Financial support from Jacob Wallenbergs Forskningsstiftelse is gratefully acknowledged.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1212). 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. (1996). C**52**, 3121–3124

Quitenidinium Ethyl Ester Ethyl Sulfate

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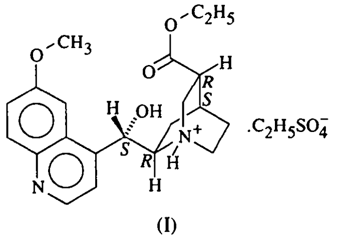
(*Received 5 March 1996; accepted 15 July 1996*)

Abstract

The crystal structure of quitenidinium ethyl ester ethyl sulfate, $C_{21}H_{27}N_2O_4^+.C_2H_5SO_4^-$ has been determined. The conformation of the cation is open in contrast to the closed conformation of the non-protonated quitenine ethyl ester. The molecules are linked by intermolecular O—H···N hydrogen bonds into chains along the *b* axis. A bifurcated hydrogen bond is present between the protonated quinuclidine N and two O atoms belonging to the ethyl sulfate anion.

Comment

The title compound, QtdEE$^+$.ES$^-$ (QtdEE = quitenidinium ethyl ester; ES = ethyl sulfate), (I), is a salt which consists of an esterified quitenidine cation and an ethyl sulfate anion. Quitenidine is a derivative of the anti-arrhythmic and antimalarial alkaloid, quinidine, in which the vinyl group at C3 of the quinuclidine moiety is oxidized to carboxyl. The positive charge of QtdEE$^+$ is due to the protonated N atom, N1, of quinuclidine.



(I)

The aim of the X-ray structure analysis of QtdEE$^+$.ES$^-$ is to determine the conformation of the cation and the mode of its interaction with the anion since these two properties are important in recognition of quinidine and its derivatives by their biological receptors. It is also interesting to compare the structures of this protonated quitenidine ester and its non-protonated quitenine analogue (QtEE) reported recently (Lewinski, Nitek, Oleksyn & Stec, 1995) in order to check the effect of protonation on the geometry of the alkaloid molecules and their ability to form intermolecular contacts. QtdEE and QtEE are diastereoisomers and differ in the absolute configuration of C8 and C9 which is *R*,*S* for QtdEE and *S*,*R* for QtEE.

The bond lengths in QtdEE$^+$ (Table 2) are comparable to within 3σ with those in QtEE with the exception of the bonds between N1 and the C atoms of quinuclidine. The significant elongation of these bonds in comparison to those in QtEE may be ascribed to the influence of the N1 protonation and was previously observed in quininium salts (Oleksyn, Sliwinski, Kowalik & Serda, 1991).

The values of bond angles in the cation are also close to those in QtEE except for the angles in the ethyl ester group (C26—O28—C29, O28—C29—C30) and the angles of the type C—N1—C and C—C—N1 in the quinuclidine moiety. The differences in the angles within the ester group are only slightly greater than 3σ and may result from packing considerations. The angles in the vicinity of the N1 atom change with its protonation according to the rule described by Oleksyn & Serda (1993) for quinine and its salts. The value of pyramidality, $P = \{[360-\Sigma(\mathrm{C—N1—C})]/(360-3\times109.47)\}\times100\%$ (Häfelinger & Mack, 1994) is 92.3% and indicates slight flattening of the approximately trigonal pyramid formed by N1 in the apex and C2, C6, C8 in the base corners. In the free base quinine (Pniewska & Suszko-Purzycka, 1989) where, in contrast with the quininium cation, the lone electron pair is not occupied by the proton, P = 110%. This is in agreement with the values observed for other free base cinchona alkaloids, *e.g.* cinchonidine, cinchonine and quinidine (P = 106–110%), while for most of their salts P varies in the range of 90–95%.

The overall shape of the cation (Fig. 1) is best described by the torsion angles around the C9—C10 and C9—C8 bonds [τ_1 (C11—C10—C9—O22) = 15.0 (3) and τ_2 (O22—C9—C8—N1) = 67.6 (2)°]. These angles indicate the so-called open (*gauche–gauche*) conformation which is different from the closed one (*gauche–trans*) observed in QtEE where τ_1 and τ_2 are −32.2 (2) and −173.5 (1)°, respectively. Thus, the protonation of N1 results in the change of the QtdEE$^+$ conformation in comparison to that of QtEE. This is consistent with our previous observation that the engagement of N1 in intermolecular hydrogen bonding favors the open conformation (Lewinski, Nitek, Oleksyn & Stec, 1995).

