

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1115). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Absolute Configuration of (*S,S*)-1,2-Dihydro-1,2-*trans*-acenaphthylene-dicarboxylic Acid

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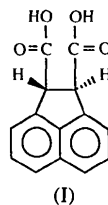
### Abstract

The naphthalene ring system in the title compound, C<sub>14</sub>H<sub>10</sub>O<sub>4</sub>, is essentially planar and the fused cyclopentane ring is in a slightly twisted conformation. The carboxylic acid groups associate *via* intermolecular hydrogen bonds [O···O 2.610 (4) and 2.655 (3) Å] forming chain structures that are held together by weak van der Waals interactions.

### Comment

In the course of our studies on the direct asymmetric introduction of the tricarbonylchromium moiety on prochiral arenes, we required pure optically active  $\eta^6$  ligands with C<sub>2</sub> symmetry. The title compound, (I), has two asymmetric centers and the absolute configuration was required for effective understanding of the mechanism of the ligand-transfer process. The X-ray structure

determination was undertaken following the inability of ordinary spectral methods to furnish the essential structural information.



A view of the molecule is shown in Fig. 1 and the unit-cell packing is shown in Fig. 2. Both asymmetric centers in the molecule have the *S* configuration. The naphthalene ring system is essentially planar [maximum deviation of atoms C(1) to C(10) is  $\pm 0.016$  (8) Å]. The fused cyclopentane ring has a slightly twisted conformation with a torsion angle C(1)—C(11)—C(12)—C(9) of 9.52 (6)°. The potential molecular C<sub>2</sub> symmetry is not retained in the solid state. The carboxylic acid groups form intermolecular hydrogen bonds resulting in zigzag chains along the *b* axis; the O(1)···O(3) ( $\frac{3}{2} - x, y - \frac{1}{2}, 2 - z$ ) and O(4)···O(2) ( $\frac{3}{2} - x, \frac{1}{2} + y, 2 - z$ ) distances are 2.610 (4) and 2.655 (3) Å, respectively.

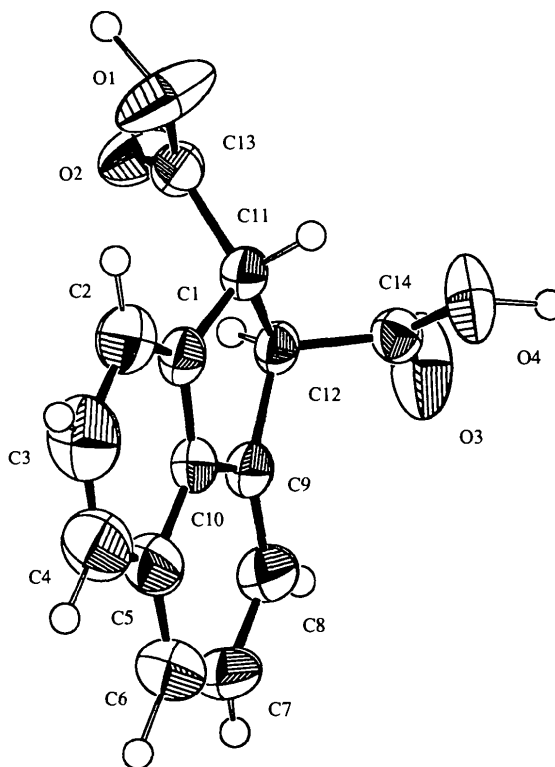


Fig. 1. Perspective drawing of the title compound with the crystallographic numbering scheme. Non-H atoms are shown with 50% probability ellipsoids.

