Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-Butylquinine tetrafluoroborate

Sana Eltaief,[a] Pascal Retailleau,[b] Leo Straver[c] and Béchir Ben Hassine[a]*

[a]Laboratoire de Synthèse Organique Asymétrique et Catalyse Homogène (01UR1201), Faculté des Sciences de Monastir, Avenue de l'Environnement, 5019 Monastir, Tunisia, [b]Institut de Chimie des Substances Naturelles-CNRS, 1 Avenue de la Terrasse, 91198 Gif sur Yvette, France, and [c]Chopinrode 8, 2717 BK Zoetermeer, The Netherlands
Correspondence e-mail: bechirbenhassine@yahoo.fr

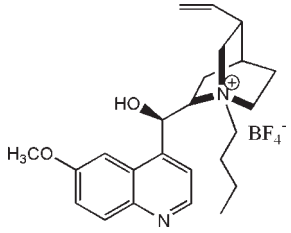
Received 9 December 2009; accepted 21 December 2009

Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.005 Å; disorder in solvent or counterion; R factor = 0.063; wR factor = 0.156; data-to-parameter ratio = 12.1.

In the title salt (2*S*,4*S*,8*R*)-1-butyl-2-[(*R*)-(hydroxy)(6-methoxyquinolin-4-yl)methyl]-8-vinylquinuclidin-1-ium tetrafluoroborate, $C_{24}H_{33}N_2O_2^+\cdot BF_4^-$, the butyl substituent at the 1-position is in an equatorial conformation with respect to the unsubstituted six-membered ring and the four butyl C atoms are almost coplanar with the ring N and vinyl C atoms (r.m.s. deviation = 0.046 Å). In the crystal, the cations are linked by O—H···N hydrogen bonds. The F atoms of the tetrafluoroborate group are disordered over two sets of site with an occupancy raitio of 0.552 (8):0.448 (8).

Related literature

For the crystal structures of similar 1-butylquinine tetrafluoroborate derivatives, see: Dijkstra *et al.* (1989); Samas *et al.* (2005). For applications of quinine salts, see: Thierry *et al.* (2001, 2003). For graph-set notation, see: Bernstein *et al.* (1994). For a description of the Cambridge Structural Database, see: Allen (2002).



Experimental

Crystal data

$C_{24}H_{33}N_2O_2^+\cdot BF_4^-$
M_r = 468.33
Orthorhombic, $P2_12_12_1$
a = 8.041 (8) Å
b = 12.597 (12) Å
c = 22.91 (2) Å
V = 2321 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 120 K
0.60 × 0.20 × 0.15 mm

Data collection

Bruker Kappa-APEX DUO diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.885, T_{max} = 0.982
25415 measured reflections
3968 independent reflections
2957 reflections with $I > 2\sigma(I)$
R_{int} = 0.072

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.156
S = 1.06
3968 reflections
329 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1*O*···N2^{i}	0.84	1.95	2.787 (4)	174

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008) and *CrystalBuilder* (Welter, 2006); molecular graphics: *PLATON* (Spek, 2009) and *Mercury* (Bruno *et al.*, 2004); software used to prepare material for publication: *SHELXL97* and *publCIF* (Westrip, 2010).

The authors thank the DGRSRT (Direction Générale de la Recherche Scientifique et de la Rénovation Technologique) of the Tunisian Ministry of Higher Education and Scientific Research and Technology for financial support of this research.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2256).

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Bernstein, J., Etter, M. C. & Leiserowitz, L. (1994). *Structure Correlation*, edited by H.-B. Bürgi & J. D. Dunitz, Vol. 2, pp. 431–507. New York: VCH.
Bruker (2008). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruno, I. J., Cole, J. C., Kessler, M., Luo, J., Motherwell, W. D. S., Purkis, L. H., Smith, B. R., Taylor, R., Cooper, R. I., Harris, S. E. & Orpen, A. G. (2004). *J. Chem. Inf. Comput. Sci.* **44**, 2133–2144.
Dijkstra, G. D. H., Kellogg, R. M., Wynberg, H., Svendsen, J. S., Marko, I. & Sharpless, K. B. (1989). *J. Am. Chem. Soc.* **111**, 8069–8076.
Samas, B., Blackburn, A. C. & Kreuchauf, K. C. (2005). *Acta Cryst.* E**61**, o3983–o3984.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Thierry, B., Plaquevent, J.-C. & Cahard, D. (2001). *Tetrahedron Asymmetry*, **12**, 983–986.
Thierry, B., Plaquevent, J.-C. & Cahard, D. (2003). *Tetrahedron Asymmetry*, **14**, 1671–1677.
Welter, R. (2006). *Acta Cryst.* A**62**, s252.
Westrip, S. P. (2010). *publCIF*. In preparation.

