$S = 1.44$
1192 reflections
109 parameters
Riding model, fixed isotropic U for H atoms
$w = 1/[\sigma^2(F) + 0.0008F^2]$
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

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

	x	y	z	U_{eq}
S	0.2685 (1)	0.3526 (1)	0.7264 (1)	0.032 (1)
O1	0.3847 (4)	0.2809 (1)	0.7717 (3)	0.040 (1)
C1	0.2587 (5)	0.3571 (2)	0.4925 (4)	0.032 (1)
C2	0.3218 (5)	0.3016 (2)	0.3919 (4)	0.034 (1)
C3	0.2953 (5)	0.3064 (2)	0.1965 (4)	0.032 (1)
O2	0.1999 (4)	0.3570 (1)	0.1212 (3)	0.040 (1)
O3	0.3870 (4)	0.2510 (1)	0.1117 (3)	0.044 (1)
C4	0.4940 (5)	0.4193 (2)	0.7489 (4)	0.038 (1)
C5	0.4642 (5)	0.4900 (2)	0.7733 (4)	0.042 (1)
C6	0.2316 (5)	0.5234 (2)	0.7727 (4)	0.042 (1)
O4	0.0623 (4)	0.4927 (1)	0.7156 (4)	0.055 (1)
O5	0.2306 (4)	0.5904 (1)	0.8383 (5)	0.063 (1)

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

S—O1	1.504 (2)	C5—C6	1.475 (4)
S—C1	1.764 (3)	C3—O2	1.213 (3)
S—C4	1.788 (3)	C6—O4	1.205 (4)
C1—C2	1.312 (4)	C3—O3	1.304 (4)
C4—C5	1.309 (4)	C6—O5	1.315 (4)
C2—C3	1.482 (4)		
O1—S—C1	106.3 (1)	O1—S—C4	103.8 (1)
S—C1—C2	122.2 (2)	S—C4—C5	125.6 (2)
C1—C2—C3	120.0 (3)	C4—C5—C6	121.5 (3)
C2—C3—O2	123.7 (3)	C5—C6—O4	123.5 (3)
C2—C3—O3	113.6 (2)	C5—C6—O5	112.6 (3)
O2—C3—O3	122.7 (3)	O4—C6—O5	123.9 (3)
C1—S—C4	94.9 (1)		

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: BK1034). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Alessio, E., Balducci, G., Calligaris, M., Costa, G., Attia, W. M. & Mestroni, G. (1991). *Inorg. Chem.* **30**, 609–618.
Ellison, R. D., Johnson, C. K. & Levy, H. A. (1971). *Acta Cryst.* B**27**, 333–344.
Feder, W., Dreizler, H., Rudolph, H. D. & Typke, V. (1969). *Z. Naturforsch. Teil A*, **24**, 266–278.
Guy, J. T. Jr, Cooper, J. C., Gilardi, R. D., Flippen-Anderson, J. L. & George, C. F. Jr (1988). *Inorg. Chem.* **27**, 635–638.
Ho, Z.-C. (1993). PhD thesis, Auburn Univ., Alabama, USA.
Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
Kisenyi, J. M., Willey, G. R. & Drew, M. G. B. (1985). *Acta Cryst.* C**41**, 700–702.
Koziol, A. E., Majewski, W. & Janczewski, M. (1985). *Acta Cryst.* C**41**, 144–146.
Pijper, W. P. (1971). *Acta Cryst.* B**27**, 344–348.
Rozsondai, B., Moore, J. H., Gregory, D. C. & Hargittai, I. (1979). *J. Mol. Struct.* **51**, 69–76.
Soriano-Garcia, M., Toscano, R. A., García, G., Larvaza, M. I. & Sánchez, I. H. (1984). *Acta Cryst.* C**40**, 887–889.
Typke, V. (1978). *Z. Naturforsh. Teil A*, **33**, 842–847.
Viswamitra, M. A. & Kannan, K. K. (1966). *Nature (London)*, **209**, 1016–1017.

