molecule *A* is 63·91°, that in molecule *B* is 65·11°, and that in molecule *C* is 75·43° (see Fig. 1).

Related literature. The crystal structures of flavone derivatives have been reported by several workers (Cantrell & Stalzer, 1982; Castleden, Hall, Nimgirawath, Thadaniti & White, 1985).

The authors wish to express their gratitude to Dr T. Taga from Kyoto University for his useful discussions and suggestions.

References

Cantrell, J. S. & Stalzer, R. A. (1982). *Acta Cryst.* **B38**, 983–984.

Castleden, I. R., Hall, S. R., Nimgirawath, S., Thadaniti, S. & White, A. H. (1985). *Aust. J. Chem.* **38**, 1177–1185.

Hirose, T., Tsuya, K., Nishigaki, T., Idaka, E. & Yano, S. (1989). *Liq. Cryst.* **4**, 653–659.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Sakurai, T. (1967). Editor. *Universal Crystallographic Computation Program System* (I). The Crystallographic Society of Japan, Tokyo, Japan.

Yao, J.-X. (1981). *Acta Cryst.* A**37**, 642–644.

Acta Cryst. (1991). C**47**, 1122–1124

10,11-Dihydroquinidine Sesquihydrate

By A. Suszko-Purzycka, T. Lipińska, Z. Karczmarzyk and B. Pniewska

Department of Chemistry, Agricultural and Teachers University in Siedlce, ul. 3 *Maja* 54, 08-110 *Siedlce, Poland*

(*Received* 23 *August* 1990; *accepted* 25 *October* 1990)

Abstract. $C_{20}H_{26}N_2O_2.1{\cdot}5H_2O$, $M_r = 353{\cdot}44$, orthorhombic, $P2_12_12_1$, $a = 12{\cdot}358\,(1)$, $b = 17{\cdot}631\,(1)$, $c = 17{\cdot}686\,(2)$ Å, $V = 3853\,(1)$ Å^3, $Z = 8$, $D_m = 1{\cdot}20\,(2)$, $D_x = 1{\cdot}217$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}54178$ Å, $\mu = 5{\cdot}91$ cm^{-1}, $F(000) = 1528$, $T = 293$ K, $R = 0{\cdot}0522$, $wR = 0{\cdot}0631$ for 4147 unique observed reflections. The asymmetric unit contains two alkaloid and three water molecules. The two independent molecules have identical geometries with the exception of the disordered ethyl chain in molecule *B*. The bond lengths and angles are in the expected ranges. The molecules are hydrogen bonded by O—H···N and O—H···O intermolecular contacts.

Experimental. 10,11-Dihydroquinidine is found with other alkaloids in *Cinchona* barks. Small amounts of this dihydrobase can be found in commercial quinidine (Suszko-Purzycka & Trzebny, 1965). The title compound was isolated from commercial quinidine by the mercuric acetate process (Thorn & Dirscherl, 1935) (based on the difference in solubility of the dihydroquinidine and the addition product of mercuric acetate to the vinyl alkaloid in aqueous ammonia). Further purification was carried out using column chromotagraphy, in the same way as for the quinine (Pniewska & Suszko-Purzycka, 1989). The alkaloid was obtained from the toluene–methanol eluate and recrystallized from acetone. Colourless prismatic crystals were grown by slow evaporation of acetone solution. D_m was determined by flotation in KI solution. A crystal 0·52 × 0·40 × 0·40 mm was used for diffractometer measurements. Data were collected with an Enraf–Nonius CAD-4 diffractometer using Cu $K\alpha$ radiation and a graphite monochromator. Unit-cell parameters were refined from the setting angles of 25 reflections having $20 < \theta < 30°$. 4426 reflections were measured with $1 < \theta < 75°$; index ranges: $0 \le h \le 15$, $0 \le k \le 22$, $0 \le l \le 22$, ω–2θ scan method, $\Delta\omega(\theta) = (1{\cdot}05 + 0{\cdot}14\tan\theta)°$. 0·5% intensity variation for three standard reflections measured every 1 h; no absorption correction. 4386 unique reflections of which 4147 with $F > 3\sigma(F)$. Structure solved by direct methods with *SHELXS*86 (Sheldrick, 1986) and refined with *SHELX*76 (Sheldrick, 1976) programs. Most non-H atoms were revealed from the first *E* map; C(10*B*) and C(11*B*) positions and the O atoms of the water molecules were located from a difference Fourier map. Anisotropic refinement of all non-H atoms gave non-positive-definite temperature tensors and implausible bond lengths for the C atoms of the ethyl group in molecule *B*. A difference Fourier map showed two alternative sites for the C(10*B*) and C(11*B*) atoms; site occupation refinement converged to 0·50 and these values and isotropic thermal parameters were used in further refinement. H atoms were located from difference maps and refined isotropically, except those for the disordered C(10*B*) and C(11*B*) atoms. Water H atoms could not be located and were omitted. $\sum w(|F_o| - |F_c|)^2$ minimized, $w = 1{\cdot}0/[\sigma^2(F_o) + 0{\cdot}007334F_o^2]$; final value for isotropic extinction parameter $g = 2{\cdot}5\,(2) \times 10^{-6}$,

