

Structure of 9-Acetylquitenine Ethyl Ester,  $C_{23}H_{28}N_2O_5$ 

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**Abstract.**  $M_r = 412$ , orthorhombic,  $P2_12_12_1$ ,  $a = 11.469$  (3),  $b = 18.575$  (6),  $c = 10.142$  (2) Å,  $V = 2160.6$  Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.27$  Mg m<sup>-3</sup>, Mo  $K\alpha$ ,  $\lambda = 0.71069$  Å,  $\mu = 0.053$  mm<sup>-1</sup>,  $F(000) = 880$ ,  $T = 293$  K,  $R = 0.0376$ , 2094 observed reflections. The conformation of the molecule is *trans* with respect to the C(8)–C(9) bond whereas *Cinchona* alkaloids and their derivatives having a non-esterified group have *gauche* conformations. Potential-energy calculations indicate that the minimum-energy conformation is *trans* and that the difference in energy between *trans* and *gauche* conformers is small. The *gauche* conformation enables N(1) of the quinuclidine group to act as an acceptor for a hydrogen bond.

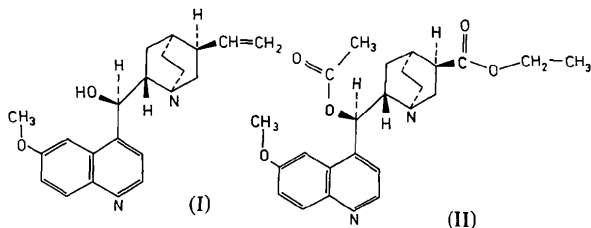
**Introduction.** In the course of a systematic study on structure–activity relationship in antimalarials related to *Cinchona* alkaloids it seems important to elucidate the influence of certain substituents on the antimalarial activity of these drugs. In particular, the activity of quinine (I) is very sensitive to some changes at C(9) and C(3). It can be completely destroyed by acylation of the carbinol group, C(9)–OH, and also by the oxidative cleavage of the vinyl group at C(3) to the carboxylic acid (quitenine), though the ester of this acid exhibits some activity (Henry, 1949). In this connection, it is interesting to compare the molecular structure of the

9-acetylquitenine ethyl ester [AQEE, (II)] with that of quinine (I) in order to correlate the changes in activity with the changes in the molecular geometry.

**Experimental.** AQEE was obtained by A. Suszko-Purzycka and E. Piotrowska, who also provided us with the crystals. From the measurements on a two-circle optical goniometer the crystals were classified as having point symmetry *mmm*, with  $a:b:c = 0.617:1:0.546$ ; they were colourless tablets with best developed faces belonging to the forms {010}, {110}, and {011}. Weissenberg photographs indicated the orthorhombic space group  $P2_12_12_1$ . The unit-cell parameters were determined and refined by least squares from the  $\theta$  angles measured with a CAD-4 diffractometer for 15 reflections. The details of the intensity measurement are given in Table 1.

Table 1. Conditions of data collection, structure determination and refinement

Crystal size (mm)	0.10 × 0.35 × 0.45
Monochromator	Graphite crystal
Diffractometer	Enraf–Nonius CAD 4
$2\theta$ range for measuring lattice parameters	$19^\circ < 2\theta < 50^\circ$
$2\theta$ range for intensity measurement	$2^\circ < 2\theta < 50^\circ$
Range of indices	$0 < h < 13, -22 < k < 0, 0 < l < 12$
Scan width ( $^\circ$ )	$0.75 + 0.42 \tan \theta$
Scan mode	$\omega/2\theta$
Standard reflections for intensity control	182, 291 (every 96 reflections)
Changes in their intensity (%)	3.6
Number of unique reflections measured	2094
Number of reflections in refinement	1904
with $ F_o  > 3\sigma(F_o)$	
Corrections applied	Lorentz, polarization
Parameters refined	384
for non-hydrogen atoms	positional and anisotropic thermal
for hydrogen atoms	positional and isotropic thermal
Weights	$w = 1/\sigma^2(F_o)$ ; $\sigma(F_o)$ based on counting statistics
Ratio of average and maximum $d/\sigma$	
for non-hydrogen atoms	0.034 0.19, $y[C(30)]$
for hydrogen atoms	0.045 0.45, $x[H(30A)]$
Maximum and minimum height in final $\Delta F$ synthesis (e Å <sup>-3</sup> )	0.014 –0.15
Final $R$	0.037
Final $wR$	0.031