supplementary materials

Acta Cryst. (2010). E**66**, o281 [**doi:10.1107/S1600536809054907**]

1-Butylquinine tetrafluoroborate

S. Eltaief, P. Retailleau, L. Straver and B. Ben Hassine

Comment

The overall utility of asymmetric catalysts can be compared by examining three main criteria: 1) the variety of reactions that the catalyst can promote, 2) the availability of both enantiomeric antipodes of the catalyst at a reasonable price, and 3) the stability of the catalyst. Alkaloids and quaternary ammonium salt fulfill all of these criteria. They make them one of the most useful catalysts to date. Alkaloids can be transformed to quaternary ammonium salt in one or two steps. Chiral 1-butylquinine cation was reacted with BF_4^- leading to a new salt. The latter could serve as a chiral catalyst for different asymmetric reactions.

The X-ray structure shows that the boron atom presents statistically two types of tetrahedral environments: E1 and E2 with occupancy rates of 55.2% and 44.8%, respectively. The first environment (E1), which consists of F1A, F2A, F3A and F4, is strongly distorted as indicated by the B—F bond lengths varying from 1.324 (5) and 1.468 (5) Å and F—B—F scattering from 99.9 (8) and 123.3 (8)°. The second environment (E2), which consists of F1B, F2B, F3B and F4, is also very distorted as revealed by the B—F distance ranging from 1.298 (5) and 1.437 (5) Å and the F—B—F angles included between 95.5 (7) and 120.3 (8)°.

Regarding the cation, the quinine skeleton displays atomic parameters, which are comparable to those of the forty-two derivatives already deposited at the Cambridge Structural Database (Version 5.30, September 2009 update), Allen, 2002, *Mogul*, Version 1.1.3; Bruno *et al.*, 2004). It commonly adopts the open conformation III described in solution by Dijkstra *et al.*,1989, and in which the butyl-substituted quinuclidine nitrogen, N1, turns away from the quinoline ring and is oriented in the same direction as the methoxy oxygen. The torsion angles, which best characterized the overall shape, C12—C11—C10—O1 and, O1—C10 —C1— C2, are -22.6 (4)° and 45.8 (3)°, respectively. The butyl substituent at N1 is in equatorial conformation with respect to the six-membered ring C3/C7—N1 and the four butyl atoms are almost coplanar with N1—C20/C23 (r.m.s. deviation of 0.046 Å), and parallel to the [001] direction.

In the crystal structure, molecules are mainly linked by intermolecular O—H···N hydrogen bonds into helical chains running along a crystallographic 2_1 axis at *y*=1/4 position in the ***a***-axis direction with graph-set notation C(7) (Bernstein *et al.* (1994). The stability of the chains also benefits from the tilted superimposition of adjacent quinolin moieties with dihedral angle of 36.2 (4)° and shortest centroid distance of 4.162 (5) Å.

Experimental

Quinine and 1-bromobutane (1.1 equiv) were dissolved in acetonitrile and refluxed overnight. The reaction mixture was concentrated and 1-butylquinine bromide was purified to 95% being washed with ethyl acetate. The desired 1-Butylquinine tetrafluoroborate,[BQ]BF4, was then produced by anion exchange with $NaBF_4$ (1.2 equiv) in biphasic CH_2Cl_2/H_2O mixture. The reaction mixture was stirred for a further 24 h. The mixture was then extracted with CH_2Cl_2 and the organic phase

was dried over $MgSO_4$. The solvent evaporation method was used to grow [BQ]BF_4 crystals in dichloromethane at room temperature. The product is a colorless single-crystal which is air stable (m.p.197- 199 °C).

