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The crystal and molecular structure of N-(17-methoxy-phenyl)-2-chloropyrrolo (2,3-*b*) quinoline

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Abstract. N-(17-methoxy-phenyl)-2-chloropyrrolo (2,3-*b*) quinoline was solved by direct methods and refined to an *R* of 0·104 for 950 observed reflections. The intensity data were collected by the multiple film equi-inclination Weissenberg technique and estimated visually. The packing of the molecule is stabilised by van der Waals interaction. The pyrrolo (2,3-*b*) quinoline ring system is planar and the methoxy phenyl ring is approximately perpendicular to the plane of this ring system with a dihedral angle of 86·5°.

Keywords. Quinoline; van der Waals interaction; dihedral angle.

1. Introduction

N-(17-methoxy-phenyl)-2-chloropyrrolo (2,3-*b*) quinoline is a derivative of pyrrolo (2,3-*b*) quinoline which has been found to possess promising antiphlogistic activity in rats (Acheson and Wollard 1975). Hydroxy quinolines are found to be bacterial inhibitors (Koshimura *et al* 1954) and precursors to a number of antimalarial and cancer drugs (Sakai *et al* 1955). The molecular diagram and numbering of atoms are shown in figure 1.

2. Experimental

Needle-shaped crystals elongated along the *b* axis were obtained from a mixture of chloroform and benzene. The cell parameters are $a = 17{\cdot}605(15)$, $b = 5{\cdot}030(4)$, $c = 17{\cdot}735(15)$ Å and $\beta = 109{\cdot}45(3)°$. Space group $P2_1/c$ with $Z = 4$, $C_{18}H_{13}N_2ClO$, Mol. Wt. = 380·1, $F(000) = 640$, $D_m = 1{\cdot}57$, $D_c = 1{\cdot}56$ g/cc. $\lambda(CuK_\alpha) = 1{\cdot}5418$ Å, $\mu = 13{\cdot}87$ cm^{-1}. Accurate values of the cell parameters were obtained by least-squares refinement of measured angle values for 30 reflections with a Picker (FACS-1) diffractometer with range $40° < 2\theta < 60°$. A crystal of size $0{\cdot}3 \times 0{\cdot}5 \times 0{\cdot}25$ mm was used for data collection. Intensity data were collected by the multiple film equi-inclination Weissenberg technique and estimated visually. The total number of observed reflections were 950 in layers

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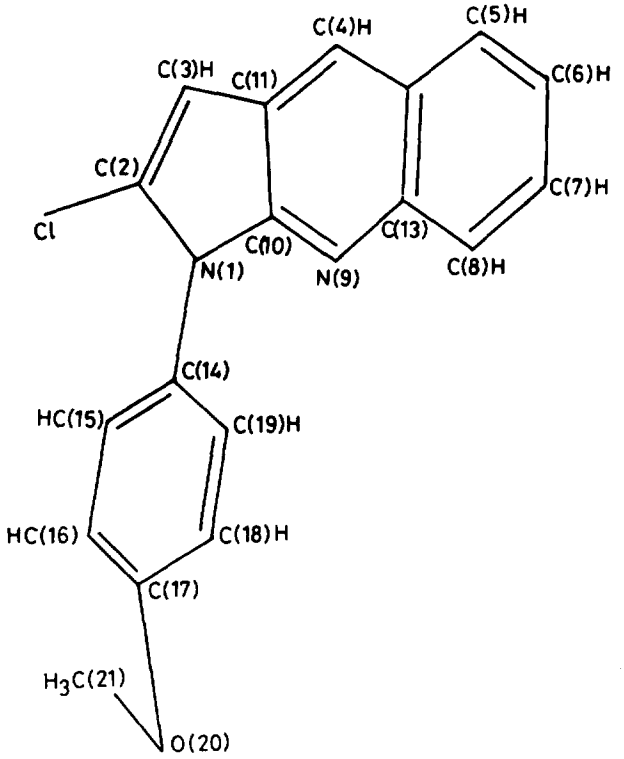

Figure 1. Molecular structure and numbering of atoms of N-(17-methoxy-phenyl)-2-chloropyrrolo (2,3-*b*) quinoline.

Table 1a. Fractional atomic parameters for the non-hydrogen atoms ($\times 10^4$) and the equivalent temperature factors with e.s.d. values in parentheses.

