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2-*tert*-Butyl-4-methyl-6-(1-piperidiniomethyl)-phenol perchlorate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.050
wR factor = 0.130
Data-to-parameter ratio = 11.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the crystal structure of the title compound, $C_{17}H_{28}NO^{+}\cdot ClO_4^{-}$, the *N*-protonated piperidine ring is present in the normal chair conformation. The perchlorate anions link the cations, through intermolecular N—H···O and O—H···O hydrogen bonds, forming a chain-like structure.

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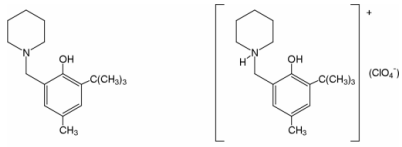
Comment

Great effort has been devoted to the syntheses of new derivatives of piperidine because they can act as anticancer drugs (Varvaresou *et al.*, 1996); they are active serotonergic agents (Radl *et al.*, 1999) and they have other clinical applications (Orjales *et al.*, 1995). However, to our knowledge, structural studies on these interesting compounds are still quite rare. Since stereochemical knowledge is so important in the rational design of pharmaceuticals, we have recently reported the synthesis and crystal structure of the piperidine derivative, 2-*tert*-butyl-4-methyl-6-(piperidyl–*N*-methyl)phenol, (I) (Deng *et al.*, 2001). As a continuation of our previous work, we report here the synthesis and X-ray crystal structure of the *N*-protonated perchlorate of (I), *viz.* 2-*tert*-butyl-4-methyl-6-(1-piperidiniomethyl)phenol perchlorate, (II).



The crystal structure of (II) consists of a discrete $C_{17}H_{28}NO^{+}$ cation and a ClO_4^{-} counter-anion, as depicted in Fig. 1. In the cation, the protonated piperidine ring has a normal chair conformation, as was found in its unprotonated analog, (I). The chair geometry for the piperidine ring is slightly distorted, as shown by the range of the ring torsion angles [55.3 (4)–56.8 (5)° for (II) compared with 60° for an

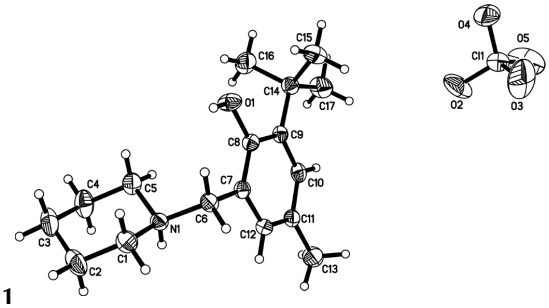


Figure 1
*ORTEP*II (Johnson, 1976) view of (II), shown with displacement ellipsoids at the 30% probability level.

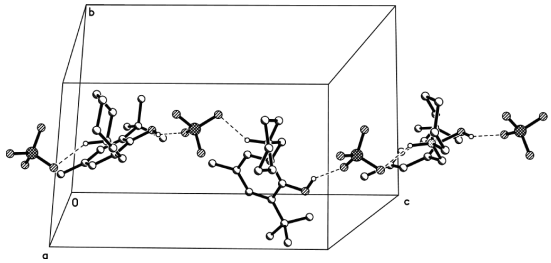


Figure 2
View of the one-dimensional hydrogen-bonding structure of (II) in the unit cell.

ideal chair conformation]; this was also observed for the piperidine ring in (I) [torsion angles ranging from 52.5 (4) to 59.8 (3)°]. The C—C and C—N bond lengths within the piperidine moiety [mean values 1.513 (5) and 1.500 (4) Å; Table 1] in the cation of (II) agree with the values in (I) [1.515 (4) and 1.461 (4) Å]. The slightly longer C—N bond distances in (II), compared with those in (I), may be due to the protonation of the piperidine ring.