Refinement

All H atoms attached to C or O atoms were placed in calculated positions (C—H = 0.95–1.00 Å; O—H = 0.84 Å (hydroxyl)) and allowed to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}($C_{aromatic\ or\ vinyl}$) or U_{iso}(H) =1.5U_{eq}(C_{others}, O). 3007 Friedel opposites were merged

Figures

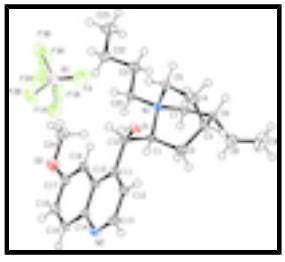


Fig. 1. An *ORTEP* diagram drawing of the title compound, with the atom-numbering scheme and 50% probability displacement ellipsoids.

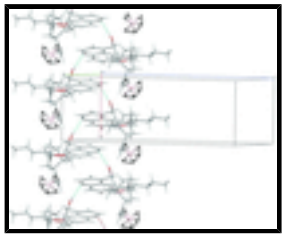


Fig. 2. Molecular packing of the title compound as viewed along the *a* axis.

(2*S*,4*S*,8*R*)-1-butyl-2-[(*R*)-(hydroxy)(6- methoxyquinolin-4-yl)methyl]-8-vinylquinuclidin-1-ium tetrafluoroborate

Crystal data

$C_{24}H_{33}N_2O_2^+ \cdot BF_4^-$	D_x = 1.340 Mg m^{-3}
M_r = 468.33	Melting point: 198 K
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation, λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 312 reflections
a = 8.041 (8) Å	θ = 2.4–23.5°
b = 12.597 (12) Å	μ = 0.11 mm^{-1}
c = 22.91 (2) Å	T = 120 K
V = 2321 (4) Å^3	Block, colourless
Z = 4	0.60 × 0.20 × 0.15 mm
F(000) = 992	

Data collection

Bruker Kappa-APEX DUO diffractometer	3968 independent reflections

Radiation source: sealed tube
graphite
Detector resolution: 8.3333 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.885, T_{max} = 0.982
25415 measured reflections

2957 reflections with $I > 2\sigma(I)$
R_{int} = 0.072
θ_{max} = 30.5°, θ_{min} = 1.8°
h = −6→11
k = −17→17
l = −32→32

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.156
S = 1.06
3968 reflections
329 parameters
0 restraints
0 constraints

Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0531P)^2 + 2.384P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.36 e $Å^{-3}$
$\Delta\rho_{min}$ = −0.38 e $Å^{-3}$

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C23	0.6730 (6)	0.0069 (3)	0.38114 (15)	0.0356 (10)	
H23A	0.6159	−0.0618	0.3825	0.053*	
H23B	0.6274	0.0537	0.4113	0.053*	
H23C	0.7922	−0.0037	0.3880	0.053*	
O1	0.4684 (3)	0.02693 (17)	0.06488 (9)	0.0199 (5)	
H1O	0.3809	0.0582	0.0546	0.030*	
O2	0.7800 (4)	0.42741 (19)	0.21386 (10)	0.0322 (7)	
C9	1.1983 (7)	−0.2286 (4)	0.0729 (2)	0.0533 (13)	
H9A	1.1473	−0.2966	0.0725	0.064*	

