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## A Diiodo-Substituted 9-Anthracenone

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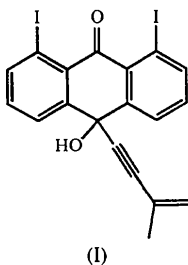
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### Abstract

The central ring of the anthrone system in 1,8-diiodo-10-hydroxy-10-(3-methylbut-3-en-1-ynyl)anthracen-9(10*H*)-one, C<sub>19</sub>H<sub>12</sub>I<sub>2</sub>O<sub>2</sub>, has a boat conformation and the two outer rings form a dihedral angle of 41.1 (2)°. The I—C bond lengths are 2.094 (4) and 2.083 (4) Å. The hydroxyl and carbonyl groups form an intermolecular hydrogen bond having an O...O distance of 2.853 (4) Å.

### Comment

The title compound, (I), was prepared as part of an ongoing project involving the structural study of key intermediates in the synthesis of substituted [3]-cumulenes (Garcia, Asfaw & Rodriguez, 1997), which have potential biological activity (Garcia, Ramos, Pratt & Rodriguez, 1995). The compound was prepared by a slight modification of a literature methodology (Garcia, Ramos, Pratt & Rodriguez, 1995) by the reaction of 1,8-diiodo-9,10-anthraquinone (Bayer, 1979) with the lithium salt of 2-methyl-1-buten-3-yne in tetrahydrofuran at 195 K.



Both the I1—C1 bond length of 2.094 (4) Å and the I2—C8 bond length of 2.083 (4) Å are comparable to the typical value of 2.095 Å for 51 measurements (Allen *et al.*, 1987). The central anthrone ring of the structure exists in a boat-like conformation having the torsion angles reported in Table 1. This conformation resembles that of 10-*tert*-butyl-9-anthracenone and almost all other 10-substituted 9-anthracenones (Sygula, Sygula, Fronczek & Rabideau, 1992). The two outer rings of

the structure are nearly planar, with the C1—C2—C3—C4—C13—C14 ring having a mean deviation of 0.0044 Å from planarity and the C5—C6—C7—C8—C11—C12 ring having a mean deviation of 0.0043 Å from planarity. The I1 atom lies 0.0772 (4) Å out of the plane of the ring to which it is bonded, while I2 lies a much greater distance of 0.1976 (4) Å out of the plane of its respective ring. The two outer rings form a dihedral angle of 41.1 (2)°. The intramolecular I1...O1 and I2...O1 distances are 3.171 (4) and 3.174 (4) Å, respectively, and are both shorter than the sum of the van der Waals radii (3.60 Å; Bondi, 1964). The I...O1...I angle is 101.4 (1)°. The intermolecular O1...O2( $\frac{3}{2} - x, 1 - y, \frac{1}{2} + z$ ) distance of 2.853 (4) Å is evidence of hydrogen bonding between the hydroxyl group and the ketone oxygen.

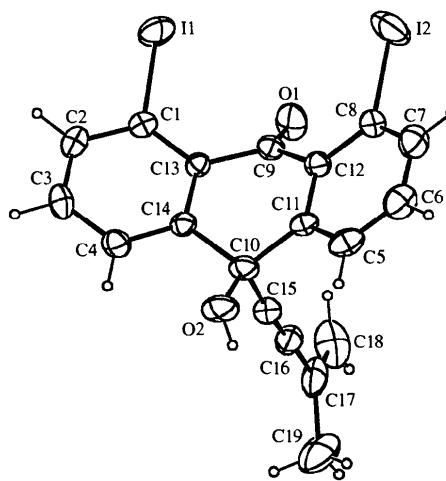


Fig. 1. The title molecule, with ellipsoids at the 40% probability level and H atoms represented with arbitrary radii.

### Experimental

The title compound was prepared by a slight modification of the method of Garcia, Ramos, Pratt & Rodriguez (1995) by the reaction of 1,8-diiodo-9,10-anthraquinone (Bayer, 1979) with the lithium salt of 2-methyl-1-buten-3-yne in tetrahydrofuran at 195 K. The resulting yellow solid was recrystallized by slow evaporation from dichloromethane [m.p. 470 K (dec.)].