0108-2701/91/051122-03$03.00

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j .$$

	x	y	z	U_{eq}
O(1)	0·3635 (2)	0·1538 (2)	0·3785 (2)	0·0917 (7)
O(2)	0·1384 (3)	0·1217 (3)	0·3815 (2)	0·1197 (13)
O(3)	0·0606 (5)	0·0098 (3)	0·4827 (4)	0·1830 (20)
Molecule *A*				
N(1*A*)	0·0246 (1)	0·8204 (1)	0·7462 (1)	0·0423 (4)
N(2*A*)	−0·2580 (2)	1·1233 (1)	0·7854 (1)	0·0523 (4)
O(1*A*)	0·0779 (1)	0·9873 (1)	0·6955 (1)	0·0520 (4)
O(2*A*	−0·1851 (2)	0·9196 (1)	1·0171 (1)	0·0637 (4)
C(2*A*)	0·0981 (2)	0·8059 (1)	0·6808 (1)	0·0447 (4)
C(3*A*)	0·0346 (2)	0·7759 (1)	0·6117 (2)	0·0490 (4)
C(4*A*)	−0·0835 (2)	0·7990 (1)	0·6243 (1)	0·0497 (4)
C(5*A*)	−0·1274 (2)	0·7496 (1)	0·6883 (2)	0·0613 (6)
C(6*A*)	−0·0482 (2)	0·7541 (1)	0·7544 (2)	0·0583 (6)
C(7*A*)	−0·0885 (2)	0·8824 (1)	0·6488 (1)	0·0413 (4)
C(8*A*)	−0·0464 (2)	0·8866 (1)	0·7304 (1)	0·0367 (4)
C(9*A*)	0·0054 (2)	0·9630 (1)	0·7528 (1)	0·0400 (4)
C(10*A*)	0·0818 (2)	0·8012 (2)	0·5370 (2)	0·0593 (4)
C(11*A*)	0·1906 (3)	0·7653 (3)	0·5204 (2)	0·0767 (8)
C(22*A*)	−0·1986 (2)	1·1229 (1)	0·7243 (2)	0·0557 (6)
C(23*A*)	−0·1118 (2)	1·0728 (1)	0·7119 (2)	0·0510 (4)
C(24*A*)	−0·0857 (2)	1·0203 (1)	0·7660 (1)	0·0410 (4)
C(25*A*)	−0·1304 (2)	0·9657 (1)	0·8932 (1)	0·0450 (4)
C(26*A*)	−0·1951 (2)	0·9664 (1)	0·9562 (1)	0·0493 (4)
C(27*A*)	−0·2811 (2)	1·0186 (2)	0·9623 (2)	0·0580 (6)
C(28*A*)	−0·3000 (2)	1·0697 (1)	0·9068 (2)	0·0560 (6)
C(29*A*)	−0·2349 (2)	1·0710 (1)	0·8404 (1)	0·0443 (4)
C(30*A*)	−0·1488 (2)	1·0179 (1)	0·8336 (1)	0·0413 (4)
C(31*A*)	−0·1024 (3)	0·8636 (2)	1·0141 (2)	0·0783 (8)
Molecule *B*				
N(1*B*)	−0·5334 (2)	0·8904 (1)	0·7433 (1)	0·0517 (4)
N(2*B*)	−0·2529 (2)	0·5850 (1)	0·7610 (1)	0·0467 (4)
O(1*B*)	−0·5820 (2)	0·73058 (9)	0·6703 (1)	0·0497 (4)
