Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

9-(*p*-Methylphenyl)fluorene

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Received 24 November 2003
Accepted 5 December 2003
Online 12 December 2003

Key indicators

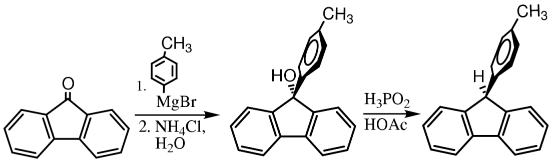
Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.006 Å
R factor = 0.038
wR factor = 0.117
Data-to-parameter ratio = 8.2

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

The title compound, $C_{20}H_{16}$, displays no unusual structural characteristics. The aryl and fluorenyl ring planes form an angle of 78.26 (14)°. Neither intermolecular hydrogen bonding nor aryl–H$\cdots\pi$(arene) interactions are exhibited, which conforms with the rapid recrystallization of the melted crystals on cooling.

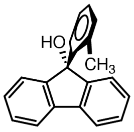
Comment

9-(*p*-Methylphenyl)fluorene, (II), was prepared from its precursor, 9-(*p*-methylphenyl)-9-fluorenol, (I) (Robinson *et al.*, 2003).



(I)

(II)



(III)

The structure of crystalline (II) with its atom numbering is shown in Fig. 1. Its geometric parameters are not grossly different from those of related fluorenes we have studied. For example, the angle between the aryl and fluorene ring planes of (II) is 78.26 (14)°, indicating a conformation in line with that generally exhibited by related fluorenes not affected by steric or rotational influences. In this connection, it should be noted that related 9-(*o*-methylphenyl)-9-fluorenol, (III), is unusual in that its aryl and fluorene rings are perpendicular (Meyers *et al.*, 2003). (II) exhibits neither intermolecular hydrogen bonding nor noteworthy aryl–H$\cdots\pi$(arene) interaction. It has a sharp melting point and the melt recrystallizes immediately at room temperature. In contrast, its precursor 9-fluorenol (I) (Robinson *et al.*, 2003) exhibits intermolecular O—H$\cdots$O—H as well as O—H$\cdots\pi$(arene) hydrogen bonds, and its melt fails to recrystallize at room temperature even after a long period, even though the NMR spectra of the melt and crystals were identical, showing that no molecular decomposition had occurred on melting. However, although the related 9-(*o*-methylphenyl)-9-fluorenol, (III), displays no hydrogen bonding, it exhibits six intermolecular aryl–H...π(arene) interactions, and its melt also recrystallized only after many hours at room temperature (Meyers *et al.*, 2003). A recent report (Robinson *et al.*, 2003) briefly summarizes our series of 9-arylfluorenes and 9-aryl-9-fluorenols and suggests the possible relationship between those exhibiting inter-

DOI: 10.1107/S1600536803028095

molecular hydrogen bonding or intermolecular aryl–H$\cdots\pi$(arene) interactions (probably van der Waals forces) and the failure of their melts to recrystallize easily on being cooled to room temperature.

Experimental

A solution of I_2 (0.025 g, 0.098 mmol), 50% aqueous H_3PO_2 solution (1.4 ml, 13.52 mmol), and glacial acetic acid (10 ml) was stirred and heated under argon until it became colorless, and 9-(*p*-methylphenyl)-9-fluorenol (0.35 g, 1.29 mmol) (Robinson *et al.*, 2003) was then added. The mixture became yellow and was refluxed under argon for 5 h, then cooled, diluted with water and extracted with ether. The combined extracts were washed with saturated aq. sodium bicarbonate, dried ($MgSO_4$), and concentrated *in vacuo* to a pale yellow solid (0.314 g, 95% yield). Recrystallization (hexanes/methanol) afforded colorless crystals [m.p. 397–398 K; literature m.p. 376 K (Cockerill & Lamper, 1971)]. This discrepancy in melting points may be explained by an error in reporting by those authors. Thus, they tabulated the melting point of their 9-(*m*-methylphenyl)-fluorene) as 401 K, which is very close to our melting point of (II), and the melting point they noted for their (II), 376 K, was very close to our melting point of 373–374 K for 9-(*m*-methylphenyl)fluorene which we recently prepared. The melt of (II) recrystallized immediately on cooling and remelted at the same temperature. ^{1}H NMR ($CDCl_3$): δ 2.31 (*s*, 3H), 5.01 (*s*, 1 H), 6.96–6.99 (*m*, 2H), 7.06–7.09 (*m*, 2H), 7.22–7.40 (*m*, 6H), 7.78–7.80 (*m*, 2H). ^{13}C-NMR ($CDCl_3$): δ 21.1, 54.1, 119.8, 125.3, 127.2, 127.3, 128.2, 129.4, 136.4, 138.5, 141.0, 148.1.

Crystal data

$C_{20}H_{16}$
$M_r = 256.33$
Monoclinic, $P2_1$
$a = 9.8828$ (19) Å
$b = 5.8836$ (17) Å
$c = 12.5231$ (12) Å
$\beta = 95.684$ (10)°
$V = 724.6$ (3) Å^3
$Z = 2$

$D_x = 1.175$ Mg m^{-3}
Mo Kα radiation
Cell parameters from 25 reflections
$\theta = 10.1–13.5°$
$\mu = 0.07$ mm^{-1}
$T = 296$ K
Elongated prism, colorless
0.38 × 0.18 × 0.15 mm

Data collection

Rigaku AFC-5S diffractometer
ω scans
Absorption correction: none
1577 measured reflections
1489 independent reflections
881 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.024$

$\theta_{\rm max} = 25.6°$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 7$
$l = -15 \rightarrow 15$
3 standard reflections every 100 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.117$
$S = 1.06$
1489 reflections
182 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0538P)^2 + 0.0314P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.14$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.14$ e Å^{-3}

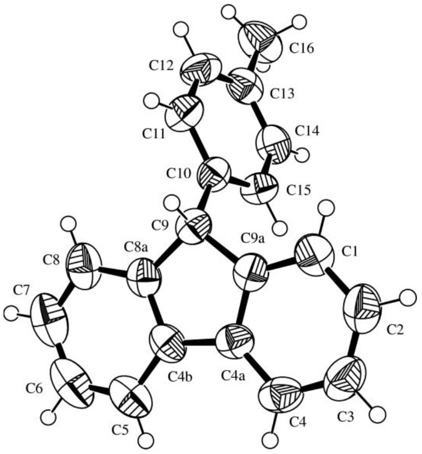


Figure 1
The molecular structure and atom-numbering scheme for (II), with displacement ellipsoids drawn at the 50% probablility level.

The rotational orientation of the methyl group was refined by the circular Fourier method available in *SHELXL*97 (Sheldrick, 1997). All H atoms were treated as riding with C—H distances ranging from 0.93 to 0.98 Å and $U_{\rm iso}$(H) values equal to 1.5 (methyl H atoms) or 1.2 (all other H atoms) times $U_{\rm eq}$ of the parent atom. The absolute configuration could not be determined because of the lack of significant anomalous scattering effects; Friedel pairs were merged.

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); software used to prepare material for publication: *TEXSAN, SHELXL*97, and *PLATON* (Spek, 2003).

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