#### Crystal data

C<sub>19</sub>H<sub>12</sub>I<sub>2</sub>O<sub>2</sub>  
*M<sub>r</sub>* = 526.11  
 Orthorhombic  
*Pbca*  
*a* = 15.117 (1) Å  
*b* = 14.4592 (6) Å  
*c* = 15.8539 (9) Å  
*V* = 3465.3 (6) Å<sup>3</sup>  
*Z* = 8  
*D<sub>x</sub>* = 2.017 Mg m<sup>-3</sup>  
*D<sub>m</sub>* not measured

Mo *K*α radiation  
 $\lambda$  = 0.71073 Å  
 Cell parameters from 25 reflections  
 $\theta$  = 8–14°  
 $\mu$  = 3.597 mm<sup>-1</sup>  
*T* = 297 K  
 Cuboidal  
 0.55 × 0.50 × 0.48 mm  
 Yellow

**Data collection**

Enraf–Nonius CAD-4 diffractometer  
 $\theta/2\theta$  scans  
 Absorption correction:  $\psi$  scans (North, Phillips & Mathews, 1968)  
 $T_{\min} = 0.127$ ,  $T_{\max} = 0.178$   
 10 786 measured reflections  
 5037 independent reflections

3815 reflections with  $I > \sigma(I)$   
 $R_{\text{int}} = 0.033$   
 $\theta_{\max} = 29.97^\circ$   
 $h = 0 \rightarrow 21$   
 $k = -20 \rightarrow 20$   
 $l = 0 \rightarrow 22$   
 3 standard reflections  
 frequency: 120 min<sup>-1</sup>  
 intensity decay: 0.82%

**Refinement**

Refinement on  $F^2$   
 $R = 0.045$   
 $wR = 0.054$   
 $S = 1.880$   
 3815 reflections  
 209 parameters  
 H atoms riding [C—H  
 $0.95 \text{ \AA}$ ;  $U(\text{H}) = 1.3U_{\text{eq}}(\text{C})$ ]  
 $w = 4F_o^2/[\sigma^2(F_o^2) + 0.0004F_o^4]$   
 $(\Delta/\sigma)_{\max} = 0.029$

$\Delta\rho_{\max} = 1.36 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.23 \text{ e \AA}^{-3}$   
 Extinction correction: isotropic (Zachariasen, 1963)  
 Extinction coefficient:  $9.2(4) \times 10^{-8}$   
 Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

I1—C1	2.094 (4)	C10—C14	1.534 (5)
I2—C8	2.083 (4)	C10—C15	1.489 (6)
O1—C9	1.209 (4)	C15—C16	1.185 (6)
O2—C10	1.423 (4)	C16—C17	1.440 (7)
C9—C12	1.479 (5)	C17—C18	1.345 (8)
C9—C13	1.479 (5)	C17—C19	1.486 (9)
C10—C11	1.527 (6)		
I1—C1—C2	116.1 (3)	C11—C10—C14	110.8 (3)
I1—C1—C13	122.6 (3)	C11—C10—C15	108.1 (3)
I2—C8—C7	116.4 (3)	C14—C10—C15	108.7 (3)
I2—C8—C12	122.2 (3)	C10—C15—C16	174.2 (4)
C12—C9—C13	115.3 (3)	C15—C16—C17	178.1 (5)
O2—C10—C11	110.4 (3)	C16—C17—C18	120.2 (5)
O2—C10—C14	107.0 (3)	C16—C17—C19	115.5 (5)
O2—C10—C15	111.9 (3)	C18—C17—C19	124.3 (5)
C13—C9—C12—C11	36.3 (5)	C11—C10—C14—C13	37.8 (5)
C12—C9—C13—C14	-37.4 (5)	C10—C11—C12—C9	2.0 (5)
C14—C10—C11—C12	-37.8 (5)	C9—C13—C14—C10	-1.8 (5)

The two largest peaks in the final difference map, both of density greater than  $1 \text{ e \AA}^{-3}$ , were located less than  $1 \text{ \AA}$  from the two I atoms.

Data collection: *CAD-4 Operations Manual* (Enraf–Nonius, 1977). Cell refinement: *CAD-4 Operations Manual*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *MULTAN80* (Main *et al.*, 1980). Program(s) used to refine structure: *MolEN*. Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *MolEN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1169). Services for accessing these data are described at the back of the journal.

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**(1S,6S)-Tetrazolo[1,5-g]-7-aza-trans-himachal-2-ene†**

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**Abstract**

In the title compound,  $\text{C}_{14}\text{H}_{22}\text{N}_4$ , the tetrazole ring is fused with the eight-membered ring of the himachal-2-ene skeleton, which is in a *trans* conformation.

† Alternative name: (7aS,11aS)-4,5,6,7,7a,10,11,11a-octahydro-7,7,9-trimethyl-2-benzazocino[1,2-d]tetrazole.