O(2*B*)	−0·3475 (2)	0·7695 (1)	1·0043 (1)	0·0613 (4)
C(2*B*)	−0·6096 (2)	0·9090 (2)	0·6808 (3)	0·0800 (12)
C(3*B*)	−0·5541 (4)	0·9435 (2)	0·6134 (3)	0·1003 (13)
C(4*B*)	−0·4341 (3)	0·9256 (2)	0·6219 (2)	0·0683 (6)
C(5*B*)	−0·3905 (2)	0·9726 (2)	0·6883 (2)	0·0607 (6)
C(6*B*)	−0·4634 (2)	0·9576 (1)	0·7562 (2)	0·0573 (6)
C(7*B*)	−0·4196 (2)	0·8413 (1)	0·6398 (1)	0·0470 (4)
C(8*B*)	−0·4617 (1)	0·8278 (1)	0·7197 (1)	0·0343 (4)
C(9*B*)	−0·5134 (2)	0·7490 (1)	0·7320 (1)	0·0387 (4)
C(10*B*)	−0·6334 (7)	0·9179 (4)	0·5524 (5)	0·0791 (17)*
C(10*B*′)	−0·5692 (6)	0·9165 (4)	0·5253 (4)	0·0783 (17)*
C(11*B*)	−0·6125 (10)	0·9556 (7)	0·4770 (7)	0·1164 (33)*
C(11*B*′)	−0·6766 (10)	0·9519 (7)	0·5028 (7)	0·1063 (27)*
C(22*B*)	−0·3102 (2)	0·5894 (1)	0·6979 (1)	0·0473 (4)
C(23*B*)	−0·3959 (2)	0·6410 (1)	0·6868 (1)	0·0447 (4)
C(24*B*)	−0·4242 (2)	0·6906 (1)	0·7428 (1)	0·0370 (4)
C(25*B*)	−0·3894 (2)	0·7346 (1)	0·8750 (1)	0·0427 (4)
C(26*B*)	−0·3304 (2)	0·7278 (1)	0·9401 (1)	0·0450 (4)
C(27*B*)	−0·2443 (2)	0·6748 (2)	0·9461 (1)	0·0563 (6)
C(28*B*)	−0·2206 (2)	0·6289 (1)	0·8868 (1)	0·0520 (4)
C(29*B*)	−0·2801 (2)	0·6337 (1)	0·8180 (1)	0·0413 (4)
C(30*B*)	−0·3652 (2)	0·6874 (1)	0·8123 (1)	0·0370 (4)
C(31*B*)	−0·4246 (4)	0·8290 (2)	0·9997 (2)	0·0753 (8)

* Isotropic thermal parameter and s.o.f. = 0·50.

where $F_c^* = F_c(1 - gF_c/\sin\theta)$; $R = 0{\cdot}0522$, $wR = 0{\cdot}0631$, max. $\Delta/\sigma = 0{\cdot}233$ (av. 0·025) for non-H atoms and 0·590 (av. 0·067) for H atoms; max. difference peak +0·38, min. difference peak $-0{\cdot}22$ e Å^{-3}. Atomic scattering factors as supplied by *SHELX*76. The geometrical calculations were carried out using *CSU*88 (Vickovic, 1988).

