

Discussion. Although no crystallographic symmetry is imposed on the molecule, it possesses approximate C_s symmetry with the mirror plane running through Os(3), Os(4) and Os(5). Os(1), Os(2), Os(3) and Os(5) are coplanar to within 0.01 Å, with Os(4) 2.39 Å above this plane. The Os—C and C—O lengths are effectively equal, with mean values of 1.89 and 1.15 Å. There are no Os...C non-bonded contacts < 2.93 Å [Os(3)...C(2)], so there are no incipient bridging carbonyls as found, for example, in Os₅(CO)₁₆ (Reichert & Sheldrick, 1977). NMR measurements by Eady, Johnson & Lewis (1977) indicate that there are two equivalent edge-bridging hydrides; from the disposition of the carbonyl ligands they probably bridge the two long Os—Os bonds [Os(1)—Os(4) and Os(2)—Os(4)]. If the two Os—H—Os bridges are considered to be two-electron three-centre bonds, a structure based on a square pyramid of Os atoms obeys the 18-electron rule overall, and would probably have

been predicted on the basis of Wade's theory. The less symmetrical structure actually adopted makes the same number of bonds, and so also obeys a net 18-electron rule.

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1-(1-Naphthyl)isoquinoline

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Abstract. C₁₉H₁₃N, triclinic, $P\bar{1}$, $Z = 4$, $M_r = 255.3$, $a = 11.363$ (3), $b = 12.243$ (3), $c = 11.305$ (3) Å, $\alpha = 100.87$ (2), $\beta = 105.38$ (2), $\gamma = 108.46$ (2)°, $D_x = 1.285$, $D_m = 1.23$ g cm⁻³, $\mu(\text{Cu}) = 5.6$ cm⁻¹, final $R = 0.040$. The sp^2 – sp^2 bond connecting the two halves is 1.501 (3) Å in one of the molecules and 1.495 (3) Å in the other. The planar halves of each molecule are inclined at 77.5 and 63.9°.

Introduction. The present study was undertaken in connection with an investigation of the steric effect of N atom lone-pair electrons performed by Dr J. Pedersen (Department of Organic Chemistry, Chalmers University of Technology). A crystal 0.3 × 0.4 × 0.5 mm was used for the data collection. Cell dimensions and

intensities were measured on a Syntex P2₁ automatic four-circle diffractometer with graphite-monochromated Cu $K\alpha$ radiation. The θ – 2θ scan procedure was used and the 2θ scan speed was allowed to vary between 1.5 and 8° min⁻¹ depending on the intensity of the reflection. Each reflection was measured in 96 steps and the intensities were evaluated by a profile-analysis procedure (Lehmann & Larsen, 1974). Of the 3449 independent reflections with $2\theta \leq 115^\circ$, 2572 had $I > 3\sigma(I)$ and were considered observed. A control reflection monitored every 50 reflections showed no significant fluctuation.

The structure was solved with the symbolic addition program system SIMPEL (Overbeek, van der Putten & Schenk, 1976). Triplet and quartet relations were

calculated between the 400 strongest normalized structure factors ($E_{\min} = 1.57$). 2606 triplets with $E_3 \geq 1$ and 117 quartets with $E_4 \geq 1.5$ were obtained. ($E_3 = N^{-1/2}|E_H \times E_K \times E_{-H-K}|$, $E_4 = N^{-1}|E_H \times E_K \times E_L \times E_{-H-K-L}|$, N is the number of atoms in the unit cell.) The program found a good starting set by means of a convergence procedure based on maximum interaction between the triplets and strengthened quartets (Schenk, 1973). Symbolic addition was then carried out with triplets only. The correct set of signs for the eight symbols used was found by calculating Harker-Kasper and negative quartet criteria (Schenk & de Jong, 1973; Schenk, 1974) for the possible 256 combinations. The E map calculated with the set of signs having the best negative quartet figure and number ten in the Harker-Kasper criterion showed all 40 non-hydrogen atoms. Anisotropic block-diagonal least-squares refinement of the non-hydrogen atoms gave $R = 0.070$ and all the H atoms could be found in the subsequent difference map. The final refinement with anisotropic C and N and isotropic H atoms converged at $R = 0.040$ for 2572 observed reflections and 466 parameters. Weights were defined as $w = (4 + |F_o| + 0.04F_o^2)^{-1}$ and the scattering factors for C and N were those of Doyle & Turner (1968) and for H those of Stewart, Davidson &

Simpson (1965). Atomic coordinates are given in Table 1.*

In addition to the program system *SIMPEL*, the following programs were used: *LELA* for profile analysis (Lindqvist & Ljungström), *SYN* for data reduction (Lindqvist & Ljungström), *BLOCK* for least squares (Lindgren), *DISTAN* for distances and angles (Zalkin), *PLANEFIT* for least-squares planes (Wengelin) and *ORTEP* (Johnson) for stereoscopic projections.