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Acta Cryst. (1995). C**51**, 306–308

Decahydro-6,6-dimethoxy-1,2,2,4-tetramethylquinolinium Picrate

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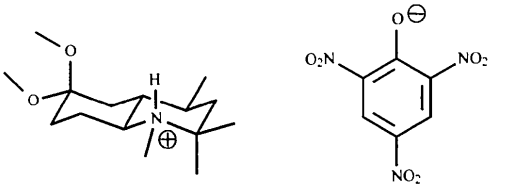
(*Received* 20 *May* 1993; *accepted* 17 *June* 1994)

Abstract

The crystal structure of the title compound, $C_{15}H_{30}NO_2^+.C_6H_2N_3O_7^-$, was determined in order to ascertain the stereochemistry of the decahydroquinoline moiety. It was found to show a normal chair–chair conformation with the 1- and 4-methyl groups in equatorial positions.

Comment

During the course of a project aimed at the development of new fungicidal compounds (Kasemann, 1993), the title compound (1) was synthesized by Birch reduction of 6-ethoxy-1,2,3,4-tetrahydro-1,2,2,4-tetramethylquinoline (Aldrich, Germany) followed by $NaCNBH_3$ reduction of the enamine moiety and subsequent conversion of the enol ether to the dimethyl ketal. As the quinoline base did not crystallize well, it was converted to the picrate, which crystallized from ethanol, after prolonged standing, as large yellow blocks.



(1)

The decahydroquinolinium moiety adopts a normal chair–chair conformation (Fig. 1*a*) with total ring puckering amplitudes (Cremer & Pople, 1975) of $Q = 0.551\ (3)$ Å [$\theta = 1.4\ (3)°$] for the piperidinium ring and $Q = 0.573\ (3)$ Å [$\theta = 4.3\ (3)°$] for the cyclohexane ring. Bond lengths and angles in the moiety are in good accord with expected values; mean values are C—C = 1.520 (11), C—N = 1.521 (17) and C—O = 1.416 (18) Å. The picrate anion (Fig. 1*b*) has dimensions comparable with those of potassium picrate (Palenik, 1972) or other organic picrates (*e.g.* Bernstein, Regev & Herbstein, 1980; Ferguson, Ruhl, Wieckowski, Lloyd & McNab, 1984). The three nitro groups are inclined at 26.9 (2), 17.3 (2) [$N(26)O_2$ and $N(32)O_2$, both *ortho*] and 1.4 (3)° [$N(29)O_2$, *para*] to the benzene ring. An N—H···O hydrogen bond links the quinoline moiety with the phenolic O atom of the picrate anion [N(1)···O(25) = 2.786 (4) Å].

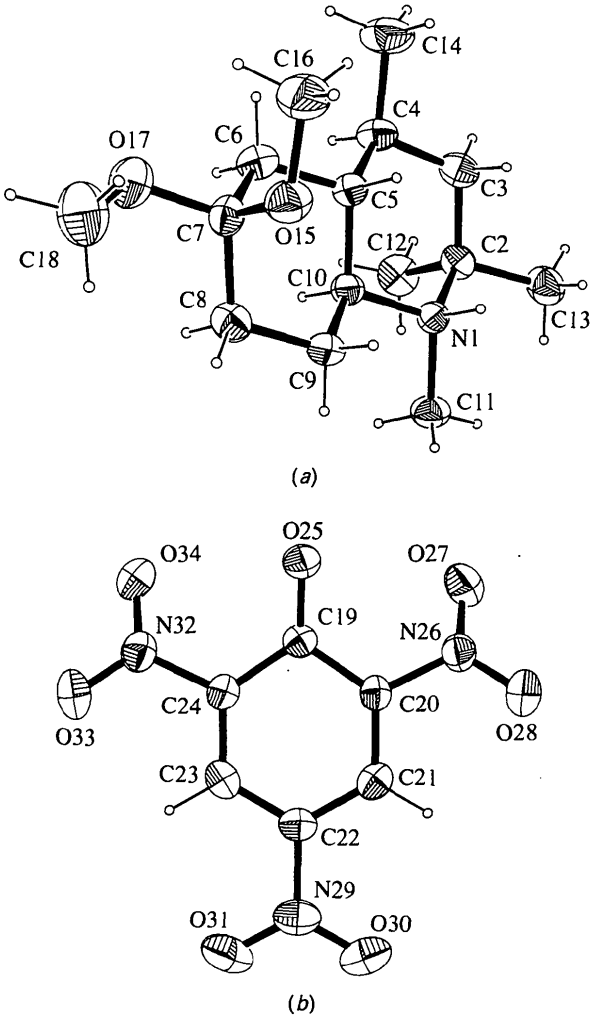


Fig. 1. Displacement ellipsoid plot (30% probability) of (*a*) the decahydroquinolinium cation and (*b*) the picrate anion.