The absolute configuration for both molecules was assigned on the basis of internal comparison with the configuration of quinidine with *R* at C(3), *S* at C(4), *R* at C(8) and *S* at C(9) atoms (Turner & Woodward, 1953; Carter, McPhail & Sim, 1967).

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

	Molecule *A*	Molecule *B*		Molecule *A*	Molecule *B*
N(1)—C(2)	1·492 (3)	1·489 (5)	C(7)—C(8)	1·535 (3)	1·524 (3)
N(1)—C(6)	1·483 (3)	1·484 (3)	C(8)—C(9)	1·543 (3)	1·545 (3)
N(1)—C(8)	1·487 (3)	1·475 (3)	C(9)—C(24)	1·531 (3)	1·520 (3)
N(2)—C(22)	1·307 (4)	1·324 (3)	C(10)—C(11)	1·515 (5)	1·513 (15)
N(2)—C(29)	1·370 (3)	1·367 (3)	C(10′)—C(11′)		1·519 (14)
O(1)—C(9)	1·419 (3)	1·419 (3)	C(22)—C(23)	1·406 (3)	1·410 (3)
O(2)—C(26)	1·361 (3)	1·368 (3)	C(23)—C(24)	1·370 (3)	1·366 (3)
O(2)—C(31)	1·423 (5)	1·420 (5)	C(24)—C(30)	1·429 (3)	1·432 (3)
C(2)—C(3)	1·546 (3)	1·504 (6)	C(25)—C(26)	1·372 (3)	1·369 (3)
C(3)—C(4)	1·532 (3)	1·524 (6)	C(25)—C(30)	1·418 (3)	1·418 (3)
C(3)—C(10)	1·511 (4)	1·525 (9)	C(26)—C(27)	1·411 (4)	1·421 (4)
C(3)—C(10′)		1·639 (9)	C(27)—C(28)	1·352 (4)	1·357 (4)
C(4)—C(5)	1·528 (4)	1·535 (4)	C(28)—C(29)	1·424 (4)	1·423 (3)
C(4)—C(7)	1·533 (3)	1·530 (4)	C(29)—C(30)	1·422 (3)	1·419 (3)
C(5)—C(6)	1·527 (4)	1·524 (4)			

	Molecule *A*	Molecule *B*
C(2)—N(1)—C(6)	108·1 (2)	107·9 (2)
C(2)—N(1)—C(8)	110·4 (2)	109·5 (2)
C(6)—N(1)—C(8)	106·2 (2)	106·9 (2)
C(22)—N(2)—C(29)	117·8 (2)	117·0 (2)
C(26)—O(2)—C(31)	117·2 (2)	116·9 (2)
N(1)—C(2)—C(3)	111·2 (2)	112·9 (3)
C(2)—C(3)—C(4)	106·1 (2)	106·4 (3)
C(2)—C(3)—C(10)	113·2 (2)	98·5 (4)
C(2)—C(3)—C(10′)		125·7 (4)
C(4)—C(3)—C(10)	114·6 (2)	129·3 (4)
C(4)—C(3)—C(10′)		98·3 (4)
C(3)—C(4)—C(5)	107·1 (2)	107·8 (3)
C(3)—C(4)—C(7)	109·5 (2)	109·6 (3)
C(5)—C(4)—C(7)	108·8 (2)	108·9 (2)
C(4)—C(5)—C(6)	108·0 (2)	107·6 (2)
N(1)—C(6)—C(5)	110·8 (2)	111·2 (2)
C(4)—C(7)—C(8)	107·4 (2)	107·7 (2)
N(1)—C(8)—C(7)	109·8 (2)	110·5 (2)
N(1)—C(8)—C(9)	113·0 (2)	112·7 (2)
C(7)—C(8)—C(9)	115·1 (2)	114·4 (2)
O(1)—C(9)—C(8)	110·1 (2)	110·1 (2)
O(1)—C(9)—C(24)	112·0 (2)	112·0 (2)
C(8)—C(9)—C(24)	108·0 (2)	109·1 (2)
C(3)—C(10)—C(11)	112·9 (3)	112·6 (7)
C(3)—C(10′)—C(11′)		103·2 (6)
N(2)—C(22)—C(23)	124·1 (2)	123·8 (2)
C(22)—C(23)—C(24)	119·7 (2)	120·3 (2)
C(9)—C(24)—C(23)	120·8 (2)	121·9 (2)
C(9)—C(24)—C(30)	120·7 (2)	120·3 (2)
C(23)—C(24)—C(30)	118·4 (2)	117·8 (2)
C(26)—C(25)—C(30)	120·2 (2)	119·6 (2)
O(2)—C(26)—C(25)	125·7 (2)	124·6 (2)
O(2)—C(26)—C(27)	113·8 (2)	114·1 (2)
C(25)—C(26)—C(27)	120·4 (2)	121·3 (2)
C(26)—C(27)—C(28)	120·7 (3)	119·8 (2)
C(27)—C(28)—C(29)	120·7 (3)	120·9 (2)
N(2)—C(29)—C(28)	118·6 (2)	117·8 (2)
N(2)—C(29)—C(30)	122·6 (2)	123·3 (2)
C(28)—C(29)—C(30)	118·8 (2)	118·9 (2)
C(24)—C(30)—C(25)	123·6 (2)	122·8 (2)
C(24)—C(30)—C(29)	117·4 (2)	117·7 (2)
C(25)—C(30)—C(29)	119·0 (2)	119·5 (2)

