

C6—C7	1.441 (2)	C17—C18	1.388 (3)
C7—C15	1.364 (2)	C18—C19	1.373 (4)
C7—C8	1.460 (2)	C19—C20	1.367 (4)
C8—O22	1.217 (2)	C20—C21	1.388 (3)
C15—S—C1	90.10 (8)	C10—N9—C16	117.23 (14)
C6—C1—C2	125.7 (2)	C8—N9—C16	119.77 (14)
C6—C1—S	112.76 (13)	N11—C10—N14	111.7 (2)
C2—C1—S	121.54 (15)	N11—C10—N9	128.1 (2)
C1—C2—C3	110.1 (2)	N14—C10—N9	120.14 (15)
C1—C2—C3'	108.3 (7)	C10—N11—N12	105.71 (14)
C4—C3—C2	111.8 (3)	C13—N12—N11	108.12 (15)
C4'—C3'—C2	112.1 (14)	N12—C13—N14	110.5 (2)
C3—C4—C5	112.7 (2)	C10—N14—C13	103.93 (14)
C3'—C4'—C5	107.6 (14)	C10—N14—C15	120.30 (14)
C6—C5—C4	111.2 (2)	C13—N14—C15	135.7 (2)
C6—C5—C4'	110.6 (6)	C7—C15—N14	120.6 (2)
C1—C6—C7	111.9 (2)	C7—C15—S	113.94 (13)
C1—C6—C5	121.8 (2)	N14—C15—S	125.40 (13)
C7—C6—C5	126.4 (2)	C17—C16—C21	121.6 (2)
C15—C7—C6	111.3 (2)	C17—C16—N9	118.9 (2)
C15—C7—C8	121.3 (2)	C21—C16—N9	119.4 (2)
C6—C7—C8	127.3 (2)	C16—C17—C18	118.8 (2)
O22—C8—N9	120.4 (2)	C19—C18—C17	120.3 (2)
O22—C8—C7	125.0 (2)	C20—C19—C18	120.1 (2)
N9—C8—C7	114.67 (15)	C19—C20—C21	120.8 (2)
C10—N9—C8	122.92 (15)	C16—C21—C20	118.3 (2)
C1—C2—C3—C4	-44.7 (3)	C1—C2—C3'—C4'	45.6 (17)
C2—C3—C4—C5	61.2 (4)	C2—C3'—C4'—C5	-67.5 (19)
C3—C4—C5—C6	-43.0 (4)	C3'—C4'—C5—C6	52.5 (16)
C4—C5—C6—C1	13.0 (3)	C4'—C5—C6—C1	-21.0 (8)
C5—C6—C1—C2	0.6 (3)	C6—C1—C2—C3'	-12.0 (9)
C6—C1—C2—C3	15.3 (3)		

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## Disordered Structure of 2-Methyl-anthraquinone

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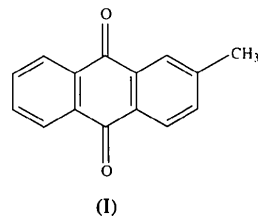
(Received 28 November 1994; accepted 25 March 1995)

### Abstract

Crystals of the title compound, C<sub>15</sub>H<sub>10</sub>O<sub>2</sub>, are monoclinic, space group *P*<sub>2</sub><sub>1</sub>/*c*. The molecules are disordered around a centre of symmetry. Packing considerations and the calculation of the intermolecular interaction energies show that the disordered structure has occurred by random packing of structures with space groups *P*<sub>2</sub><sub>1</sub> and *Pc*, the maximal subgroups of *P*<sub>2</sub><sub>1</sub>/*c*.

### Comment

A certain kind of structural disorder has been observed in homomolecular crystals like those of azulene (Robertson, Shearer, Sim & Watson, 1962) and 1-bromo-4-chlorobenzene (Klug, 1947). In these crystals, each molecule is apparently disordered in two orientations related by a  $\bar{1}$  symmetry operation. Here, we comment on such a structural disorder as found in 2-methylanthraquinone, (I).



