1* lists the final fractional coordinates and the equivalent isotropic thermal parameters. In Table 2 the bond lengths and bond angles are listed. The molecular conformation with the numbering scheme and the crystal packing are shown in Figs. 1 and 2.

Related literature. The conformation observed in this work differs notably from that of the *m*-nitrobenzoyl derivative of praziquantel (Toscano, Rubio, Cetina & Pérez-Ibarra, 1991), while it correlates well with the predicted minimum-energy conformation calculated by molecular mechanics for praziquantel and some alkyl and cycloalkyl derivatives (Rubio, Escobar-Valderrama & Cetina, 1990).

References

Rubio, M., Escobar-Valderrama, J. L. & Cetina, R. (1990). *Rev. Latinoam. Quím.* **21**, 130–133.

Sheldrick, G. M. (1983). *SHELXTL*. Revision 4.1. *An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data.* Nicolet XRD Corporation, Madison, Wisconsin, USA.

Toscano, R. A., Rubio, M., Cetina, R. & Pérez-Ibarra, B. M. (1991). *Acta Cryst.* C**47**, 2492–2494.

Vicković, I. (1988). *J. Appl. Cryst.* **21**, 987–990.

---

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55175 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH0582]

Acta Cryst. (1992). C**48**, 1870–1872

Structure of 10-Acetyloxy-10-methyl-9(10*H*)-anthracenone

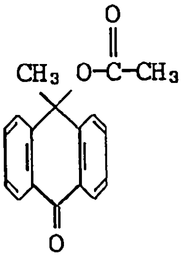
By Tzuen-Yeuan Lin,* Yuh-Lin Mao* and Chih-Ming Chuo

Department of Chemistry, Soochow University, Taipei, Taiwan

(*Received* 20 *August* 1991; *accepted* 11 *February* 1992)

Abstract. 10-Methyl-9-oxo-9,10-dihydroanthracen-10-yl acetate, $C_{17}H_{14}O_3$, $M_r = 266.30$, monoclinic, $P2_1/c$, $a = 7.912$ (1), $b = 27.749$ (8), $c = 13.402$ (3) Å, $\beta = 107.09$ (2)°, $V = 2812$ (1) Å^3, $Z = 8$, $D_x = 1.258$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.70930 Å, $\mu = 0.08$ mm^{-1}, $F(000) = 1119.87$, $T = 298$ K, final $R = 0.046$ for 1825 reflections with $I > 2\sigma(I)$. There are two crystallographically independent molecules in the asymmetric unit, but they are structurally similar. The structure determination confirms that the title compound is one of the products of oxidation of 9-methylanthracene by potassium dichromate.

Experimental. A solution of 9-methylanthracene (3.85 g, 20 mmol) in acetic acid/acetic anhydride (100 ml, 1:1) was cooled to 273 K. Potassium dichromate (15.95 g, 54 mmol) was dissolved in equal volumes of acetic acid and acetic anhydride (100 ml), then added dropwise to the prepared solution of 9-methylanthracene at 273 K. Stirring was continued for 24 h at room temperature. The mixture was poured into 1000 ml of stirred cold water and extracted with hot chloroform. The combined extracts were distilled to yield pale-yellow needles (4.17 g) which were separated on a column chromatograph (silica gel). The two products obtained were recrystallized from ethanol separately. One is 9,10-anthraquinone (54%), the other is 10-acetyloxy-10-methyl-9(10*H*)-anthracenone (I) (37%) whose crystal structure is described.



(I)

A thin colourless plate crystal of dimensions 0.15 × 0.40 × 0.40 mm was selected for study. Cell constants were derived from least-squares refinement of 25 reflections having $19 < 2\theta < 24°$. Intensity data were collected with the $\omega/2\theta$-scan technique with $0 < 2\theta < 45°$ ($h = -8$ to 8, $k = 0$ to 29, $l = 0$ to 14) on a Nonius CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ radiation. Scan rate was

---

* To whom correspondence should be addressed.