The phenol O atom and the protonated piperidine N atom act as donors in intermolecular O—H···O and N—H···O hydrogen bonds to the O atoms of the perchlorate. Thus, the perchlorate anions act as bridges linking the cations, through hydrogen bonds, to form a chain-like structure along the *c* direction in the unit cell, as shown in Fig. 2. The hydrogen-bond parameters, listed in Table 2, are in the normal range for hydrogen-bonded interactions.

Experimental

2-*tert*-Butyl-4-methyl-6-(*N*-piperidylmethyl)phenol was prepared according to the procedure detailed in our previous work (Deng *et al.*, 2001). Colorless single crystals of (II) suitable for X-ray diffraction were obtained by slow diffusion of diethyl ether into an acetonitrile/methanol solution of 2-*tert*-butyl-4-methyl-6-(*N*-piperidylmethyl)-phenol in the presence of $HClO_4$. Yield: 90%. Analysis calculated for the title compound: C 56.42, H 7.80, N 3.87%; found: C 56.31, H 8.06, N 3.72%. FT–IR (KBr pellet, cm^{-1}): 3536 (*s*), 3116 (*m*), 3013 (*w*), 2946 (*s*), 2869 (*m*), 2805 (*w*), 2752 (*w*), 2540 (*w*), 2037 (*w*), 1778 (*w*), 1637 (*w*), 1596 (*w*), 1481 (*s*), 1474 (*s*), 1455 (*s*), 1409 (*s*), 1363 (*m*), 1316 (*m*), 1283 (*m*), 1261 (*m*), 1232 (*m*), 1177 (*s*), 1134 (*vs*), 1070 (*vs*), 1032 (*s*), 933 (*m*), 875 (*s*), 760 (*m*), 624 (*vs*), 588 (*w*), 536 (*m*), 529 (*w*).

Crystal data

$C_{17}H_{28}NO^+\cdot ClO_4^-$
M_r = 361.85
Monoclinic, $P2_1/c$
a = 11.639 (5) Å
b = 10.390 (4) Å
c = 16.466 (6) Å
β = 107.297 (7)°
V = 1901.1 (13) Å^3
Z = 4
D_x = 1.264 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5481 reflections
θ = 2.4–25.0°
μ = 0.23 mm^{-1}
T = 293 (2) K
Prism, colorless
0.20 × 0.15 × 0.10 mm

Data collection

Bruker SMART 1000 diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
T_{min} = 0.956, T_{max} = 0.978
5523 measured reflections
3256 independent reflections
1555 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
θ_{max} = 25.0°
$h = -7 \rightarrow 12$
$k = -11 \rightarrow 11$
$l = -18 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.130
S = 1.03
2656 reflections
226 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.06P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C8	1.378 (4)	N1—C6	1.506 (4)
N1—C1	1.492 (4)	N1—C5	1.506 (4)
C1—N1—C6	111.0 (3)	O1—C8—C7	121.8 (3)
C1—N1—C5	111.2 (3)	O1—C8—C9	116.8 (3)
C6—N1—C5	112.3 (2)	C7—C8—C9	121.5 (3)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O5^{i}	0.82	2.05	2.761 (4)	145
N1—H2···O4^{ii}	0.91	2.17	2.998 (5)	151

Symmetry codes: (i) $1+x, y, z$; (ii) $1+x, \frac{3}{2}-y, \frac{1}{2}+z$.

All H atoms were placed in calculated positions at distances of 0.97, 0.93 and 0.96 Å from their parent C atoms for methylene C—H, phenyl C—H and methyl C—H bonds, respectively, and included in the final refinement in the riding-model approximation, with displacement parameters derived from the atoms to which they were bonded [U_{iso}(H) = 1.2U_{eq}(C,N) for N—H, phenyl C—H and methyl C—H, and 1.5U_{eq}(C,O) for O—H and methyl C—H]. The crystal gradually decomposed during data collection making it necessary to discard some reflections during refinement.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Bruker, 1998); software used to prepare material for publication: *SHELXL*97.

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