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Key indicators

Single-crystal X-ray study
 $T = 150$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.045
 wR factor = 0.137
Data-to-parameter ratio = 14.8For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

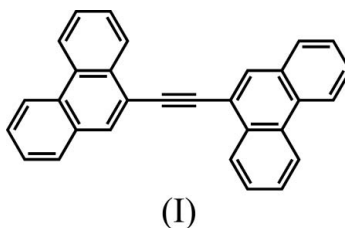
Bis(9-phenanthryl)ethyne

The molecule of the title compound, $\text{C}_{30}\text{H}_{18}$, is located on an inversion center and has an almost completely planar geometry.

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Comment

Diarylethyne have been extensively investigated with respect to the wavelength–conjugation length relationship using UV spectroscopy (Akiyama *et al.*, 1971; Nakagawa *et al.*, 1971). Most of the compounds, however, have not been studied crystallographically, so the detailed structure–property relationship is still open for discussion. We report here the crystal structure of bis(9-phenanthryl)ethyne, (I).



The molecule of (I), located an inversion center, has an almost completely planar geometry (Fig. 1). The bond lengths of the linker unit between the two phenanthrene rings, C1–C1(1 – x , – y , 2 – z) and C1–C2 (Table 1), are typical for $\text{C}_{sp}-\text{C}_{sp}$ and $\text{C}_{sp}-\text{C}_{sp}^2$ bonds, respectively (Bastiansen & Traetteberg, 1962). The molecules form a chain running along the c axis through a $\pi-\pi$ interaction between neighboring C2–C4/C9/C10/C15 rings. The distance between the centroids [Cg1...Cg1(1 – x , – y , 1 – z)] is 3.7566 (6) Å. The chains are connected by C–H... π interactions (Fig. 2 and Table 2).

Experimental

The title compound was prepared according to the procedure of Mio *et al.* (2002). A Schlenk tube was charged with 9-bromophenanthrene (514 mg, 2.00 mmol), $\text{PdCl}_2(\text{PPh}_3)_2$ (42 mg, 0.060 mmol), CuI (38 mg, 0.20 mmol), 1,8-diazabicyclo[5.4.0]undec-7-ene (1.79 ml, 12.0 mmol), water (9.0 μl , 0.50 mmol) and benzene (10 ml). Trimethylsilylacetylene (0.14 ml, 1.0 mmol) was added to the mixture, which was degassed three times, and the reaction tube was purged with dry argon. The reaction tube was capped tightly and heated at 353 K for 2 d. After cooling, the reaction mixture was extracted with CH_2Cl_2 , washed with 0.5 N HCl (twice) and saturated aqueous NaCl, and dried over Na_2SO_4 . The crude product was purified by silica gel column chromatography (benzene–hexane, 1:1). Single crystals were obtained from a benzene solution (yield 106 mg, 28%; m.p. 516–518 K).

Crystal data

C₃₀H₁₈
M_r = 378.47
 Monoclinic, *P*2₁/*c*
a = 9.3403 (8) Å
b = 15.2266 (11) Å
c = 6.9007 (5) Å
 β = 102.0338 (13)°
V = 959.86 (13) Å³

Z = 2
D_x = 1.309 Mg m⁻³
 Mo *K*α radiation
 μ = 0.07 mm⁻¹
T = 150 (2) K
 Prism, colorless
 0.45 × 0.30 × 0.20 mm

Data collection

Rigaku/MSC Mercury CCD
 diffractometer
 ω scans
 Absorption correction: none
 9274 measured reflections

2151 independent reflections
 2036 reflections with *I* > 2σ(*I*)
*R*_{int} = 0.019
 θ_{\max} = 27.5°

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.045
wR (*F*²) = 0.137
S = 1.10
 2151 reflections
 145 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0872P)^2 + 0.1528P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.33 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.19 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

C1—C1 ⁱ	1.197 (2)	C2—C3	1.3648 (14)
C1—C2	1.4351 (13)	C2—C15	1.4538 (13)
C1 ⁱ —C1—C2	178.37 (14)		

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

Table 2

Hydrogen-bond geometry (Å, °).

Cg2 and Cg3 are the centroids of the C4–C9 and C10–C15 rings, respectively.

<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>
C7—H4...Cg2 ⁱⁱ	0.95	2.94	3.6512 (12)	133
C11—H6...Cg3 ⁱⁱ	0.95	2.82	3.5932 (11)	140

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

All H atoms were placed in geometrically idealized positions (C—H = 0.95 Å) and constrained to ride on their parent atoms, with *U*_{iso}(H) = 1.2*U*_{eq}(C).

Data collection: *CrystalClear* (Rigaku/MSC, 2002); cell refinement: *CrystalClear*; data reduction: *TEXSAN* (Rigaku/MSC, 2004); 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: *TEXSAN*.

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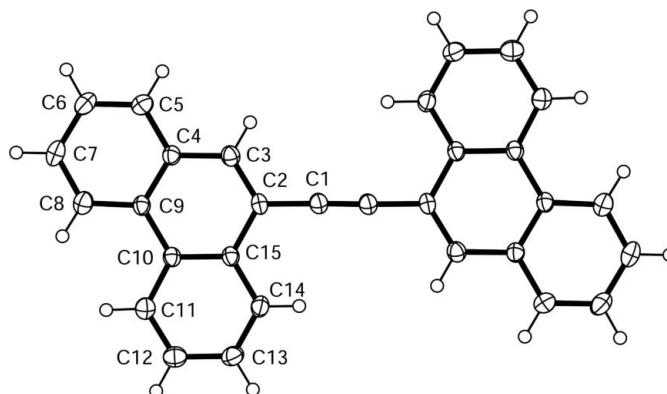


Figure 1

Drawing of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. Unlabeled atoms are related to labeled atoms by $1 - x, -y, 2 - z$.

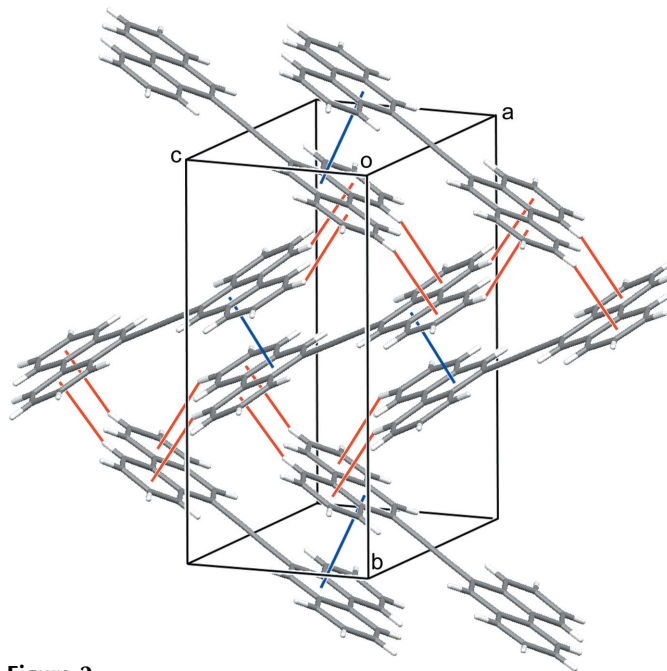


Figure 2

The crystal packing of (I). π - π and C—H... π interactions are indicated by blue and red lines, respectively.

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