Experimental

Crystal data

$C_{15}H_{30}NO_2^+.C_6H_2N_3O_7^-$
$M_r = 484.51$
Monoclinic
$P2_1/n$
$a = 9.109\ (3)$ Å
$b = 16.546\ (5)$ Å
$c = 15.703\ (5)$ Å
$\beta = 93.38\ (2)°$
$V = 2363\ (1)$ Å^3
$Z = 4$
$D_x = 1.362$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 18 reflections
$\theta = 18$–$20°$
$\mu = 0.10$ mm^{-1}
$T = 294$ K
Block
0.62 × 0.57 × 0.26 mm
Yellow

Data collection

Philips PW100 four-circle diffractometer
θ–2θ scans
Absorption correction: none
4478 measured reflections
4162 independent reflections
2646 observed reflections [$F > 4\sigma(F)$]
$R_{int} = 0.029$
$\theta_{max} = 25°$
$h = -10 \rightarrow 10$
$k = 0 \rightarrow 19$
$l = 0 \rightarrow 18$
3 standard reflections
frequency: 120 min
intensity decay: 0.8%

Refinement

Refinement on F
$R = 0.060$
$wR = 0.057$
$S = 1.93$
2646 reflections
326 parameters
$w = 1/[\sigma^2(F_o) + 0.0002F^2]$
$(\Delta/\sigma)_{max} = 0.01$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ e Å^{-3}
Extinction correction: $F^* = F(1 - 0.0001\chi F^2/\sin\theta)$
Extinction coefficient: $\chi = 0.0017\ (3)$
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

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

	x	y	z	U_{eq}
N(1)	0.3591 (2)	0.3163 (1)	0.2605 (1)	0.044 (1)
C(2)	0.4997 (3)	0.3276 (2)	0.2120 (2)	0.051 (1)
C(3)	0.6264 (3)	0.2945 (2)	0.2687 (2)	0.059 (1)
C(4)	0.6121 (3)	0.2086 (2)	0.2974 (2)	0.057 (1)
C(5)	0.4700 (3)	0.1995 (2)	0.3447 (2)	0.048 (1)
C(6)	0.4406 (3)	0.1139 (2)	0.3715 (2)	0.057 (1)
C(7)	0.3070 (3)	0.1055 (2)	0.4226 (2)	0.051 (1)
C(8)	0.1726 (3)	0.1405 (2)	0.3732 (2)	0.056 (1)
C(9)	0.1992 (3)	0.2262 (2)	0.3433 (2)	0.052 (1)
C(10)	0.3354 (3)	0.2303 (2)	0.2916 (2)	0.044 (1)
C(11)	0.2245 (3)	0.3468 (2)	0.2112 (2)	0.061 (1)
C(12)	0.4860 (4)	0.2843 (2)	0.1261 (2)	0.071 (1)
C(13)	0.5235 (4)	0.4181 (2)	0.2003 (2)	0.071 (1)
C(14)	0.7488 (4)	0.1840 (3)	0.3523 (3)	0.091 (2)
O(15)	0.3185 (2)	0.1481 (1)	0.4996 (1)	0.067 (1)
C(16)	0.4451 (4)	0.1297 (3)	0.5562 (2)	0.083 (2)
O(17)	0.2945 (3)	0.0219 (1)	0.4372 (2)	0.075 (1)
C(18)	0.1897 (5)	−0.0006 (3)	0.4956 (3)	0.102 (2)
C(19)	0.3517 (3)	0.4295 (2)	0.4751 (2)	0.046 (1)
C(20)	0.3660 (3)	0.4082 (2)	0.5644 (2)	0.046 (1)
C(21)	0.2909 (3)	0.4448 (2)	0.6267 (2)	0.052 (1)
C(22)	0.1974 (3)	0.5079 (2)	0.6056 (2)	0.051 (1)
C(23)	0.1808 (3)	0.5347 (2)	0.5232 (2)	0.053 (1)
C(24)	0.2533 (3)	0.4975 (2)	0.4607 (2)	0.047 (1)
O(25)	0.4113 (3)	0.3911 (1)	0.4184 (1)	0.063 (1)