Discussion. Bond lengths and angles are given in Figs. 1 and 2. The molecular packing is shown in Fig. 3. The asymmetric unit consists of two molecules. Each bond length is thus determined twice and corresponding values show good agreement. However, the e.s.d.'s would appear to be slightly underestimated. The two planar halves of each molecule are linked by an sp^2-sp^2 C—C bond of 1.495 (3) and 1.501 (3) Å.

Least-squares planes fitted to the two planar residues

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33270 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. Final atomic parameters ($\times 10^4$; for H $\times 10^3$) with e.s.d.'s in parentheses

	x	y	z		x	y	z
C(1)	8973 (2)	6598 (2)	4659 (2)	C(20)	7361 (2)	2801 (2)	67 (2)
C(2)	8443 (2)	5398 (2)	4548 (2)	C(21)	8387 (2)	3903 (2)	651 (2)
C(3)	7436 (2)	4947 (2)	5048 (2)	C(22)	9683 (2)	4013 (2)	1290 (2)
C(4)	6978 (2)	5703 (2)	5648 (2)	C(23)	9930 (2)	3008 (2)	1334 (2)
C(5)	7504 (2)	6950 (2)	5795 (2)	C(24)	8905 (2)	1849 (2)	768 (2)
C(6)	7066 (2)	7764 (2)	6433 (2)	C(25)	9147 (2)	796 (2)	842 (3)
C(7)	7615 (2)	8969 (2)	6591 (3)	C(26)	8150 (3)	-315 (2)	313 (3)
C(8)	8627 (2)	9431 (2)	6117 (3)	C(27)	6852 (2)	-425 (2)	-317 (3)
C(9)	9058 (2)	8669 (2)	5483 (2)	C(28)	6579 (2)	563 (2)	-414 (2)
C(10)	8535 (2)	7414 (2)	5313 (2)	C(29)	7598 (2)	1736 (2)	123 (2)
C(11)	14 (2)	7044 (2)	4084 (2)	C(30)	6010 (2)	2730 (2)	-633 (2)
N(1)	9574 (2)	6851 (2)	2827 (2)	N(2)	5530 (2)	2164 (2)	-1882 (2)
C(12)	491 (2)	7206 (2)	2257 (2)	C(31)	4306 (2)	2089 (3)	-2573 (2)
C(13)	1807 (2)	7732 (2)	2890 (2)	C(32)	3561 (2)	2546 (3)	-2069 (2)
C(14)	2299 (2)	7969 (2)	4233 (2)	C(33)	4034 (2)	3141 (2)	-741 (2)
C(15)	3663 (2)	8543 (2)	4978 (2)	C(34)	3302 (2)	3639 (2)	-137 (2)
C(16)	4081 (2)	8776 (3)	6278 (2)	C(35)	3792 (2)	4198 (2)	1146 (2)
C(17)	3170 (2)	8433 (2)	6897 (2)	C(36)	5041 (2)	4282 (2)	1894 (2)
C(18)	1844 (2)	7859 (2)	6208 (2)	C(37)	5774 (2)	3811 (2)	1350 (2)
C(19)	1382 (2)	7619 (2)	4857 (2)	C(38)	5300 (2)	3234 (2)	7 (2)
H(2)	876 (2)	487 (2)	409 (2)	H(21)	822 (2)	464 (2)	61 (2)
H(3)	704 (2)	409 (2)	493 (2)	H(22)	961 (2)	514 (2)	833 (3)
H(4)	626 (2)	538 (2)	595 (2)	H(23)	1082 (2)	304 (2)	174 (2)
H(6)	632 (2)	743 (2)	676 (2)	H(25)	1004 (2)	88 (2)	124 (3)
H(7)	732 (2)	955 (2)	710 (3)	H(26)	831 (3)	-107 (3)	34 (3)
H(8)	914 (2)	1040 (3)	663 (3)	H(27)	612 (2)	-127 (2)	-68 (3)
H(9)	981 (2)	900 (2)	522 (2)	H(28)	565 (2)	48 (2)	-86 (2)
H(12)	11 (2)	705 (2)	131 (2)	H(31)	398 (2)	163 (2)	-352 (3)
H(13)	244 (2)	794 (2)	243 (2)	H(32)	271 (2)	244 (2)	-260 (3)
H(15)	429 (2)	872 (2)	453 (2)	H(34)	243 (2)	361 (2)	-67 (2)
H(16)	508 (2)	920 (2)	681 (3)	H(35)	327 (2)	455 (2)	157 (2)
H(17)	351 (2)	857 (2)	787 (2)	H(36)	535 (2)	468 (2)	283 (2)
H(18)	120 (2)	765 (2)	666 (2)	H(37)	665 (2)	389 (2)	188 (2)

