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1,3-Bi-9-anthrylpropane

Mustafa Arslan
Sakarya University, Adapazari, Turkey

Erol Asker
Balikesir University, Balikesir, Turkey

John Masnovi
Cleveland State University, j.masnovi@csuohio.edu

Ronald J. Baker

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Mustafa Arslan,^{a*} Erol Asker,^b
John Masnovi^c and Ronald J.
Baker^c

^aDepartment of Chemistry, Faculty of Arts and Sciences, Sakarya University, 54140 Esentepe/ Adapazari, Turkey, ^bNecatibey Faculty of Education, Balikesir University, 10100 Balikesir, Turkey, and ^cDepartment of Chemistry, Cleveland State University, Cleveland, OH 44115, USA

Correspondence e-mail: marslan@sakarya.edu.tr

Key indicators

Single-crystal X-ray study

$T = 295$ K

Mean $\sigma(\text{C}-\text{C}) = 0.006$ Å

R factor = 0.044

wR factor = 0.103

Data-to-parameter ratio = 7.4

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

1,3-Bi-9-anthrylpropane

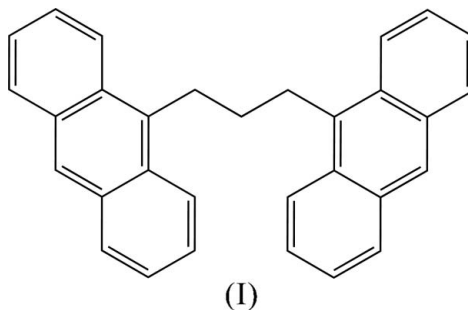
The title compound, $\text{C}_{31}\text{H}_{24}$, with three molecules in the asymmetric unit. The crystal packing is mainly stabilized by weak $\text{C}-\text{H}\cdots\pi$ interactions in addition to van der Waals forces.

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Comment

Bichromophoric model compounds of polyvinylanthracenes are extensively used in studies concerning the electrophotographic properties of polymers (Hayashi *et al.*, 1976; Becker & Andersson, 1987; Becker *et al.*, 1992). In pendant-type polymeric systems, photoconductivity depends largely on the spacing and orientation of the pendant units in the polymer backbone. The molecular geometries of 9,9'-bianthryl, bis-9-anthrylmethane (Becker *et al.*, 1992) and 1,2-bis(9-anthryl)ethane (Becker *et al.*, 1984) were determined in order to understand their photochemical and photophysical properties. We report here the crystal structure of the title compound, (I) (Fig. 1), as the third member of this family of bichromophoric model compounds of polyvinylanthracenes.



The crystal structure of compound (I) (Fig. 1) was solved in the non-centrosymmetric space group $P2_12_12_1$, with three independent molecules (*A*, *B*, and *C*) in the asymmetric unit. All six anthracene skeletons are essentially planar, the largest deviation from planarity of the fitted atoms being 0.088 (4) Å for atom C29*b*. The bond distances and angles of the anthracene rings in all three molecules are comparable with each other and with those of related molecules (Becker *et al.*, 1984, 1992). The anthryl groups exhibit an *anti-anti* conformation along the connecting aliphatic chains. The dihedral angles formed by the anthracene planes are 61.00 (6)° in molecule *A*, 59.79 (6)° in molecule *B* and 69.85 (5)° in molecule *C*.

The crystal packing diagram (Fig. 2) reveals that the crystal structure of (I) is stabilized only by van der Waals forces and weak $\text{C}-\text{H}\cdots\pi$ interactions (Table 1). Although molecules with aromatic groups often pack in the solid state with parallel planar π systems, there are neither intra- nor intermolecular

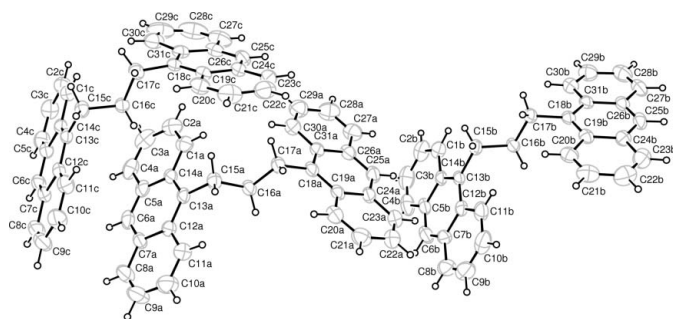


Figure 1
The three independent molecules of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

