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## Key indicators

Single-crystal X-ray study  
 $T = 293$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.014$  Å  
 $R$  factor = 0.063  
 $wR$  factor = 0.122  
Data-to-parameter ratio = 17.3For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.2-*tert*-Butyl-4-methyl-6-(1-piperidinio-methyl)phenol bromide

In the crystal structure of the title compound,  $\text{C}_{17}\text{H}_{28}\text{NO}^+\cdot\text{Br}^-$ , the *N*-protonated piperidine ring adopts the normal chair conformation. Each bromide anion acts as an acceptor in  $\text{N}-\text{H}\cdots\text{Br}$  and  $\text{O}-\text{H}\cdots\text{Br}$  hydrogen bonds, resulting in an  $R_2^1(8)$  ring pattern.

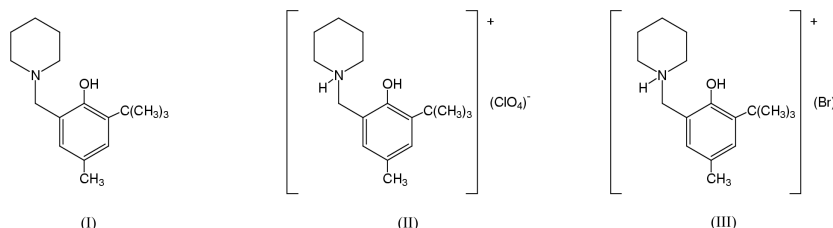
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## Comment

The design and synthesis of new piperidine derivatives have attracted much interest owing to their application in anti-cancer drugs (Varvaresou *et al.*, 1996), high active serotonergic agents (Radl *et al.*, 1999) and other areas of clinical medicine (Orjales *et al.*, 1995). Recently, we have reported the synthesis and crystal structures of 2-*tert*-butyl-4-methyl-6-(piperidyl-*N*-methyl)phenol, (I) (Deng *et al.*, 2001), and its *N*-protonated perchlorate, (II) (Zhao & Du, 2003). In the present contribution, we report the synthesis and crystal structure of the *N*-protonated bromide of (I), namely 2-*tert*-butyl-4-methyl-6-(1-piperidiniomethyl)phenol bromide, (III).

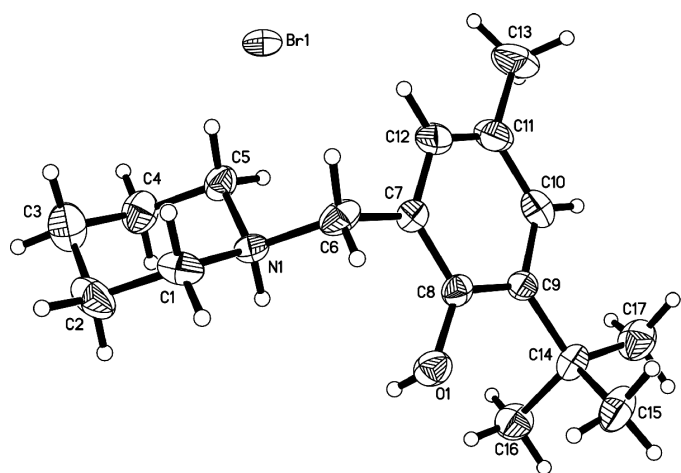


The crystal structure of (III) consists of a  $\text{C}_{17}\text{H}_{28}\text{NO}^+$  cation and a  $\text{Br}^-$  counter-anion, as shown in Fig. 1. As in its analogs, (I) and (II), the piperidine ring adopts a normal chair conformation. The chair geometry is slightly distorted from ideal, the ring torsion angles lying in the range 56.3 (10)–58.5 (13)°. In (I) and (II), the angles are in the ranges 52.5 (4)–59.8 (3) and 55.3 (4)–56.8 (5)°, respectively.

