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Acta Cryst. (1975). B**31**, 2517

20-Methylcholanthrene (a New Refinement)

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(*Received* 4 *June* 1975; *accepted* 12 *June* 1975)

Abstract. 20-Methylcholanthrene, $C_{21}H_{16}$, M.W. 268·36, monoclinic $P2_1/c$, $a=4·898$ (3), $b=11·36$ (1), $c=25·16$ (2) Å, $\beta=95·3$ (1)°, $U=1393·94$ Å^3, $D_m=1·277$ g cm^{-3}, $Z=4$, $D_x=1·294$, λ(Cu $K\alpha$) = 1·5418 Å. The structure was reported previously [Iball & MacDonald, *Z. Kristallogr.* (1960). **114**, 439–446]. New intensities (1974 reflexions) have been refined by least-squares calculations to a final R of 0·060. (A new c and β were chosen to make the cell more nearly rectangular.)

Introduction. The earlier determination and refinement of the structure of this carcinogenic hydrocarbon was carried out by differential Fourier syntheses (Iball & MacDonald, 1960). In the present work new and more extensive reflexion data were collected from equi-inclination Weissenberg photographs. The camera was fitted with one-dimensional integration and the spots were measured by densitometer. There were 1974 reflexions with non-zero intensities and the new value of R (=0·060) compares with 0·15 for the earlier refinement. The accuracy of the bond lengths and angles is correspondingly increased.

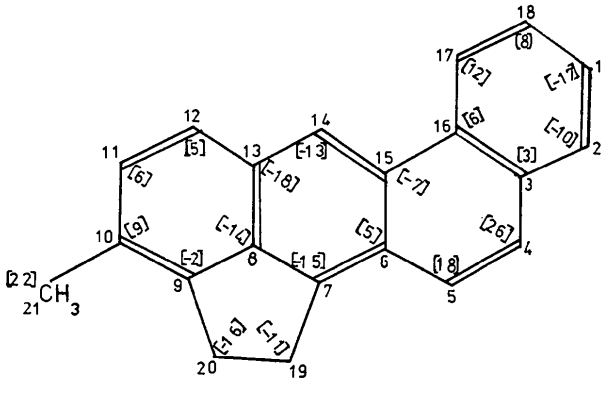

Fig. 1. 20-Methylcholanthrene: the numbering system and deviations (Å × 10^3) of C atoms from the mean plane.

Refinement. The atomic parameters were refined by block-diagonal least-squares calculations and the weighting scheme was $\sqrt{w}=1/\{1+[(|F_o|-F^*)/G^*]^2\}^{1/2}$; F^* and G^* were 15 and 35 respectively. Scattering factors were taken from *International Tables for X-ray Crystallography* (1962). C atoms were refined anisotropically. The H atoms were included in the structure factor calculation but not refined until near the end when the C atoms were fixed and the H atoms (positions only, not temperature parameters) allowed to refine. In the final three cycles the H atoms were again fixed and only the C atoms allowed to refine. The final value of R was 0·060.†

Final parameters with their standard deviations are listed in Tables 1, 2, 3 and bond lengths and angles in Table 4. Fig. 1 shows the molecule with deviations from the mean plane in parentheses. The mean plane of the C atoms is given by $-0·71275X+0·57671Y-0·39924Z=1·4845$ (X is parallel to **a**, Y to **b**, and Z is perpendicular to **a** and **b**). The shortest intermolecular distance, 3·308 (12) Å, is between C(20) at (x,y,z) and C(20) of the molecule at $(1-x,1-y,-z)$.

Discussion. Methylcholanthrene (Iball, 1936) is a transformation product of deoxycholic acid (Cook & Haslewood, 1934) and of cholic acid (Fieser & Newman, 1935) both of which are present in bile; it is a very potent carcinogen. The refinement confirms the essential planarity of the molecule and the short bonds

† A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31197 (14 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Atomic positions* ($\times 10^4$) *for C atoms (e.s.d.'s in parentheses)*

