Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5,11,11-Trimethyl-11*H*-benzo[*a*]fluorene

Shu-Hong Li[a] and Cai-Hong Xu[b]*

[a]School of Chemical and Environmental Engineering, Beijing Technology and Business University, Beijing 100037, People's Republic of China, and [b]Laboratory of Advanced Polymer Materials, Institute of Chemistry, Chinese Academy of Sciences, Beijing 100080, People's Republic of China
Correspondence e-mail: caihong@iccas.ac.cn

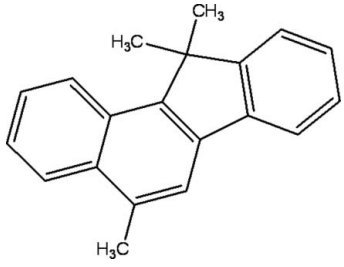
Received 21 May 2007; accepted 13 July 2007

Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.004 Å; R factor = 0.038; wR factor = 0.067; data-to-parameter ratio = 9.3.

The title compound, $C_{20}H_{18}$, contains four approximately coplanar fused rings: three six- and one five-membered. In the crystal structure, molecules form columnar stacks along the *a* axis. Molecules in adjacent stacks along the [010] direction are oriented approximately perpendicular to each other, with a dihedral angle of 81.38 (3)°. There is a short intermolecular C—H···C contact of 2.72 Å between a methyl group and an aromatic ring.

Related literature

For the synthesis of similar compounds, see: Mitra & Ray (1981); Bradsher & Burhans (1940).



Experimental

Crystal data

$C_{20}H_{18}$
M_r = 258.34
Orthorhombic, $Pca2_1$
a = 9.5632 (19) Å
b = 9.3715 (19) Å
c = 16.089 (3) Å
V = 1441.9 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 295 (2) K
0.31 × 0.26 × 0.22 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: none
3084 measured reflections
1702 independent reflections
1216 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.067
S = 1.07
1702 reflections
183 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.11 e Å^{-3}

Table 1
Short contact (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C20—H20A···C8^{i}	0.96	2.72	3.563 (4)	147

Symmetry code: (i) $x+\frac{1}{2}, -y+2, z$.

Data collection: *RAPID-AUTO* (Rigaku, 2001); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1994) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97*.

The authors thank Professor Ru-Ji Wang of Tsinghua University for his help.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2079).

References

Bradsher, C. K. & Burhans, A. S. (1940). *J. Am. Chem. Soc.* **62**, 3140–3141.
Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
Mitra, A. & Ray, R. M. (1981). *Indian J. Chem.* **20B**, 68–70.
Rigaku (2001). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (1994). *SHELXTL/PC*. Version 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

  doi:10.1107/S1600536807034320

supplementary materials

Acta Cryst. (2007). E**63**, o3658 [**doi:10.1107/S1600536807034320**]

5,11,11-Trimethyl-11*H*-benzo[*a*]fluorene

S.-H. Li and C.-H. Xu

Comment

During our investigation of new intramolecular reductive cyclization the title compound has been synthesized unexpectedly as the product of an attempted synthesis of 1,2 -bis (2-(2-(bromopropan-2-yl)phenyl)ethyne in the reaction of 2,2'-(2,2'-(ethyne-1,2-diyl)bis(2,1-phenylene)dipropan-2-ol and chlorotrimethylsilane catalyzed by LiBr in refluxed MeCN (Scheme 2). The molecule consists of four fused rings, three six-membered rings and one five-membered ring, which show a nearly planar structure (Fig. 1). Synthesis of a similar benzofluorene derivative has been reported previously (Mitra & Ray, 1981; Bradsher & Burhans, 1940)

In the crystal structure (Fig. 2), the molecules exhibit a T-packing mode with a few intermolecular CH_2—H···π interactions having H···π distances of about 2.7 Å.