H9B	1.3072	−0.2197	0.0577	0.064*
C22	0.6473 (6)	0.0576 (3)	0.32091 (13)	0.0287 (8)
H22A	0.5272	0.0701	0.3143	0.034*
H22B	0.7048	0.1271	0.3195	0.034*
N2	0.6884 (4)	0.3676 (2)	−0.02267 (11)	0.0217 (6)
C10	0.5780 (4)	0.1006 (2)	0.09150 (12)	0.0158 (6)
H10	0.5270	0.1271	0.1285	0.019*
N1	0.7366 (4)	−0.0274 (2)	0.16071 (10)	0.0166 (5)
C24	0.7511 (5)	0.3433 (3)	0.25445 (13)	0.0256 (8)
H24A	0.6405	0.3129	0.2477	0.038*
H24B	0.7573	0.3711	0.2943	0.038*
H24C	0.8357	0.2881	0.2492	0.038*
C17	0.7533 (5)	0.4053 (2)	0.15683 (14)	0.0228 (7)
C16	0.6980 (5)	0.3092 (2)	0.13639 (13)	0.0189 (6)
H16	0.6765	0.2531	0.1631	0.023*
C15	0.6724 (4)	0.2928 (2)	0.07579 (12)	0.0162 (6)
C11	0.6174 (4)	0.1949 (2)	0.05190 (12)	0.0167 (6)
C1	0.7426 (4)	0.0434 (2)	0.10566 (11)	0.0153 (6)
H1	0.8281	0.0995	0.1129	0.018*
C2	0.8059 (5)	−0.0253 (3)	0.05386 (13)	0.0205 (7)
H2A	0.7304	−0.0168	0.0201	0.025*
H2B	0.9181	−0.0012	0.0420	0.025*
C3	0.8128 (5)	−0.1425 (2)	0.07171 (14)	0.0220 (7)
H3	0.8413	−0.1873	0.0371	0.026*
C6	0.9446 (4)	−0.1570 (2)	0.11938 (13)	0.0198 (7)
H6	0.9325	−0.2300	0.1359	0.024*
C8	1.1178 (5)	−0.1465 (3)	0.09467 (14)	0.0263 (8)
H8	1.1703	−0.0789	0.0948	0.032*
C21	0.7152 (5)	−0.0141 (3)	0.27310 (13)	0.0249 (8)
H21A	0.6534	−0.0821	0.2728	0.030*
H21B	0.8339	−0.0298	0.2807	0.030*
C20	0.6969 (5)	0.0408 (2)	0.21390 (12)	0.0200 (7)
H20A	0.5811	0.0667	0.2102	0.024*
H20B	0.7708	0.1037	0.2134	0.024*
C18	0.7836 (5)	0.4904 (3)	0.11797 (16)	0.0280 (8)
H18	0.8210	0.5568	0.1325	0.034*
C19	0.7590 (5)	0.4769 (2)	0.05954 (15)	0.0236 (7)
H19	0.7789	0.5344	0.0337	0.028*
C14	0.7041 (4)	0.3780 (2)	0.03679 (13)	0.0186 (6)
C13	0.6389 (5)	0.2759 (3)	−0.04317 (13)	0.0221 (7)
H13	0.6283	0.2685	−0.0843	0.027*
C12	0.6002 (4)	0.1872 (3)	−0.00788 (12)	0.0195 (7)
H12	0.5627	0.1229	−0.0252	0.023*
C4	0.6443 (5)	−0.1761 (3)	0.09637 (16)	0.0286 (8)
H4A	0.6440	−0.2535	0.1039	0.034*
H4B	0.5555	−0.1602	0.0678	0.034*
C5	0.6115 (5)	−0.1157 (3)	0.15346 (14)	0.0231 (7)
H5A	0.4977	−0.0857	0.1529	0.028*
H5B	0.6195	−0.1652	0.1869	0.028*