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The structure was solved by direct methods using the *MULTAN77* system of programs (Main, Lessinger, Woolfson, Germain & Declercq, 1977). Full-matrix least-squares refinement (on  $F$ ) of positions and isotropic temperature factors gave  $R = 0.16$ , which decreased to 0.095 after anisotropic refinement. H atoms, which were found on successive difference Fourier maps, were gradually introduced to the refinement which was carried out in two, partially overlapping, structural fragments;  $R = 0.05$ . In the last few cycles 17 reflections, which seemed to be influenced by the extinction effect, were suppressed from further refinement which was terminated when the shifts of most coordinates for non-hydrogen atoms were less than 0.1 of their e.s.d.'s and  $R = 0.037$ . The refinement was performed with *SHELX76* (Sheldrick, 1976) using neutral-atom scattering factors. For geometrical calculations *XANADU* (Roberts & Sheldrick, 1972) and *ORTEP* (Johnson, 1965) were used. The calculations were carried out on an IBM 370/158 computer at the Computing Center of the University of Liège and on a CYBER 72 computer at the Regional Computing Center, Kraków.

**Discussion.** The final atomic coordinates together with equivalent isotropic  $U$  values are given in Table 2.\* The molecule is depicted in Fig. 1, in which the numbering of the atoms is also shown. Bond lengths and angles are listed in Table 3.

The enantiomorph was chosen so that the absolute configuration at C(9), C(8), and C(3) (namely *R*, *S* and *R*), is the same as for quinine.

The bond lengths and angles in the quinoline and quinuclidine moieties are very similar (within  $3\sigma$ ) to the corresponding values in a quinine (I) derivative, 10-hydroxy-10-methylidihydroquinine (Suszko-Purzycka, Lipińska, Piotrowska & Oleksyn, 1983) and in cinchonidine (Oleksyn, 1982) as well as in quinidine base (Kashino & Haisa, 1983) and quinidine sulphate (Karle & Karle, 1981).

#### Quinoline geometry

The quinoline ring is planar within experimental error, the largest deviation from the least-squares plane being that of C(19) ( $-0.017 \text{ \AA}$ ). The bending of the C(16)–O(20) bond, which is revealed by the angles C(15)–C(16)–O(20)  $114.4(2)$  and C(17)–C(16)–O(20)  $125.5(3)^\circ$ , corresponds well to that observed and explained by Colapietro & Domenicano (1978) in *p*-methoxybenzoic acid ( $115.5$  and  $124.3^\circ$ ). The torsion angles around the bond O(20)–C(16) (Table 3)

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, and bond lengths and angles involving H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39895 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Fractional atomic coordinates ( $\times 10^4$ ) for non-hydrogen atoms and equivalent isotropic thermal parameters ( $\text{\AA}^2 \times 10^4$ ) with e.s.d.'s in parentheses