Experimental

The mixture of 135 mg 2,2'-(2,2'-(ethyne-1,2-diyl)bis(2,1-phenylene)dipropan-2-ol, 0.3 ml chlorotrimethylsilane, 163.9 mg LiBr and 7 ml MeCN was refluxed for 34 h under nitrogen atomsphere. The mixture was extracted with Et_2O, the organic layer was washed with brine, dried over $MgSO_4$, filtered and concentrated under reduced pressure to give 163.7 mg of product in 85% yield. Recrystallization from hexane/ethyl acetate (4:1) gave a single-crystal which was used for X-ray analysis.

Refinement

H atoms were positioned geometrically and refined using a riding model with C—H bonds 0.93–0.96Å and with U_{iso}(H)=1.2U_{eq}(C) for benzene H atoms or U_{iso}(H)=1.5U_{eq}(C) for methyl groups. In the absence of signifcant anomalous scattering effects, Friedel pairs were averaged.

Figures

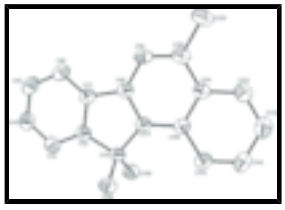


Fig. 1. The molecular structure of the title compound, with atom labels and 50% probability displacement ellipsoids for non-H atoms.

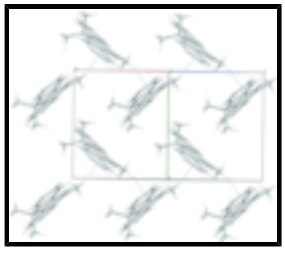


Fig. 2. The crystal packing of the title compound, view along the [101] direction.

Fig. 3. The formation of (I).

5,11,11-Trimethyl-11*H*-benzo[*a*]fluorene

Crystal data

$C_{20}H_{18}$	F_{000} = 552
M_r = 258.34	D_x = 1.190 Mg m^{-3}
Orthorhombic, $Pca2_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2c -2ac	Cell parameters from 3084 reflections
a = 9.5632 (19) Å	θ = 2.2–27.5º
b = 9.3715 (19) Å	μ = 0.07 mm^{-1}
c = 16.089 (3) Å	T = 295 (2) K
V = 1441.9 (5) Å^3	Platelet, colorless
Z = 4	0.31 × 0.26 × 0.22 mm

Data collection

Rigaku R-AXIS RAPID diffractometer	1216 reflections with $I > 2\sigma(I)$
Radiation source: Rotating anode	R_{int} = 0.019
Monochromator: graphite	θ_{max} = 27.5º
T = 295(2) K	θ_{min} = 2.2º
ω scans	h = −12→12
Absorption correction: none	k = −12→12
3084 measured reflections	l = −20→20
1702 independent reflections	

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.007P)^2 + 0.2P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.038	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.067	$\Delta\rho_{max}$ = 0.12 e Å^{-3}
S = 1.07	$\Delta\rho_{min}$ = −0.11 e Å^{-3}

1702 reflections

183 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0309 (11)

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 $F^2 > 2\text{sigma}(F^2)$ 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}
C1	0.3492 (3)	0.6555 (3)	0.74283 (17)	0.0494 (6)
C2	0.3398 (3)	0.7348 (3)	0.82441 (17)	0.0479 (6)
C3	0.2341 (3)	0.7335 (3)	0.88295 (17)	0.0604 (8)
H3A	0.1547	0.6778	0.8750	0.072*
C4	0.2484 (4)	0.8161 (3)	0.9532 (2)	0.0683 (8)
H4A	0.1783	0.8144	0.9933	0.082*
C5	0.3630 (3)	0.9005 (3)	0.9655 (2)	0.0710 (9)
H5A	0.3700	0.9562	1.0131	0.085*
C6	0.4688 (3)	0.9028 (3)	0.90671 (19)	0.0632 (8)
H6A	0.5471	0.9601	0.9143	0.076*
C7	0.4563 (3)	0.8193 (3)	0.83708 (17)	0.0493 (7)
C8	0.5481 (2)	0.8018 (3)	0.76460 (15)	0.0468 (6)
C9	0.6766 (2)	0.8676 (3)	0.74735 (19)	0.0543 (7)
H9A	0.7160	0.9291	0.7862	0.065*
C10	0.7438 (3)	0.8426 (2)	0.67483 (18)	0.0530 (7)
C11	0.6831 (3)	0.7487 (3)	0.61546 (17)	0.0486 (6)
C12	0.7497 (4)	0.7194 (3)	0.5390 (2)	0.0621 (7)
H12A	0.8336	0.7647	0.5264	0.075*
C13	0.6939 (3)	0.6266 (4)	0.4837 (2)	0.0734 (9)
H13A	0.7390	0.6099	0.4335	0.088*
C14	0.5699 (3)	0.5561 (4)	0.50155 (19)	0.0684 (9)
H14A	0.5338	0.4903	0.4639	0.082*
C15	0.5011 (3)	0.5827 (3)	0.57354 (18)	0.0576 (7)
H15A	0.4169	0.5363	0.5840	0.069*
C16	0.5541 (3)	0.6789 (3)	0.63294 (16)	0.0473 (6)

