

Refinement

Refinement on *F**R* = 0.061*wR* = 0.058*S* = 2.21

589 reflections

85 parameters

Only H-atom *U*'s refined*w* = 1/*σ*²(*F*) $(\Delta/\sigma)_{\max} = 0.02$ $\Delta\rho_{\max} = 0.23 \text{ e } \text{Å}^{-3}$ $\Delta\rho_{\min} = -0.25 \text{ e } \text{Å}^{-3}$

Extinction correction: none

Atomic scattering factors
from *International Tables*
for *X-ray Crystallography*
(1974, Vol. IV)Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$B_{\text{eq}} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq}
O(1)	-0.3451 (8)	0.0355 (2)	-0.2166 (2)	6.4 (2)
C(1)	-0.125 (1)	0.2097 (4)	-0.0757 (4)	4.8 (2)
C(2)	-0.019 (1)	0.2955 (4)	-0.0034 (5)	5.5 (2)
C(3)	0.154 (1)	0.2739 (4)	0.1101 (4)	5.6 (3)
C(4)	0.217 (1)	0.1719 (4)	0.1477 (4)	5.1 (2)
C(5)	0.109 (1)	0.0879 (3)	0.0748 (4)	3.8 (2)
C(6)	-0.063 (1)	0.1066 (3)	-0.0381 (3)	3.9 (2)
C(7)	-0.184 (1)	0.0198 (3)	-0.1176 (4)	4.2 (2)
C(8)	-0.091 (2)	0.4038 (6)	-0.0407 (8)	6.0 (5)

Table 2. Selected geometric parameters (Å, °)

O(1)—C(7)	1.232 (4)	C(3)—C(4)	1.383 (5)
C(1)—C(2)	1.396 (5)	C(4)—C(5)	1.385 (5)
C(1)—C(6)	1.396 (5)	C(5)—C(6)	1.388 (5)
C(2)—C(3)	1.402 (6)	C(5)—C(7 ⁱⁱ)	1.476 (5)
C(2)—C(8)	1.464 (8)	C(6)—C(7)	1.467 (5)
C(2)—C(1)—C(6)	122.3 (4)	C(6)—C(5)—C(7 ⁱⁱ)	121.2 (3)
C(1)—C(2)—C(3)	117.0 (4)	C(1)—C(6)—C(5)	119.3 (4)
C(1)—C(2)—C(8)	122.6 (6)	C(1)—C(6)—C(7)	119.7 (4)
C(3)—C(2)—C(8)	120.4 (5)	C(5)—C(6)—C(7)	121.0 (4)
C(2)—C(3)—C(4)	121.0 (4)	O(1)—C(7)—C(5 ⁱⁱ)	120.7 (4)
C(3)—C(4)—C(5)	121.1 (4)	O(1)—C(7)—C(6)	121.5 (4)
C(4)—C(5)—C(6)	119.3 (4)	C(5 ⁱⁱ)—C(7)—C(6)	117.8 (3)
C(4)—C(5)—C(7 ⁱⁱ)	119.5 (4)		

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

The crystals of the title compound have a tendency to grow as twinned crystals. The H atoms of the disordered methyl group were not included in the refinement. The other H atoms were fixed at calculated positions and only their *B*_{iso}'s were refined. The structure refinement was carried out at the X-ray Laboratory of Okayama University.

Data collection: RASAI (Rigaku Corporation, 1990). Cell refinement: RASAI. Data reduction: TEXSAN (Molecular Structure Corporation, 1985). Program(s) used to solve structure: MITHRIL (Gilmore, 1984). Program(s) used to refine structure: TEXSAN. Molecular graphics: ORTEPII (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: AS1161). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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4,5-Dihydrobenzo[*a*]pyrene-4,5-trans-(*e,e*)-diol

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Abstract

The title compound, C₂₀H₁₄O₂, is a metabolite of the chemical carcinogen benzo[*a*]pyrene. The crystal structure consists of groups of molecules in each of which both hydroxy groups donate and accept hydrogen bonds. Stacking of dihydrobenzo[*a*]pyrene ring systems occurs along the crystallographic *c* axis.