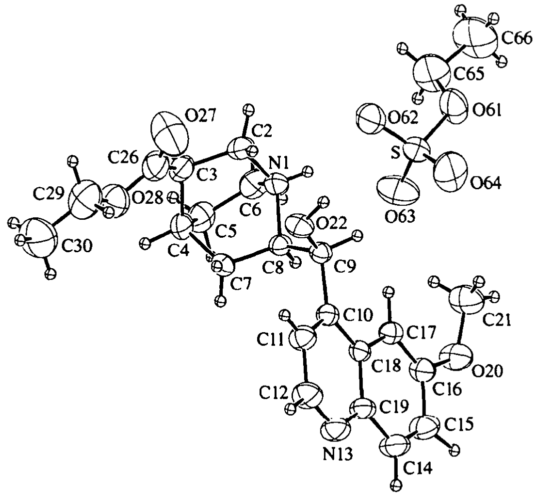


Fig. 1. A *ZORTEP* (Zsolnai, 1995) projection of the quitenidinium ethyl ester cation and the ethyl sulfate anion with atom numbering. Displacement ellipsoids are shown at the 50% probability level.

The conformation of the quinuclidine moiety, expressed by the torsion angles C—C—N1—C and N1—C—C—C is similar to that in the quinidinium cation in other crystal structures. Thus, the bulky ester group does not additionally deform the quinidine skeleton.

The quinoline moiety has a slightly concave shape, with the methoxyl substituent twisted from the mean plane of the ring [C17—C16—O20—C21 = 12.1 (4)°].

The conformation of the ester group at C3 differs from that in QtEE, the torsion angle C26—O28—C29—C30 being 175.4 (3)° while in QtEE it is 92.7 (3)°. The approximately planar group defined by the atoms C3, C26, O27, O28 and C29 forms an angle of 56.34 (8)° with the least-squares plane of the quinoline moiety. This angle is much larger than in the case of QtEE [27.08 (2)°], most probably for packing reasons.

In the ethyl sulfate anion, S—O61 is much longer than the three other S—O bonds because of the O61 linkage with the ethyl group. In consequence, the flattening of the pyramid (within the distorted tetrahedron SO_4) with S in the apex and O62, O63, O64 forming the base, is significant (P = 74%) which also suggests a certain excess of positive charge on O61 in comparison with the other O atoms. The negative charge seems to be localized in the vicinity of O63, the pyramidality of the group S, O64, O62, O61 being 117.4%.

The packing (Fig. 2) is dominated by intermolecular hydrogen bonds: O22—H221 = 0.87 (3), O22···N13^{i} = 2.708 (3), N13^{i}···H221 = 1.88 (3) Å, O22—H221···N13^{i} = 157 (3)° [(i) = $1-x, y+0.5, 0.5-z$]; N1—H11 = 0.83 (3), N1···O62 = 2.821 (3), O62···H11 = 2.05 (3), N1—H11···O62 = 155 (3)°; N1···O63 = 3.174 (3), O63···H11 = 2.47 (3) Å, N1—H11···O63 = 143 (3)°. The first of these links the carbinol group of one molecule with the quinoline N atom of another, so that chains in the *b*-axis direction are formed, very similar to those observed in the structure of QtEE (Lewinski, Nitek, Oleksyn & Stec, 1995). The second and third hydrogen bonds connect the proton of N1 in a bifurcated fashion (Olovsson & Jönsson, 1976) with two O atoms of the ethyl sulfate anion. The pairs of anions occupy the channels along the 2_1 axes parallel to **c** causing an enlargement of their diameters in comparison to those observed in the structure of QtEE. This leads to about a 30% enlargement of the unit-cell volume with some change in the channel-wall structure.