C17	0.4877 (2)	0.7098 (2)	0.70901 (16)	0.0433 (6)
C18	0.2245 (3)	0.6957 (3)	0.68713 (18)	0.0633 (8)
H18A	0.2254	0.7966	0.6769	0.095*
H18B	0.1389	0.6699	0.7144	0.095*
H18C	0.2314	0.6455	0.6353	0.095*
C19	0.3511 (3)	0.4956 (3)	0.75899 (18)	0.0643 (8)
H19A	0.2629	0.4667	0.7820	0.096*
H19B	0.4246	0.4733	0.7975	0.096*
H19C	0.3670	0.4458	0.7077	0.096*
C20	0.8835 (3)	0.9111 (3)	0.6573 (2)	0.0769 (10)
H20A	0.9137	0.9633	0.7054	0.115*
H20B	0.8747	0.9751	0.6110	0.115*
H20C	0.9509	0.8385	0.6444	0.115*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0484 (14)	0.0450 (14)	0.0547 (16)	−0.0066 (11)	−0.0079 (13)	0.0020 (13)
C2	0.0528 (14)	0.0417 (13)	0.0491 (14)	0.0040 (12)	−0.0030 (13)	0.0052 (13)
C3	0.0603 (18)	0.0589 (17)	0.0620 (18)	0.0018 (15)	0.0006 (15)	0.0069 (17)
C4	0.078 (2)	0.0657 (17)	0.0608 (17)	0.0128 (19)	0.0070 (16)	0.0010 (17)
C5	0.087 (2)	0.0637 (19)	0.0619 (19)	0.0132 (18)	−0.0064 (19)	−0.0148 (17)
C6	0.0653 (19)	0.0585 (18)	0.0660 (19)	0.0021 (15)	−0.0086 (16)	−0.0107 (16)
C7	0.0539 (15)	0.0404 (13)	0.0537 (16)	0.0041 (12)	−0.0081 (14)	−0.0004 (14)
C8	0.0463 (13)	0.0389 (13)	0.0553 (17)	0.0026 (11)	−0.0093 (13)	0.0006 (13)
C9	0.0502 (15)	0.0397 (14)	0.0730 (19)	−0.0017 (11)	−0.0102 (15)	−0.0076 (14)
C10	0.0452 (14)	0.0392 (13)	0.075 (2)	0.0031 (13)	−0.0024 (14)	0.0029 (14)
C11	0.0494 (14)	0.0383 (13)	0.0580 (16)	0.0053 (11)	−0.0028 (13)	0.0057 (13)
C12	0.0584 (16)	0.0615 (18)	0.0664 (17)	0.0051 (16)	0.0034 (15)	0.0059 (17)
C13	0.076 (2)	0.086 (2)	0.059 (2)	0.0133 (19)	0.0049 (17)	−0.0053 (19)
C14	0.075 (2)	0.074 (2)	0.0560 (18)	0.0074 (19)	−0.0087 (17)	−0.0119 (16)
C15	0.0607 (16)	0.0585 (16)	0.0535 (16)	−0.0005 (15)	−0.0084 (14)	−0.0052 (15)
C16	0.0485 (14)	0.0424 (13)	0.0510 (16)	0.0040 (12)	−0.0097 (13)	0.0028 (13)
C17	0.0453 (13)	0.0359 (12)	0.0488 (14)	0.0014 (11)	−0.0078 (12)	0.0030 (13)
C18	0.0505 (15)	0.0747 (18)	0.065 (2)	−0.0061 (14)	−0.0101 (14)	0.0042 (17)
C19	0.0790 (18)	0.0483 (14)	0.0657 (19)	−0.0099 (14)	0.0001 (16)	0.0009 (16)
C20	0.0545 (17)	0.0645 (19)	0.112 (3)	−0.0106 (15)	0.0074 (18)	−0.007 (2)