	$x$	$y$	$z$	$U_{eq}^*$
N(1)	875 (2)	2078 (1)	8066 (2)	407 (12)
C(2)	189 (3)	1514 (1)	8733 (3)	501 (19)
C(3)	775 (2)	1237 (1)	10006 (2)	448 (15)
C(4)	2013 (2)	1558 (1)	10032 (3)	437 (16)
C(5)	2612 (3)	1346 (2)	8734 (3)	584 (19)
C(6)	1989 (3)	1756 (2)	7627 (3)	510 (19)
C(7)	1950 (2)	2375 (1)	10109 (2)	414 (13)
C(8)	1081 (2)	2645 (1)	9055 (2)	345 (13)
C(9)	1464 (2)	3346 (1)	8409 (2)	356 (13)
C(10)	544 (2)	3685 (1)	7545 (2)	357 (14)
C(11)	-359 (2)	4057 (1)	8107 (3)	482 (17)
C(12)	-1206 (2)	4397 (1)	7328 (3)	562 (20)
N(13)	-1206 (2)	4388 (1)	6032 (2)	548 (16)
C(14)	-287 (3)	4019 (2)	4043 (3)	584 (20)
C(15)	551 (3)	3668 (2)	3377 (3)	630 (21)
C(16)	1442 (3)	3301 (1)	4065 (2)	537 (19)
C(17)	1446 (2)	3293 (1)	5416 (2)	423 (16)
C(18)	575 (2)	3652 (1)	6137 (2)	369 (13)
C(19)	-321 (2)	4017 (1)	5435 (2)	441 (16)
O(20)	2252 (2)	2984 (1)	3279 (2)	778 (16)
C(21)	3153 (3)	2579 (2)	3866 (2)	794 (30)
O(22)	1700 (1)	3815 (1)	9528 (1)	437 (10)
C(23)	2433 (2)	4369 (1)	9303 (3)	478 (17)
O(24)	2814 (2)	4505 (1)	8247 (2)	735 (15)
C(25)	2675 (5)	4754 (2)	10564 (4)	775 (28)
C(26)	152 (2)	1445 (1)	11256 (3)	487 (17)
O(27)	-580 (2)	1904 (1)	11376 (2)	666 (14)
O(28)	562 (2)	1057 (1)	12265 (2)	603 (13)
C(29)	56 (3)	1207 (2)	13550 (3)	735 (30)
C(30)	488 (5)	630 (3)	14449 (4)	1018 (49)

$$* U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33}).$$

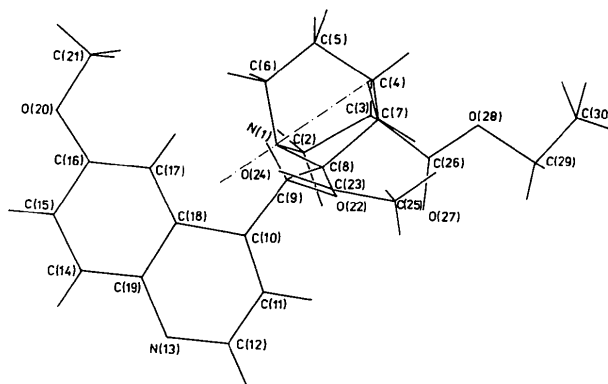


Fig. 1. The molecule of AQEE projected on the plane of the quinoline moiety.

show that the bonds C(21)–O(20) and C(15)–C(16) are in mutual *trans* positions, which was found in all similar compounds mentioned above except for the quinidine base, where this position is *cis* (Kashino & Haisa, 1983).

#### Quinuclidine geometry

The conformation of the quinuclidine moiety is skewed as in other cases as shown by torsion angles around the line N(1)···C(4) (Table 3). The quinuclidine ring has no exact mirror symmetry as does that of the quinuclidinium cation described by Kurahashi, Engel & Nowacki (1980), but the C–C bond lengths in both

Table 3. Bond lengths (Å), bond angles (°) and selected torsion angles (°) involving non-hydrogen atoms with *e.s.d.*'s in parentheses