	x	*y*	*z*
C(1)	−4419 (15)	4121 (7)	3325 (3)
C(2)	−2580 (14)	4847 (7)	3117 (3)
C(3)	−1765 (12)	4643 (6)	2601 (2)
C(4)	148 (13)	5421 (6)	2383 (2)
C(5)	1002 (12)	5242 (6)	1895 (2)
C(6)	−11 (11)	4269 (5)	1570 (2)
C(7)	856 (11)	4061 (5)	1068 (2)
C(8)	−222 (11)	3103 (5)	767 (2)
C(9)	826 (11)	3035 (5)	261 (2)
C(10)	−43 (13)	2155 (6)	−81 (2)
C(11)	−1974 (14)	1339 (6)	91 (3)
C(12)	−3013 (13)	1406 (6)	579 (2)
C(13)	−2122 (11)	2315 (5)	948 (2)
C(14)	−2983 (12)	2511 (6)	1455 (2)
C(15)	−1963 (11)	3464 (5)	1770 (2)
C(16)	−2867 (11)	3678 (5)	2301 (2)
C(17)	−4782 (13)	2954 (6)	2526 (2)
C(18)	5567 (14)	3173 (7)	3031 (3)
C(19)	2857 (11)	4725 (6)	748 (2)
C(20)	2839 (11)	4029 (6)	215 (2)
C(21)	992 (16)	2028 (7)	−628 (3)

Table 2. *Atomic positions* ($\times 10^3$) *and isotropic temperature factors* (Å^2) *for H atoms (e.s.d.'s in parentheses)*

	x	*y*	*z*	*B*
H(1)	−503 (14)	428 (6)	369 (3)	4·0
H(2)	−158 (15)	548 (7)	332 (3)	4·5
H(4)	79 (14)	611 (7)	260 (3)	4·0
H(5)	235 (14)	580 (6)	175 (3)	4·0
H(11)	−267 (14)	66 (6)	−16 (3)	4·0
H(12)	−447 (14)	81 (6)	68 (3)	4·0
H(14)	−428 (14)	194 (7)	159 (3)	4·0
H(17)	−560 (14)	223 (7)	233 (3)	4·0
H(18)	−706 (14)	262 (6)	318 (3)	4·0
H(19*a*)	476 (14)	472 (7)	96 (3)	4·5
H(19*b*)	228 (15)	557 (7)	69 (3)	4·5
H(20*a*)	475 (15)	368 (7)	16 (3)	4·5
H(20*b*)	217 (15)	457 (7)	−12 (3)	4·5
H(21*a*)	275 (18)	173 (8)	−58 (3)	7·0
H(21*b*)	−12 (18)	163 (8)	−85 (3)	7·0
H(21*c*)	151 (17)	278 (8)	−75 (3)	7·0

C(4)–C(5) = 1·348, C(9)–C(10) = 1·362 Å. The former is 0·022 Å smaller than in the earlier work and this bond is the 'K' region of the phenanthrene nucleus. It is expected to have almost full double-bond character. However, the other short bond is at the other end of the molecule and there is not the same theoretical basis for such a small value. These two sites could play an

Table 4. *Bond lengths* (Å) *and angles* (°)

C(1)–C(2)	1·361 (10)	C(9)—C(20)	1·510 (8)
C(1)–C(18)	1·396 (10)	C(9)—C(10)	1·362 (8)
C(2)–C(3)	1·412 (9)	C(10)–C(11)	1·419 (9)
C(3)–C(4)	1·434 (9)	C(10)–C(21)	1·518 (9)
C(3)–C(16)	1·410 (8)	C(11)–C(12)	1·375 (9)
C(4)–C(5)	1·348 (9)	C(12)–C(13)	1·429 (8)
C(5)–C(6)	1·436 (8)	C(13)–C(14)	1·399 (8)
C(6)–C(7)	1·390 (8)	C(14)–C(15)	1·400 (8)
C(6)–C(15)	1·446 (8)	C(15)–C(16)	1·466 (8)
C(7)–C(19)	1·525 (8)	C(16)–C(17)	1·406 (9)
C(7)–C(8)	1·401 (8)	C(17)–C(18)	1·382 (9)
C(8)–C(13)	1·397 (8)	C(19)–C(20)	1·557 (8)
C(8)–C(9)	1·419 (8)		