Table 1. *Atomic parameters and equivalent isotropic thermal parameters* (Å^2)

B_{eq} is defined as one third of the trace of the orthogonalized **B** tensor.

	x	*y*	*z*	B_{eq}
C(1*A*)	0.4811 (6)	0.84580 (19)	0.0696 (3)	3.7 (3)
C(2*A*)	0.4578 (7)	0.79827 (21)	0.0370 (4)	4.7 (3)
C(3*A*)	0.5940 (9)	0.77187 (21)	0.0213 (4)	5.6 (3)
C(4*A*)	0.7580 (8)	0.79192 (24)	0.0374 (4)	5.4 (4)
C(5*A*)	0.7875 (7)	0.83876 (22)	0.0731 (4)	4.9 (3)
C(6*A*)	0.6485 (7)	0.86592 (20)	0.0892 (4)	4.2 (3)
C(7*A*)	0.6839 (7)	0.91623 (21)	0.1250 (4)	4.9 (3)
C(8*A*)	0.5315 (7)	0.94705 (22)	0.1211 (4)	4.3 (3)
C(9*A*)	0.5553 (7)	0.99673 (24)	0.1339 (4)	5.2 (3)
C(10*A*)	0.4147 (9)	1.02652 (22)	0.1260 (5)	6.2 (4)
C(11*A*)	0.2474 (8)	1.00757 (24)	0.1042 (5)	5.9 (3)
C(12*A*)	0.2212 (7)	0.95843 (23)	0.0933 (4)	5.2 (3)
C(13*A*)	0.3627 (7)	0.92743 (19)	0.0995 (4)	3.8 (3)
C(14*A*)	0.3329 (6)	0.87417 (19)	0.0904 (4)	3.8 (3)
C(15*A*)	0.2965 (7)	0.85525 (20)	0.1895 (4)	5.4 (3)
C(16*A*)	0.1532 (7)	0.87554 (18)	−0.0902 (4)	3.9 (3)
C(17*A*)	−0.0262 (7)	0.86408 (18)	−0.1592 (4)	4.7 (3)
O(1*A*)	0.8355 (5)	0.93178 (14)	0.1546 (3)	7.4 (3)
O(2*A*)	0.1669 (4)	0.86324 (11)	0.00983 (25)	3.94 (18)
O(3*A*)	0.2710 (5)	0.89314 (13)	−0.1168 (3)	5.09 (21)
C(1*B*)	0.0076 (7)	0.90651 (17)	0.5592 (4)	3.47 (24)
C(2*B*)	0.0715 (7)	0.94243 (19)	0.6330 (4)	4.7 (3)
C(3*B*)	−0.0398 (9)	0.96762 (20)	0.6748 (4)	5.4 (3)
C(4*B*)	−0.2184 (8)	0.95875 (21)	0.6435 (5)	5.5 (3)
C(5*B*)	−0.2846 (7)	0.92293 (21)	0.5720 (4)	4.9 (3)
C(6*B*)	−0.1733 (7)	0.89668 (17)	0.5288 (4)	3.7 (3)
C(7*B*)	−0.2474 (7)	0.85832 (19)	0.4534 (4)	4.5 (3)
C(8*B*)	−0.1266 (7)	0.83020 (17)	0.4102 (4)	3.47 (25)
C(9*B*)	−0.1982 (7)	0.79392 (20)	0.3399 (4)	4.7 (3)
C(10*B*)	−0.0887 (9)	0.76544 (20)	0.3018 (4)	5.6 (4)
C(11*B*)	0.0926 (9)	0.77260 (21)	0.3343 (4)	5.3 (3)
C(12*B*)	0.1631 (7)	0.80946 (20)	0.4041 (4)	4.6 (3)
C(13*B*)	0.0532 (7)	0.83867 (17)	0.4423 (4)	3.6 (3)
C(14*B*)	0.1310 (6)	0.88164 (18)	0.5083 (4)	3.55 (25)
C(15*B*)	0.1918 (7)	0.91814 (19)	0.4412 (4)	5.0 (3)
C(16*B*)	0.2876 (8)	0.83437 (20)	0.6569 (4)	4.4 (3)
C(17*B*)	0.4685 (8)	0.82517 (22)	0.7315 (4)	6.6 (3)
O(1*B*)	−0.4065 (5)	0.84872 (14)	0.4292 (3)	6.60 (22)
O(2*B*)	0.2961 (4)	0.86881 (12)	0.5878 (3)	4.28 (17)
O(3*B*)	0.1559 (5)	0.81387 (13)	0.6584 (3)	5.53 (21)