$B_{eq} = 8\pi^2/3(U_{11} + U_{22} + U_{33} + 2U_{13}\cos\beta)$

Atom	*X*	*Y*	*Z*	B_{eq} (Å)
Cl	2207(2)	2013(8)	2908(2)	3.3
N(1)	2305(6)	5462(24)	4089(2)	2.5
C(2)	2703(8)	4073(24)	3663(8)	2.6
C(3)	3509(8)	4595(32)	3975(9)	2.8
C(4)	4237(9)	7734(31)	5126(9)	2.6
C(5)	4712(9)	11111(35)	6240(10)	3.3
C(6)	4543(10)	12919(39)	6752(10)	3.8
C(7)	3771(12)	13395(34)	6727(10)	4.1
C(8)	3145(10)	11839(31)	6219(9)	2.9
N(9)	2646(7)	8657(24)	5162(7)	2.3
C(10)	2823(9)	6940(33)	4680(9)	2.7
C(11)	3605(8)	6474(28)	4617(8)	1.9
C(12)	4103(8)	9619(30)	5679(8)	2.1
C(13)	3285(10)	9965(31)	5661(8)	2.5
C(14)	1424(8)	5219(30)	3954(8)	1.9
C(15)	1198(10)	3214(39)	4349(12)	5.1
C(16)	0365(10)	2960(36)	4235(10)	4.1
C(17)	−0162(9)	4579(33)	3715(9)	2.5

(*Contd.*)

Table 1a. (*Contd.*)

Atom	*X*	*Y*	*Z*	B_{eq} (Å)
C(18)	0068(10)	6533(41)	3317(10)	4.3
C(19)	0912(10)	6872(36)	3446(11)	4.0
O(20)	−1000(6)	4513(23)	3545(6)	3.4
C(21)	−1245(9)	2329(35)	3925(11)	4.0

Table 1b. Anisotropic thermal parameters ($\times 10^3$) for the non-hydrogen atoms with e.s.d. values in parentheses.
$T = -2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cl	039(2)	046(2)	053(2)	−022(2)	020(2)	−025(2)
N(1)	027(7)	021(7)	050(9)	−011(6)	003(6)	−014(6)
C(2)	074(9)	007(7)	042(9)	−006(6)	033(8)	−028(6)
C(3)	032(9)	037(10)	039(10)	−015(7)	004(8)	−022(8)
C(4)	050(10)	021(10)	030(10)	−009(8)	002(9)	−005(8)
C(5)	049(11)	025(10)	054(13)	−003(8)	006(10)	−003(9)
C(6)	056(12)	051(12)	049(13)	−011(4)	016(10)	000(11)
C(7)	088(15)	017(10)	060(14)	−004(11)	016(11)	−001(9)
C(8)	063(11)	008(8)	056(12)	−003(8)	025(10)	−001(9)
N(9)	048(8)	015(7)	033(8)	000(6)	014(7)	001(6)
C(10)	033(9)	022(10)	045(11)	001(8)	−006(8)	−001(9)
C(11)	025(8)	015(8)	035(10)	007(7)	003(7)	−004(4)
C(12)	030(8)	021(9)	032(10)	−002(7)	004(7)	001(8)
C(13)	063(11)	021(9)	018(10)	011(8)	012(9)	−003(8)
C(14)	030(9)	026(10)	023(9)	−006(7)	008(7)	−010(8)
C(15)	046(11)	059(13)	101(17)	001(10)	017(11)	051(13)
C(16)	058(12)	040(12)	073(13)	004(10)	023(10)	045(11)
C(17)	043(11)	031(10)	037(11)	−002(9)	020(9)	−004(9)
C(18)	041(11)	059(13)	063(13)	−001(10)	−003(10)	007(11)
C(19)	063(13)	032(11)	068(14)	002(10)	015(11)	011(11)
O(20)	040(6)	053(7)	045(7)	003(6)	012(5)	016(6)
C(21)	037(10)	044(11)	101(15)	−018(8)	045(10)	012(11)

Table 1c. Fractional co-ordinates ($\times 10^4$) of the hydrogen atoms and isotropic thermal parameters.

Atom	*X*	*Y*	*Z*	Bi_{so} (Å^2)
H(3)	3807	4031	3731	3.61
H(4)	4805	6834	5165	2.89
H(5)	5232	10549	6321	3.52
H(6)	4974	14065	7226	2.91
H(7)	3667	15468	7157	3.63
H(8)	2550	12309	6282	2.89
H(15)	1502	2201	4696	3.94
H(16)	0225	1801	4548	3.22
H(18)	−0307	7460	2967	3.69
H(19)	1089	8435	3176	2.76

from 0 to 5 along the *b* axis. Corrections for Lorentz and polarisation factors were made to the intensities of these reflections but none for absorption effects.

