

10-(4-Bromostyryl)anthracene

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The title compound, C₂₂H₁₅Br, contains two crystallographically independent molecules in the asymmetric unit, with similar geometry. The crystal packing is stabilized mainly by van der Waals forces.

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Key indicators

Single-crystal X-ray study

T = 273 K

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

R factor = 0.043

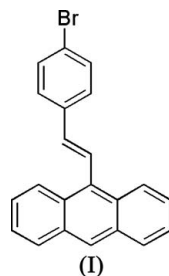
wR factor = 0.101

Data-to-parameter ratio = 13.9

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

Comment

Nonlinear optical effects in organic crystals have been exploited extensively during the past decade (Prasad & Williams, 1991). In these organic materials, π -conjugated organic molecules have been investigated with great interest owing to their potential applications in optical image processing, all-optical switching and integrated optical devices (Nishihara *et al.*, 1989; Tian *et al.*, 1999; Roberto *et al.*, 2000). As a part of our continuing studies of the synthesis and characterization of nonlinear optical materials, we have prepared a new anthracene derivative, (I), containing an anthracenyl group as donor and a Br atom as acceptor, and have investigated its crystal structure.



There are two crystallographically independent molecules in the asymmetric unit (Fig. 1), both molecules adopting an *E* configuration about the central C=C bond. Bond lengths and angles in the two molecules are similar and are in agreement with the values reported in the literature (Allen *et al.*, 1987). The anthracene rings are essentially planar, with maximum displacements from the mean plane of 0.091 (4) and 0.120 (5) Å for C20 and C42, respectively. The dihedral angles formed by the C1–C6 and C23–C28 benzene rings with the C9–C22 and C31–C44 anthracene ring systems are 76.2 (2) and 67.7 (2)°, respectively. The crystal packing is stabilized mainly by van der Waals forces (Fig. 2).

Experimental

For the preparation of (4-bromobenzyl)triphenylphosphonium bromide, a flask was charged with a mixture of 1-bromo-4-methylbenzene (8.55 g, 50 mmol), *N*-bromosuccinimide (9.2 g, 51 mmol) and a catalytic amount of benzoyl peroxide, which was vigorously stirred

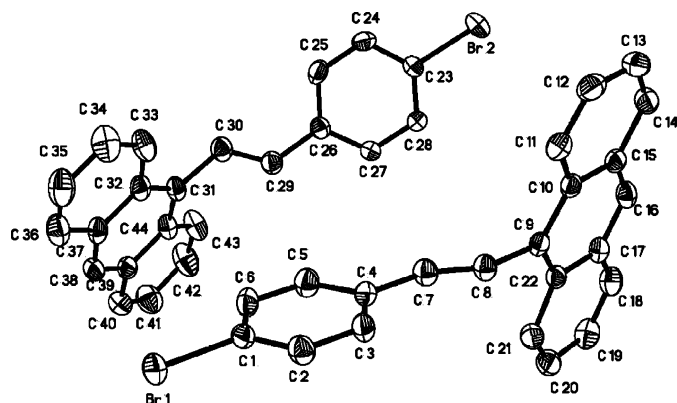


Figure 1
The asymmetric unit of the title compound, showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

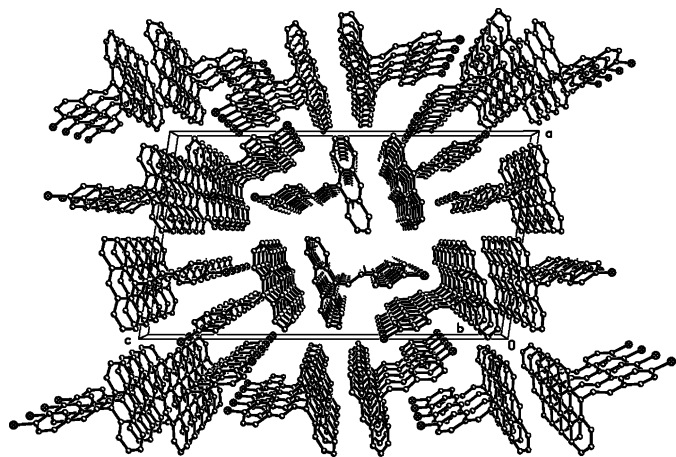


Figure 2
A packing diagram of the title compound, viewed along the *b* axis. H atoms have been omitted for clarity.