C(2)—C(1)–C(18)	120·8 (6)	C(9)—C(10)–C(11)	117·8 (6)
C(1)—C(2)–C(3)	120·5 (6)	C(9)—C(10)–C(21)	122·1 (6)
C(2)—C(3)–C(4)	120·1 (6)	C(11)–C(10)–C(21)	120·1 (6)
C(2)—C(3)–C(16)	119·5 (6)	C(10)–C(11)–C(12)	123·3 (6)
C(4)—C(3)–C(16)	120·4 (5)	C(11)–C(12)–C(13)	120·2 (6)
C(3)—C(4)–C(5)	121·6 (6)	C(8)—C(13)–C(12)	115·2 (5)
C(4)—C(5)–C(6)	120·9 (6)	C(8)—C(13)–C(14)	117·4 (5)
C(5)—C(6)–C(7)	122·0 (5)	C(12)–C(13)–C(14)	127·4 (5)
C(5)—C(6)–C(15)	119·6 (5)	C(13)–C(14)–C(15)	121·0 (5)
C(7)—C(6)–C(15)	118·4 (5)	C(6)—C(15)–C(14)	120·2 (5)
C(6)—C(7)–C(8)	119·5 (5)	C(6)—C(15)–C(16)	118·5 (5)
C(6)—C(7)–C(19)	131·4 (5)	C(14)–C(15)–C(16)	121·3 (5)
C(8)—C(7)–C(19)	109·1 (5)	C(3)—C(16)–C(15)	118·9 (5)
C(7)—C(8)–C(9)	112·2 (5)	C(3)—C(16)–C(17)	118·5 (5)
C(7)—C(8)–C(13)	123·5 (5)	C(15)–C(16)–C(17)	122·6 (5)
C(9)—C(8)–C(13)	124·3 (5)	C(16)–C(17)–C(18)	121·2 (6)
C(8)—C(9)–C(10)	119·3 (5)	C(1)—C(18)–C(17)	119·5 (6)
C(8)—C(9)–C(20)	108·7 (5)	C(7)—C(19)–C(20)	104·5 (5)
C(10)–C(9)–C(20)	132·0 (5)	C(9)—C(20)–C(19)	105·4 (5)

Table 3. *Anisotropic temperature factors* ($\times 10^5$) *for C atoms (e.s.d.'s in parentheses)*

$$T = \exp\left[-(h^2b_{11} + k^2b_{12} + l^2b_{33} + hkb_{12} + hlb_{13} + klb_{23})\right].$$

	b_{11}	b_{12}	b_{13}	b_{22}	b_{23}	b_{33}
C(1)	6018 (346)	706 (295)	485 (102)	1122 (74)	−44 (50)	149 (10)
C(2)	5134 (309)	583 (271)	169 (96)	980 (67)	−102 (48)	152 (10)
C(3)	3705 (239)	413 (216)	45 (81)	667 (50)	−32 (40)	141 (9)
C(4)	4434 (274)	−34 (234)	−38 (94)	744 (58)	−105 (45)	167 (11)
C(5)	4029 (255)	−512 (215)	11 (85)	662 (52)	−23 (42)	150 (9)
C(6)	3160 (217)	−122 (192)	−21 (76)	603 (48)	8 (36)	126 (9)
C(7)	2778 (206)	−103 (189)	−34 (75)	593 (47)	40 (37)	134 (9)
C(8)	3171 (217)	139 (195)	81 (73)	597 (49)	16 (37)	121 (8)
C(9)	3134 (220)	149 (201)	131 (75)	676 (52)	−5 (38)	130 (9)
C(10)	4029 (247)	328 (219)	215 (85)	758 (56)	−46 (42)	146 (10)
C(11)	4793 (293)	217 (244)	37 (96)	775 (57)	−69 (45)	155 (10)
C(12)	4519 (274)	−634 (225)	71 (90)	634 (51)	−41 (42)	157 (10)
C(13)	3526 (233)	−157 (203)	45 (77)	611 (50)	14 (38)	129 (9)
C(14)	3683 (240)	−399 (204)	76 (81)	622 (50)	49 (38)	139 (9)
C(15)	3152 (218)	12 (190)	7 (74)	598 (49)	17 (37)	124 (8)
C(16)	3519 (231)	340 (199)	104 (78)	576 (46)	49 (38)	132 (9)
C(17)	4079 (249)	275 (218)	297 (83)	757 (58)	28 (41)	146 (9)
C(18)	5236 (306)	312 (266)	572 (97)	928 (67)	76 (49)	179 (11)
C(19)	3275 (224)	−422 (206)	160 (78)	726 (54)	38 (40)	140 (9)
C(20)	3048 (216)	−140 (210)	157 (76)	814 (56)	46 (41)	136 (9)
C(21)	6484 (369)	−109 (317)	543 (108)	1224 (80)	−204 (52)	166 (11)

important role if the cancer-producing property of methylcholanthrene depended on intercalation with nucleic acid.

We thank the Cancer Research Campaign and S.R.C. for financial support. We are grateful to Dr W. K. Grant for assistance with collecting some of the data and to Dr J. S. Rollett for help with the earlier stages of the refinement.