Comment

A comparison of the present results for the title compound, (I), with the results for benzo[*a*]pyrene (Iball, Scrimgeour & Young, 1976), benzo[*a*]pyrene 4,5-oxide (Glusker, Zacharias, Fu & Harvey, 1976) and (±)-7α,8β-dihydroxy-7,8-dihydrobenzo[*a*]pyrene (Neidle, Subbiah & Osborne, 1981) shows mainly a lengthening in the *K* region [*i.e.* the C(4)—C(5) bond], where the ring is now partially saturated and dihydroxylation

has occurred. Both hydroxy groups are equatorial, as expected in view of the lack of steric hindrance in this area of the molecule.

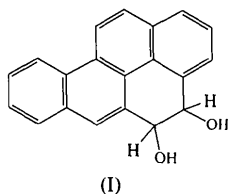


Fig. 1 shows the atomic numbering scheme used; the packing in the unit cell of the title compound is shown by a stereoview in Fig. 2. The crystal structure consists of a hydrogen-bonded stacks of 4,5-dihydroxy-4,5-dihydrobenzo[*a*]pyrene molecules in which both hydroxy groups participate as donor and acceptor. This hydrogen-bonding scheme is illustrated in Fig. 3, which shows a view down the *c* axis (Fig. 3*a*) and the hydrogen bonding between molecules running parallel to the *c* direction. Molecules also stack along this direction.

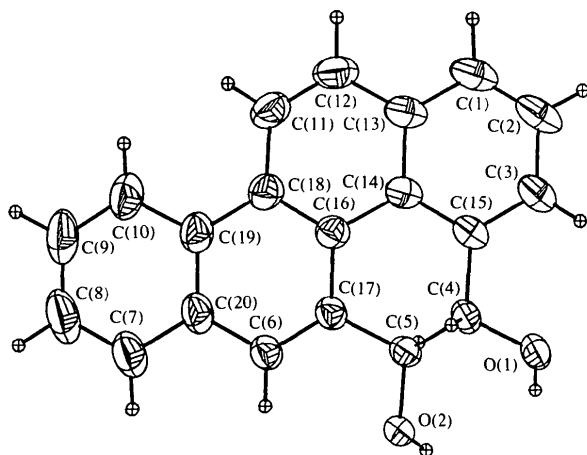


Fig. 1. Displacement ellipsoid (50%) representation of the title compound, drawn using *SNOOPI* (Davies, 1983) and *ORTEP* (Johnson, 1965), showing the atomic numbering scheme.

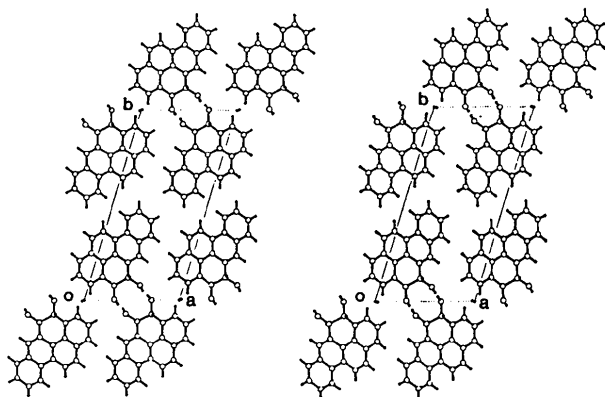


Fig. 2. Stereoview of the packing in the unit cell viewed along *c* (Erlebacher & Carrell, 1992)