N(26)	0.4664 (3)	0.3427 (2)	0.5917 (2)	0.064 (1)
O(27)	0.5730 (3)	0.3282 (1)	0.5521 (2)	0.080 (1)
O(28)	0.4396 (3)	0.3066 (2)	0.6569 (2)	0.114 (1)
N(29)	0.1186 (3)	0.5472 (2)	0.6712 (2)	0.068 (1)
O(30)	0.1346 (3)	0.5219 (2)	0.7443 (2)	0.084 (1)
O(31)	0.0375 (3)	0.6038 (2)	0.6501 (2)	0.095 (1)
N(32)	0.2280 (3)	0.5312 (2)	0.3750 (2)	0.061 (1)
O(33)	0.1215 (3)	0.5748 (2)	0.3608 (2)	0.082 (1)
O(34)	0.3141 (3)	0.5159 (2)	0.3217 (2)	0.105 (1)

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

N(1)—C(2)	1.540 (3)	O(17)—C(18)	1.412 (6)
N(1)—C(10)	1.524 (4)	C(19)—C(20)	1.444 (4)
N(1)—C(11)	1.498 (3)	C(19)—C(24)	1.448 (4)
C(2)—C(3)	1.518 (4)	C(19)—O(25)	1.244 (4)
C(2)—C(12)	1.526 (4)	C(20)—C(21)	1.368 (4)
C(2)—C(13)	1.526 (5)	C(20)—N(26)	1.466 (4)
C(3)—C(4)	1.499 (5)	C(21)—C(22)	1.375 (4)
C(4)—C(5)	1.537 (4)	C(22)—C(23)	1.368 (4)
C(4)—C(14)	1.527 (5)	C(22)—N(29)	1.444 (4)
C(5)—C(6)	1.506 (5)	C(23)—C(24)	1.362 (4)
C(5)—C(10)	1.529 (4)	C(24)—N(32)	1.462 (4)
C(6)—C(7)	1.503 (4)	N(26)—O(27)	1.208 (4)
C(7)—C(8)	1.524 (4)	N(26)—O(28)	1.222 (5)
C(7)—O(15)	1.398 (4)	N(29)—O(30)	1.222 (4)
C(7)—O(17)	1.408 (4)	N(29)—O(31)	1.226 (4)
C(8)—C(9)	1.518 (5)	N(32)—O(33)	1.219 (4)
C(9)—C(10)	1.524 (4)	N(32)—O(34)	1.207 (4)
O(15)—C(16)	1.446 (4)		
C(2)—N(1)—C(10)	114.2 (2)	C(5)—C(10)—C(9)	110.3 (3)
C(2)—N(1)—C(11)	112.5 (2)	C(7)—O(15)—C(16)	116.2 (2)
C(10)—N(1)—C(11)	110.7 (2)	C(7)—O(17)—C(18)	115.4 (3)
N(1)—C(2)—C(3)	107.0 (2)	C(20)—C(19)—C(24)	111.1 (3)
N(1)—C(2)—C(12)	110.7 (2)	C(20)—C(19)—O(25)	123.5 (3)
N(1)—C(2)—C(13)	107.9 (2)	C(24)—C(19)—O(25)	125.3 (3)
C(3)—C(2)—C(12)	111.6 (3)	C(19)—C(20)—C(21)	124.5 (3)
C(3)—C(2)—C(13)	108.4 (3)	C(19)—C(20)—N(26)	118.9 (3)
C(12)—C(2)—C(13)	111.0 (3)	C(21)—C(20)—N(26)	116.6 (3)
C(2)—C(3)—C(4)	116.2 (2)	C(20)—C(21)—C(22)	119.5 (3)
C(3)—C(4)—C(5)	109.2 (3)	C(21)—C(22)—C(23)	120.4 (3)
C(3)—C(4)—C(14)	109.9 (3)	C(21)—C(22)—N(29)	119.8 (3)
C(5)—C(4)—C(14)	112.5 (3)	C(23)—C(22)—N(29)	119.8 (3)
C(4)—C(5)—C(6)	113.3 (3)	C(22)—C(23)—C(24)	120.3 (3)
C(4)—C(5)—C(10)	112.0 (3)	C(19)—C(24)—C(23)	124.1 (3)
C(6)—C(5)—C(10)	108.4 (2)	C(19)—C(24)—N(32)	120.2 (3)
C(5)—C(6)—C(7)	113.5 (3)	C(23)—C(24)—N(32)	115.7 (3)
C(6)—C(7)—C(8)	110.1 (3)	C(20)—N(26)—O(27)	120.2 (3)
C(6)—C(7)—O(15)	113.2 (2)	C(20)—N(26)—O(28)	117.0 (3)
C(6)—C(7)—O(17)	104.7 (3)	O(27)—N(26)—O(28)	122.7 (3)
C(8)—C(7)—O(15)	105.4 (2)	C(22)—N(29)—O(30)	118.6 (3)
C(8)—C(7)—O(17)	112.7 (3)	C(22)—N(29)—O(31)	117.9 (3)
O(15)—C(7)—O(17)	110.9 (3)	O(30)—N(29)—O(31)	123.5 (3)
C(7)—C(8)—C(9)	112.0 (2)	C(24)—N(32)—O(33)	118.3 (3)
C(8)—C(9)—C(10)	110.9 (3)	C(24)—N(32)—O(34)	119.0 (3)
N(1)—C(10)—C(5)	111.2 (2)	O(33)—N(32)—O(34)	122.7 (3)
N(1)—C(10)—C(9)	110.3 (2)		

