

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)
$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	x	y	z	<i>U</i> _{eq}
O1	0.0535 (3)	0.1891 (3)	-0.2387 (2)	0.0756 (9)
C1	0.0728 (3)	0.0358 (3)	-0.0994 (2)	0.0390 (7)
C2	0.0021 (4)	0.0987 (3)	-0.1903 (2)	0.0491 (8)
C3	-0.1324 (5)	0.0417 (6)	-0.2210 (3)	0.0758 (13)
C4	-0.2199 (4)	-0.0062 (6)	-0.1333 (3)	0.0701 (12)
C5	-0.1646 (4)	0.0368 (5)	-0.0290 (3)	0.0598 (10)
C6	-0.0236 (3)	-0.0186 (3)	-0.0147 (2)	0.0390 (7)
C7	0.0481 (3)	0.0110 (3)	0.0905 (2)	0.0446 (8)
C8	0.1922 (3)	0.0537 (3)	0.0566 (2)	0.0449 (8)
C9	0.2890 (3)	-0.0637 (4)	0.0318 (3)	0.0526 (9)
C10	0.2338 (4)	-0.1533 (3)	-0.0541 (3)	0.0547 (9)
C11	0.1640 (3)	-0.0781 (4)	-0.1413 (3)	0.0497 (8)
C12	0.1641 (4)	0.1329 (3)	-0.0415 (3)	0.0452 (8)
C13	0.4296 (4)	-0.0111 (6)	0.0071 (5)	0.0752 (13)
C14	-0.0143 (5)	0.1283 (4)	0.1492 (3)	0.0671 (11)
C15	0.0432 (4)	-0.1097 (4)	0.1624 (3)	0.0590 (10)

Table 2. Selected geometric parameters (Å, °)

O1—C2	1.210 (5)	C6—C7	1.569 (5)
C1—C2	1.509 (5)	C7—C15	1.522 (6)
C1—C12	1.526 (6)	C7—C14	1.528 (7)
C1—C6	1.557 (6)	C7—C8	1.560 (6)
C1—C11	1.552 (6)	C8—C12	1.524 (6)
C2—C3	1.508 (7)	C8—C9	1.549 (6)
C3—C4	1.511 (8)	C9—C13	1.529 (7)
C4—C5	1.522 (8)	C9—C10	1.530 (7)
C5—C6	1.520 (6)	C10—C11	1.525 (6)
C2—C1—C6	114.1 (3)	C1—C6—C7	105.6 (3)
C12—C1—C6	103.9 (3)	C15—C7—C14	106.6 (4)
C12—C1—C11	106.7 (3)	C8—C7—C6	103.0 (3)
O1—C2—C1	121.1 (4)	C12—C8—C9	109.4 (4)
O1—C2—C3	121.2 (4)	C12—C8—C7	102.0 (3)
C1—C2—C3	117.6 (4)	C10—C9—C8	111.6 (4)
C4—C3—C2	115.6 (4)	C11—C10—C9	114.7 (4)
C3—C4—C5	111.9 (4)	C10—C11—C1	111.4 (3)
C4—C5—C6	109.9 (4)	C8—C12—C1	101.1 (3)
C5—C6—C1	110.9 (3)		

Symmetry-related reflections for the tetragonal system were checked but were not found to be equivalent, so orthorhombic symmetry was assumed. The similarity of the unit-cell parameters *a* and *b* was concluded to be fortuitous. The structure was solved by direct methods (*MULTAN88*; Debaerdemaeker *et al.*, 1988) followed by Fourier methods and refined by anisotropic full-matrix least-squares on *F*² using *SHELXL93* (Sheldrick, 1993). H atoms were located from difference Fourier maps and were refined isotropically. Data collection: AFC-5 software. Software used for geometric calculations: *PARST* (Nardelli, 1983). Software used to prepare material for publication: *SHELXL93*. Molecular graphics: *ORTEPII* (Johnson, 1976). Calculations were performed using a VAX3400 computer at the Computer Center, Indian Association for the Cultivation of Science.

Two of the authors (SD & DM) wish to acknowledge DST, Government of India, for financial assistance.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: LI1123). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1995). **C51**, 282–285

Quitenine Ethyl Ester

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(Received 8 July 1994; accepted 8 September 1994)

Abstract

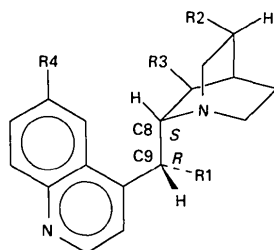
The crystal structure of 6'-methoxy-(8 α ,9*R*)-cinchonan-9-ol-3-carboxylic acid ethyl ester, C₂₁H₂₆N₂O₄, has been determined by X-ray diffraction. The conformation of the molecule is closed, in contrast to the open conformation of quinine. The N atom of the quinuclidine moiety is not involved in hydrogen bonding. The molecules linked by intermolecular N13···H—O22 hydrogen bonds form chains along the *y* axis.

