FRENZ, B. A. (1982). *The Enraf–Nonius CAD4-SDP. A Real-Time System for Concurrent X-ray Data Collection and Crystal Structure Solution.* In *Computing in Crystallography*, edited by H. SCHENK, R. OLTHOF-HAZEKAMP, H. VAN KONINGSVELD & G. C. BASSI. Delft Univ. Press.
JAIN, S., ZON, G. & SUNDARALINGAM, M. (1989). *Biochemistry*, **28**, 2360–2364.
KARLE, J. M. & KARLE, I. L. (1988). *Acta Cryst.* C**44**, 1605–1608.
OLENICK, J. G. (1975). *Antibiotics*, **3**, 516–520.
OLENICK, J. G. & HAHN, F. E. (1972). *Antimicrobial Agents Chemotherapy*, **1**, 259–264.
PRESTON, H. S. & STEWART, J. M. (1970). *Chem Commun.* pp. 1142–1143.
STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
WEBSTER, L. T. (1985). *The Pharmacological Basis of Therapeutics*, 7th edition, edited by G. GILMAN, L. S. GOODMAN, T. W. RALL & F. MURAD, pp. 1051–1054.
WHICHARD, L. P., MORRIS, C. R., SMITH, J. M. & HOLBROOK, D. J. (1968). *Mol. Pharmacol.* **4**, 630–639.
WOO, N. H., SEEMAN, N. C. & RICH, A. (1979). *Biopolymers*, **18**, 539–545.

Acta Cryst. (1992). C**48**, 382–384

Structure of 2-Chloro-7,12-dihydropyrido[3,2-*b*:5,4-*b'*]diindole

BY MARIA B. SZKARADZINSKA AND ALEKSANDER W. ROSZAK

Departments of Chemistry and of Pharmacology and Therapeutics, University of Calgary, Calgary, Alberta, Canada T2N 1N4

MARK L. TRUDELL AND JAMES M. COOK

Department of Chemistry, University of Wisconsin-Milwaukee, Milwaukee, Wisconsin 53201, *USA*

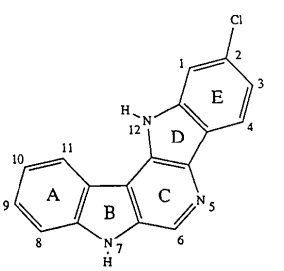
AND PENELOPE W. CODDING*

Departments of Chemistry and of Pharmacology and Therapeutics, University of Calgary, Calgary, Alberta, Canada T2N 1N4

(*Received* 16 *May* 1991; *accepted* 12 *August* 1991)

Abstract. $C_{17}H_{10}ClN_3$, $M_r = 291.74$, trigonal, $P3_1$, $a = 8.3291\ (5)$, $c = 17.1142\ (14)$ Å, $V = 1028.2\ (1)$ Å^3, $Z = 3$, $D_x = 1.413$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.54178 Å, $\mu = 2.44$ mm^{-1}, $F(000) = 450$, $T = 293$ K, $R = 0.038$ for 1287 unique observed reflections. The pyridodiindole skeleton is planar (r.m.s. = 0.039 Å and $\chi^2 = 0.62$ for 20 atoms). In the crystal, the molecules stack in pairs with the terminal *A* and *E* rings of adjacent molecules (x, y, z and $x+1$, $y+1$, z) above one another at a distance of 3.44 (7) Å. The other intermolecular interaction in this structure is a hydrogen bond between the indole N(12)—H(12) group and the pyridine N(5) atom from a symmetry-related molecule at $-x+y$, $1-x$, $-\frac{1}{3}+z$; the N···N distance is 2.874 (5) Å, the H···N distance is 1.92 (5) Å and the angle N—H···N is 162 (4)°. Interestingly, the other hydrogen-donor group, the indole N(7)—H(7) group, is not involved in hydrogen bonding. The C—Cl bond length is 1.728 (4) Å.

Experimental. The title compound (I) was prepared by Trudell, Basile, Shannon, Skolnick & Cook (1987). Single crystals were obtained by slow evaporation from methanol/ethyl acetate solution. A rectangular solid of dimensions 0.44 × 0.20 × 0.08 mm was used for data collection on an Enraf–Nonius CAD-4F diffractometer with Ni-filtered Cu $K\alpha$ radiation. Accurate unit-cell parameters were obtained from a least-squares refinement of the angles of 25 reflections with $31 < \theta < 45°$. An ω–2θ scan mode was used; three standards measured every 2000 s indicated no crystal deterioration [003 682 (17), $2\bar{4}5$ 482 (18), $3\bar{1}2$ 237 (7)]; intensities for 4360 reflections were collected [h: $-10\rightarrow10$, k: $-10\rightarrow10$, l: $-21\rightarrow0$; maximum $(\sin\theta/\lambda) = 0.6257$ Å^{-1}]; 1470 unique reflections ($R_{int} = 0.076$) of which 183 were regarded as unobserved [$I < 2.5\sigma(I)$]. No absorption correction was applied.