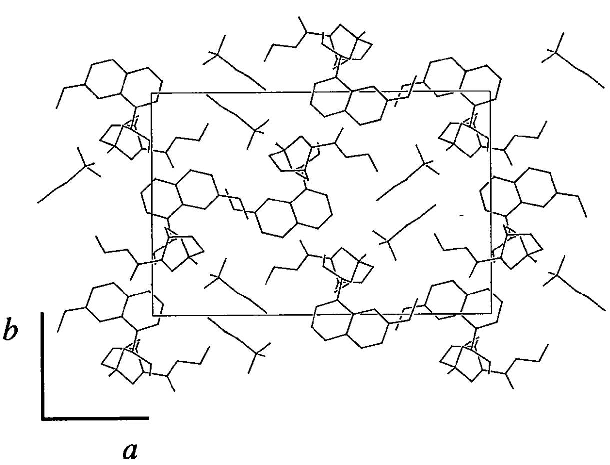


Fig. 2. *ORTEP*II (Johnson, 1976) projection of the unit cell along the *z* axis. The intermolecular hydrogen bonds are shown as dotted lines.

Experimental

Solution of freshly dried quitenidine (2 g) in absolute ethanol (20 cm^3) was treated with concentrated H_2SO_4 (3.2 cm^3) and refluxed for 8 h under a drying tube containing $CaCl_2$. Then water with ice (40 cm^3) was added to the previously cooled reaction mixture. The pH of the stirred resultant solution was adjusted to 7±0.1 (Merck pH-Box) with 10% solution of $NaHCO_3$. The white precipitate was separated, washed with cold water and dried. The crude product was recrystallized from water to yield 2.2 g (80%) of colorless needles, m.p. 490–491 K. Found: C 55.68, H 6.75, N 5.78, S 6.68%; $C_{23}H_{32}N_2O_8S$ requires C 55.61, H 6.50, N 5.64, S 6.45%; IR(KBr): 3200–2800, 2800–2650, 1730, 1620, 1600, 1520, 1475, 1450, 1380, 1270, 1200, 1120, 950, 890, 800, 750, 625, 590 cm^{-1}. The IR spectra is consistent with the protonation of the quinuclidine N atom, N1.

Crystal data

$C_{21}H_{27}N_2O_4^+.C_2H_5SO_4^-$
$M_r = 496.57$
Orthorhombic
$P2_12_12_1$
$a = 21.562\,(3)$ Å
$b = 14.055\,(2)$ Å
$c = 8.0720\,(10)$ Å
$V = 2446.3\,(6)$ Å^3
$Z = 4$
$D_x = 1.348$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 25 reflections
$\theta = 30$–$35°$
$\mu = 1.608$ mm^{-1}
$T = 293\,(2)$ K
Needle
0.3 × 0.1 × 0.1 mm
Colorless

Data collection

Automatic single-crystal KM-4 diffractometer
$\omega/1.3\theta$ scans
Absorption correction: none
4467 measured reflections
3967 independent reflections
3965 observed reflections [$I > 2\sigma(I)$]

$R_{\rm int} = 0.0451$
$\theta_{\rm max} = 75.16°$
$h = -26 \rightarrow 26$
$k = 0 \rightarrow 17$
$l = 0 \rightarrow 10$
3 standard reflections monitored every 40 reflections intensity decay: 2.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0356$
$wR(F^2) = 0.0984$
$S = 1.060$
3967 reflections
400 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0646P)^2 + 0.4119P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.302$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.212$ e Å^{-3}

Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0029 (3)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983)
Flack parameter = 0.00 (2)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