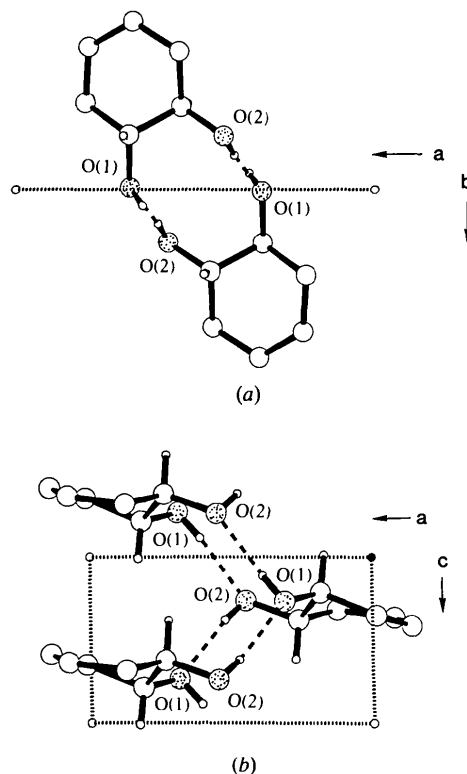


Fig. 3. Detailed views of hydrogen bonding (Erlebacher & Carrell, 1992) viewed (a) down the *c* axis and (b) in the *b* direction. In both views only the ring bearing the two hydroxy groups is shown.

Experimental

Crystal data

$C_{20}H_{14}O_2$
 $M_r = 286.33$
 Triclinic
 $P\bar{1}$
 $a = 9.3901(15) \text{ \AA}$
 $b = 18.497(3) \text{ \AA}$
 $c = 4.2114(5) \text{ \AA}$
 $\alpha = 90.65(1)^\circ$
 $\beta = 102.86(1)^\circ$
 $\gamma = 73.09(1)^\circ$
 $V = 681.1(2) \text{ \AA}^3$
 $Z = 2$
 $D_x = 1.396 \text{ Mg m}^{-3}$

Cu $K\alpha$ radiation
 $\lambda = 1.5418 \text{ \AA}$
 Cell parameters from 16 reflections
 $\theta = 20.6\text{--}28.8^\circ$
 $\mu = 0.621 \text{ mm}^{-1}$
 $T = 294 \text{ K}$
 Prismatic
 $0.45 \times 0.12 \times 0.10 \text{ mm}$
 Amber

Data collection

Nicolet Siemens *P3m* diffractometer
 Variable ω - 2θ scans
 Absorption correction:
 ψ scan (North, Phillips & Mathews, 1968)
 $T_{\min} = 0.942$, $T_{\max} = 1.000$
 2902 measured reflections
 2528 independent reflections

2241 observed reflections
 $[I > 3\sigma(I)]$
 $R_{\text{int}} = 0.014$
 $\theta_{\text{max}} = 69.5^\circ$
 $h = -11 \rightarrow 11$
 $k = -22 \rightarrow 22$
 $l = 0 \rightarrow 5$
 4 standard reflections
 frequency: 120 min
 intensity decay: < 1%

Refinement

Refinement on *F**R* = 0.045*wR* = 0.058*S* = 0.85

2241 reflections

255 parameters

All H-atom parameters refined

$$w = 1/\sigma^2(F)$$

$$(\Delta/\sigma)_{\max} = 0.13$$

$$\Delta\rho_{\max} = 0.22 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.25 \text{ e } \text{\AA}^{-3}$$

Atomic scattering factors

from *International Tables*for *X-ray Crystallography*

(1974, Vol. IV)

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

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

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
O(1)	0.3141 (1)	0.00280 (5)	0.2616 (3)	0.0422 (4)
O(2)	0.5321 (1)	0.07655 (5)	0.1683 (3)	0.0378 (4)
C(1)	-0.1605 (2)	0.2114 (1)	0.3101 (5)	0.0601 (9)
C(2)	-0.1188 (2)	0.1343 (1)	0.3540 (5)	0.0641 (9)
C(3)	0.0210 (2)	0.0897 (1)	0.2918 (4)	0.0507 (7)
C(4)	0.2670 (1)	0.07779 (7)	0.1185 (3)	0.0350 (5)
C(5)	0.3897 (1)	0.11777 (7)	0.2345 (3)	0.0343 (5)
C(6)	0.4301 (2)	0.22868 (7)	-0.0332 (4)	0.0401 (5)
C(7)	0.4780 (2)	0.3383 (1)	-0.2701 (5)	0.0595 (8)
C(8)	0.4317 (3)	0.4136 (1)	-0.3715 (7)	0.076 (1)
C(9)	0.2851 (3)	0.4586 (1)	-0.3578 (7)	0.073 (1)
C(10)	0.1881 (3)	0.42907 (9)	-0.2437 (6)	0.0594 (9)
C(11)	-0.0182 (2)	0.36009 (9)	0.0185 (5)	0.0539 (8)
C(12)	-0.1090 (2)	0.3273 (1)	0.1342 (5)	0.0569 (8)
C(13)	-0.0652 (2)	0.2476 (1)	0.2080 (4)	0.0490 (7)
C(14)	0.0802 (1)	0.20346 (8)	0.1650 (4)	0.0396 (6)
C(15)	0.1192 (1)	0.12352 (8)	0.2016 (3)	0.0393 (6)
C(16)	0.1814 (2)	0.24022 (7)	0.0716 (3)	0.0376 (5)
C(17)	0.3364 (1)	0.19708 (7)	0.0773 (3)	0.0353 (5)
C(18)	0.1314 (2)	0.31807 (8)	-0.0185 (4)	0.0434 (6)
C(19)	0.2310 (2)	0.35181 (8)	-0.1363 (4)	0.0461 (7)
C(20)	0.3797 (2)	0.30630 (8)	-0.1508 (4)	0.0448 (6)

