Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

9,9′-Bifluorene-9,9′-diol ethyl acetate solvate

Jinyu Sun,[a] Lianmei Hu[b] and Zhao Zhang[b]*

[a]School of Chemistry and Chemical Engineering, Shanxi University, Taiyuan 030006, People's Republic of China, , and, , Department of Chemistry, Xinzhou Teachers University, Xinzhou 034000, People's Republic of China, and [b]School of Chemistry and Chemical Engineering, Shanxi University, Taiyuan 030006, People's Republic of China

Correspondence e-mail: z.zhang@sxu.edu.cn

Key indicators

Single-crystal X-ray study
T = 203 K
Mean σ(C–C) = 0.003 Å
R factor = 0.046
wR factor = 0.120
Data-to-parameter ratio = 13.3

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

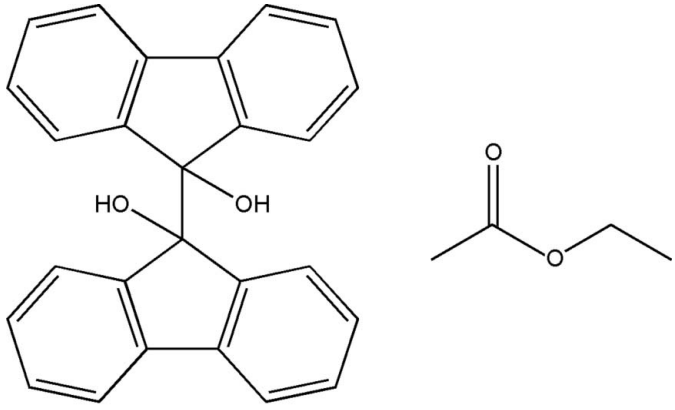
© 2007 International Union of Crystallography
All rights reserved

Received 24 October 2006
Accepted 13 November 2006

The 9,9′-bifluorene-9,9′-diol molecules in the title compound, $C_{26}H_{18}O_2\cdot C_4H_8O_2$, adopt a *gauche* conformation stabilized by intramolecular O—H···O hydrogen bonds between hydroxyl groups. There are two formula units per asymmetric unit. The molecules are linked into one-dimensional chains by intermolecular O—H···O hydrogen bonds; ethyl acetate molecules are bound to these chains, also by O—H···O hydrogen bonds.

Comment

Organic electroluminescent materials have attracted much attention because of their potential application in large-area flat-panel display devices (Thomas *et al.*, 2001). The compound 9,9′-bifluorene-9,9′-diol is an important intermediate in the synthesis of a variety of organic electroluminescent materials, (Hou *et al.*, 1998; Hou *et al.*, 1999). We report here a 1:1 solvate of 9,9′-bifluorene-9,9′-diol with ethyl acetate.



(I)

There are two independent formula units in the asymmetric unit (Fig. 1); the two diol molecules are approximate mirror images of each other (with the mirror plane passing approximately through atoms O2/C13/C26 and O3/C39/C52. All fluorenyl rings are essentially planar (maximum mean deviation from the least-squares plane 0.079 Å for C14–C26), and dihedral angles of 55.6 (1) and 58.7 (1)° are formed between the two ring planes in each molecule. The central C13—C26 [1.570 (2) Å] and C39—C52 [1.569 (2) Å] bonds are slightly longer than the standard C—C distance of 1.53 Å (Allen *et al.*, 1987), probably on account of steric hindrance. The molecules adopt a *gauche* conformation which is stabilized by intramolecular O—H···O hydrogen bonding (Table 1). Intermolecular O—H···O hydrogen bonds are also formed between 9,9′-bifluorene-9,9′-diol molecules (Table 1), linking

them into one-dimensional chains (Fig. 2). The ethyl acetate molecules are bound to these chains, also by O—H···O hydrogen bonds.

Experimental

A mixture of fluorene (10 g), Zn (50 g), $ZnCl_2$ (10 g), THF (50 ml) and water (50 ml) was stirred at room temperature for 3 h (Tanaka *et al.*, 1990; Liu *et al.*, 2005). The reaction mixture was combined with 3 *N* HCl (25 ml) and stirred for 20 min, then THF (50 ml) and toluene (50 ml) were added and stirred for a further 10 min. The mixture was filtered to remove Zn powder, and the filtrate was treated a further 3 times with HCl followed by THF/toluene, as described above. The oil layer was then separated and evaporated to provide crude 9,9′-bifluorene-9,9′-diol. Crystals of the title compound were obtained by recrystallization from ethyl acetate.