N(1)–C(2)	1.474 (3)	C(12)–N(13)	1.315 (4)
N(1)–C(6)	1.479 (4)	N(13)–C(19)	1.367 (3)
N(1)–C(8)	1.473 (3)	C(14)–C(15)	1.344 (4)
C(2)–C(3)	1.543 (4)	C(14)–C(19)	1.413 (4)
C(3)–C(4)	1.540 (4)	C(15)–C(16)	1.413 (4)
C(3)–C(26)	1.506 (4)	C(16)–C(17)	1.370 (3)
C(4)–C(5)	1.535 (4)	C(16)–O(20)	1.358 (3)
C(4)–C(7)	1.522 (4)	C(17)–C(18)	1.406 (3)
C(5)–C(6)	1.534 (4)	C(18)–C(19)	1.422 (3)
C(7)–C(8)	1.545 (3)	O(20)–C(21)	1.411 (5)
C(8)–C(9)	1.522 (3)	O(22)–C(23)	1.349 (3)
C(9)–C(10)	1.510 (3)	C(23)–O(24)	1.184 (3)
C(9)–O(22)	1.456 (3)	C(23)–C(25)	1.492 (5)
C(10)–C(11)	1.370 (3)	C(26)–O(27)	1.203 (3)
C(10)–C(18)	1.429 (3)	C(26)–O(28)	1.338 (3)
C(11)–C(12)	1.403 (4)	O(28)–C(29)	1.454 (4)
		C(29)–C(30)	1.491 (7)
C(2)–N(1)–C(6)	108.2 (2)	C(12)–N(13)–C(19)	116.7 (2)
C(2)–N(1)–C(8)	106.3 (2)	C(15)–C(14)–C(19)	121.4 (3)
C(6)–N(1)–C(8)	110.8 (2)	C(14)–C(15)–C(16)	120.2 (3)
C(2)–C(3)–C(4)	106.6 (2)	C(15)–C(16)–C(17)	120.1 (3)
C(2)–C(3)–C(26)	114.4 (2)	C(15)–C(16)–O(20)	114.4 (2)
C(4)–C(3)–C(26)	108.9 (2)	C(17)–C(16)–O(20)	125.5 (3)
C(3)–C(4)–C(5)	107.4 (2)	C(16)–C(17)–C(18)	120.8 (2)
C(3)–C(4)–C(7)	110.0 (2)	C(10)–C(18)–C(17)	123.9 (2)
C(5)–C(4)–C(7)	108.7 (2)	C(10)–C(18)–C(19)	117.5 (2)
C(4)–C(5)–C(6)	107.0 (2)	C(17)–C(18)–C(19)	118.6 (2)
N(1)–C(6)–C(5)	112.5 (2)	N(13)–C(19)–C(14)	117.5 (2)
C(4)–C(7)–C(8)	108.6 (2)	N(13)–C(19)–C(18)	123.7 (2)
N(1)–C(8)–C(7)	110.1 (2)	C(14)–C(19)–C(18)	118.8 (2)
N(1)–C(8)–C(9)	111.4 (2)	C(16)–O(20)–C(21)	119.0 (2)
C(7)–C(8)–C(9)	112.9 (2)	C(9)–O(22)–C(23)	116.1 (2)
C(8)–C(9)–C(10)	113.9 (2)	O(22)–C(23)–O(24)	123.1 (2)
C(8)–C(9)–O(22)	103.3 (2)	O(22)–C(23)–C(25)	109.7 (3)
C(10)–C(9)–O(22)	109.5 (2)	O(24)–C(23)–C(25)	127.2 (3)
C(9)–C(10)–C(11)	119.8 (1)	C(3)–C(26)–O(27)	126.7 (3)
C(9)–C(10)–C(18)	123.0 (2)	C(3)–C(26)–O(28)	109.8 (2)
C(11)–C(10)–C(18)	117.1 (2)	O(27)–C(26)–O(28)	123.4 (3)
C(10)–C(11)–C(12)	121.1 (2)	C(26)–O(28)–C(29)	116.2 (2)
C(11)–C(12)–N(13)	123.9 (3)	O(28)–C(29)–C(30)	106.1 (3)
C(15)–C(16)–O(20)–C(21)	–177.4 (3)		
C(17)–C(16)–O(20)–C(21)	3.3 (3)		
C(2)–N(1)–C(4)–C(3)	–5.8 (3)		
C(2)–N(1)–C(4)–C(5)	112.0 (3)		
C(2)–N(1)–C(4)–C(7)	–127.6 (3)		
C(6)–N(1)–C(4)–C(5)	–8.0 (3)		
C(6)–N(1)–C(4)–C(7)	112.4 (3)		
C(6)–N(1)–C(4)–C(3)	–125.9 (3)		
C(8)–N(1)–C(4)–C(7)	–10.5 (3)		
C(8)–N(1)–C(4)–C(3)	111.3 (3)		
C(8)–N(1)–C(4)–C(5)	–130.9 (3)		

O(28), and C(29) lie in a plane which is tilted by 15.5° from that of the quinoline ring.