Comment

Quitenine ethyl ester, (II), is a derivative of the anti-malarial alkaloid quinine, (I), in which the vinyl group at C3 of the quinuclidine moiety is oxidized to carboxyl

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and then esterified with ethanol. The crystal structure investigation of (II) was undertaken as a part of systematic studies on the relationship between biological activity and molecular properties of cinchona alkaloids and their derivatives. In particular, it seems interesting to compare the molecular geometry of (II) in the crystalline state with that of (I) and its two other ester derivatives: acetylquitenine ethyl ester, (III), and the synthetic compound (IV), which is similar to substituted quinine but without the vinyl group on the quinuclidine fragment and methoxy group on the quinoline fragment.



	R1	R2	R3	R4
(I)	—OH	—CH=CH ₂	—H	—O—CH ₃
(II)	—OH	—C(=O)—O—C ₂ H ₅	—H	—O—CH ₃
(III)	—O—C(=O)—CH ₃	—C(=O)—O—C ₂ H ₅	—H	—O—CH ₃
(IV)	—O—C(=O)—CH ₃	—H	—O—C(=O)—CH ₃	—H

The bond lengths in (II) (Table 2) are comparable to within 3σ with those of (III) (Dupont, Konsur, Lewinski & Oleksyn, 1985) and (IV) (South, Kashyap, Minter, Krawiec & Watson, 1993), except for C9—O22 and C8—C9. The first of these bonds is longer [1.456 (3) Å in (III) and 1.466 (4) Å in (IV)] and the second shorter [1.522 (3) Å in (III) and 1.520 (4) Å in (IV)] in the molecules with an esterified —C9—O22H group than in (II). The bond angles in (II) (Table 2) are in good agreement with those in (III) and (IV); some differences occur in the vicinity of C9 and in the ester group at C3.

The molecular shape of the compounds (I)–(IV) is determined by two torsion angles, $\tau_1 = \text{C11—C10—C9—O22}$ and $\tau_2 = \text{O22—C9—C8—N1}$, around the bonds connecting the quinuclidine and quinoline moieties with C9. The values in (II) are $\tau_1 = -32.2(2)^\circ$ and $\tau_2 = -173.5(1)^\circ$. These angles determine the mutual orientation of the quinine pharmacophore fragments, *i.e.* the basic quinuclidine N1 atom, the hydroxyl at C9 and the hydrophobic quinoline ring. The overall conformation of (II) (Fig. 1) can be characterized as *gauche-trans* or closed [the term introduced

by Dijkstra *et al.* (1989)] and is different from that of the quinine base (I) (Pniewska & Suszko-Purzycka, 1989), which has a *gauche-gauche* or open conformation (Fig. 2). The open conformation was observed in the crystalline state for all *erythro* cinchona alkaloids in the form of free bases and N1-protonated cations, with the torsion angles τ_1 and τ_2 varying within the ranges -23 to -12° and -82 to -72° , respectively, for the diastereoisomers C8(*S*),C9(*R*) (cinchonidine and quinine), and within the ranges 15 to 23° and 46 to 78° , respectively, for the C8(*R*),C9(*S*) isomers (cinchonine and quinidine) (Oleksyn, Śliwiński, Kowalik & Serda, 1991). The closed conformation has hitherto been reported for two derivatives of the natural alkaloids, *i.e.* for (III) and (*p*-chlorobenzoyl)dihydroquinidine (V) (Dijkstra *et al.*, 1989), and for the synthetic compound (IV). The torsion angle τ_1 observed for (II)–(IV) changes in a wide range, from -32 to -99° , while τ_2 is close to 180° . In (V), τ_1 is positive (54°) because of the absolute configuration C8(*R*),C9(*S*), but τ_2 is also approximately 180° .