The structure was solved by MULTAN (Main *et al* 1980). All hydrogen atoms except the methoxy hydrogens were geometricaly fixed and their positions were checked in the difference Fourier. They were included in the structure factor calculation but were not refined. Full matrix least-squares refinement (Gantzel *et al* 1961) with anisotropic temperature factors to non-hydrogen atoms converged at an R value of 0·104.

The weighting scheme $W = 1/(10{\cdot}0 + |F_0| + 0{\cdot}02|F_0|^2)$ (Cruickshank *et al* 1961) was applied. The atomic scattering factors were taken from the *International tables for X-ray crystallography* (1962). The final positional and thermal parameters are listed in table 1.

3. Discussion

The bond lengths and bond angles of non-hydrogen atoms are given in table 2. The average standard deviation in bond lengths and bond angles are 0·03 Å and 1·35 °

Table 2a. Bond lengths (Å) involving non-hydrogen atoms with e.s.d. values in parentheses

Cl −C(2)	1·68(1)	N(9) −C(10)	1·32(2)
N(1) −C(2)	1·37(2)	N(9) −C(13)	1·35(1)
N(1) −C(10)	1·35(1)	C(10)−C(11)	1·44(2)
N(1) −C(14)	1·49(1)	C(12)−C(11)	1·44(2)
C(2) −C(3)	1·36(1)	C(14)−C(15)	1·36(2)
C(3) −C(11)	1·44(2)	C(14)−C(19)	1·33(2)
C(4) −C(11)	1·33(1)	C(15)−C(16)	1·42(2)
C(4) −C(12)	1·44(2)	C(16)−C(17)	1·34(2)
C(5) −C(6)	1·38(2)	C(17)−C(18)	1·35(2)
C(5) −C(12)	1·41(1)	C(17)−O(20)	1·40(1)
C(6) −C(7)	1·37(2)	C(18)−C(19)	1·43(2)
C(7) −C(8)	1·40(2)	O(20)−C(21)	1·43(2)
C(8) −C(13)	1·45(2)		

Table 2b. Bond angles (°) involving non-hydrogen atoms with e.s.d. values in parentheses

Cl −C(2) −N(1)	121·5(9)	C(5) −C(12)−C(13)	118·0(11)
Cl −C(2) −N(3)	129·2(9)	C(6) −C(7) −C(8)	118·8(13)
N(1) −C(2) −C(3)	109·3(9)	C(7) −C(8) −C(13)	121·7(12)
C(2) −N(1) −C(10)	111·5(9)	C(8) −C(13)−N(9)	118·1(11)
N(1) −C(10)−N(9)	127·8(11)	C(8) −C(13)−C(12)	117·5(11)
N(1) −C(10)−C(11)	105·3(10)	N(9) −C(10)−C(11)	126·5(11)
C(2) −N(1) −C(14)	124·1(9)	C(10)−N(9) −C(13)	114·7(10)
C(10)−N(1) −C(14)	124·2(9)	N(9) −C(13)−C(12)	124·4(11)
N(1) −C(14)−C(15)	116·4(11)	C(4) −C(15)−C(16)	117·4(13)
N(1) −C(14)−C(19)	119·3(11)	C(15)−C(14)−C(19)	124·2(12)
C(2) −C(4) −C(11)	106·1(10)	C(14)−C(19)−C(18)	117·6(13)
C(3) −C(11)−C(4)	133·9(11)	C(15)−C(16)−C(17)	119·2(13)
C(3) −C(11)−C(10)	107·7(10)	C(16)−C(17)−C(18)	122·8(12)
C(4) −C(11)−C(10)	118·2(10)	C(16)−C(17)−O(20)	124·6(11)
C(11)−C(4) −C(12)	118·9(11)	C(17)−C(18)−C(19)	118·6(11)
C(4) −C(12)−C(5)	124·9(11)	C(18)−C(17)−O(20)	112·5(11)
C(4) −C(12)−C(13)	117·1(11)	C(17)−O(20)−C(21)	112·2(9)
C(5) −C(6) −C(7)	121·5(13)		
C(6) −C(5) −C(12)	122·1(12)		