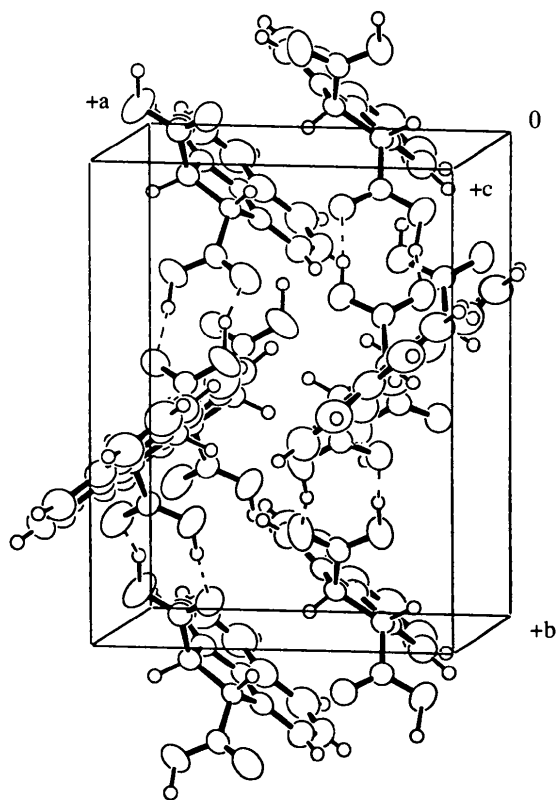


Fig. 2. A view of the unit cell showing intermolecular hydrogen bonding.

A search of the Cambridge Structural Database (Allen, Kennard & Taylor, 1983) revealed that the structures of the *trans*-dichloro and *trans*-dibromo derivatives of acenaphthylene (LeBihan & Perucaud, 1972), along with several *cis* derivatives, have been determined.

## Experimental

The compound was prepared according to the literature by slow carboxylation of the sodium radical anion obtained by addition of one equivalent of metal to a tetrahydrofuran solution of acenaphthylene (Canceill & Jacques, 1973). Subsequent resolution of its brucine salt, followed by acidification and recrystallization, gave almost pure (+)-acenaphthylene-*trans*-1,2-dicarboxylic acid. Recrystallization was from benzene-acetic acid (9:1) solution.

### Crystal data

$C_{14}H_{10}O_4$   
 $M_r = 242.23$   
 Orthorhombic  
 $P2_12_12$   
 $a = 9.987(4) \text{ \AA}$   
 $b = 12.937(4) \text{ \AA}$   
 $c = 8.921(2) \text{ \AA}$   
 $V = 1152.7(6) \text{ \AA}^3$   
 $Z = 4$   
 $D_x = 1.396 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation  
 $\lambda = 0.71069 \text{ \AA}$   
 Cell parameters from 25 reflections  
 $\theta = 10\text{--}15^\circ$   
 $\mu = 0.103 \text{ mm}^{-1}$   
 $T = 295 \text{ K}$   
 Prismatic  
 $0.40 \times 0.32 \times 0.30 \text{ mm}$   
 Colourless

### Data collection

Rigaku AFC-6S diffractometer  
 $\omega$ - $2\theta$  scans  
 Absorption correction: empirical, three  $\psi$  scans  
 $T_{\min} = 0.9159$ ,  $T_{\max} = 0.9989$   
 2292 measured reflections  
 2187 independent reflections

1616 observed reflections [ $I > 3.0\sigma(I)$ ]  
 $R_{\text{int}} = 0.039$   
 $\theta_{\max} = 25^\circ$   
 $h = -11 \rightarrow 11$   
 $k = 0 \rightarrow 15$   
 $l = 0 \rightarrow 10$   
 3 standard reflections  
 frequency: 120 min  
 intensity decay: none

### Refinement

Refinement on  $F$   
 $R = 0.0414$   
 $wR = 0.0457$   
 $S = 1.97$   
 1616 reflections  
 194 parameters  
 Only coordinates of H atoms refined  
 $w = 1/\sigma^2(F_o)$

$(\Delta/\sigma)_{\max} = 0.15$   
 $\Delta\rho_{\max} = 0.01 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.30 \text{ e \AA}^{-3}$   
 Atomic scattering factors from Cromer & Mann (1968) and Stewart, Davidson & Simpson (1965)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$$