The molecular arrangement is characterized by the formation of a molecular sheet in (10 $\bar{2}$ ) and molecular stacking along the *a* axis with an interplanar spacing

Disorder of atoms C3 and C4 was observed after anisotropic refinement of the non-H atoms. C3 and C4 were assigned occupancies of *p* and C3' and C4' were assigned occupancies of 1–*p*, with *p* = 0.850 (6) at convergence. All the H atoms belonging to the major conformer were located from difference Fourier maps and refined isotropically.

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990a). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1990b). Software used to prepare material for publication: *SHELXL93*. Geometric calculations: *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1178). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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of 3.493 (4) Å. Stacked molecules are displaced relative to one another by 1.64 Å along the dipole axes of the carbonyl groups. The weak intermolecular C—H...O interactions observed in the sheet are C(3)···O(1<sup>iii</sup>) 3.578 (7) and C(8<sup>v</sup>)···O(1<sup>iv</sup>) 3.66 (1) Å (Fig. 2). The non-bonding distance C(8)···C(8<sup>vi</sup>) of 2.69 (2) Å is too short for a normal van der Waals contact. Thus, the two methyl groups cannot actually coexist as seen in the apparent  $P2_1/c$  structure. It means that the  $\bar{1}$  symmetry at (0,  $\frac{1}{2}$ , 0), and thus at (0, 0, 0), is generated as a result of disordering.

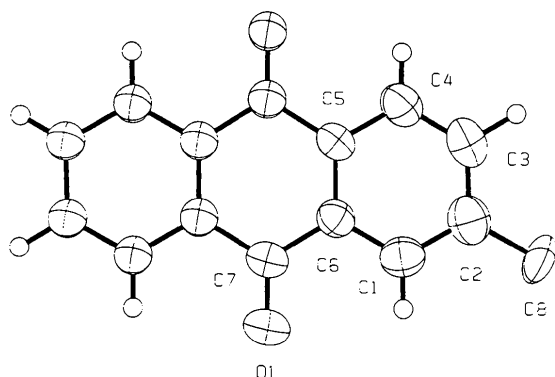


Fig. 1. The displacement ellipsoids, with atomic numbering, plotted at 50% probability for the non-H atoms. H atoms are represented as spheres equivalent to  $B = 1.0 \text{ \AA}^2$ . The molecule has  $\bar{1}$  symmetry by disordering. One of the disordered methyl groups has been omitted to show an actual molecular unit.

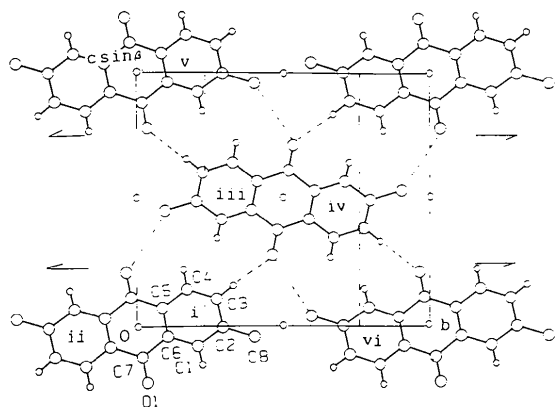


Fig. 2. The crystal packing viewed along the  $a$  axis with the atomic numbering. Symmetry codes: (i)  $x, y, z$ ; (ii)  $-x, -y, -z$ ; (iii)  $x, \frac{1}{2} - y, \frac{1}{2} + z$ ; (iv)  $-x, \frac{1}{2} + y, \frac{1}{2} - z$ ; (v)  $x, y, 1 + z$ ; (vi)  $-x, 1 - y, -z$ .

The disordered  $P2_1/c$  structure can be built from random packing of  $Pc$  and  $P2_1$  structures. The energy contributions of each structure,  $P2_1$  and  $Pc$ , are equivalent; the sum total of the van der Waals interaction energy for each structure has been estimated to be  $-107.9 \text{ kJ mol}^{-1}$  for eight neighbouring molecules by assuming Kitaigorosky's universal potential (Kita-

gorosky, 1973). An antiparallel stacking of molecules along the  $a$  axis, which may occur on disordering, is more stable by  $0.5 \text{ kJ mol}^{-1}$  per molecular pair than a parallel stacking by a genuine translation along the  $a$  axis.

