

SCN, dont le rôle est déterminant dans ces liaisons H, il existe une distorsion directement liée à celles-ci: l'angle de liaison S-C-N n'est plus que de 177,5° en moyenne, lorsque le groupement fait pont, dans la structure, alors qu'il demeure très voisin de 180° lorsque son atome de soufre reste libre.

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Références

- BROUTY, C., WHULER, A., SPINAT, P. & HERPIN, P. (1975). *Bull. Soc. Fr. Minér. Crist.* **98**, 218-222.
 BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1962). *ORFLS*. Oak Ridge National Laboratory Report ORNL-TM-305.
 CHURCHILL, M. R. (1973). *Inorg. Chem.* **12**, 1213-1214.

- DUESLER, E. N. & RAYMOND, K. N. (1971). *Inorg. Chem.* **10**, 1486-1492.
 GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* **A27**, 368-376.
 HAMILTON, W. C. & IBERS, J. A. (1968). *Hydrogen Bonding in Solids*, p. 16. New York: Benjamin.
International Tables for X-ray Crystallography (1962). Vol. III. Birmingham: Kynoch Press.
 IUPAC (1970). *Inorg. Chem.* **9**, 1-5.
 IWATA, M., NAKATSU, K. & SAITO, Y. (1969). *Acta Cryst.* **B25**, 2562-2571.
 JAEGER, F. M. (1915). *Z. Kristallogr.* **55**, 209-248.
 JOHNSON, C. K. (1965). *ORTEP*. Oak Ridge National Laboratory Report ORNL-3794.
 MOORE, F. H. (1963). *Acta Cryst.* **16**, 1169-1175.
 RAYMOND, K. N., CORFIELD, P. W. R. & IBERS, J. A. (1968). *Inorg. Chem.* **7**, 1362-1373.
 WELLS, A. F. (1962). *Structural Inorganic Chemistry*, p. 723. Oxford: Clarendon Press.
 WHULER, A., BROUTY, C., SPINAT, P. & HERPIN, P. (1975). *Acta Cryst.* **B31**, 2069-2076.

Acta Cryst. (1976). **B32**, 2159

The Crystal and Molecular Structure of 4-Methyl-4-hydroxy-1,2,3,4-tetrahydro-6-methoxyacridine

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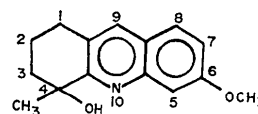
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Crystals of 4-methyl-4-hydroxy-1,2,3,4-tetrahydro-6-methoxyacridine, $C_{15}H_{17}NO_2$, are monoclinic, space group $P2_1/c$ with $a=9.28$ (3), $b=15.36$ (1), $c=9.23$ 2(Å); $\beta=105.0$ (5)°; $q_o=1.274$, $q_c=1.271$ g cm⁻³ for $Z=4$. The structure was solved by direct methods using visually estimated data, and refined by the full-matrix least-squares method to an R value of 0.101 for 1698 observed reflexions. The e.s.d.'s in bond lengths not involving H and disordered C atoms are 0.005-0.007 Å and in bond angles, 0.3-0.5°. The dimensions of the two aromatic rings of the molecule show small but significant and expected changes from the corresponding dimensions in acridine. There is disorder in the cyclohexene ring with the two terminal C atoms out of the aromatic plane, in opposite directions, in two ways; the two configurations are not symmetrical with respect to the aromatic plane. Pairs of molecules are held together mainly by two centrosymmetrically related OH...N hydrogen bonds of length 2.91 Å; the H-O-N angle is 9.6°.

Introduction

During studies on the cyclo-dehydration of 2-arylaminomethylene cycloalkanones under acidic conditions in this laboratory (Vankar, Gogte & Tilak, 1976), the reaction of *m*-anisidine hydrochloride with 2-*m*-anisidinomethylene-6-methylcyclohexanone gave a white crystalline solid of formula $C_{15}H_{17}NO_2$ as one of the main products; the NMR and IR spectra of this compound indicated the presence of an OH group, a methyl group centred on a tertiary centre and four aromatic H atoms. The present study has shown that the compound is 4-methyl-4-hydroxy-

1,2,3,4-tetrahydro-6-methoxyacridine; its structural formula is given below.