Final positional parameters and equivalent isotropic temperature factors are given in Table 1,* bond distances and angles in Table 2 and intermolecular short contacts in Table 3. The molecular structure and atomic numbering scheme are shown in the *PLUTO*78 (Motherwell & Clegg, 1978) drawing in Fig. 1.

---

* Lists of structure factors, anisotropic thermal parameters, least-squares planes, dihedral angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53698 (36 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Short intermolecular contacts* (Å,°)

O—H···*A*	O—H	H···*A*	O···*A*	O—H···*A*
O(1A^{i})—H(1*A*)···N(2B^{ii})	0·87 (3)	2·02 (3)	2·869 (3)	164 (2)
O(1B^{i})—H(1*B*)···N(2A^{iii})	0·86 (3)	2·00 (3)	2·846 (3)	166 (3)
O(1^{i})···N(1A^{iv})			2·756 (3)	
O(2^{i})···N(1B^{iv})			2·775 (4)	
O(1^{i})···O(2^{i})			2·839 (5)	
O(2^{i})···O(3^{i})			2·832 (7)	

Symmetry code: (i) x, y, z; (ii) $-x, \frac{1}{2}+y, \frac{1}{2}+1-z$; (iii) $-1-x, \frac{1}{2}+y, \frac{1}{2}+1-z$; (iv) $\frac{1}{2}-x, 1-y, -\frac{1}{2}+z$.

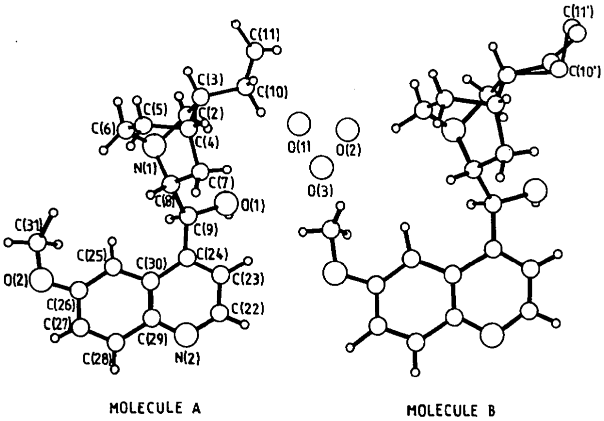

Fig. 1. A view of the molecules with the atomic labelling.