(I)

* Author to whom correspondence should be addressed.

0108-2701/92/020382-03$03.00

Table 1. *Fractional coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^2$) *for the non-H atoms (e.s.d.'s in parentheses)*

The z coordinate of Cl(2) was held invariant to define the cell origin.

$$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Cl(2)	393 (2)	−402 (1)	2517	659 (6)
C(1)	2445 (6)	3270 (5)	2286 (3)	430 (17)
C(2)	1212 (5)	1892 (5)	2762 (3)	432 (16)
C(3)	561 (5)	2235 (5)	3458 (3)	416 (16)
C(4)	1192 (5)	4039 (5)	3700 (2)	380 (14)
N(5)	3399 (4)	8556 (4)	3872 (2)	376 (12)
C(6)	4426 (6)	10386 (6)	3786 (2)	403 (15)
N(7)	6729 (5)	13030 (4)	2952 (3)	411 (13)
C(8)	8951 (6)	14720 (5)	1870 (3)	443 (17)
C(9)	9650 (5)	14452 (6)	1189 (3)	440 (17)
C(10)	9059 (6)	12689 (6)	879 (3)	417 (16)
C(11)	7705 (5)	11118 (5)	1259 (2)	348 (13)
N(12)	4317 (4)	6729 (4)	2159 (2)	354 (12)
C(13)	3048 (4)	5086 (5)	2525 (2)	323 (13)
C(14)	2452 (5)	5486 (4)	3225 (2)	320 (12)
C(15)	3433 (4)	7495 (5)	3288 (2)	331 (13)
C(16)	4520 (5)	8165 (4)	2610 (2)	308 (12)
C(17)	5653 (4)	10103 (4)	2518 (2)	313 (12)
C(18)	5560 (5)	11164 (4)	3129 (2)	359 (14)
C(19)	7613 (5)	13151 (5)	2261 (3)	375 (14)
C(20)	6972 (5)	11357 (4)	1957 (2)	333 (13)

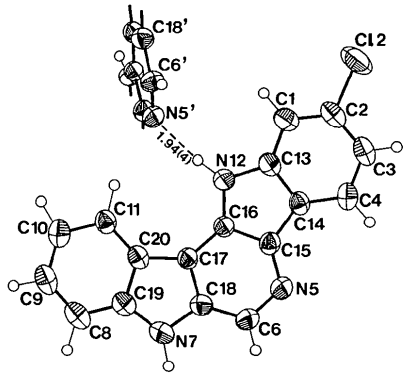


Fig. 1. A view of the title molecule showing the labeling of the non-H atoms and the hydrogen bond to the neighboring molecule related by the symmetry operation $-x+y, 1-x, -\frac{1}{3}+z$. Thermal ellipsoids are drawn at the 50% probability level.

Structure was solved by direct methods with *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and refined on F values by full-matrix least-squares techniques with *XRAY*76 (Stewart, 1976). All H atoms were located in a difference Fourier synthesis and refined with isotropic thermal parameters. All non-H atoms in the final cycles were refined with anisotropic thermal parameters; altogether, 230 parameters were refined.

The final $R = 0.038$, $wR = 0.031$ $[w^{-1} = \sigma^2(F)]$ and $S = 1.164$ were obtained for 1287 observed reflections; an empirical isotropic extinction parameter (g) converged at $0.67\,(2) \times 10^{-3}$ (Zachariasen, 1967; Larson, 1967); maximum (Δ/σ) was 0.0036 in the final cycle; residual densities in difference maps ranged from 0.22 to -0.29 e $Å^{-3}$, the extreme values were associated with the Cl atom. The scattering factors for non-H atoms were taken from Cromer & Mann (1968) and for H atoms from Stewart, Davidson & Simpson (1965).