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}$
O(1)	0.8934 (3)	-0.1033 (2)	0.8456 (4)	0.095 (2)
O(2)	0.6875 (2)	-0.0908 (2)	0.9294 (3)	0.061 (1)
O(3)	0.5768 (3)	0.2391 (2)	0.9748 (4)	0.090 (2)
O(4)	0.7885 (2)	0.2363 (2)	0.9124 (3)	0.069 (2)
C(1)	0.7609 (3)	0.0261 (2)	0.5986 (3)	0.042 (2)
C(2)	0.8369 (4)	-0.0315 (3)	0.5016 (4)	0.060 (2)
C(3)	0.8043 (5)	-0.0223 (4)	0.3468 (5)	0.081 (3)
C(4)	0.7083 (5)	0.0390 (4)	0.2922 (5)	0.078 (3)
C(5)	0.6269 (4)	0.0977 (3)	0.3920 (4)	0.062 (2)
C(6)	0.5203 (5)	0.1622 (4)	0.3544 (6)	0.080 (3)
C(7)	0.4531 (5)	0.2140 (4)	0.4643 (7)	0.081 (3)
C(8)	0.4842 (4)	0.2045 (3)	0.6194 (4)	0.059 (2)
C(9)	0.5879 (3)	0.1402 (2)	0.6563 (4)	0.046 (2)
C(10)	0.6584 (3)	0.0877 (2)	0.5449 (4)	0.044 (2)
C(11)	0.7710 (3)	0.0424 (2)	0.7686 (3)	0.040 (2)
C(12)	0.6461 (3)	0.1082 (2)	0.8074 (4)	0.038 (2)
C(13)	0.7820 (3)	-0.0567 (2)	0.8554 (4)	0.043 (2)
C(14)	0.6719 (3)	0.2002 (2)	0.9075 (4)	0.041 (2)

Table 2. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

C(13)—O(1)	1.268 (4)	C(10)—C(9)	1.395 (4)
C(14)—O(3)	1.231 (3)	C(13)—O(2)	1.233 (3)
C(11)—C(1)	1.534 (4)	C(14)—O(4)	1.256 (3)
C(12)—C(11)	1.550 (4)	C(12)—C(9)	1.526 (4)
C(14)—C(12)	1.510 (4)	C(13)—C(11)	1.502 (4)
C(2)—C(1)	1.371 (4)	C(10)—C(1)	1.383 (4)
C(3)—C(2)	1.424 (4)	C(4)—C(3)	1.336 (6)
C(5)—C(4)	1.425 (6)	C(6)—C(5)	1.394 (6)
C(10)—C(5)	1.406 (5)	C(7)—C(6)	1.364 (6)
C(8)—C(7)	1.424 (6)	C(9)—C(8)	1.368 (5)
C(11)—C(1)—C(2)	131.5 (3)	C(11)—C(12)—C(9)	105.0 (2)
C(12)—C(9)—C(8)	131.8 (3)	C(14)—C(12)—C(11)	115.3 (2)
C(12)—C(11)—C(1)	104.1 (2)	C(11)—C(13)—O(1)	115.7 (3)
C(13)—C(11)—C(12)	114.3 (2)	O(4)—C(14)—O(3)	123.1 (3)
C(14)—C(12)—C(9)	111.9 (2)	C(12)—C(14)—O(4)	118.2 (3)
O(2)—C(13)—O(1)	122.5 (3)	C(3)—C(2)—C(1)	116.1 (4)
C(11)—C(13)—O(2)	121.7 (3)	C(5)—C(4)—C(3)	119.9 (4)
C(12)—C(14)—O(3)	118.6 (3)	C(10)—C(5)—C(4)	115.4 (4)
C(10)—C(1)—C(2)	120.3 (3)	C(7)—C(6)—C(5)	119.8 (4)
C(4)—C(3)—C(2)	124.5 (4)	C(8)—C(7)—C(6)	123.2 (4)
C(6)—C(5)—C(4)	127.2 (4)	C(10)—C(9)—C(8)	120.5 (3)