Crystal data

$C_{26}H_{18}O_2\cdot C_4H_8O_2$
M_r = 450.51
Triclinic, $P\overline{1}$
a = 10.5498 (18) Å
b = 12.464 (2) Å
c = 19.203 (3) Å
α = 73.437 (2)°
β = 85.872 (2)°
γ = 77.195 (2)°
V = 2359.9 (7) Å^3
Z = 4
D_x = 1.268 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 203 (2) K
Block, colourless
0.60 × 0.50 × 0.30 mm

Data collection

Bruker SMART CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.830, T_{max} = 0.976
11565 measured reflections
8153 independent reflections
6351 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.120
S = 1.02
8153 reflections
614 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0573P)^2 + 0.4614P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0050 (7)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···O4^{i}	0.83	1.94	2.7513 (16)	164
O2—H2A···O1	0.83	2.28	2.7006 (17)	112
O2—H2A···O5^{i}	0.83	2.12	2.8597 (18)	148
O3—H3B···O2^{ii}	0.83	1.98	2.7598 (16)	157
O4—H4B···O3	0.83	2.23	2.6518 (17)	111
O4—H4B···O8^{iii}	0.83	2.21	2.9521 (17)	149

Symmetry codes: (i) $x, y+1, z$; (ii) $x+1, y-1, z$; (iii) $x, y-1, z$.

H atoms were placed in idealized positions and allowed to ride on their parent atoms with O—H = 0.83 Å and C—H = 0.94–0.98 Å, and with U_{iso}(H) = 1.2U_{eq}(C,O).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

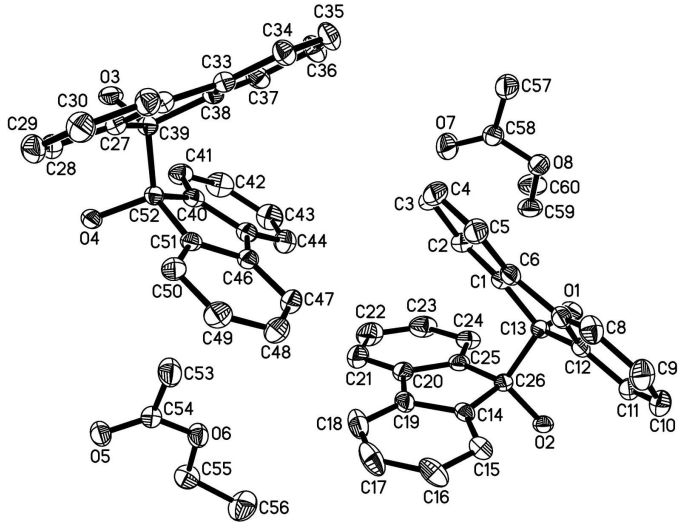


Figure 1
The asymmetric unit of (I), showing displacement ellipsoids drawn at the 30% probability level. H atoms are omitted.

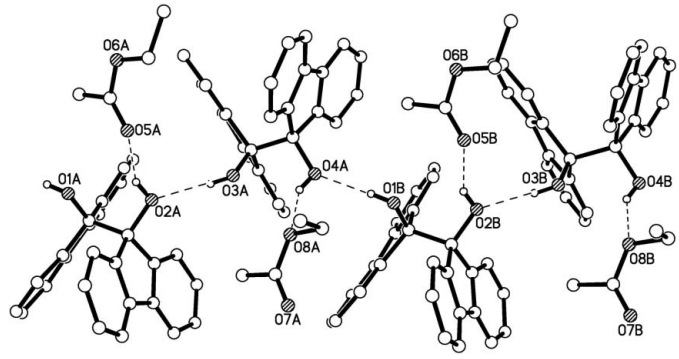


Figure 2
One-dimensional chain formed by intermolecular O—H···O hydrogen bonds (dashed lines). H atoms not involved in hydrogen bonding have been omitted. Atoms labelled with the suffix B are related to those labelled with the suffix A by the symmetry operator $x+1, y, z$.

This work was supported by the Natural Science Foundation of Shanxi Province (grant No. 2006011014).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–S19.
Bruker (2000). *SMART* (Version 5.0), *SAINT* (Version 6.02) and *SHELXTL* (Version 6.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Hou, Z., Fujita, A., Koizumi, T.-A., Yamazaki, H. & Wakatsuki, Y. (1999). *Organometallics*, **18**, 1979–1985.
Hou, Z., Fujita, A., Zhang, Y., Miyano, T., Yamazaki, H. & Wakatsuki, Y. (1998). *J. Am. Chem. Soc.* **120**, 754–766.
Liu, J.-C., Jin, S., Liu, B. & Guo, J.-P. (2005). *Acta Cryst.* E**61**, o2035–o2037.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Tanaka, K., Kishigami, S. & Toda, F. (1990). *J. Org. Chem.* **55**, 2981–2983.
Thomas, K. R. J., Lin, J. T., Tao, Y.-T. & Ko, C.-W. (2001). *J. Am. Chem. Soc.* **123**, 9404–9411.