The present study is likely to be of some biological interest since it is known that acridines often show mutagenic and anti-tumour properties (Albert, 1966).

Experimental

Colourless crystals, grown from methanol, are monoclinic, space group $P2_1/c$ with $a=9.28$ (3), $b=15.36$ (1),

* Communication No. 1979.

$c=9.23$ (2) Å; $\beta=105.0$ (5)°; $\rho_o=1.274$, $\rho_c=1.271$ g cm⁻³ for $Z=4$. Cell dimensions were measured with the help of high-angle reflexions in zero-layer Weissenberg photographs with the films mounted in the Straumanis arrangement. Data were collected with unfiltered Cu radiation from zero to eighth layer Weissenberg photographs about the c axis and from zero to fourth layer photographs about the a axis; the crystals used for the two sets of photographs had cross sections 0.8×0.9 and 1.0×0.6 mm respectively. The data were processed in the usual way; absorption was neglected.

Determination and refinement of the structure

The signs of 182 structure factors ($E \geq 1.5$) were determined with the help of the Sayre-equation program written by Long (1965); the E map obtained on the basis of these signs revealed the molecule fairly clearly. The structure was refined by the block-diagonal least-squares method and in the later stages of refinement, H atoms were included and anisotropic temperature factors were introduced and the refinement continued with Cruickshank's weighting scheme; R came down to 0.110 for 1698 observed reflexions. At this stage, the molecular dimensions were all reasonable except that the C(2)–C(3) bond was too short for a single C–C bond (~ 1.32 Å); the observed r.m.s. amplitudes across the molecular plane were also too large for C(2) and C(3). Disorder in the positions of C(2) and C(3) was suspected; therefore a Fourier map from which all atoms except C(2) and C(3) were subtracted was calculated; this showed two clear peaks separated by more than 1 Å for C(2) and a markedly ellipsoidal peak for C(3), thus indicating disorder. Further refinement was performed taking two half C atoms C(2) and C'(2), and two half atoms C(3) and C'(3) at the positions in-

dicated by the Fourier map. Finally, a full-matrix least-squares refinement was carried out using program *LALS* of Gantzel, Sparks & Trueblood (1961); the scattering factors for C(valence), N and O were taken from *International Tables for X-ray Crystallography*, (1962) and for H from Stewart, Davidson & Simpson (1965). All H atoms except those attached to C(1), C(2), C'(2), C(3) and C'(3) were included in the refinement; however, their thermal parameters were kept fixed and assumed to be the same as those of the attached C (or O) atoms. The full-matrix refinement was carried out in three stages owing to the limitation on computer memory: firstly the atoms of the cyclohexene ring were refined (two cycles), next the remaining non-hydrogen atoms were refined (two cycles), and finally, the positional parameters of the H atoms were refined (one cycle). Final R is 0.101 for 1698 observed reflexions. The final atomic and thermal parameters along with their e.s.d.'s are given in Table 1.*

The intramolecular bond lengths and angles are shown in Fig. 1(a) and (b) and the equation of the aromatic plane referred to the a', b, c orthogonal axes is given in Table 2 along with the deviations of the atoms from this plane. The packing of the molecules in the crystal is shown in Fig. 2.

Description of the structure and discussion

The atoms of the two aromatic rings of the molecule are coplanar within about 0.012 Å. The bond lengths

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31639 (17 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. Final atomic and thermal parameters and their estimated standard deviations (in parentheses)

Anisotropic thermal parameters are of the form $T = \exp [-(b_{11}h^2 + b_{22}k^2 + b_{33}l^2 + b_{12}hk + b_{23}kl + b_{13}hl)]$.