The structure was solved by direct methods using the *Xtal*3.0 suite of programs (Hall & Stewart, 1990). Structure refinement was carried out with the program *SHELX*76 (Sheldrick, 1976) using anisotropic displacement factors for non-H atoms. H atoms were generated in idealized positions (CN—H = 0.96 Å) and were then refined either as riding with the atoms to which they are bonded (CH, CH_2, NH) or as parts of rigid CH_3 groups. The isotropic temperature factors of the H atoms were fixed at either 1.0 × U_{eq} (CH, CH_2, NH) or 1.36 × U_{eq} (CH_3) of their carrier atoms.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SH1070). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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References

Bernstein, J., Regev, H. & Herbstein, F. H. (1980). *Acta Cryst.* B**36**, 1170–1175.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Ferguson, G., Ruhl, B. L., Wieckowski, T., Lloyd, D. & McNab, H. (1984). *Acta Cryst.* C**40**, 1740–1742.
Hall, S. R. & Stewart, J. M. (1990). Editors. *Xtal*3.0 *Reference Manual*. Univs. of Western Australia, Australia, and Maryland, USA.
Kasemann, O. (1993). PhD thesis, Technical Univ. of Vienna, Austria.
Palenik, G. J. (1972). *Acta Cryst.* B**28**, 1633–1635.
Sheldrick, G. M. (1976). *SHELX*76. *Program for Crystal Structure Determination*. Univ. of Cambridge, England.

Acta Cryst. (1995). C**51**, 308–311

Ergotamine Tartrate Bis(ethanol) Solvate

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(*Received* 6 *December* 1993; *accepted* 20 *April* 1994)

Abstract

The molecule of the title compound 12′-hydroxy-2′-methyl-3′,6′,18-trioxo-5′α-(phenylmethyl)ergotamanium tartrate bis(ethanol) solvate, $C_{33}H_{36}N_5O_5^+.0.5C_4H_4O_6^{2-}.2C_2H_6O$, consists of two different substituted polycyclic systems connected by an amide linkage. The partial double-bond character of the C16—N3 amide bond results in conformational rigidity of the molecule. The ergotamine *C*, *F* and *G* rings have regular envelope conformations and the *D* ring possesses a predominant half-chair conformation. An intramolecular O5—HO5···O1 hydrogen bond was found in the structure. The ergotamine and tartrate molecules are joined together through N2—HN2···O6(x, $y+1$, z) and N3—HN3···O8($-x$, $-y+1$, z) hydrogen bonds.