Geometric parameters (Å, °)

C1—C2	1.511 (3)	C11—C12	1.412 (4)
C1—C17	1.519 (3)	C11—C16	1.424 (3)
C1—C19	1.522 (4)	C12—C13	1.353 (4)
C1—C18	1.539 (3)	C12—H12A	0.9300
C2—C7	1.382 (4)	C13—C14	1.387 (4)
C2—C3	1.382 (4)	C13—H13A	0.9300
C3—C4	1.377 (4)	C14—C15	1.355 (4)
C3—H3A	0.9300	C14—H14A	0.9300
C4—C5	1.366 (4)	C15—C16	1.409 (4)

C4—H4A	0.9300	C15—H15A	0.9300
C5—C6	1.385 (4)	C16—C17	1.409 (3)
C5—H5A	0.9300	C18—H18A	0.9600
C6—C7	1.372 (4)	C18—H18B	0.9600
C6—H6A	0.9300	C18—H18C	0.9600
C7—C8	1.469 (3)	C19—H19A	0.9600
C8—C17	1.370 (3)	C19—H19B	0.9600
C8—C9	1.402 (3)	C19—H19C	0.9600
C9—C10	1.353 (4)	C20—H20A	0.9600
C9—H9A	0.9300	C20—H20B	0.9600
C10—C11	1.422 (4)	C20—H20C	0.9600
C10—C20	1.509 (3)		
C2—C1—C17	101.4 (2)	C13—C12—C11	121.3 (3)
C2—C1—C19	109.7 (2)	C13—C12—H12A	119.4
C17—C1—C19	112.4 (2)	C11—C12—H12A	119.4
C2—C1—C18	109.9 (2)	C12—C13—C14	120.5 (3)
C17—C1—C18	112.7 (2)	C12—C13—H13A	119.7
C19—C1—C18	110.5 (2)	C14—C13—H13A	119.7
C7—C2—C3	119.6 (3)	C15—C14—C13	120.2 (3)
C7—C2—C1	111.2 (2)	C15—C14—H14A	119.9
C3—C2—C1	129.1 (3)	C13—C14—H14A	119.9
C4—C3—C2	118.8 (3)	C14—C15—C16	121.5 (3)
C4—C3—H3A	120.6	C14—C15—H15A	119.2
C2—C3—H3A	120.6	C16—C15—H15A	119.2
C5—C4—C3	121.6 (3)	C17—C16—C15	124.0 (2)
C5—C4—H4A	119.2	C17—C16—C11	117.9 (2)
C3—C4—H4A	119.2	C15—C16—C11	118.2 (2)
C4—C5—C6	119.8 (3)	C8—C17—C16	120.4 (2)
C4—C5—H5A	120.1	C8—C17—C1	110.2 (2)
C6—C5—H5A	120.1	C16—C17—C1	129.4 (2)
C7—C6—C5	119.0 (3)	C1—C18—H18A	109.5
C7—C6—H6A	120.5	C1—C18—H18B	109.5
C5—C6—H6A	120.5	H18A—C18—H18B	109.5
C6—C7—C2	121.2 (3)	C1—C18—H18C	109.5
C6—C7—C8	131.3 (3)	H18A—C18—H18C	109.5
C2—C7—C8	107.5 (2)	H18B—C18—H18C	109.5
C17—C8—C9	121.1 (2)	C1—C19—H19A	109.5
C17—C8—C7	109.7 (2)	C1—C19—H19B	109.5
C9—C8—C7	129.2 (2)	H19A—C19—H19B	109.5
C10—C9—C8	120.7 (3)	C1—C19—H19C	109.5
C10—C9—H9A	119.6	H19A—C19—H19C	109.5
C8—C9—H9A	119.6	H19B—C19—H19C	109.5
C9—C10—C11	119.5 (2)	C10—C20—H20A	109.5
C9—C10—C20	120.5 (3)	C10—C20—H20B	109.5
C11—C10—C20	119.9 (3)	H20A—C20—H20B	109.5
C12—C11—C10	121.4 (3)	C10—C20—H20C	109.5
C12—C11—C16	118.2 (2)	H20A—C20—H20C	109.5
C10—C11—C16	120.3 (2)	H20B—C20—H20C	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C20—H20A···C8^{i}	0.96	2.72	3.563 (4)	147