Table 2. *Bond distances* (Å) *and bond angles* (°)

C(1*A*)—C(2*A*)	1.385 (8)	C(1*B*)—C(2*B*)	1.390 (7)
C(1*A*)—C(6*A*)	1.390 (7)	C(1*B*)—C(6*B*)	1.395 (7)
C(1*A*)—C(14*A*)	1.505 (7)	C(1*B*)—C(14*B*)	1.512 (7)
C(2*A*)—C(3*A*)	1.370 (9)	C(2*B*)—C(3*B*)	1.367 (9)
C(3*A*)—C(4*A*)	1.369 (9)	C(3*B*)—C(4*B*)	1.373 (9)
C(4*A*)—C(5*A*)	1.381 (9)	C(4*B*)—C(5*B*)	1.373 (9)
C(5*A*)—C(6*A*)	1.401 (8)	C(5*B*)—C(6*B*)	1.393 (8)
C(6*A*)—C(7*A*)	1.476 (8)	C(6*B*)—C(7*B*)	1.466 (7)
C(7*A*)—C(8*A*)	1.467 (8)	C(7*B*)—C(8*B*)	1.478 (8)
C(7*A*)—O(1*A*)	1.225 (6)	C(7*B*)—O(1*B*)	1.233 (7)
C(8*A*)—C(9*A*)	1.395 (9)	C(8*B*)—C(9*B*)	1.382 (8)
C(8*A*)—C(13*A*)	1.392 (7)	C(8*B*)—C(13*B*)	1.380 (7)
C(9*A*)—C(10*A*)	1.365 (10)	C(9*B*)—C(10*B*)	1.377 (9)
C(10*A*)—C(11*A*)	1.374 (9)	C(10*B*)—C(11*B*)	1.385 (10)
C(11*A*)—C(12*A*)	1.381 (9)	C(11*B*)—C(12*B*)	1.388 (9)
C(12*A*)—C(13*A*)	1.395 (8)	C(12*B*)—C(13*B*)	1.391 (8)
C(13*A*)—C(14*A*)	1.496 (7)	C(13*B*)—C(14*B*)	1.505 (7)
C(14*A*)—C(15*A*)	1.532 (7)	C(14*B*)—C(15*B*)	1.523 (7)
C(14*A*)—O(2*A*)	1.466 (5)	C(14*B*)—O(2*B*)	1.466 (5)
C(16*A*)—C(17*A*)	1.483 (7)	C(16*B*)—C(17*B*)	1.508 (8)
C(16*A*)—O(2*A*)	1.357 (6)	C(16*B*)—O(2*B*)	1.346 (7)
C(16*A*)—O(3*A*)	1.196 (7)	C(16*B*)—O(3*B*)	1.193 (8)