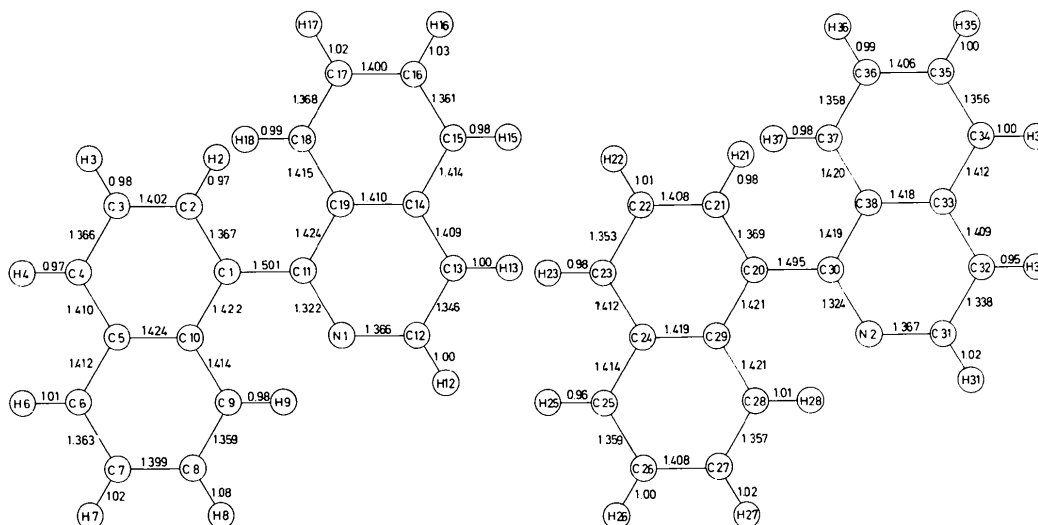


Fig. 1. Bond distances (Å) in the two independent molecules. The e.s.d. of a non-hydrogen bond is normally 0.003 and never exceeds 0.004 Å. For a C–H bond the e.s.d. is normally 0.02 and never exceeds 0.03 Å.

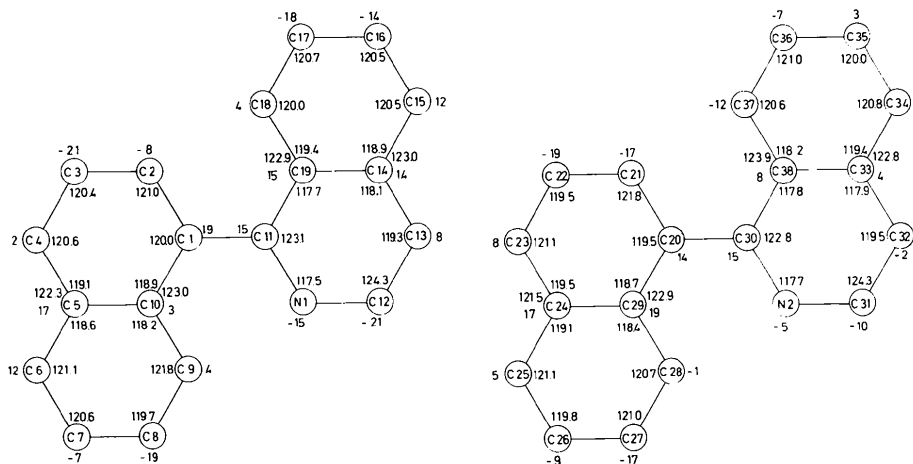


Fig. 2. Angles (°) and deviations ($\times 10^3$ Å) from the least-squares planes through the planar halves of the molecules. The e.s.d. of an angle is normally 0.3 and never exceeds 0.4°.