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

	x	y	z	$U_{\rm eq}$
N1	0.60862 (9)	0.23825 (13)	−0.1777 (2)	0.0365 (4)
C2	0.55870 (13)	0.3009 (2)	−0.2459 (3)	0.0438 (5)
C3	0.52773 (12)	0.2506 (2)	−0.3928 (3)	0.0437 (5)
C4	0.54890 (13)	0.1462 (2)	−0.3946 (3)	0.0426 (5)
C5	0.61817 (14)	0.1443 (2)	−0.4326 (3)	0.0523 (7)
C6	0.65224 (13)	0.2125 (2)	−0.3154 (3)	0.0490 (6)
C7	0.53828 (12)	0.10316 (15)	−0.2233 (3)	0.0385 (5)
C8	0.58505 (10)	0.14624 (13)	−0.1015 (2)	0.0308 (4)
C9	0.55972 (9)	0.16147 (14)	0.0748 (2)	0.0303 (4)
C10	0.54509 (10)	0.06459 (13)	0.1500 (3)	0.0315 (4)
C11	0.48438 (11)	0.0368 (2)	0.1606 (3)	0.0425 (5)
C12	0.46877 (13)	−0.0519 (2)	0.2264 (4)	0.0510 (6)
N13	0.51007 (10)	−0.11289 (14)	0.2818 (3)	0.0480 (5)
C14	0.61467 (12)	−0.1505 (2)	0.3451 (4)	0.0500 (6)
C15	0.67575 (12)	−0.1282 (2)	0.3505 (4)	0.0499 (6)
C16	0.69649 (11)	−0.03978 (15)	0.2861 (3)	0.0394 (5)
C17	0.65594 (10)	0.02279 (15)	0.2161 (3)	0.0338 (4)
C18	0.59170 (10)	0.00170 (13)	0.2107 (2)	0.0311 (4)
C19	0.57123 (11)	−0.08667 (15)	0.2771 (3)	0.0386 (5)
O20	0.75866 (8)	−0.02504 (12)	0.3065 (3)	0.0573 (5)
C21	0.78182 (13)	0.0674 (2)	0.2815 (5)	0.0640 (8)
O22	0.50525 (8)	0.21681 (11)	0.0668 (2)	0.0420 (4)
C26	0.45876 (14)	0.2684 (2)	−0.3859 (3)	0.0549 (6)
O27	0.43604 (12)	0.3365 (2)	−0.3220 (4)	0.0966 (9)
O28	0.42667 (9)	0.20249 (15)	−0.4661 (3)	0.0629 (5)
C29	0.36005 (15)	0.2155 (3)	−0.4721 (5)	0.0744 (9)
C30	0.3339 (2)	0.1397 (3)	−0.5775 (7)	0.0997 (15)
S	0.69550 (3)	0.32861 (4)	0.16001 (7)	0.0424 (2)
O61	0.75083 (10)	0.3982 (2)	0.1895 (3)	0.0637 (5)
O62	0.65289 (10)	0.37496 (14)	0.0492 (3)	0.0601 (5)
O63	0.71814 (12)	0.24351 (15)	0.0848 (4)	0.0804 (7)
O64	0.67036 (12)	0.3165 (2)	0.3218 (3)	0.0836 (7)
C65	0.7862 (2)	0.4294 (3)	0.0489 (5)	0.0789 (11)
C66	0.8365 (2)	0.4895 (4)	0.1104 (7)	0.106 (2)

Table 2. *Selected geometric parameters (Å, °)*

N1—C2	1.496 (3)	C14—C15	1.354 (4)
N1—C6	1.501 (3)	C14—C19	1.408 (3)
N1—C8	1.519 (3)	C15—C16	1.419 (3)
C2—C3	1.534 (3)	C16—C17	1.363 (3)
C3—C26	1.509 (4)	C16—O20	1.366 (3)
C3—C4	1.536 (3)	C17—C18	1.417 (3)
C4—C5	1.525 (4)	C18—C19	1.423 (3)
C4—C7	1.527 (3)	O20—C21	1.406 (3)
C5—C6	1.534 (4)	C26—O27	1.192 (4)
C7—C8	1.533 (3)	C26—O28	1.325 (4)
C8—C9	1.539 (3)	O28—C29	1.449 (4)
C9—O22	1.410 (3)	C29—C30	1.475 (6)
C9—C10	1.524 (3)	S—O64	1.424 (2)