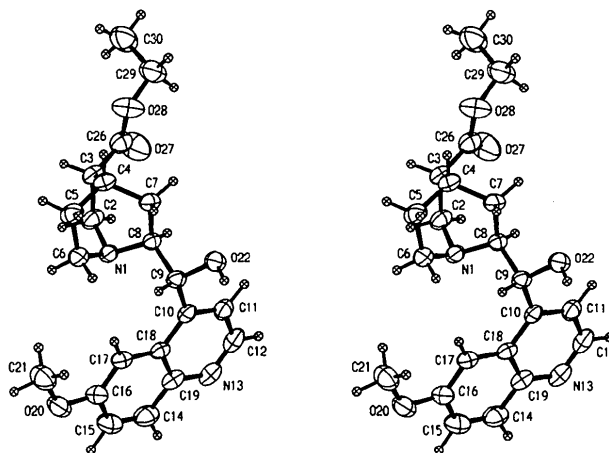


Fig. 1. A stereoscopic projection of the molecule of quitenine ethyl ester (II) with atom numbers. Displacement ellipsoids are shown at the 50% probability level.

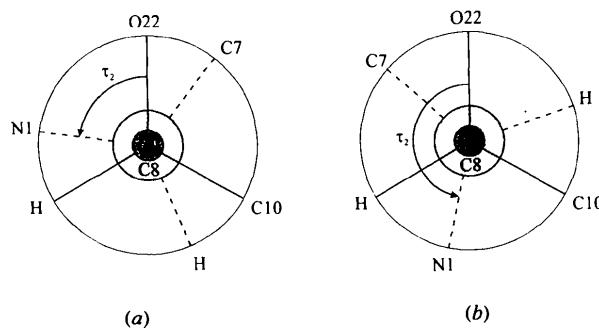


Fig. 2. Newman projections along the C9—C8 bond for (a) the open conformation of (I) and (b) the closed conformation of (II).

According to molecular-mechanics calculations (Dijkstra *et al.*, 1989; Oleksyn, Suszko-Purzycka, Dive & Lamotte-Brasseur, 1992), the potential-energy values of the closed and open conformations of the isolated alkaloid-base molecules are very similar. Thus, the occurrence of one of these conformations in a given crystal structure seems to be the result of an interplay between the influence of substituents and packing conditions. Among the latter factors, the most important are intermolecular hydrogen bonds involving N1, which are observed in all the structures in which the alkaloid molecule has the open conformation. In the closed conformation, the atom N1 is inaccessible for hydrogen bonding, its free-electron pair being directed towards the quinoline ring and approximately parallel to its plane. These facts suggest that the engagement of N1 in the hydrogen bonding favours the open conformation and, indeed, the non-protonated alkaloid derivatives (III)–(V) which do not contain any proton donors have closed conformations in their crystalline states. The closed conformation of (II), observed in spite of the presence of the proton donor –O22H in its structure, is an apparent exception from the above rule, but can be explained by the influence of the substituent, R2, at C3. The bulky ethyl ester group may hinder the formation of the intermolecular hydrogen bond, N1···H—O22, and another, N13···H—O22, is formed instead. This reasoning is confirmed by the occurrence of the open conformation in the structure of quitenidine methyl ester (VI) (Ciechanowicz-Rutkowska, Oleksyn, Suszko-Purzycka & Lipinska, 1992), in which N1 is linked by a hydrogen bond to a water molecule and the ester substituent at C3 is less bulky than in (II).

The absolute value of the torsion angle τ_1 around the C10—C9 bond in (II) is comparable to those in (III) and (VI), but is lower than those of (IV) and (V), which may be explained by the effect of substituents R3 and R1 at C7 and C9, respectively, in the latter two molecules. The conformation of the quinuclidine moiety is similar to that described earlier for (III). The ethyl ester group defined by atoms C26, O27, O28 and C29 is planar and forms an angle of 27.08 (2)° with the best plane of the quinoline moiety; thus, the mutual orientation of these two fragments is similar to that in (III), in which this angle is 15.5°.

The packing of the molecules of (II) in the unit cell is determined by intermolecular hydrogen bonds, O22—H221···N13ⁱ [symmetry code: (i) $-x + 2, y - \frac{1}{2}, -z + \frac{1}{2}$], which lead to the formation of chains along the *y* axis. The parameters of these bonds, N13ⁱ···O22 = 2.830 (2), N13ⁱ···H221 = 1.89 (3) Å, O22—H221···N13ⁱ = 178 (2)°, are close to those observed in the structure of 10-hydroxy-10-methyl-10,11-dihydroquinine (Suszko-Purzycka, Lipinska, Piotrowska & Oleksyn, 1985). The chains interact with one another through van der Waals forces.

Experimental

Quitenine ethyl ester was obtained from natural quinine by oxidation to quitenine followed by esterification with ethanol (Suszko-Purzycka, Lipinska, Piotrowska & Oleksyn, 1985). Crystals were obtained by slow evaporation from ethanol solution.