C(2*A*)—C(1*A*)—C(6*A*)	118.2 (5)	C(2*B*)—C(1*B*)—C(6*B*)	118.4 (5)
C(2*A*)—C(1*A*)—C(14*A*)	121.2 (4)	C(2*B*)—C(1*B*)—C(14*B*)	120.0 (4)
C(6*A*)—C(1*A*)—C(14*A*)	120.4 (5)	C(6*B*)—C(1*B*)—C(14*B*)	121.5 (4)
C(1*A*)—C(2*A*)—C(3*A*)	121.4 (5)	C(1*B*)—C(2*B*)—C(3*B*)	121.0 (5)
C(2*A*)—C(3*A*)—C(4*A*)	120.7 (5)	C(2*B*)—C(3*B*)—C(4*B*)	120.8 (5)
C(3*A*)—C(4*A*)—C(5*A*)	119.5 (5)	C(3*B*)—C(4*B*)—C(5*B*)	119.3 (5)
C(4*A*)—C(5*A*)—C(6*A*)	120.0 (5)	C(4*B*)—C(5*B*)—C(6*B*)	120.7 (5)
C(1*A*)—C(6*A*)—C(5*A*)	120.2 (5)	C(1*B*)—C(6*B*)—C(5*B*)	119.7 (5)
C(1*A*)—C(6*A*)—C(7*A*)	121.4 (5)	C(1*B*)—C(6*B*)—C(7*B*)	120.8 (5)
C(5*A*)—C(6*A*)—C(7*A*)	118.4 (5)	C(5*B*)—C(6*B*)—C(7*B*)	119.5 (5)
C(6*A*)—C(7*A*)—C(8*A*)	117.7 (5)	C(6*B*)—C(7*B*)—C(8*B*)	118.7 (5)
C(6*A*)—C(7*A*)—O(1*A*)	120.8 (5)	C(6*B*)—C(7*B*)—O(1*B*)	120.6 (5)
C(8*A*)—C(7*A*)—O(1*A*)	121.5 (5)	C(8*B*)—C(7*B*)—O(1*B*)	120.7 (5)
C(7*A*)—C(8*A*)—C(9*A*)	119.5 (5)	C(7*B*)—C(8*B*)—C(9*B*)	118.0 (5)
C(7*A*)—C(8*A*)—C(13*A*)	120.5 (5)	C(7*B*)—C(8*B*)—C(13*B*)	121.0 (5)
C(9*A*)—C(8*A*)—C(13*A*)	119.9 (5)	C(9*B*)—C(8*B*)—C(13*B*)	121.0 (5)
C(8*A*)—C(9*A*)—C(10*A*)	120.8 (5)	C(8*B*)—C(9*B*)—C(10*B*)	119.6 (5)
C(9*A*)—C(10*A*)—C(11*A*)	119.8 (6)	C(9*B*)—C(10*B*)—C(11*B*)	120.5 (5)
C(10*A*)—C(11*A*)—C(12*A*)	120.4 (5)	C(10*B*)—C(11*B*)—C(12*B*)	119.5 (5)
C(11*A*)—C(12*A*)—C(13*A*)	120.6 (5)	C(11*B*)—C(12*B*)—C(13*B*)	120.4 (5)
C(8*A*)—C(13*A*)—C(12*A*)	118.4 (5)	C(8*B*)—C(13*B*)—C(12*B*)	119.0 (5)
C(8*A*)—C(13*A*)—C(14*A*)	121.5 (5)	C(8*B*)—C(13*B*)—C(14*B*)	121.6 (5)
C(12*A*)—C(13*A*)—C(14*A*)	120.0 (5)	C(12*B*)—C(13*B*)—C(14*B*)	119.1 (4)
C(1*A*)—C(14*A*)—C(13*A*)	114.8 (4)	C(1*B*)—C(14*B*)—C(13*B*)	114.8 (4)
C(1*A*)—C(14*A*)—C(15*A*)	109.7 (4)	C(1*B*)—C(14*B*)—C(15*B*)	108.7 (4)
C(1*A*)—C(14*A*)—O(2*A*)	109.5 (4)	C(1*B*)—C(14*B*)—O(2*B*)	109.8 (4)
C(13*A*)—C(14*A*)—C(15*A*)	109.4 (4)	C(13*B*)—C(14*B*)—C(15*B*)	109.0 (4)
C(13*A*)—C(14*A*)—O(2*A*)	110.5 (4)	C(13*B*)—C(14*B*)—O(2*B*)	111.0 (4)
C(15*A*)—C(14*A*)—O(2*A*)	102.2 (4)	C(15*B*)—C(14*B*)—O(2*B*)	102.8 (4)
C(17*A*)—C(16*A*)—O(2*A*)	110.3 (5)	C(17*B*)—C(16*B*)—O(2*B*)	110.3 (5)
C(17*A*)—C(16*A*)—O(3*A*)	126.1 (5)	C(17*B*)—C(16*B*)—O(3*B*)	124.9 (5)
O(2*A*)—C(16*A*)—O(3*A*)	123.7 (4)	O(2*B*)—C(16*B*)—O(3*B*)	124.8 (5)
C(14*A*)—O(2*A*)—C(16*A*)	118.0 (4)	C(14*B*)—O(2*B*)—C(16*B*)	117.3 (4)