C10—C11	1.368 (3)	S—O63	1.427 (2)
C10—C18	1.425 (3)	S—O62	1.439 (2)
C11—C12	1.396 (3)	S—O61	1.561 (2)
C12—N13	1.315 (3)	O61—C65	1.435 (4)
N13—C19	1.370 (3)	C65—C66	1.463 (6)
C2—N1—C6	108.7 (2)	C12—N13—C19	117.8 (2)
C2—N1—C8	114.1 (2)	C15—C14—C19	120.8 (2)
C6—N1—C8	107.7 (2)	C14—C15—C16	119.8 (2)
C2—N1—H11	112 (2)	C17—C16—O20	125.6 (2)
C6—N1—H11	106 (2)	C17—C16—C15	121.0 (2)
C8—N1—H11	108 (2)	O20—C16—C15	113.4 (2)
N1—C2—C3	109.1 (2)	C16—C17—C18	120.3 (2)
C26—C3—C2	108.9 (2)	C17—C18—C19	118.3 (2)
C26—C3—C4	116.9 (2)	C17—C18—C10	124.8 (2)
C2—C3—C4	108.6 (2)	C19—C18—C10	116.9 (2)
C5—C4—C7	108.8 (2)	N13—C19—C14	117.3 (2)
C5—C4—C3	108.1 (2)	N13—C19—C18	123.0 (2)
C7—C4—C3	109.0 (2)	C14—C19—C18	119.8 (2)
C4—C5—C6	109.5 (2)	C16—O20—C21	118.1 (2)
N1—C6—C5	107.9 (2)	O27—C26—O28	123.9 (3)
C4—C7—C8	109.0 (2)	O27—C26—C3	123.6 (3)
N1—C8—C7	107.3 (2)	O28—C26—C3	112.4 (2)
N1—C8—C9	112.0 (2)	C26—O28—C29	116.5 (3)
C7—C8—C9	114.5 (2)	O28—C29—C30	107.9 (3)
O22—C9—C10	109.8 (2)	O64—S—O63	114.8 (2)
O22—C9—C8	109.3 (2)	O64—S—O62	112.4 (2)
C10—C9—C8	108.5 (2)	O63—S—O62	109.48 (15)
C11—C10—C18	118.4 (2)	O64—S—O61	103.06 (14)
C11—C10—C9	118.5 (2)	O63—S—O61	109.18 (14)
C18—C10—C9	123.0 (2)	O62—S—O61	107.42 (12)
C10—C11—C12	120.6 (2)	C65—O61—S	118.4 (2)
N13—C12—C11	123.3 (2)	O61—C65—C66	107.6 (4)

Most H atoms were found on difference Fourier maps and refined with individual positional parameters. The positions of those on C21, C29, C30, C65 and C66 were calculated and included as riding atoms in the refinement. Isotropic displacement parameters for H atoms were used in the refinement.

Data collection: *KM*-4 *Software*. Cell refinement: *KM*-4 *Software*. Data reduction: *KM*-4 *Software*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ZORTEP* (Zsolnai, 1995), *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93, *PARST* (Nardelli, 1983).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SK1021). 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. (1996). C**52**, 3124–3126

Columnar Stacking of 1,3-Diphenyl-1,2,4-benzotriazin-4(1*H*)-yl Radicals

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(*Received 2 July 1996; accepted 9 September 1996*)

Abstract

The title radicals, $C_{19}H_{14}N_3$, are arranged in columns along the *b* axis in which the almost planar 1,2,4-triazin-4(1*H*)-yl ring and the phenyl ring on C3 of adjacent molecules lie alternately on top of one another. Within the array, the mean interplanar distance is 3.45 Å. However, the centers of the 1,2,4-triazin-4(1*H*)-yl rings, bearing most of the spin population, are 5.50 Å apart. Consequently, there are only very weak intermolecular interactions along the columns, which is in agreement with the results of static magnetic susceptibility measurements.

Comment

Recent studies of magnetic properties of 1,2,4-benzotriazin-4(1*H*)-yl radicals (Mukai, Inoue, Achiwa, Jamali, Krieger & Neugebauer, 1994) directed our attention towards the crystal structure of 1,3-diphenyl-1,2,4-benzotriazin-4(1*H*)-yl which can be considered to be a basic compound in this group of stable free radicals (Blatter & Lukaszewski, 1968; Blatter, 1969; Neugebauer & Umminger, 1980). Moreover, it is of interest to compare the crystal structure of the title compound, (I), with that of the 1-(4-chlorophenyl)-3-phenyl derivative. This stable radical is packed in columns comprising two kinds of radical pairs and shows an extraordinarily large antiferromagnetic exchange interaction, whereas the susceptibility of the title compound follows the Curie–Weiss law (Mukai *et al.*, 1994).

Acta Crystallographica Section C
ISSN 0108-2701 © 1996