATON* (Spek, 2000).

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