varied from 1.65 to 8.24° min^{-1} with scan width $(0.65 + 0.35\tan\theta)°$. Three standard reflections, measured every 3600 s, showed small (< 3%) random variations. Data were corrected for Lorentz and polarization effects. No absorption correction was applied. 3676 unique reflections were collected. The space group was $P2_1/c$ (systematic absences: $h0l$, l = odd; $0k0$, k = odd). Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). All computations were carried out using the *NRC* MicroVAX III crystallographic package (Gabe & Lee, 1981). Direct methods were used to find the positions of all non-H atoms. All non-H atoms were refined anisotropically and H atoms were fixed in an idealized geometry. $\sum w(|F_o| - |F_c|)^2$ was minimized, where $w = [\sigma^2(F_o) + 0.0001F_o^2]^{-1}$. Refinement converged to $R = 0.046$, $wR = 0.049$ and $S = 1.16$, based on 362 variables and 1825 observed reflections. The largest shift/e.s.d. in the final least-squares cycle was 0.006. Maximum and minimum residual electron densities in the difference Fourier map were 0.15 and −0.18 e Å^{-3}.

Final positional and equivalent isotropic thermal parameters are presented in Table 1. Bond lengths and angles are given in Table 2. One of the two

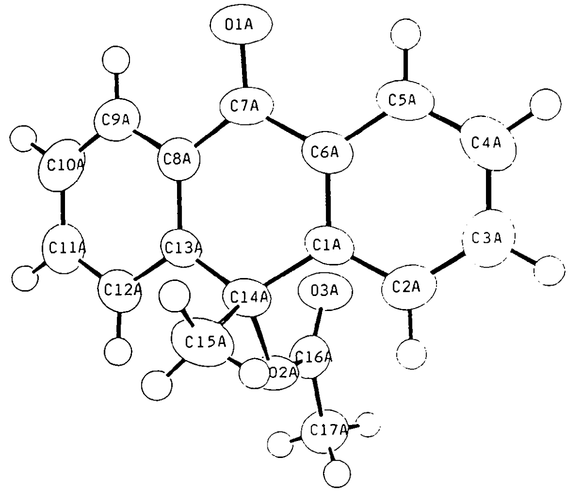


Fig. 1. *ORTEP*II (Johnson, 1976) drawing and labelling of one of the two independent molecules.

independent molecules with the atomic labelling scheme is shown in Fig. 1.*

Related literature. Polycyclic aromatic hydrocarbons (PAH) are the most widespread particulate genoxic environmental pollutants (Gelboin & Ts'o, 1978; Cho & Harvey, 1974). Epoxides have been proved to be the initial products of the oxidation of polycyclic aromatic hydrocarbons (Fu & von Tungeln, 1989). They subsequently metabolize to the corresponding quinones, diols or phenolic derivatives. Harvey and co-workers reported *K*-region oxidized metabolites from PAH (Goh & Harvey, 1973; Harvey, Goh & Cortez, 1975).

References

CHO, H. & HARVEY, R. G. (1974). *Tetrahedron Lett.* **16**, 1491–1494.

FU, P. P. & VON TUNGELN, L. S. (1989). *J. Chromatogr.* **461**, 315–326.

GABE, E. J. & LEE, F. L. (1981). *Acta Cryst.* A**37**, C-339.

GELBOIN, H. V. & TS'O, P. O. P. (1978). Editors. *Polycyclic Hydrocarbons and Cancer*, Vols. 1 and 2. New York: Academic Press.

GOH, S. H. & HARVEY, R. G. (1973). *J. Am. Chem. Soc.* **95**, 242–243.