Crystal data

C ₂₁ H ₂₆ N ₂ O ₄	Cu K α radiation
$M_r = 370.44$	$\lambda = 1.54178 \text{ \AA}$
Orthorhombic	Cell parameters from 25 reflections
$P2_12_12_1$	$\theta = 17.9\text{--}34.0^\circ$
$a = 9.325 (1) \text{ \AA}$	$\mu = 0.744 \text{ mm}^{-1}$
$b = 11.474 (2) \text{ \AA}$	$T = 293 (2) \text{ K}$
$c = 17.418 (3) \text{ \AA}$	Needle
$V = 1863.6 (5) \text{ \AA}^3$	$0.3 \times 0.2 \times 0.2 \text{ mm}$
$Z = 4$	Colourless
$D_x = 1.320 \text{ Mg m}^{-3}$	

Data collection

CAD-4 diffractometer	$\theta_{\max} = 75.68^\circ$
$\omega/2\theta$ scans	$h = -11 \rightarrow 0$
Absorption correction: none	$k = -14 \rightarrow 0$
	$l = -21 \rightarrow 21$
4061 measured reflections	2 standard reflections
3662 independent reflections	monitored every 22 reflections
3344 observed reflections	intensity decay: 3.5%
$[I > 2\sigma(I)]$	
$R_{\text{int}} = 0.0150$	

Refinement

Refinement on F^2	Extinction correction: <i>SHELXL93</i> (Sheldrick, 1993)
$R[F^2 > 2\sigma(F^2)] = 0.0335$	Extinction coefficient: 0.0033 (4)
$wR(F^2) = 0.0914$	Atomic scattering factors from <i>International Tables for Crystallography</i> (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
$S = 1.045$	Absolute configuration: $\chi = -0.2 (2)$ (Flack, 1983)
3662 reflections	
349 parameters	
All H-atom parameters refined	
$w = 1/[\sigma^2(F_o^2) + (0.0612P)^2 + 0.1359P]$	
where $P = (F_o^2 + 2F_c^2)/3$	
$(\Delta/\sigma)_{\max} = 0.025$	
$\Delta\rho_{\max} = 0.263 \text{ e \AA}^{-3}$	
$\Delta\rho_{\min} = -0.131 \text{ e \AA}^{-3}$	

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

	$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$			
	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
N1	0.50109 (13)	0.88686 (12)	0.21795 (8)	0.0376 (3)
C2	0.3742 (2)	0.9532 (2)	0.24271 (11)	0.0446 (4)
C3	0.2777 (2)	0.88238 (14)	0.29839 (10)	0.0404 (3)
C4	0.3374 (2)	0.75821 (14)	0.30208 (10)	0.0410 (4)
C5	0.3412 (2)	0.7120 (2)	0.21979 (12)	0.0513 (4)
C6	0.4512 (2)	0.7841 (2)	0.17461 (11)	0.0491 (4)
C7	0.4908 (2)	0.76025 (14)	0.33341 (10)	0.0397 (3)
C8	0.5788 (2)	0.85031 (13)	0.28769 (8)	0.0320 (3)
C9	0.7321 (2)	0.80773 (13)	0.26981 (9)	0.0349 (3)
C10	0.8268 (2)	0.90613 (13)	0.24078 (9)	0.0354 (3)

C11	0.8944 (2)	0.9763 (2)	0.29332 (11)	0.0443 (4)
C12	0.9798 (2)	1.0694 (2)	0.26900 (12)	0.0495 (4)
N13	1.0007 (2)	1.09721 (13)	0.19693 (10)	0.0490 (4)
C14	0.9521 (2)	1.0614 (2)	0.06480 (13)	0.0564 (5)
C15	0.8899 (3)	0.9985 (2)	0.00826 (12)	0.0601 (6)
C16	0.8067 (2)	0.8995 (2)	0.02591 (10)	0.0463 (4)
C17	0.7868 (2)	0.86631 (15)	0.10045 (9)	0.0389 (3)
C18	0.8479 (2)	0.93167 (14)	0.16128 (10)	0.0363 (3)
C19	0.9344 (2)	1.02994 (15)	0.14293 (11)	0.0427 (4)
O20	0.7504 (2)	0.84474 (14)	-0.03643 (7)	0.0590 (4)
C21	0.6583 (3)	0.7478 (2)	-0.02329 (12)	0.0647 (6)
O22	0.78629 (13)	0.76352 (11)	0.34025 (7)	0.0469 (3)
C26	0.2602 (2)	0.9395 (2)	0.37587 (11)	0.0467 (4)
O27	0.2900 (2)	1.03828 (15)	0.39006 (10)	0.0808 (5)
O28	0.2032 (2)	0.86660 (15)	0.42692 (9)	0.0685 (4)
C29	0.1717 (3)	0.9094 (3)	0.50414 (13)	0.0688 (7)
C30	0.0219 (3)	0.9510 (3)	0.5082 (2)	0.0787 (8)