Table 2. *Bond lengths* (Å) *and bond angles* (°) *for the non-H atoms (e.s.d.'s in parentheses)*

Cl(2)—C(2)	1.728 (4)	C(9)—C(10)	1.398 (7)
C(1)—C(2)	1.363 (5)	C(10)—C(11)	1.389 (5)
C(1)—C(13)	1.396 (6)	C(11)—C(20)	1.400 (6)
C(2)—C(3)	1.396 (7)	N(12)—C(13)	1.391 (4)
C(3)—C(4)	1.384 (6)	N(12)—C(16)	1.362 (5)
C(4)—C(14)	1.397 (5)	C(13)—C(14)	1.399 (6)
N(5)—C(6)	1.332 (5)	C(14)—C(15)	1.453 (5)
N(5)—C(15)	1.345 (5)	C(15)—C(16)	1.403 (5)
C(6)—C(18)	1.402 (6)	C(16)—C(17)	1.414 (4)
N(7)—C(18)	1.394 (4)	C(17)—C(18)	1.394 (6)
N(7)—C(19)	1.370 (6)	C(17)—C(20)	1.440 (4)
C(8)—C(9)	1.371 (7)	C(19)—C(20)	1.411 (5)
C(8)—C(19)	1.393 (5)		

C(2)—C(1)—C(13)	116.7 (4)	C(13)—C(14)—C(15)	106.0 (3)
C(1)—C(2)—Cl(2)	120.0 (4)	N(5)—C(15)—C(14)	128.8 (3)
C(1)—C(2)—C(3)	122.9 (4)	N(5)—C(15)—C(16)	125.1 (3)
Cl(2)—C(2)—C(3)	117.0 (3)	C(14)—C(15)—C(16)	106.1 (3)
C(2)—C(3)—C(4)	120.1 (3)	N(12)—C(16)—C(15)	110.3 (3)
C(3)—C(4)—C(14)	118.4 (4)	N(12)—C(16)—C(17)	131.2 (3)
C(6)—N(5)—C(15)	117.2 (3)	C(15)—C(16)—C(17)	118.4 (3)
N(5)—C(6)—C(18)	121.1 (4)	C(16)—C(17)—C(18)	115.0 (3)
C(18)—N(7)—C(19)	108.6 (3)	C(16)—C(17)—C(20)	137.2 (4)
C(9)—C(8)—C(19)	117.5 (4)	C(18)—C(17)—C(20)	107.7 (3)
C(8)—C(9)—C(10)	122.5 (4)	C(6)—C(18)—N(7)	128.5 (4)
C(9)—C(10)—C(11)	120.4 (4)	C(6)—C(18)—C(17)	123.1 (3)
C(10)—C(11)—C(20)	118.1 (4)	N(7)—C(18)—C(17)	108.3 (3)
C(13)—N(12)—C(16)	107.9 (3)	N(7)—C(19)—C(8)	129.2 (4)
C(1)—C(13)—N(12)	128.2 (4)	N(7)—C(19)—C(20)	109.6 (3)
C(1)—C(13)—C(14)	122.1 (3)	C(8)—C(19)—C(20)	121.2 (4)
N(12)—C(13)—C(14)	109.7 (3)	C(11)—C(20)—C(17)	134.0 (3)
C(4)—C(14)—C(13)	119.7 (3)	C(11)—C(20)—C(19)	120.3 (3)
C(4)—C(14)—C(15)	134.3 (4)	C(17)—C(20)—C(19)	105.6 (3)

The fractional coordinates and B_{eq} values for the non-H atoms are given in Table 1.* The atomic labeling scheme and the hydrogen bond observed in this structure are shown in Fig. 1 (*ORTEP*II; Johnson, 1976). Bond lengths and angles are listed in Table 2. The C—H bond distances range from 0.95 to 1.07 Å with an average of 1.00 Å (σ of sample 0.04 Å); the N(7)—H(7) bond length is 1.05 (7) and N(12)—H(12) is 0.98 (5) Å,

Related literature. The title compound was synthesized as a one of a novel class of rigid, planar benzodiazepine receptor ligands and was found to have high affinity for the receptor ($IC_{50} = 10$ nM) and an antagonist pharmacological profile (Trudell *et al.*, 1987; Trudell, Lifer, Tan, Martin, Deng,

* Lists of anisotropic thermal parameters, H-atom parameters, bond distances and angles for the H atoms, least-squares plane data and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54584 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Skolnick & Cook, 1990). This is the first X-ray structure determination of a 7,12-dihydropyrido[3,2-*b*:5,4-*b*']diindole.

This work was supported by the Alberta Heritage Foundation for Medical Research and the Medical Research Council of Canada.