#### Overall shape of the molecule

If we consider the mutual positions of the atoms N(1) and O(22), the overall geometry of the AQEE molecule may be described as a *trans* conformation with respect to the C(9)–C(8) bond, while all *erythro* epimers of *Cinchona* alkaloids or their derivatives studied hitherto in the crystalline state are *gauche* conformers (Carter, McPhail & Sim, 1967; Oleksyn, Stadnicka & Hodorowicz, 1978; Oleksyn, Lebioda & Ciechanowicz-Rutkowska, 1979; Oleksyn, 1982; Karle & Karle, 1981; Suszko-Purzycka, Lipińska, Piotrowska & Oleksyn, 1983; Kashino & Haisa, 1983). In order to explain, at least qualitatively, this difference in conformational behaviour, we calculated the potential energy of the AQEE molecule as a function of three torsion angles,  $\tau_1 = C(11)–C(10)–C(9)–O(22)$ ,  $\tau_2 = O(22)–C(9)–C(8)–C(7)$ , and  $\tau_3 = C(10)–C(9)–O(22)–C(23)$ , using the atom–atom potential approximation (program *EENY* written by Motherwell, 1974). As shown in Table 4, the energy maps revealed five minima, one of which corresponds approximately to the *trans* conformation observed for AQEE.

In the same table the observed and calculated values of  $\tau_1$  and  $\tau_2$  in some *Cinchona* alkaloids are given for comparison. For all these compounds the energetically preferred conformation is *trans*, although the energy differences between the *trans* conformers and those *gauche* conformers which are the most similar to those observed in crystals are rather small. On the other hand, the similarity of the calculated values of  $\tau_1$  and  $\tau_2$  for the alkaloids without such bulky substituents at C(9) and C(3) as those in AQEE shows that the substituents themselves seem not to have an important influence on the conformational behaviour. Hence, the most probable factor which stabilizes the *gauche* conformation observed in the *Cinchona* alkaloids should be connected with packing conditions. In fact, these conditions for AQEE are different from those for such *Cinchona* alkaloids as cinchonidine (Oleksyn, 1982), cinchonine (Oleksyn, Lebioda & Ciechanowicz-Rutkowska, 1979; Oleksyn, Stadnicka & Hodorowicz, 1978), quinine (Suszko-Purzycka, Lipińska, Piotrowska & Oleksyn, 1983), and quinidine (Karle & Karle 1981; Carter, McPhail & Sim, 1967; Kashino & Haisa, 1983), all four occurring as *gauche* conformers. The packing in AQEE, whose molecule has no potential donor for hydrogen bonds, is determined only by van der Waals interactions (Fig. 2), while in the alkaloids which are not substituted at C(9) intermolecular hydrogen bonds, O–H...A, where O is the oxygen atom of the carbinol group, are formed. The acceptor, A, is either an N atom of an adjacent molecule or an O atom of some other co-crystallizing molecule. Formation of such a bond seems to favour the *gauche*

cases compare well. The N–C bonds in the quinuclidinium cation are longer by 0.03 Å than those observed in AQEE, which was noted previously as an effect of the protonation of N(1) (Oleksyn, Lebioda, Ciechanowicz-Rutkowska & Story, 1977). The non-bonded C(4)...N(1) distance in AQEE [2.571 (4) Å] is longer than that in the quinuclidinium cation [2.534 (8) Å], but is in very good agreement with the value found in 10-hydroxy-10-methyldihydroquinine (Suszko-Purzycka, Lipińska, Piotrowska & Oleksyn, 1983).

#### Ester substituents

Both acetyl and ethyl ester groups have normal geometry as compared with other esters investigated recently (*e.g.* Drew, Vickery & Willey, 1982). The first is planar and forms an angle of 94.0° with the plane of the quinoline ring. In the other, the atoms C(26), O(27),

Table 4. Comparison of torsion angles [ $\tau_1 = \text{C}(11) - \text{C}(10) - \text{C}(9) - \text{O}(22)$ ,  $\tau_2 = \text{O}(22) - \text{C}(9) - \text{C}(8) - \text{C}(7)$ , and  $\tau_3 = \text{C}(10) - \text{C}(9) - \text{O}(22) - \text{C}(23)$ ] observed and calculated for Cinchona alkaloids and their derivatives