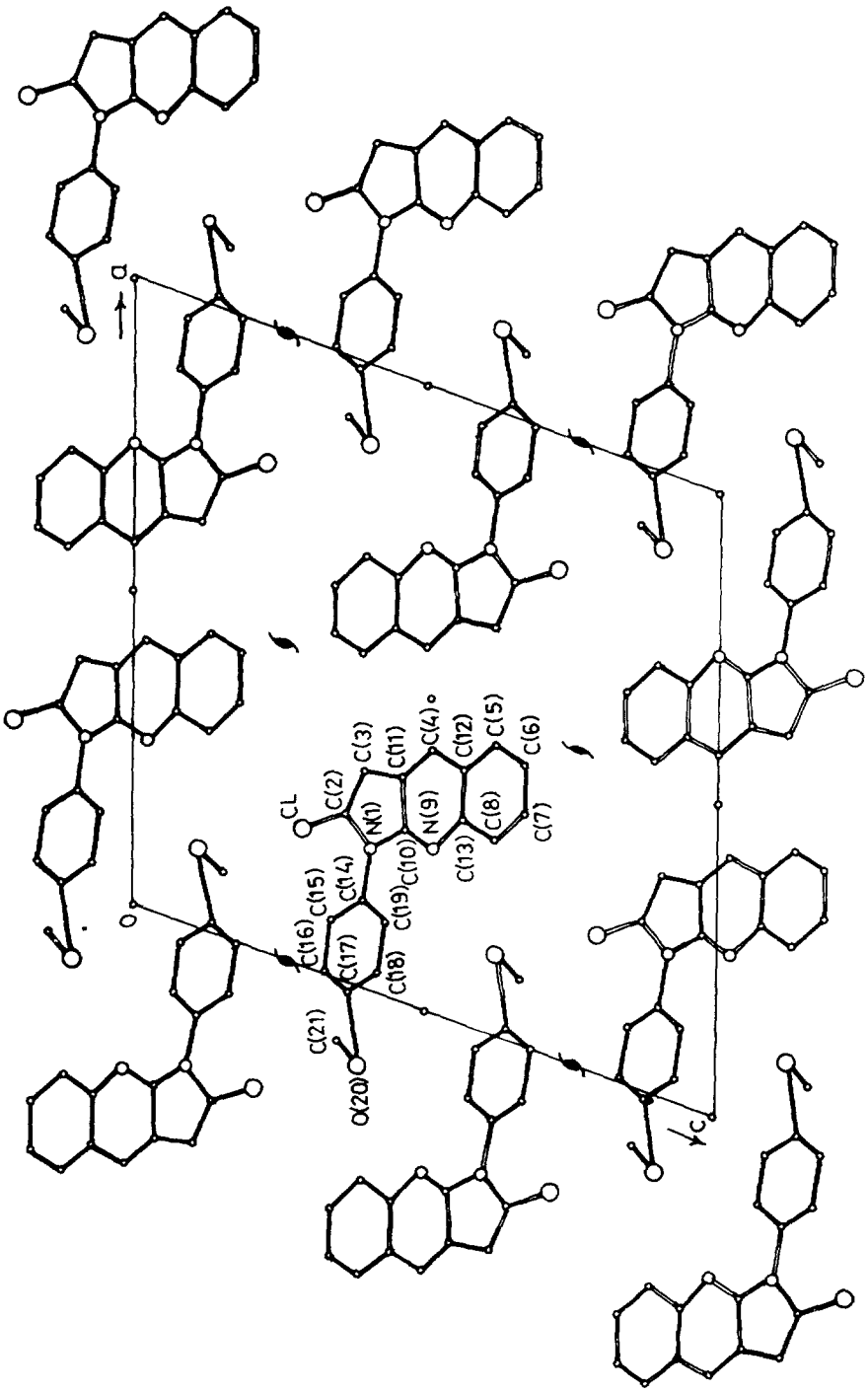

Figure 2. The crystal structure of the compound as viewed along the *b* axis.

respectively. The quinoline moiety is planar. The chloropyrrolo ring is also planar and lies nearly in the same plane as the quinoline moiety with a dihedral angle of 3·4°. The bond lengths and bond angles agree within 3σ level of the corresponding bonds in pyrrolo (2,3-*b*) quinoline derivatives (Szmuzkoviz *et al* 1976; Berman and Glusker 1972; Abbott *et al* 1976). The phenyl ring is approximately perpendicular to the plane of the chloropyrrolo (2,3-*b*) quinoline ring system with a dihedral angle of 86·5°. The crystal structure viewed along the *b* axis is shown in figure 2. An intermolecular contact of 2·99 Å is observed in the crystal structure between Cl and C(2). The structure is stabilised by van der Waals interaction.

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