References

CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* A**24**, 321–324.
JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
LARSON, A. C. (1967). *Acta Cryst.* **23**, 664–665.
MAIN, P., HULL, S. E. LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1978). *MULTAN*78. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.
STEWART, J. M. (1976). *The XRAY*76 *System of Crystallographic Programs.* Computer Science Center, Univ. of Maryland, College Park, MD, USA.
STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
TRUDELL, M. L., BASILE, A. S., SHANNON, H. E., SKOLNICK, P. & COOK, J. M. (1987). *J. Med. Chem.* **30**, 456–458.
TRUDELL, M. L., LIFER, S. L., TAN, Y.-C., MARTIN, M. J., DENG, L., SKOLNICK, P. & COOK, J. M. (1990). *J. Med. Chem.* **33**, 2412–2420.
ZACHARIASEN, W. H. (1967). *Acta Cryst.* **23**, 558–564.

Acta Cryst. (1992). C**48**, 384–385

Structure of 1-(β-D-Arabinofuranosyl)-6-methylcytosine

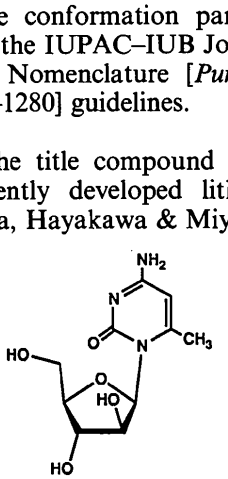
BY KENTARO YAMAGUCHI, GO MATSUMURA, MIHO SHIMIZU, HIROMICHI TANAKA AND TADASHI MIYASAKA

School of Pharmaceutical Sciences, Showa University 1–5–8, *Hatanodai, Shinagawa-ku, Tokyo* 142, *Japan*

(*Received* 7 *June* 1991; *accepted* 15 *July* 1991)

Abstract. $C_{10}H_{15}N_3O_5$, $M_r = 257.25$, orthorhombic, $P2_12_12_1$, $a = 10.235$ (2), $b = 10.833$ (1), $c = 10.129$ (3) Å, $V = 1123.0$ (3) Å^3, $Z = 4$, $D_x = 1.521$ Mg m^{-3}, λ(Cu $K\alpha_1$) = 1.54050 Å, $\mu = 1.002$ mm^{-1}, $F(000) = 544$, $T = 295$ K, final $R = 0.035$ for 950 reflections. The sugar conformation and puckering parameters are ^3E with $P = 18.5°$ and $\psi_m = 21.4°$. The *N*-glycosidic torsion angle χ has a value of -72.7 (3)° in the *syn* range. The C4′—C5′ side-chain conformation is $-ap$ with $\gamma = -174.5$ (4)°. The conformation parameters are in accordance with the IUPAC–IUB Joint Commission on Biochemical Nomenclature [*Pure Appl. Chem.* (1983), **55**, 1273–1280] guidelines.

Experimental. The title compound (I) was synthesized by a recently developed lithiation method (Shimizu, Tanaka, Hayakawa & Miyasaka, 1990).



(I)

Crystal dimensions 0.30 × 0.10 × 0.40 mm, by recrystallization from CH_3OH, having m.p. 492–493 K. Rigaku AFC-5 four-circle diffractometer used with ω–2θ-scan method, ω-scan width $(1.3 + 0.14\tan\theta)°$ and scan speed 16° min^{-1}. Lattice parameters obtained from least-squares analysis of 20 reflections with 2θ values ranging from 57 to 60°. Of 1026 reflections scanned (within index range h 0–11, k 0–12, l 0–11 up to $\sin\theta/\lambda < 0.56$ Å^{-1}), 986 unique reflections [$F > \sigma(F)$] classified as observed. Three standard reflections measured every 150 reflections, intensity variation <3%. Intensities corrected for Lorentz and polarization factors, but absorption correction not applied. Structure solved using program package *SAPI*85 (Yao, Zheng, Qian, Han, Gu & Fan, 1985). The refinement was carried out by the full-matrix least-squares method with anisotropic temperature factors for non-H atoms. The function minimized was $\sum w[|(|F_o|)^2 - (|F_c|)^2|]^2$ with $w = 1/[\sigma^2(F_o) + 0.02(F_c)]$, $\sigma(F_o)$ was determined from counting statistics. All H atoms located from the difference map and theoretical calculations were refined, initial thermal parameters set at equivalent isotropic thermal parameter of each bonded atom. Final discrepancy indices $R = 0.035$, $wR = 0.044$, $S = 1.535$ for 298 variables and 950 reflections with $F > 2.5\sigma(F)$. Maximum $\Delta/\sigma = 0.37$ in final least-squares cycle. Final difference Fourier excursions 0.14 and -0.21 e Å^{-3}. All major computations performed on PANAFACOM computer with *RCRYSTAN* (Rigaku Corporation, 1985) X-ray analysis program system. The atomic scattering factors were

0108-2701/92/020384-02$03.00