C7	0.9085 (4)	−0.0766 (2)	0.16817 (13)	0.0183 (6)	
H7A	0.9148	−0.1127	0.2065	0.022*	
H7B	0.9938	−0.0200	0.1675	0.022*	
B1	0.2102 (6)	0.1749 (3)	0.21354 (18)	0.0298 (10)	
F4	0.2425 (5)	0.0825 (2)	0.18461 (12)	0.0708 (11)	
F1A	0.3521 (8)	0.2072 (5)	0.2369 (4)	0.078 (3)	0.552 (8)
F2A	0.1287 (7)	0.2640 (4)	0.18703 (19)	0.0410 (15)	0.552 (8)
F3A	0.0935 (13)	0.1528 (5)	0.2613 (3)	0.085 (3)	0.552 (8)
F1B	0.3276 (11)	0.2428 (5)	0.1845 (4)	0.066 (3)	0.448 (8)
F2B	0.0631 (9)	0.1900 (7)	0.1900 (3)	0.056 (3)	0.448 (8)
F3B	0.2247 (13)	0.1795 (5)	0.2699 (3)	0.049 (2)	0.448 (8)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C23	0.045 (3)	0.045 (2)	0.0171 (15)	−0.006 (2)	0.0005 (16)	0.0078 (14)
O1	0.0225 (14)	0.0132 (10)	0.0241 (11)	−0.0040 (10)	−0.0072 (10)	0.0040 (8)
O2	0.0489 (19)	0.0218 (12)	0.0258 (12)	−0.0070 (12)	−0.0034 (12)	−0.0064 (9)
C9	0.034 (3)	0.069 (3)	0.058 (3)	0.004 (3)	0.003 (2)	−0.028 (3)
C22	0.039 (2)	0.0311 (18)	0.0162 (14)	0.0057 (17)	0.0041 (14)	0.0043 (12)
N2	0.0248 (17)	0.0199 (13)	0.0202 (12)	0.0009 (12)	0.0044 (11)	0.0079 (10)
C10	0.0197 (18)	0.0124 (13)	0.0151 (12)	−0.0009 (12)	0.0003 (12)	0.0043 (10)
N1	0.0216 (16)	0.0138 (11)	0.0144 (11)	0.0030 (11)	0.0001 (10)	0.0030 (9)
C24	0.027 (2)	0.0300 (18)	0.0200 (14)	−0.0028 (15)	−0.0006 (14)	−0.0055 (12)
C17	0.029 (2)	0.0153 (14)	0.0237 (15)	0.0020 (14)	0.0013 (14)	−0.0024 (11)
C16	0.0259 (19)	0.0134 (13)	0.0175 (13)	0.0022 (13)	−0.0005 (12)	0.0025 (10)
C15	0.0202 (18)	0.0123 (13)	0.0159 (13)	0.0011 (12)	0.0007 (12)	0.0029 (10)
C11	0.0194 (18)	0.0142 (14)	0.0166 (13)	0.0017 (13)	0.0007 (12)	0.0032 (10)
C1	0.0217 (18)	0.0132 (13)	0.0111 (11)	−0.0020 (12)	0.0001 (11)	0.0037 (9)
C2	0.0266 (19)	0.0202 (14)	0.0148 (13)	0.0042 (14)	0.0012 (12)	−0.0007 (11)
C3	0.027 (2)	0.0144 (14)	0.0244 (15)	−0.0011 (14)	−0.0011 (14)	−0.0042 (11)
C6	0.0255 (19)	0.0110 (13)	0.0228 (14)	0.0019 (12)	0.0020 (14)	−0.0001 (11)
C8	0.028 (2)	0.0289 (17)	0.0221 (15)	−0.0003 (16)	−0.0015 (14)	−0.0016 (13)
C21	0.035 (2)	0.0229 (16)	0.0172 (14)	0.0070 (15)	−0.0005 (14)	0.0069 (11)
C20	0.0296 (19)	0.0160 (14)	0.0144 (12)	0.0069 (13)	0.0020 (13)	0.0019 (10)
C18	0.037 (2)	0.0119 (14)	0.0348 (18)	−0.0024 (14)	0.0053 (17)	−0.0017 (12)
C19	0.028 (2)	0.0117 (14)	0.0311 (16)	−0.0004 (14)	0.0078 (15)	0.0041 (11)
C14	0.0201 (18)	0.0139 (13)	0.0218 (14)	0.0017 (13)	0.0041 (12)	0.0053 (10)
C13	0.025 (2)	0.0260 (16)	0.0153 (13)	−0.0010 (14)	0.0009 (13)	0.0078 (12)
C12	0.0227 (19)	0.0191 (14)	0.0167 (13)	−0.0005 (13)	−0.0007 (12)	0.0022 (11)
C4	0.031 (2)	0.0164 (15)	0.0379 (19)	−0.0060 (14)	−0.0064 (16)	−0.0028 (13)
C5	0.027 (2)	0.0158 (14)	0.0269 (15)	−0.0038 (14)	−0.0017 (14)	0.0062 (12)
C7	0.0183 (18)	0.0165 (14)	0.0201 (14)	0.0031 (13)	−0.0019 (12)	0.0018 (11)
B1	0.041 (3)	0.0181 (17)	0.0301 (19)	−0.0058 (18)	−0.0086 (19)	0.0032 (14)
F4	0.108 (3)	0.0438 (15)	0.0606 (17)	−0.0434 (18)	0.0418 (18)	−0.0335 (13)
F1A	0.046 (4)	0.046 (3)	0.142 (8)	0.018 (3)	−0.054 (5)	−0.054 (5)
F2A	0.059 (4)	0.025 (2)	0.039 (2)	0.007 (2)	−0.009 (2)	0.0031 (17)
F3A	0.120 (8)	0.058 (4)	0.077 (5)	0.052 (5)	0.068 (5)	0.036 (3)