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Acta Cryst. (1975). B**31**, 2519

6-Azathymine

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(*Received* 26 *December* 1974; *accepted* 16 *May* 1975)

Abstract. $C_4H_5N_3O_2$, orthorhombic, *Pnam*, $a=6{\cdot}7313$ (7), $b=13{\cdot}0361$ (12), $c=6{\cdot}3309$ (10) Å, $Z=4$, $D_c=1{\cdot}513$, $D_m=1{\cdot}50$ (2) g cm^{-3}, $U=555{\cdot}5$ Å^3. The azapyrimidine ring is planar, and the molecular geometry is similar to that of 6-azauracil. The hydrogen-bonding scheme is different, however, involving N(1)–H(1)···O(4) and N(3)–H(3)···O(2) interactions of length 2·79 Å which give rise to a sheet-like structure perpendicular to the crystallographic *c* axis.

Introduction. Plate-like crystals of 6-azathymine were grown from aqueous solution, and the sample used had dimensions $0{\cdot}71\times0{\cdot}64\times0{\cdot}13$ mm. The data showed systematic absences of $k+l=2n+1$ for $0kl$ and $h=2n+1$ for $h0l$. The intensity data were collected using Mo $K\alpha$ radiation on a four-circle Picker automatic diffractometer with a scintillation counter and a pulse height analyzer. The incident beam was filtered through a 3·0 mil niobium foil. The cell constants were determined by the least-squares procedure of Busing & Levy (1967) using data obtained with Cu $K\alpha$ radiation filtered through 0·5 mil nickel foil. The data were corrected for Lorentz and polarization effects and for background, but not for absorption. 1113 reflections [$513>3\sigma(I)$] were measured.

Solution of the structure by direct methods (Hauptman & Karle, 1953) proved to be difficult, and so the structure was solved by means of a three-dimensional Patterson function. All hydrogen atoms were located unambiguously in a difference Fourier synthesis, and least-squares refinements were carried out in which the hydrogen atoms were refined isotropically and all other atoms were refined anisotropically. The function minimized was $\sum w(|F_o|-|F_c|)^2$, and the weights w were taken as $4F_o^2/\sigma^2(F_o)^2$. Examination of the data at a late stage of refinement suggested to us that no correction for secondary extinction was necessary.

The final agreement indices, $R_1=\sum||F_o-F_c||/\sum|F_o|$ and $R_2=[\sum w(|F_o|-|F_c|)^2/\sum w|F_o|^2]^{1/2}$ were 0·062 and 0·083, respectively, based on 513 observations and 68 variables. Refinement in the non-centrosymmetric space group $Pna2_1$ led to a model which did not differ significantly from the centrosymmetric model and which yielded values of 0·059 and 0·076 for R_1 and R_2,

Table 1. *Positional and thermal parameters for* 6-*azathymine*

All parameters (except hydrogen isotropic thermal parameters) have been multiplied by 10^4. All anisotropically refined atoms are constrained to lie on a mirror plane in *Pnam*, so for all atoms $\beta_{13}=\beta_{23}=0{\cdot}0$ and $z=\frac{1}{4}$. The form of the anisotropic thermal ellipsoid is exp $\{-[\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk]\}$.

	x	y	z	β_{11} (B, Å^2)	β_{22}	β_{33}	β_{12}
N(1)	−281 (6)	321 (3)	¼	95 (7)	48 (2)	233 (11)	3 (3)
C(2)	731 (6)	1219 (3)	¼	126 (8)	41 (2)	205 (11)	7 (4)
N(3)	2776 (4)	1119 (3)	¼	106 (6)	31 (2)	362 (12)	−16 (3)
C(4)	3743 (5)	199 (3)	¼	37 (7)	43 (2)	226 (11)	3 (3)
C(5)	2456 (6)	−711 (3)	¼	123 (8)	35 (2)	196 (10)	−2 (4)
N(6)	575 (5)	−628 (2)	¼	128 (8)	43 (2)	244 (10)	5 (3)
C(7)	3367 (11)	−1743 (4)	¼	243 (14)	32 (3)	398 (22)	23 (5)
O(2)	−70 (4)	2052 (2)	¼	152 (7)	47 (2)	462 (13)	37 (3)
O(4)	5586 (4)	151 (2)	¼	94 (6)	69 (2)	369 (11)	−13 (3)
H(1)	−1516 (61)	351 (24)	¼	1·2 (7)			
H(3)	3257 (70)	1750 (35)	¼	4·1 (11)			
H(71)	2517 (80)	−2219 (46)	¼	5·4 (16)			
H(72)	4208 (71)	−1822 (27)	1440 (62)	7·1 (11)			