in tetrachloromethane (150 ml) and refluxed for 5 h. The reaction was monitored by thin-layer chromatography. After completion of the reaction, the mixture was cooled to room temperature and extracted with chloroform several times. The organic layer was washed with water and saturated brine, and then the organic extracts were dried over MgSO_4 . After removing the solvent under reduced pressure, the residue was collected and dried *in vacuo*. This intermediate was added to triphenylphosphine (13.1 g, 50 mmol) in benzene (150 ml), and the resulting solution was refluxed for 4 h. After cooling to room temperature, a white solid was collected by filtration and air-dried (yield 16.1 g, 63%). For the preparation of 10-(4-bromostyryl)-anthracene, anthraldehyde (3.09 g, 15 mmol), (4-bromobenzyl)-triphenylphosphonium bromide (7.9 g, 15 mmol) and powdered NaOH (2.4 g, 60 mmol) were crushed together with a pestle and mortar for 2 h (Yang *et al.*, 2005). The mixture was extracted three times with dichloromethane. The organic layer was washed with saturated brine and the organic extracts were dried over MgSO_4 . After removing the solvent under reduced pressure, the residue was purified by flash column chromatography to give the product as a yellow solid. Single crystals suitable for X-ray analysis were obtained by slow evaporation of a dichloromethane–2-propanol (5:1) solution.

Crystal data

$\text{C}_{22}\text{H}_{15}\text{Br}$
 $M_r = 359.25$
 Monoclinic, $P2_1/c$
 $a = 17.966$ (4) Å
 $b = 5.8413$ (11) Å
 $c = 31.627$ (6) Å
 $\beta = 98.582$ (3)°
 $V = 3281.9$ (11) Å³
 $Z = 8$

$D_x = 1.454$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 3657 reflections
 $\theta = 2.3\text{--}22.1^\circ$
 $\mu = 2.50$ mm⁻¹
 $T = 273$ (2) K
 Block, pale yellow
 $0.35 \times 0.33 \times 0.31$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.427$, $T_{\max} = 0.460$
 16373 measured reflections

5778 independent reflections
 3717 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.037$
 $\theta_{\max} = 25.0^\circ$
 $h = -21 \rightarrow 20$
 $k = -6 \rightarrow 6$
 $l = -26 \rightarrow 37$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.101$
 $S = 1.02$
 5778 reflections
 415 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.043P)^2 + 1.3248P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.43$ e Å⁻³
 $\Delta\rho_{\min} = -0.49$ e Å⁻³

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C–H = 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

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

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References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
 Bruker (1997). SMART and SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.
 Nishihara, H., Haruna, M. & Suhara, T. (1989). *Optical Intergrated Circuits*. New York: McGraw–Hill.
 Prasad, P. N. & Williams, D. J. (1991). *Introduction to Nonlinear Optical Effects in Molecules and Polymers*. New York: Wiley.
 Roberto, D., Ugo, R., Bruni, S., Cariati, E., Cariati, F., Fantucci, P., Invernizzi, I., Quici, S., Ledoux, I. & Zyss, J. (2000). *Organometallics*, **19**, 1775–1788.
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
 Sheldrick, G. M. (1997a). SHELXS97 and SHELXL97. University of Göttingen, Germany.
 Sheldrick, G. M. (1997b). SHELXTL. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
 Tian, Y.-P., Lu, Z.-L. & You, X.-Z. (1999). *Acta Chim. Sin.* **57**, 1068–1074.
 Yang, J.-X., Tao, X.-T., Yuan, C.-X., Yan, Y.-X., Wang, L., Liu, Z., Ren, Y. & Jiang, M.-H. (2005). *J. Am. Chem. Soc.* **127**, 3278–3279.