F1B	0.069 (6)	0.022 (3)	0.106 (7)	−0.026 (3)	0.051 (5)	−0.020 (3)
F2B	0.037 (4)	0.084 (7)	0.046 (3)	0.027 (4)	−0.006 (3)	−0.008 (4)
F3B	0.077 (7)	0.043 (3)	0.026 (3)	0.014 (4)	−0.013 (3)	−0.015 (2)

Geometric parameters (Å, °)

C23—C22	1.535 (5)	C1—C2	1.555 (4)
C23—H23A	0.9800	C1—H1	1.0000
C23—H23B	0.9800	C2—C3	1.533 (5)
C23—H23C	0.9800	C2—H2A	0.9900
O1—C10	1.417 (4)	C2—H2B	0.9900
O1—H1O	0.8400	C3—C4	1.528 (6)
O2—C17	1.353 (4)	C3—C6	1.533 (5)
O2—C24	1.429 (4)	C3—H3	1.0000
C9—C8	1.317 (6)	C6—C8	1.509 (5)
C9—H9A	0.9500	C6—C7	1.536 (4)
C9—H9B	0.9500	C6—H6	1.0000
C22—C21	1.522 (5)	C8—H8	0.9500
C22—H22A	0.9900	C21—C20	1.530 (4)
C22—H22B	0.9900	C21—H21A	0.9900
N2—C13	1.310 (5)	C21—H21B	0.9900
N2—C14	1.374 (4)	C20—H20A	0.9900
C10—C11	1.528 (4)	C20—H20B	0.9900
C10—C1	1.541 (5)	C18—C19	1.364 (5)
C10—H10	1.0000	C18—H18	0.9500
N1—C5	1.509 (4)	C19—C14	1.421 (5)
N1—C7	1.524 (4)	C19—H19	0.9500
N1—C20	1.525 (4)	C13—C12	1.414 (4)
N1—C1	1.545 (4)	C13—H13	0.9500
C24—H24A	0.9800	C12—H12	0.9500
C24—H24B	0.9800	C4—C5	1.536 (5)
C24—H24C	0.9800	C4—H4A	0.9900
C17—C16	1.372 (4)	C4—H4B	0.9900
C17—C18	1.415 (5)	C5—H5A	0.9900
C16—C15	1.419 (4)	C5—H5B	0.9900
C16—H16	0.9500	C7—H7A	0.9900
C15—C14	1.419 (4)	C7—H7B	0.9900
C15—C11	1.421 (4)	B1—F4	1.364 (5)
C11—C12	1.380 (4)		
C17—O2—C24	116.7 (3)	C8—C6—C7	112.9 (3)
C8—C9—H9A	120.0	C3—C6—C7	108.0 (3)
C8—C9—H9B	120.0	C8—C6—H6	108.2
H9A—C9—H9B	120.0	C3—C6—H6	108.2
C21—C22—C23	110.6 (3)	C7—C6—H6	108.2
C21—C22—H22A	109.5	C9—C8—C6	121.8 (4)
C23—C22—H22A	109.5	C9—C8—H8	119.1
C21—C22—H22B	109.5	C6—C8—H8	119.1
C23—C22—H22B	109.5	C22—C21—C20	109.6 (3)
H22A—C22—H22B	108.1	C22—C21—H21A	109.8