Related literature. For structures of quinidine and its derivatives, see Carter, McPhail & Sim (1967); Doherty, Benson, Maienthal & Stewart (1978); Karle & Karle (1981); Kashino & Haisa (1983); Suszko-Purzycka, Lipińska, Karczmarzyk & Pniewska (1987, 1990).

The intensities were measured at The Institute of Techinal Biochemistry, Technical University in Łódź. This research was supported by Project RP.II.10 from MEN.

References

Carter, O. L., McPhail, A. T. & Sim, G. A. (1967). *J. Chem. Soc. A*, pp. 365–373.

Doherty, R., Benson, W. R., Maienthal, M. & Stewart, J. McD. (1978). *J. Pharm. Sci.* **67**, 1698–1701.

Karle. I. L. & Karle J. (1981). *Proc. Natl Acad. Sci. USA*, **78**, 5938–5947.

Kashino, S. & Haisa, M. (1983). *Acta Cryst.* **C39**, 310–312.

Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ of Cambridge, England.

Pniewska, B. & Suszko-Purzycka, A. (1989). *Acta Cryst.* **C45**, 638–642.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Sheldrick, G. M. (1986). *SHELXS86*. Program for the solution of crystal structures. Univ. of Göttingen, Germany.

Suszko-Purzycka, A., Lipińska, T., Karczmarzyk, Z. & Pniewska, B. (1987). *J. Crystallogr. Spectrosc. Res.* **17**, 31–40.

Suszko-Purzycka, A., Lipińska, T., Karczmarzyk, Z. & Pniewska, B. (1990). *Acta Cryst.* **C46**, 2124–2126.

Suszko-Purzycka, A. & Trzebny, W. (1965). *J. Chromatogr.* **17**, 114–119.

Thorn, H. & Dirscherl, W. (1935). *Ann. Chem.* **515**, 252–260.

Turner, R. B. & Woodward, R. B. (1953). *The Alkaloids*, Vol. III, edited by R. H. F. Manske & H. L. Holmes, pp. 1–63. New York: Academic Press.

Vickovic, I. (1988). *CSU*. Program for crystal structure utility. Univ. of Zagreb, Yugoslavia.

Acta Cryst. (1991). **C47**, 1124–1126

Diethylammonium Tetrafluoro-4-pyridinolate

By R. G. Pritchard, A. J. Adamson, R. E. Banks, W. Jondi and A. E. Tipping

Department of Chemistry, University of Manchester Institute of Science and Technology, PO Box 88,
Manchester M60 1QD, England

(*Received* 27 *September* 1990; *accepted* 29 *October* 1990)

Abstract. $C_4H_{12}N^+.C_5F_4NO^-$, $M_r = 240·2$, triclinic, $P\bar{1}$, $a = 9·268$ (6), $b = 9·637$ (7), $c = 7·209$ (4) Å, $\alpha = 107·06$ (5), $\beta = 95·22$ (5), $\gamma = 63·70$ (4)°, $V = 551·3$ (7) Å^3, $Z = 2$, $D_x = 1·45$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0·13$ mm^{-1}, $F(000) = 248$, $T = 293$ K, $R = 0·049$ for 1455 unique reflexions [$F \geq 3\sigma(F)$]. The title compound, $H_2NEt_2^+.NC_5F_4O^-$, forms as an unexpected by-product during the synthesis of *N*,*N*-diethyl-*O*-(tetrafluoro-4-pyridyl)-hydroxylamine. Strong hydrogen bonds link two anion/cation pairs into a cyclic centrosymmetric dimer [N···O 2·741 (8), 2·765 (7); H···O 1·60 (6), 2·08 (6) Å; N—H···O 172 (5), 161 (7)°], which may in part explain the salt's stability and hence ease of formation.

Experimental. Reaction of sodium diethylnitroxide in benzene with pentafluoropyridine yielded several products, including a white solid consisting predominantly of diethylammonium tetrafluoro-4-pyridinolate and sodium fluoride. Removal of the latter by boiling in ethyl acetate followed by repeated recrys-

0108-2701/91/051124-03$03.00