Compound; absolute configuration at C(8), C(9)	$\tau_1$ ( $^\circ$ )	$\tau_2$ ( $^\circ$ )	$\tau_3$ ( $^\circ$ )	Potential energy (kJ) referred to global minimum	Conformation with respect to bond C(8)–C(9)
AQEE: <i>S,R</i> in crystal (a)	-37.1	-51.8	-81.3		<i>trans</i>
	-50.2	-76.9	-70.3	0.0	<i>trans</i>
	-35.1	45.6	-70.2	5.9	<i>gauche</i>
calculated (c)	126.2	-100.9	-154.7	8.4	<i>trans</i>
	-51.0	-96.9	-154.0	10.5	<i>trans</i>
	-15.9	145.1	-159.9	51	<i>gauche</i>
Quinine: <i>S,R</i> in crystal (b)	-21.2	55.8	—		<i>gauche</i>
	-66.8	-68.6	—	0.0	<i>trans</i>
calculated (c)	130.9	-57.0	—	0.4	<i>trans</i>
	-27.5	40.4	—	12	<i>gauche</i>
Cinchonidine: <i>S,R</i> in crystal (d)	-22.6	48.0	—		<i>gauche</i>
	-68.9	-69.0	—	0.0	<i>trans</i>
calculated (c)	130.9	-60.7	—	0.8	<i>trans</i>
	-27.9	40.6	—	11	<i>gauche</i>
Quinidine: <i>R,S</i> (e)	17.8	-73.8	—		<i>gauche</i>
in crystals (f)	15	-62	—		<i>gauche</i>
	21	-42	—		<i>gauche</i>
(g)	23	-50.3	—		<i>gauche</i>
	57.2	64.5	—	0.0	<i>trans</i>
calculated (c)	-133.4	60.0	—	1.7	<i>trans</i>
	25.5	-44.0	—	5.9	<i>gauche</i>
	29.5	-135.6	—	19	<i>gauche</i>
Cinchonine: <i>R,S</i> in crystal (h)	23.4	-52.2	—		<i>gauche</i>
	70.4	67.0	—	0.0	<i>trans</i>
calculated (c)	-133.0	58.3	—	2.9	<i>trans</i>
	29.0	-43.4	—	6.3	<i>gauche</i>
	29.8	-136.2	—	28	<i>gauche</i>

Notes: (a) this work; (b) in the form of 10-hydroxy-10-methylidihydroquinine (Suszko-Purzycka, Lipińska, Piotrowska & Oleksyn, 1983); (c) the calculated torsion angles, except those from (a), are taken from Oleksyn & Lebioda (1980) with corrections applied to these values of  $\tau_1$  which were previously erroneous; (d) Oleksyn (1982); (e) Carter, McPhail & Sim (1967); (f) Karle & Karle (1981); (g) Kashino & Haisa (1983); (h) Oleksyn, Lebioda & Ciechanowicz-Rutkowska (1979).

conformation in which the lone pair of electrons at N(1) is available for the H atom of a donor molecule. In contrast, in the *trans* conformer of AQEE the lone pair at N(1) is directed towards the quinoline ring and is almost perpendicular ( $92^\circ$ ) to its  $\pi$  orbitals; thus, any donor group approaching N(1) along the direction of its lone pair would have to encounter the  $\pi$ -electron cloud on its way, as shown in Fig. 1.

The above considerations lead to a plausible conclusion that whenever a molecule of a Cinchona alkaloid or its derivative is able to form an intermolecular hydrogen bond it will occur in the crystalline state as the *gauche* rather than the *trans* conformer. This behaviour may be especially important in the case of interactions of these molecules with structure-specific biological receptors.

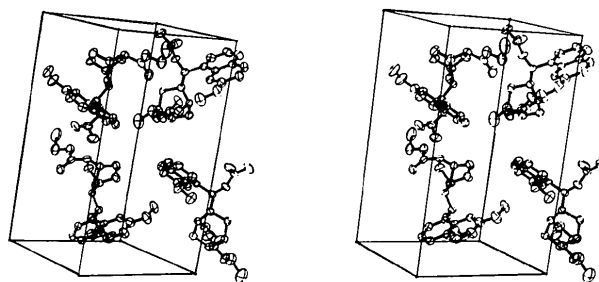


Fig. 2. Stereoview of the packing.

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