C13—N2—C14	117.8 (3)	C20—C21—H21A	109.8
O1—C10—C11	112.5 (2)	C22—C21—H21B	109.8
O1—C10—C1	108.6 (2)	C20—C21—H21B	109.8
C11—C10—C1	108.1 (3)	H21A—C21—H21B	108.2
O1—C10—H10	109.2	N1—C20—C21	115.7 (2)
C11—C10—H10	109.2	N1—C20—H20A	108.4
C1—C10—H10	109.2	C21—C20—H20A	108.4
C5—N1—C7	108.5 (2)	N1—C20—H20B	108.4
C5—N1—C20	111.3 (3)	C21—C20—H20B	108.4
C7—N1—C20	109.2 (2)	H20A—C20—H20B	107.4
C5—N1—C1	110.9 (2)	C19—C18—C17	119.9 (3)
C7—N1—C1	107.4 (2)	C19—C18—H18	120.1
C20—N1—C1	109.5 (2)	C17—C18—H18	120.1
O2—C17—C16	124.3 (3)	C18—C19—C14	121.0 (3)
O2—C17—C18	115.1 (3)	C18—C19—H19	119.5
C16—C17—C18	120.6 (3)	C14—C19—H19	119.5
C17—C16—C15	120.6 (3)	N2—C14—C15	122.4 (3)
C17—C16—H16	119.7	N2—C14—C19	118.4 (3)
C15—C16—H16	119.7	C15—C14—C19	119.2 (3)
C16—C15—C14	118.7 (3)	N2—C13—C12	124.0 (3)
C16—C15—C11	123.3 (3)	N2—C13—H13	118.0
C14—C15—C11	118.0 (3)	C12—C13—H13	118.0
C12—C11—C15	118.3 (3)	C11—C12—C13	119.4 (3)
C12—C11—C10	120.9 (3)	C11—C12—H12	120.3
C15—C11—C10	120.7 (3)	C13—C12—H12	120.3
C10—C1—N1	114.5 (3)	C3—C4—C5	109.3 (3)
C10—C1—C2	112.4 (2)	C3—C4—H4A	109.8
N1—C1—C2	108.2 (2)	C5—C4—H4A	109.8
C10—C1—H1	107.1	C3—C4—H4B	109.8
N1—C1—H1	107.1	C5—C4—H4B	109.8
C2—C1—H1	107.1	H4A—C4—H4B	108.3
C3—C2—C1	110.2 (3)	N1—C5—C4	110.2 (3)
C3—C2—H2A	109.6	N1—C5—H5A	109.6
C1—C2—H2A	109.6	C4—C5—H5A	109.6
C3—C2—H2B	109.6	N1—C5—H5B	109.6
C1—C2—H2B	109.6	C4—C5—H5B	109.6
H2A—C2—H2B	108.1	H5A—C5—H5B	108.1
C4—C3—C6	108.5 (3)	N1—C7—C6	111.0 (3)
C4—C3—C2	109.4 (3)	N1—C7—H7A	109.4
C6—C3—C2	109.3 (3)	C6—C7—H7A	109.4
C4—C3—H3	109.9	N1—C7—H7B	109.4
C6—C3—H3	109.9	C6—C7—H7B	109.4
C2—C3—H3	109.9	H7A—C7—H7B	108.0
C8—C6—C3	111.1 (3)		
C24—O2—C17—C16	1.4 (6)	C7—C6—C8—C9	150.3 (4)
C24—O2—C17—C18	−179.9 (3)	C23—C22—C21—C20	−176.9 (3)
O2—C17—C16—C15	179.7 (4)	C5—N1—C20—C21	66.9 (4)
C18—C17—C16—C15	1.0 (6)	C7—N1—C20—C21	−52.9 (4)
C17—C16—C15—C14	−0.4 (5)	C1—N1—C20—C21	−170.2 (3)

C17—C16—C15—C11	179.2 (3)	C22—C21—C20—N1	−171.6 (3)
C16—C15—C11—C12	−178.6 (3)	O2—C17—C18—C19	−179.4 (4)
C14—C15—C11—C12	1.0 (5)	C16—C17—C18—C19	−0.5 (6)
C16—C15—C11—C10	1.5 (5)	C17—C18—C19—C14	−0.4 (6)
C14—C15—C11—C10	−178.9 (3)	C13—N2—C14—C15	1.4 (5)
O1—C10—C11—C12	−22.6 (4)	C13—N2—C14—C19	179.5 (3)
C1—C10—C11—C12	97.2 (4)	C16—C15—C14—N2	177.6 (3)
O1—C10—C11—C15	157.3 (3)	C11—C15—C14—N2	−2.1 (5)
C1—C10—C11—C15	−82.9 (4)	C16—C15—C14—C19	−0.5 (5)
O1—C10—C1—N1	−78.1 (3)	C11—C15—C14—C19	179.9 (3)
C11—C10—C1—N1	159.6 (2)	C18—C19—C14—N2	−177.2 (4)
O1—C10—C1—C2	45.8 (3)	C18—C19—C14—C15	0.9 (6)
C11—C10—C1—C2	−76.4 (3)	C14—N2—C13—C12	0.2 (6)
C5—N1—C1—C10	62.0 (3)	C15—C11—C12—C13	0.5 (5)
C7—N1—C1—C10	−179.7 (2)	C10—C11—C12—C13	−179.6 (3)
C20—N1—C1—C10	−61.2 (3)	N2—C13—C12—C11	−1.2 (6)
C5—N1—C1—C2	−64.1 (3)	C6—C3—C4—C5	53.4 (3)
C7—N1—C1—C2	54.2 (3)	C2—C3—C4—C5	−65.7 (3)
C20—N1—C1—C2	172.7 (3)	C7—N1—C5—C4	−65.4 (3)
C10—C1—C2—C3	−117.9 (3)	C20—N1—C5—C4	174.4 (3)
N1—C1—C2—C3	9.5 (4)	C1—N1—C5—C4	52.2 (3)
C1—C2—C3—C4	53.0 (4)	C3—C4—C5—N1	11.5 (4)
C1—C2—C3—C6	−65.7 (4)	C5—N1—C7—C6	51.3 (3)
C4—C3—C6—C8	168.6 (3)	C20—N1—C7—C6	172.8 (2)
C2—C3—C6—C8	−72.2 (3)	C1—N1—C7—C6	−68.5 (3)
C4—C3—C6—C7	−67.0 (3)	C8—C6—C7—N1	136.1 (3)
C2—C3—C6—C7	52.2 (4)	C3—C6—C7—N1	12.8 (3)
C3—C6—C8—C9	−88.1 (4)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1O···N2^{i}	0.84	1.95	2.787 (4)	174.