(a) Non-hydrogen atoms ($\times 10^4$)

	x	y	z	b_{11}	b_{22}	b_{33}	b_{12}	b_{23}	b_{13}
C(1)	8806 (6)	2484 (3)	8078 (5)	210 (8)	51 (2)	186 (7)	11 (7)	68 (12)	-58 (7)
C(2)	10130 (10)	1973 (5)	9166 (9)	180 (13)	44 (4)	126 (11)	-29 (11)	9 (19)	-16 (10)
C'(2)	10488 (14)	2353 (8)	8397 (17)	196 (19)	62 (5)	333 (25)	-14 (16)	61 (36)	-106 (19)
C(3)	11122 (29)	1682 (9)	8257 (28)	199 (18)	45 (7)	220 (23)	-38 (24)	-68 (30)	-104 (24)
C'(3)	10913 (33)	1425 (10)	8437 (36)	213 (36)	46 (34)	322 (8)	35 (32)	-140 (27)	-110 (47)
C(4)	10288 (5)	908 (3)	7130 (5)	140 (6)	46 (2)	147 (6)	19 (5)	10 (9)	-19 (5)
C(5)	5804 (5)	263 (3)	3298 (4)	131 (6)	54 (2)	146 (6)	3 (5)	59 (9)	0 (5)
C(6)	4367 (5)	430 (3)	2515 (5)	134 (6)	63 (2)	152 (6)	-16 (6)	30 (10)	15 (6)
C(7)	3595 (5)	1160 (4)	2866 (6)	128 (6)	74 (3)	221 (9)	35 (7)	-9 (12)	21 (8)
C(8)	4279 (5)	1704 (3)	4002 (6)	141 (6)	60 (2)	242 (8)	43 (6)	47 (12)	6 (7)
C(9)	6546 (5)	2084 (3)	6032 (5)	162 (6)	43 (2)	175 (6)	33 (5)	113 (10)	6 (5)
C(10)	7985 (5)	1911 (2)	6791 (4)	161 (6)	40 (2)	149 (5)	12 (5)	100 (10)	-2 (5)
C(11)	8674 (4)	1161 (2)	6344 (4)	142 (6)	39 (2)	133 (5)	1 (5)	62 (9)	-7 (5)
C(12)	6534 (4)	828 (3)	4467 (4)	129 (5)	45 (2)	131 (5)	9 (5)	66 (9)	13 (5)
C(13)	5773 (5)	1557 (3)	4832 (4)	144 (6)	45 (2)	155 (6)	17 (5)	72 (9)	16 (5)
C(14)	4208 (6)	-831 (3)	997 (5)	201 (8)	65 (3)	177 (7)	-36 (8)	53 (12)	0 (7)
C(15)	10314 (5)	-15 (3)	7766 (5)	174 (7)	58 (2)	184 (7)	25 (6)	54 (12)	49 (6)
O(1)	3546 (4)	-67 (2)	1370 (4)	147 (5)	76 (2)	195 (5)	-22 (5)	-25 (8)	-32 (5)
O(2)	11156 (3)	937 (2)	6061 (4)	142 (4)	48 (1)	228 (5)	9 (4)	105 (8)	13 (4)
N	7991 (3)	650 (2)	5231 (3)	128 (4)	43 (1)	141 (5)	13 (4)	37 (7)	-11 (4)