Table 2. Selected geometric parameters (Å, °)

N1—C2	1.472 (2)	C11—C12	1.398 (3)
N1—C8	1.475 (2)	C12—N13	1.310 (3)
N1—C6	1.476 (2)	N13—C19	1.365 (2)
C2—C3	1.552 (2)	C14—C15	1.351 (3)
C3—C26	1.509 (3)	C14—C19	1.418 (3)
C3—C4	1.531 (2)	C15—C16	1.410 (3)
C4—C5	1.529 (3)	C16—O20	1.360 (2)
C4—C7	1.531 (2)	C16—C17	1.365 (2)
C5—C6	1.535 (3)	C17—C18	1.418 (2)
C7—C8	1.541 (2)	C18—C19	1.422 (2)
C8—C9	1.542 (2)	O20—C21	1.424 (3)
C9—O22	1.421 (2)	C26—O27	1.193 (2)
C9—C10	1.520 (2)	C26—O28	1.331 (2)
C10—C11	1.372 (2)	O28—C29	1.462 (3)
C10—C18	1.429 (2)	C29—C30	1.477 (4)
C2—N1—C8	107.51 (13)	C10—C11—C12	120.5 (2)
C2—N1—C6	108.04 (14)	N13—C12—C11	124.2 (2)
C8—N1—C6	110.42 (13)	C12—N13—C19	117.08 (15)
N1—C2—C3	112.20 (14)	C15—C14—C19	120.9 (2)
C26—C3—C4	113.95 (14)	C14—C15—C16	120.5 (2)
C26—C3—C2	113.22 (15)	O20—C16—C17	125.3 (2)
C4—C3—C2	107.64 (13)	O20—C16—C15	114.2 (2)
C5—C4—C7	108.55 (14)	C17—C16—C15	120.4 (2)
C5—C4—C3	107.0 (2)	C16—C17—C18	120.6 (2)
C7—C4—C3	109.90 (13)	C17—C18—C19	118.6 (2)
C4—C5—C6	108.01 (14)	C17—C18—C10	124.08 (15)
N1—C6—C5	112.28 (14)	C19—C18—C10	117.3 (2)
C4—C7—C8	108.88 (13)	N13—C19—C14	117.7 (2)
N1—C8—C7	110.75 (12)	N13—C19—C18	123.4 (2)
N1—C8—C9	112.28 (12)	C14—C19—C18	118.9 (2)
C7—C8—C9	112.67 (12)	C16—O20—C21	117.72 (14)
O22—C9—C10	110.22 (13)	O27—C26—O28	123.5 (2)
O22—C9—C8	105.58 (12)	O27—C26—C3	124.9 (2)
C10—C9—C8	111.74 (12)	O28—C26—C3	111.6 (2)
C11—C10—C18	117.6 (2)	C26—O28—C29	118.9 (2)
C11—C10—C9	118.75 (15)	O28—C29—C30	110.1 (2)
C18—C10—C9	123.68 (14)		

Data collection: CAD-4 diffractometer software. Cell refinement: CAD-4 diffractometer software. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *SHELXL93*; *PARST* (Nardelli, 1983).

The authors are grateful to Professor Alina Suszko-Purzycka and Dr Emilia Piotrowska for providing us with crystals of the title compound and to Regional Laboratory of Physicochemical Analysis and Structure Research for making the diffractometer available. This work was supported by grant WCH1/9, and in part by the Polish State Committee for Science Research (KBN) under project No. 226229102.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: AB1208). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3-(4-Pyridyl)-5,6,7,8-tetrahydro-9H-1,2,4-triazolo[4,3-a][1,3]diazepine Monohydrate

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(Received 7 February 1994; accepted 7 June 1994)

Abstract

The 1,2,4-triazole ring is approximately planar while the saturated 1,3-diazepine ring adopts a twist-chair conformation. The three intermolecular hydrogen bonds form a complicated network consisting of racemic dimers spanned by water molecules.

Comment

Interest in the structure of diazepine rings arises from their presence in many psychoactive benzo-diazepines, some of them also having a fused five-