The observed stacking does not show a well defined induced dipole-dipole interaction as observed in naphthoquinone and benzoquinone (Gaultier & Hauw, 1965), where a polar carbonyl group of one molecule is placed at the centre of the polarizable system of a six-membered ring (Wright, 1988). However, some degree of dipole-dipole contribution can be deduced from a calculation of charge distribution in the molecule by AM1 methods (Dewar, Zoebisch, Healy & Stewart, 1985). The carbonyl atom O(1) with the highest negative net atomic charge of  $-0.29$  is next to carbonyl C(7) ( $1 + x, y, z$ ) with the highest positive net atomic charge,  $+0.31$ . The charge distribution shows that there exist more attractive pairs of atoms than repulsive pairs between the overlapping molecules. This shows an inherent, yet minimal, electrostatic contribution to the stacking.

The packing coefficient (Kitaigorosky, 1961) for the ordered structure model is 0.706 for both the  $P2_1$  and  $Pc$  structures. This figure is lower than those of most of the non-disordered analogues: 0.78 for benz[*a*]anthracene-7,12-dione (Kuroda, Neidle & Subbiah, 1982), 0.75 for 1,4-dihydroxyanthraquinone (Nigam & Deppisch, 1980) and 0.72 for anthralin (Ahmed, 1980) and benzanthrone (Tafeenko, Solodar & Medvedev, 1988).

## Experimental

The title compound was purchased from Aldrich Chemical Co. and crystals suitable for X-ray analysis were obtained by slow evaporation from 2-butanone.

### Crystal data

$\text{C}_{15}\text{H}_{10}\text{O}_2$   
 $M_r = 222.24$   
 Monoclinic  
 $P2_1/c$   
 $a = 3.86 (1) \text{ \AA}$   
 $b = 12.771 (6) \text{ \AA}$   
 $c = 10.994 (4) \text{ \AA}$   
 $\beta = 93.93 (8)^\circ$   
 $V = 541 (2) \text{ \AA}^3$   
 $Z = 2$   
 $D_x = 1.365 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation  
 $\lambda = 0.71073 \text{ \AA}$   
 Cell parameters from 25 reflections  
 $\theta = 9-11^\circ$   
 $\mu = 0.084 \text{ mm}^{-1}$   
 $T = 295 \text{ K}$   
 Prismatic  
 $0.50 \times 0.25 \times 0.15 \text{ mm}$   
 Yellow

### Data collection

Rigaku AFC-5R diffractometer  
 $\omega/2\theta$  scans  
 Absorption correction: none  
 1102 measured reflections  
 961 independent reflections  
 589 observed reflections  
 $[I > 3\sigma(I)]$

$R_{\text{int}} = 0.017$   
 $\theta_{\text{max}} = 25^\circ$   
 $h = 0 \rightarrow 4$   
 $k = 0 \rightarrow 14$   
 $l = -13 \rightarrow 13$   
 3 standard reflections monitored every 97 reflections  
 intensity decay: 1%

## 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*<sup>2</sup>(*F*) $(\Delta/\sigma)_{\max} = 0.02$  $\Delta\rho_{\max} = 0.23 \text{ e } \text{\AA}^{-3}$  $\Delta\rho_{\min} = -0.25 \text{ e } \text{\AA}^{-3}$ 

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 ( $\text{\AA}^2$ )

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

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> <sub>eq</sub>
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 ( $\text{\AA}$ , °)

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 <sup>ii</sup> )	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 <sup>ii</sup> )	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 <sup>ii</sup> )	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 <sup>ii</sup> )—C(7)—C(6)	117.8 (3)
C(4)—C(5)—C(7 <sup>ii</sup> )	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*<sub>iso</sub>'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, C<sub>20</sub>H<sub>14</sub>O<sub>2</sub>, 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 (±)-7 $\alpha$ ,8 $\beta$ -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