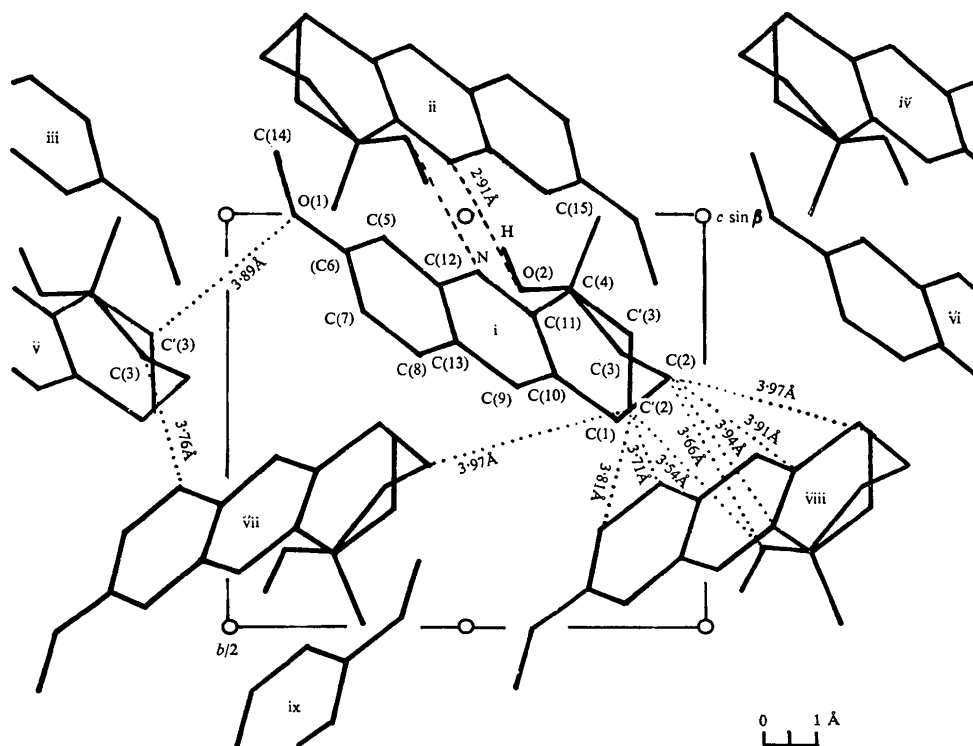


Fig. 2. [100] projection of the structure. Symmetry code: (i) x, y, z ; (ii) $\bar{x}, \bar{y}, 1-z$; (iii) $\bar{x}, \bar{y}, \bar{z}$; (iv) $\bar{x}, \bar{y}, 2-z$; (v) $x, y, z-1$; (vi) $x, y, z+1$; (vii) $x, \frac{1}{2}-y, z-\frac{1}{2}$; (viii) $x, \frac{1}{2}-y, \frac{1}{2}+z$; (ix) $\bar{x}, \frac{1}{2}+y, \frac{1}{2}-z$. Besides hydrogen bonds, the only intermolecular contacts, $< 4 \text{ \AA}$, shown are from the disordered atoms C(2), C'(2) and C(3), C'(3).

hexene ring is puckered to a greater extent [*i.e.* C(2)–C(3)] has the greater occupancy.

Pairs of molecules in the present structure are held together mainly by two centrosymmetrically related $\text{OH}\cdots\text{N}$ hydrogen bonds of length 2.91 \AA ; the H-O-N angle is 9.6° .

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References

- ALBERT, A. (1966). *The Acridines*. 2nd ed. London: Arnold.
- GANTZEL, P. K., SPARKS, R. A. & TRUEBLOOD, K. N. (1961). *LALS. Full-matrix Least-squares Refinement of Positional and Thermal Parameters and Scale Factors*.
- HAMANAKA, T., MITSUI, T., ASHIDA, T. & KAKUDO, M. (1972). *Acta Cryst. B* **28**, 214–222.
- International Tables for X-ray Crystallography* (1962). Vol. III. Birmingham: Kynoch Press.
- LONG, R. E. (1965). Univ. of California Los Angeles Dissertation, Parts II and III.
- PHILLIPS, D. C. (1956). *Acta Cryst.* **9**, 237–250.
- PHILLIPS, D. C., AHMED, F. R. & BARNES, W. H. (1960). *Acta Cryst.* **13**, 365–377.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
- THACKERAY, M. M. & GAFNER, G. (1975). *Acta Cryst. B* **31**, 335–338.
- VANKAR, Y. D., GOGTE, V. N. & TILAK, B. D. (1976). To be published.