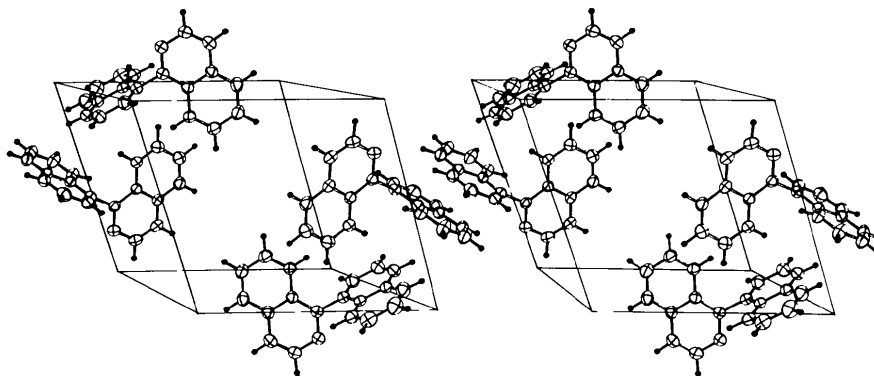


Fig. 3. Stereoscopic drawing of the unit cell (a is horizontal and b is directed away from the viewer). For clarity the ac plane has been rotated about 15° out of the plane of the paper.

of each molecule are inclined at angles of 77.5 and 63.9°. The sp^2-sp^2 linking bond is of comparable length to that found in 8,8'-biquinolyl, 1.495 (2) Å (Lenner & Lindgren, 1976), where the molecular halves are inclined at an angle of 96.8°. The conformation of the present molecule and of 8,8'-biquinolyl effectively prevents overlap of π orbitals and there would thus seem to be no π contribution to this bond. Comparable bonds in the completely planar perylene, 1.471 (5) Å (Camerman & Trotter, 1964), and in a perylene fluoranil complex, 1.473 (5) Å (Hanson, 1963), are significantly shorter.

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3-Methylcytosine Hemihydrate: The Aminooxo Tautomer

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Abstract. $C_5H_7N_3O \cdot \frac{1}{2}H_2O$, FW 134, orthorhombic, *Pba*2, $a = 8.132$ (2), $b = 13.333$ (2), $c = 5.976$ (1) Å ($t = 22 \pm 3^\circ C$), D_m (floatation) = 1.37, $D_x = 1.36$ g cm⁻³, $Z = 4$, $\mu(Cu K\alpha) = 6.5$ cm⁻¹, $F(000) = 284$, $\lambda(Cu K\alpha) = 1.54051$ Å, $R = 0.06$ for 763 reflections. The cytosine moiety exhibits the aminooxo form in the solid state with no H atom on N(1). The water molecules lie on twofold axes. The structure consists of layers of molecules joined by N–H...N and N–H...O hydrogen bonds. There is only partial stacking of the cytosine rings in the crystal structure.

Introduction. The crystal structure of 3-methylcytosine (m^3 cyt) was undertaken to study its tautomerism and its hydrogen bonding. Crystals of m^3 cyt were grown by slow evaporation from a methanol–water mixture as well formed square plates. A crystal $0.3 \times 0.35 \times 0.35$ mm was used for data collection. The crystals are orthorhombic and the space group, as deduced from the systematic absences ($0kl$, k odd; $h0l$, h odd), could be *Pba*2 or *Pbam*. The refined unit-cell parameters and

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other cell data are in the *Abstract*. Three-dimensional intensities were collected on a GE XRD-6 diffractometer, using a 5° take-off angle, by the stationary-crystal–stationary-counter technique (Furnas & Harker, 1955). Ni–Co balanced filters were used for monochromatization. 885 reflections were measured, of which 122 had intensities less than twice the background in that $\sin \theta/\lambda$ range. The difference in absorption as a function of φ was measured for the axial reflections and this was used for correcting for absorption. Lorentz and polarization corrections were applied and the data were processed in the usual way.

The crystal structure was solved by the application of the multiresolution technique using *MULTAN* (Germain, Main & Woolfson, 1971). After futile attempts to solve the structure in the centrosymmetric (as suggested by the near centric distribution of normalized structure factors) space group *Pbam*, the structure was obtained readily from the noncentrosymmetric space group *Pba*2 and refined by a least-squares procedure with block-diagonal approximation. The water O atoms lie on twofold axes. The location of the water H atoms is not accurate. All the other H atoms were refined isotropically and the non-hydrogen anisotropically. The

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