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9-(Bromoacetyl)anthracene dimer

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Key indicators

Single-crystal X-ray study
T = 153 K
Mean σ(C–C) = 0.003 Å
R factor = 0.036
wR factor = 0.074
Data-to-parameter ratio = 16.9

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

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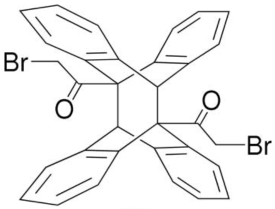
The title compound [systematic name: 5,6,11,12-tetrahydro-5,11-bis(bromoacetyl)-5,12[1′,2′]:6,11[1″,2″]dibenzenodibenzo-[*a*,*e*]cyclooctene], $C_{32}H_{22}Br_2O_2$, is a [4+4] photodimer of 9-(bromoacetyl)anthracene. The molecule is centrosymmetric and contains a long Csp^3—Csp^3 bond of 1.619 (3) Å. Intermolecular C—H···O interactions are observed.

Comment

Anthracene and its derivatives have been studied extensively in the fields of photochemistry and photobiology. Photodimerization of anthracene is one of the oldest known photochemical reactions and a classical example of photocycloaddition (Becker, 1993; Bouas-Laurent *et al.*, 2000). Recently, the photochemical reaction (Matsumoto *et al.*, 1974), laser flash photolysis (Suzuki *et al.*, 2004) and crystal structure (Kubo *et al.*, 2007) of 9-(bromoacetyl)anthracene have been reported, but its photodimer has not been obtained. The title compound, (I), was obtained unexpectedly when growing single crystals of 9-(bromoacetyl)anthracene from a chloroform solution under sunlight. We now report the structure of (I), with the aim of contributing to a deeper understanding of the [4+4] photodimerization of 9-(bromoacetyl)anthracene.



(I)

Compound (I) is centrosymmetric and a highly strained butterfly-shaped anthracene dimer (Fig. 1). The angle of the intersection between the least-squares plane defined by C1–C6/C13/C14 and the plane defined by C6–C13 is 43.1 (1)°, which is similar to that of the intramolecular photocyclomer of 9-anthroic anhydride [47.6 (1)°; Cicogna *et al.*, 2002]. The two bromoacetyl groups are *anti* with respect to one another across the anthracene dimer. Compound (I) contains a long central C—C bond connecting the anthracene units, compared with the standard C—C single-bond length of 1.54 Å (Battersby *et al.*, 1995). The C—C bond length [1.619 (3) Å] is close to that of the anthracene dimer [1.618 (3) Å; Abboud *et al.*, 1990].

Intermolecular C—H···O interactions with typical geometric parameters (Takahashi *et al.*, 2005; Kubo *et al.*, 1997, 2006, 2007) are observed in the crystal structure (Table 1).

Experimental

Compound (I) was prepared by sunlight irradiation of a chloroform solution (1.25 mmol l^{-1}) of 9-(bromoacetyl)anthracene. Crystals of (I) were grown from a chloroform solution by slow evaporation.

Crystal data

$C_{32}H_{22}Br_2O_2$
M_r = 598.33
Triclinic, $P\bar{1}$
a = 7.9044 (18) Å
b = 9.1861 (17) Å
c = 9.4138 (17) Å
α = 66.648 (9)°
β = 75.696 (10)°
γ = 83.656 (11)°
V = 608.0 (2) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 3.37 mm^{-1}
T = 153.1 K
0.18 × 0.08 × 0.07 mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan (*REQAB*; Jacobson, 1998)
$T_{\min}$ = 0.634, $T_{\max}$ = 0.790
7481 measured reflections
2777 independent reflections
2444 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.074
S = 1.05
2777 reflections
164 parameters
H-atom parameters constrained
$\Delta\rho_{\max}$ = 0.56 e Å^{-3}
$\Delta\rho_{\min}$ = −0.48 e Å^{-3}

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C9—H7$\cdots$O1^{ii}	0.95	2.59	3.318 (4)	134

Symmetry code: (ii) $x, y-1, z$.

H atoms were positioned geometrically and refined as riding, with C—H = 0.95 Å for aromatic, C—H = 0.99 Å for methylene and C—H = 1.00 Å for methine H atoms, and with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C).

Data collection: *CrystalClear* (Rigaku, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2006); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *Mercury* (Version 1.3; Bruno *et al.*, 2002); software used to prepare material for publication: *CrystalStructure*.

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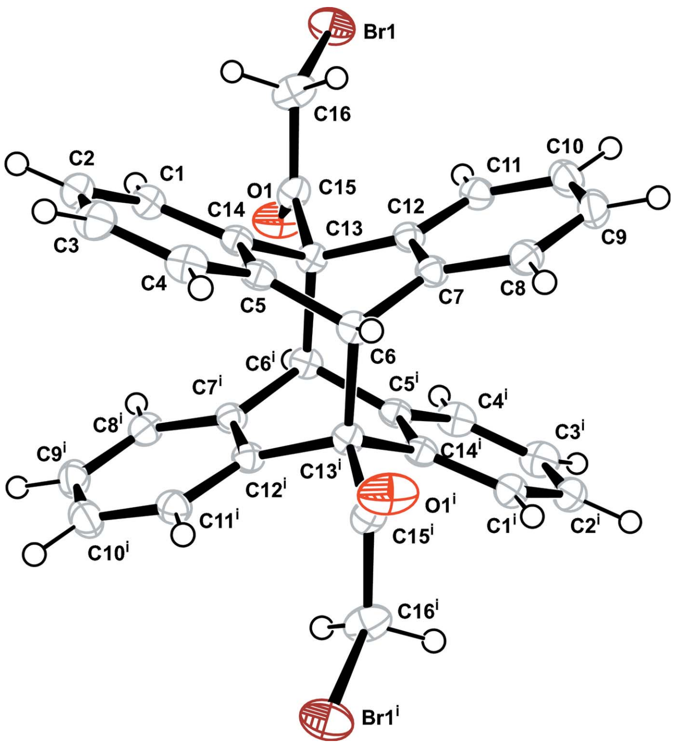

Figure 1
The molecular structure of (I), showing the atom-numbering scheme and 50% probability displacement ellipsoids. [Symmetry code: (i) $1-x, -y, 2-z$.]

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