HARVEY, R. G., GOH, S. H. & CORTEZ, C. (1975). *J. Am. Chem. Soc.* **97**, 3468–3479.

JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

---

* Lists of structure factors, anisotropic thermal parameters for non-H atoms, and atomic coordinates and thermal parameters for H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55186 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR0385]

Acta Cryst. (1992). C**48**, 1872–1873

Structure of 8,10-Diacetyl-2,4,6-trinitro-2,4,6,8,10-pentaazabicyclo[5.3.0]decane

BY RICHARD GILARDI, JUDITH L. FLIPPEN-ANDERSON AND CLIFFORD F. GEORGE

Laboratory for the Structure of Matter, Naval Research Laboratory, Washington, DC 20375, *USA*

(*Received* 4 *November* 1991; *accepted* 5 *February* 1992)

Abstract. 2,4,6-Trinitro-2,4,6,8,10-pentaazabicyclo-[5.3.0]decane-8,10-diyl dimethyl diketone, $C_9H_{14}N_8O_8$, $M_r = 362.3$, orthorhombic, $P2_12_12_1$, $a = 8.145\,(2)$, $b = 12.238\,(2)$, $c = 14.186\,(3)$ Å, $V = 1413.9\,(4)$ Å^3, $Z = 4$, $D_x = 1.702$ Mg m^{-3}, $\lambda(\text{Cu } K\alpha) = 1.54184$ Å, $\mu = 1.31$ mm^{-1}, $F(000) = 752$, $T = 295$ K, final $R = 0.046$, $wR = 0.053$ for 1302 independent observed reflections. The two rings in this polyamine are fused *cis* to one another, imparting a folded shape to the bicyclic system. The parameters of the five-membered ring fit a flattened envelope conformation, with one N atom lying 0.25 Å from the (±0.01 Å) plane of the other four atoms. The torsion angles of the seven-membered ring are intermediate between those of the ideal chair and twist-chair forms of cycloheptane, but are closer to a chair form. Of the five tri-substituted amine atoms, one is essentially flat, and the other four are slightly pyramidal (the angles between the exocyclic amine bonds and the adjacent CNC planes range from 16.2 to 24.0°).

Experimental. A clear colorless prism (0.08 × 0.10 × 0.10 mm) data crystal was provided by Clifford L. Coon of Lawrence Livermore National Laboratory. Data were collected on an automated Siemens *R*3*m*/*V* diffractometer with incident-beam monochromator. 25 centered reflections within $40 \le 2\theta \le 98°$ were used for determining lattice parameters. $(\sin\theta/\lambda)_{\max} = 0.588$ Å^{-1}, range of *hkl*: $0 \le h \le 9$, $0 \le k \le 12$, $0 \le l \le 16$. Three standards (301, 042, 013) monitored every 97 reflections showed linear decline of 10.0% over data collection. Data were collected using the $\theta/2\theta$-scan mode with scan width $[2\theta(K\alpha_1) - 1.4]$ to $[2\theta(K\alpha_2) + 1.4]°$ and ω-scan rate a function of count rate (minimum 3.0, maximum 12.0° min^{-1}). Of 1505 reflections measured, 1413 were unique ($R_{\text{int}} = 2.3\%$), of which 1302 were observed with $F_o > 3\sigma(F_o)$. Data were corrected for Lorentz and polarization effects. No absorption correction was applied. The structure solution, by direct methods, and the full-matrix least-squares refinement used programs in *SHELXTL-Plus* (Sheldrick, 1987). The function $\sum w(|F_o| - |F_c|)^2$ was minimized, where $w = 1/[\sigma^2(|F_o|) + g(F_o)^2]$, $g = 0.000225$. A secondary-extinction parameter $p = 0.005\,(1)$, in $F_c^* = F_c/[1.0 + 0.002(p)F_o^2/\sin(2\theta)]^{0.25}$. 257 parameters were refined: atom coordinates and anisotropic thermal param-

0108-2701/92/101872-02$06.00