Symmetry codes: (i) *x*−1/2, −*y*+1/2, −*z*.

Fig. 1

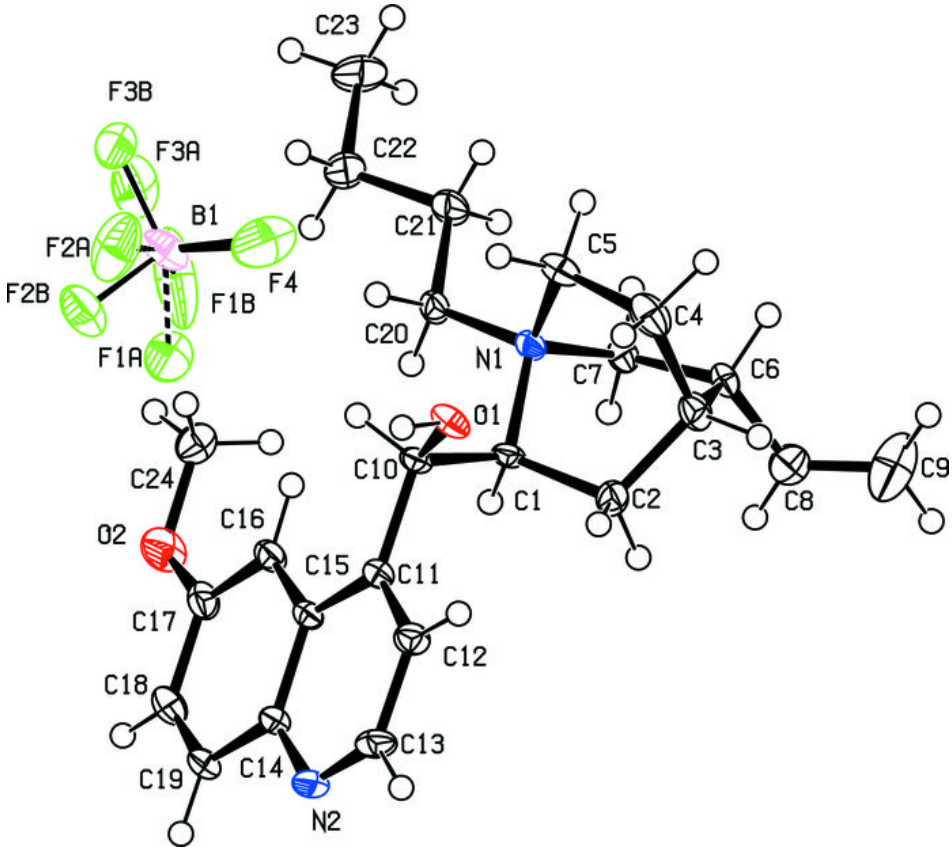

C23
F3B
F3A
C22
B1
C21
C5
F2A
F4
F2B
F1B
C20
N1
C4
C7
C6
F1A
O1
C3
C24
C10
C1
C2
C8
C9
O2
C16
C15
C11
C17
C12
C18
C13
C19
C14
N2

Fig. 2