C(10)—C(5)—C(6)	117.3 (4)	C(9)—C(10)—C(1)	114.1 (3)	
C(11)—C(1)—C(10)	108.2 (3)	C(9)—C(8)—C(7)	116.8 (4)	
C(12)—C(9)—C(10)	107.8 (2)	C(5)—C(10)—C(1)	123.7 (3)	
C(13)—C(11)—C(1)	113.4 (2)	C(9)—C(10)—C(5)	122.3 (3)	
<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>
O(1)—H(O1)...O(3 <sup>1</sup> )	1.12 (4)	1.55 (4)	2.610 (4)	155 (1)
O(4)—H(O4)...O(2 <sup>1</sup> )	1.01 (4)	1.65 (4)	2.655 (3)	175 (3)

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

Based on the systematic absences ( $h00: h \neq 2n$  and  $0k0: k \neq 2n$ ), the space group was uniquely determined to be  $P2_12_12$ . Lorentz-polarization and empirical absorption corrections were applied. The structure was solved by direct methods using *SAPI91* (Fan, 1991) and refined by full-matrix least-squares calculations with the non-H atoms anisotropic. Allowance was made for anomalous dispersion (Cromer & Liberman, 1970). H atoms were located from a  $\Delta F$  map and were included in the refinement cycles allowing an overall isotropic temperature factor to refine [final value 0.074 (3) Å]. Reflections were collected in two quadrants ( $hkl$  and  $-hkl$ ) and were not merged. At the final stages of refinement, parallel and independent calculations on the two stereoisomers of the molecule converged with  $R = 0.0414$  and  $0.0438$  and  $wR = 0.0457$  and  $0.0587$ . A statistical test on the  $wR$ -factor ratio (Hamilton, 1965) indicated that the latter stereoisomer could be rejected at the 0.005 significance level as being the configuration present in this crystal. Accordingly, all coordinates reported in this paper refer to the statistically favoured configuration. Computer programs used in this study were *TEXSAN* (Molecular Structure Corporation, 1993) and *SHELX76* (Sheldrick, 1976). The figures were drawn using *ORTEPII* (Johnson, 1976).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, bond distances and angles involving H atoms and least-squares-planes data have been deposited with the IUCr (Reference: FG1008). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Steric Effects in Heteroboranes. III. 1-Ph-2-Me-1,2-*closo*-C<sub>2</sub>B<sub>10</sub>H<sub>10</sub>

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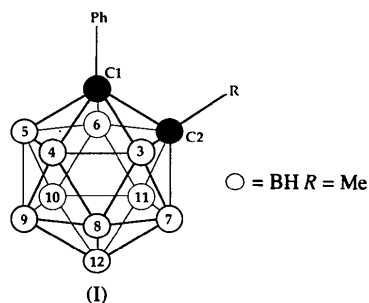
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### Abstract

The crystallographically determined structure of 2-methyl-1-phenyl-1,2-dicarba-*closo*-dodecaborane(12), C<sub>9</sub>H<sub>18</sub>B<sub>10</sub>, is reported. The 12-vertex polyhedron is relatively undistorted although the phenyl substituent is twisted by *ca* 73° from its electronically preferred orientation by steric pressure from the methyl group, resulting in a C—C(cage) separation of 1.695 (5) Å.

### Comment

In the first two papers in this series (Lewis & Welch, 1993; Baghurst *et al.*, 1993) we have demonstrated that unusual polyhedral structures and unusually facile polyhedral isomerizations can result as a consequence of intramolecular crowding in carbametalaboranes. These findings have prompted us to study the stereochemical consequences of systematically varying the amount of crowding within a homologous series of heteroboranes. In this and subsequent papers we report the molecular structures of a series of *closo* carbaboranes 1-Ph-2-*R*-1,2-C<sub>2</sub>B<sub>10</sub>H<sub>10</sub>, in which the steric demand of the changing group *R* is gradually increased. In the present contribution *R* = Me.



The title compound, (I), has been synthesized in good yield by direct reaction between  $\text{MeC}\equiv\text{CPh}$  and  $\text{B}_{10}\text{H}_{14}$  in the presence of *N,N*-dimethylaniline.

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