Symmetry codes: (i) *x*+1/2, −*y*+2, *z*.

Fig. 1

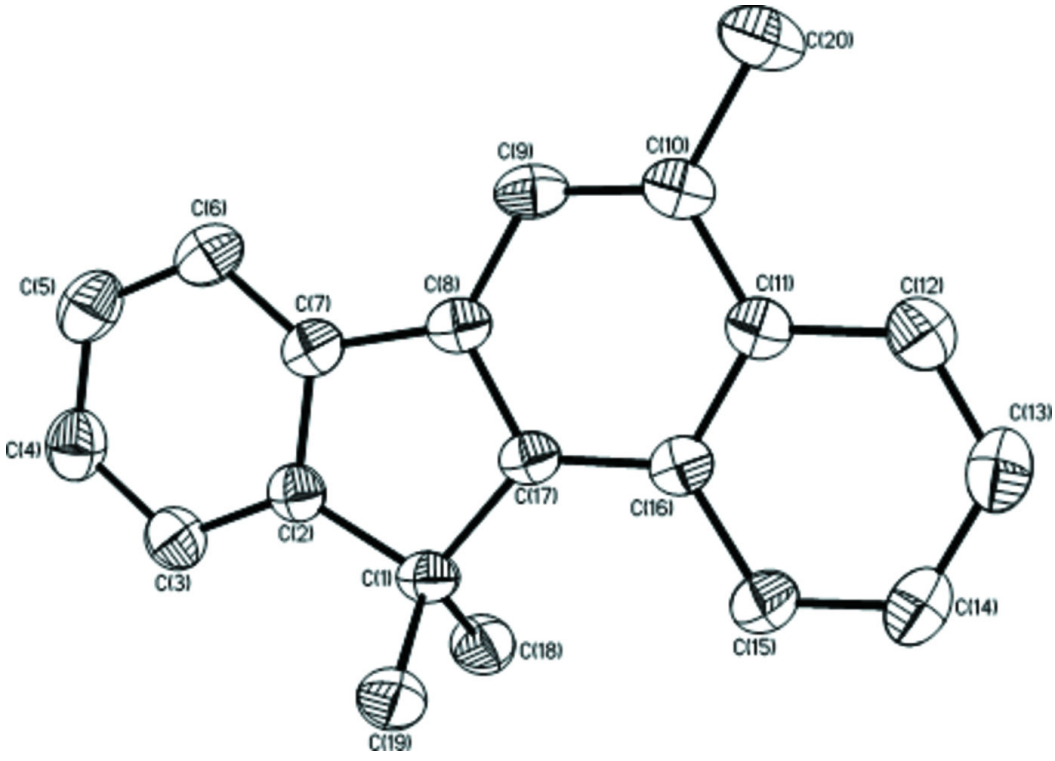

C(20)
C(10)
C(9)
C(6)
C(5)
C(7)
C(8)
C(11)
C(12)
C(13)
C(4)
C(17)
C(16)
C(2)
C(3)
C(1)
C(14)
C(15)
C(18)
C(19)