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

O(1)—C(4)	1.423 (2)	C(8)—C(9)	1.400 (4)
O(2)—C(5)	1.420 (2)	C(9)—C(10)	1.359 (4)
C(1)—C(2)	1.367 (3)	C(10)—C(19)	1.417 (2)
C(1)—C(13)	1.402 (3)	C(11)—C(12)	1.351 (3)
C(2)—C(3)	1.411 (3)	C(11)—C(18)	1.436 (2)
C(3)—C(15)	1.372 (2)	C(12)—C(13)	1.427 (3)
C(4)—C(5)	1.533 (2)	C(13)—C(14)	1.421 (2)
C(4)—C(15)	1.513 (2)	C(14)—C(15)	1.417 (2)
C(5)—C(17)	1.513 (2)	C(14)—C(16)	1.438 (2)
C(6)—C(17)	1.349 (2)	C(16)—C(17)	1.437 (2)
C(6)—C(20)	1.431 (2)	C(16)—C(18)	1.406 (2)
C(7)—C(8)	1.376 (3)	C(18)—C(19)	1.436 (2)
C(7)—C(20)	1.409 (2)	C(19)—C(20)	1.419 (2)
O(1)—C(4)—C(5)	111.3 (1)	O(2)—C(5)—C(4)	111.7 (1)
O(1)—C(4)—C(15)	110.6 (1)	O(2)—C(5)—C(17)	110.7 (1)
C(5)—C(4)—C(15)	109.2 (1)	C(4)—C(5)—C(17)	109.9 (1)
O(1)—C(4)—C(5)—O(2)	56.6 (1)		
O(1)—C(4)—C(5)—C(17)	179.8 (1)		
C(15)—C(4)—C(5)—O(2)	178.9 (1)		
C(15)—C(4)—C(5)—C(17)	-57.8 (1)		

<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...O(2) ⁱ	0.83 (2)	1.89 (2)	2.718 (1)	175 (2)
O(2)—H...O(1) ⁱⁱ	0.78 (3)	1.93 (3)	2.703 (1)	170 (3)

Symmetry codes: (i) 1 - *x*, -*y*, -*z*; (ii) 1 - *x*, -*y*, 1 - *z*.

The structure was solved using *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). For all other crystallographic calculations, in-house programs were used (Carrell, Shieh & Takusagawa, 1981). The structure refinement was carried out using a least-squares procedure. H atoms were located from difference Fourier maps and included in the final refinements.

This work was supported by grants CN10 (to JPG) and CN-22 (to RGH) from the American Cancer Society, grants CA-10925 and CA-06927 from the National Institutes of Health, and an appropriation from the Commonwealth of Pennsylvania.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, least-squares-planes data with r.m.s deviations, along with comparison of the molecular geometry of various benzo[*a*]pyrene moieties reported in the literature have been deposited with the IUCr (Reference: SZ1006). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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DL-Proline Monohydrate

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Abstract

In the crystal of the title compound, C₅H₉NO₂·H₂O, hydrogen-bonded centrosymmetric dimers of proline molecules stack along *a* to form columns stabilized by S1-type head-to-tail sequences. These columns are interconnected by water molecules to give rise to