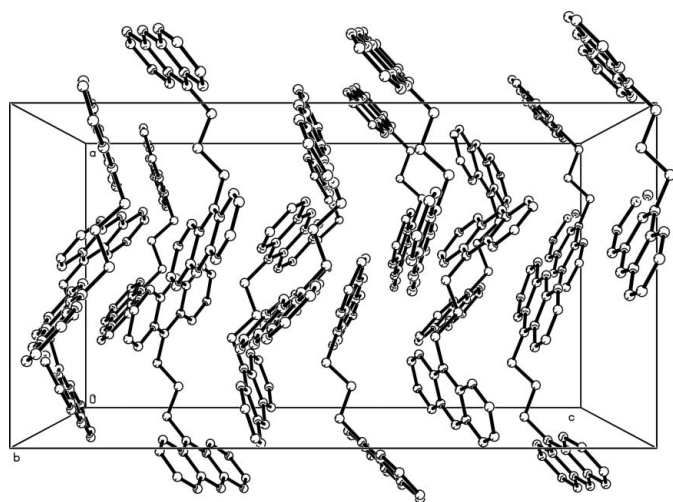


Figure 2
The crystal packing of (I), viewed down the *b* axis. H atoms have been omitted for clarity.

π - π interactions observed among the anthracene ring systems of (I).

Experimental

The title compound was prepared *via* $\text{LiAlH}_4/\text{AlCl}_3$ reduction of a mixture of 1,3-bis(9-anthryl)propan-1-one and 1,3-bis(9-anthryl)propan-1-ol, which were prepared according to the literature procedure (Becker & Andersson, 1983). First, AlCl_3 (3.3 g, 25 mmol) in diethyl ether (40 ml) was added to a stirred solution of LiAlH_4 (0.95 g, 25 mmol) in diethyl ether (40 ml) in an ice bath, which was removed after the addition was complete. A mixture (2.15 g, 5.25 mmol) of 1,3-bis(9-anthryl)propan-1-ol and 1,3-bis(9-anthryl)propan-1-one (2:1 molar ratio) was dissolved in diethyl ether (50 ml) and a minimum amount of tetrahydrofuran (to increase solubility), and the solution was then added dropwise to the above $\text{LiAlH}_4/\text{AlCl}_3$ mixture and the reaction mixture refluxed for 2 h. After cooling, ethyl acetate (60 ml) and 20% H_2SO_4 (70 ml) were added to the reaction mixture to deactivate unreacted LiAlH_4 . The mixture was extracted with dichloromethane and dried over sodium sulfate. The residue obtained after vacuum evaporation of the solvents was purified by column chromatography on silica gel using hexane-dichloromethane (9:1 v/v) and crystallized from hexane-dichloromethane (1:1 v/v) to give 1.77 g (4.47 mmol, 82% yield) of (I) as yellow crystals (m.p. 468–469 K).

Crystal data

$\text{C}_{31}\text{H}_{24}$
 $M_r = 396.5$
Orthorhombic, $P2_12_12_1$
 $a = 13.478$ (2) Å
 $b = 19.1167$ (14) Å
 $c = 25.2951$ (17) Å

$V = 6517.5$ (12) Å³
 $Z = 12$
Mo $K\alpha$ radiation
 $\mu = 0.07$ mm⁻¹
 $T = 295$ (2) K
 $0.50 \times 0.50 \times 0.50$ mm

Data collection

Enraf-Nonius CAD-4 diffractometer
Absorption correction: none
6228 measured reflections
6228 independent reflections

4061 reflections with $I > 2\sigma(I)$
3 standard reflections
frequency: 120 min
intensity decay: 0.7%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.103$
 $S = 1.03$
6228 reflections

838 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.12$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.10$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
<i>C2a</i> —H2 <i>a</i> ... <i>Cg1</i>	0.93	2.86	3.5722 (5)	135
<i>C6c</i> —H6 <i>c</i> ... <i>Cg2</i> ⁱ	0.93	2.95	3.8708 (4)	170
<i>C8b</i> —H8 <i>b</i> ... <i>Cg3</i> ⁱⁱ	0.93	2.82	3.7438 (6)	173
<i>C8c</i> —H8 <i>c</i> ... <i>Cg4</i> ⁱ	0.93	2.80	3.6815 (5)	159
<i>C17b</i> —H17 <i>d</i> ... <i>Cg5</i> ⁱⁱⁱ	0.97	2.90	3.8021 (4)	156
<i>C17c</i> —H17 <i>e</i> ... <i>Cg6</i> ⁱⁱ	0.97	2.90	3.8054 (4)	155
<i>C23a</i> —H23 <i>a</i> ... <i>Cg7</i> ^{iv}	0.93	2.59	3.5045 (5)	167

Symmetry codes: (i) $x + 1, y, z$; (ii) $x - \frac{1}{2}, -y + \frac{1}{2}, -z$; (iii) $-x + 1, y - \frac{1}{2}, -z + \frac{1}{2}$; (iv) $-x, y + \frac{1}{2}, -z + \frac{1}{2}$.

All H atoms were positioned geometrically and allowed to ride on their corresponding parent atoms, with C—H = 0.93 and 0.97 Å for aromatic and methylene H atoms, respectively, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection and cell refinement: *CAD-4-PC Software* (Enraf-Nonius, 1993); data reduction: *DATRD2* in *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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