Fig. 2

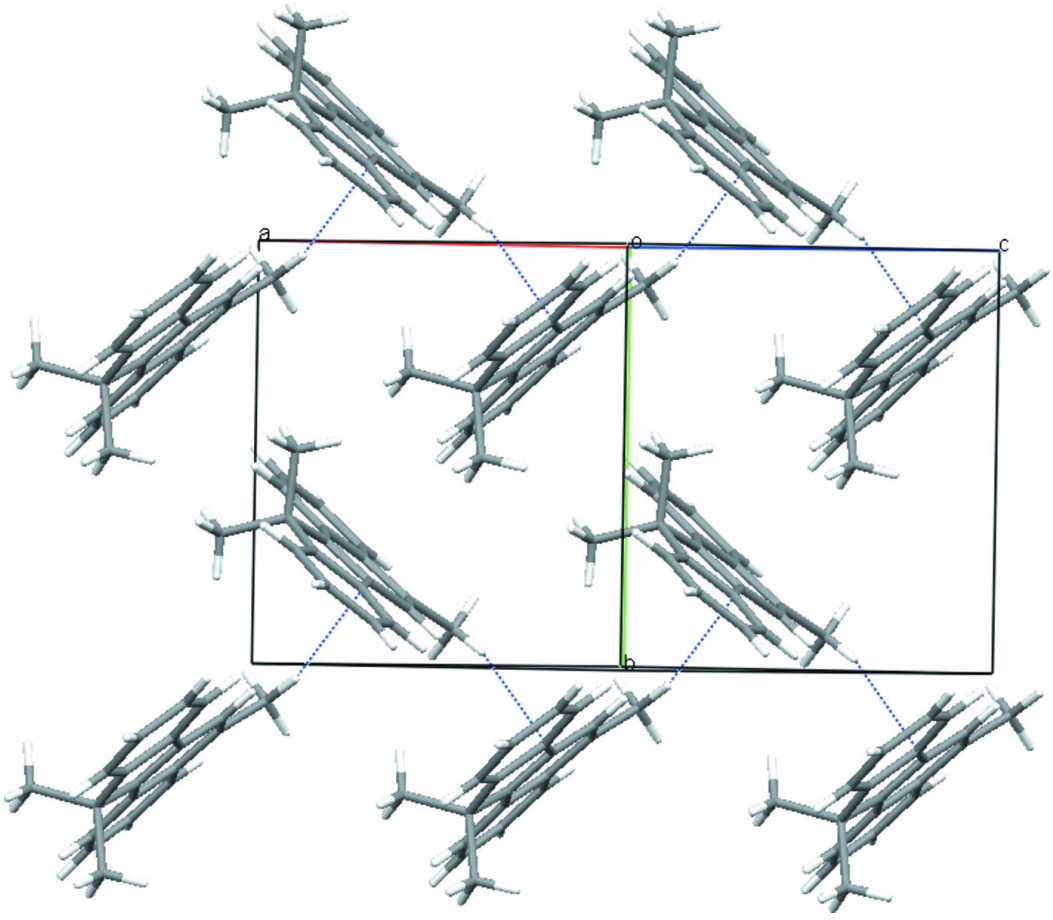
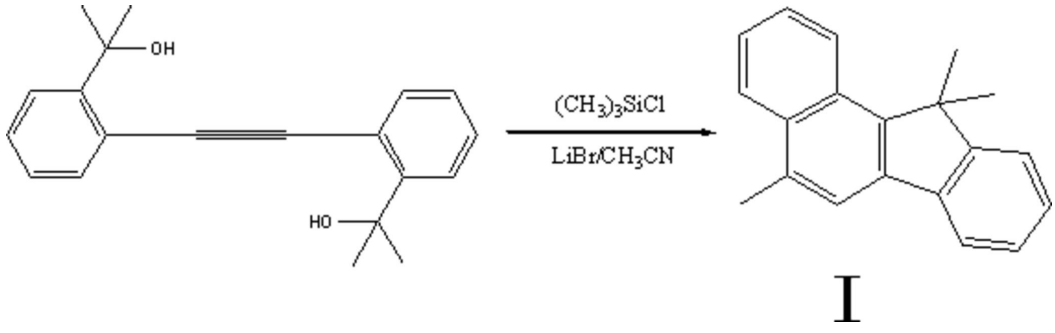


Fig. 3



I