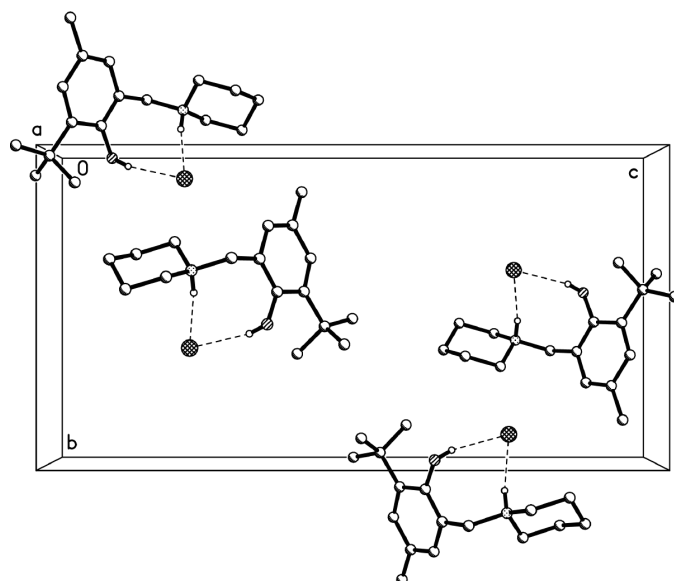
The C–C and C–N bond lengths in the piperidine ring (mean values 1.524 and 1.513 Å; Table 1) can be compared with the values in (I) (1.515 and 1.461 Å) and (II) (1.513 and 1.500 Å). The slightly longer C–N bond distances in (II) and (III), compared with those in (I), may be due to the protonation of the piperidine ring.

It should be noted that the orientations of the piperidine ring and the benzene ring in (II) and (III) are different; this is reflected by the torsion angles  $\text{N1}-\text{C6}-\text{C7}-\text{C8}$  [103.6 (4)° for (II) and –84.1 (10)° for (III)] and  $\text{N1}-\text{C6}-\text{C7}-\text{C12}$  [–77.9 (4)° for (II) and 100.2 (10)° for (III)].

Analysis of the crystal packing of the title compound reveals the existence of an  $\text{O}-\text{H}\cdots\text{Br}$  hydrogen bond between the phenol O atom and the bromide anion. An  $\text{N}-$



**Figure 1**  
View of the asymmetric unit of (III), with displacement ellipsoids drawn at the 30% probability level. H atoms are represented by spheres of arbitrary radius.



**Figure 2**  
A view of the hydrogen-bonding pattern in the unit cell. Hydrogen bonds are indicated by dashed lines.

H $\cdots$ Br hydrogen bond is also present, involving the protonated piperidine N atom and the Br $^-$  anion. According to the formalism of graph-set patterns (Etter, 1990), the resulting motif (Fig. 2) is characterized as an  $R_2^1(8)$  ring pattern. The relevant geometrical details are listed in Table 2; these values are in the normal range for weak hydrogen-bonding interactions. It is interesting to compare this pattern with that in (II), where the perchlorate anions link the protonated piperidine rings, through O—H $\cdots$ O and N—H $\cdots$ O interactions, thereby forming a one-dimensional chain motif. This indicates that the counter-anions play an important role in the development of hydrogen bonding in the two structures. Examination of the structure with *PLATON* (Spek, 2003) indicates that there are no solvent-accessible voids nor  $\pi$ – $\pi$  stacking interactions in the crystal structure of (III).

## Experimental

2-*tert*-Butyl-4-methyl-6-(piperidyl-*N*-methyl)phenol was prepared as in previous work (Deng *et al.*, 2001). Colorless single crystals of the title compound, (III), suitable for X-ray diffraction were obtained, in 95% yield, by slow diffusion of diethyl ether into a methanol solution of 2-*tert*-butyl-4-methyl-6-(piperidyl-*N*-methyl)phenol in the presence of HBr.

### Crystal data

$C_{17}H_{28}NO^+ \cdot Br^-$   
 $M_r = 342.30$   
 Orthorhombic,  $P2_12_12_1$   
 $a = 5.987$  (4) Å  
 $b = 12.604$  (8) Å  
 $c = 24.469$  (15) Å  
 $V = 1846$  (2) Å $^3$   
 $Z = 4$   
 $D_x = 1.232$  Mg m $^{-3}$

Mo  $K\alpha$  radiation  
 Cell parameters from 673 reflections  
 $\theta = 2.3$ – $19.5^\circ$   
 $\mu = 2.22$  mm $^{-1}$   
 $T = 293$  (2) K  
 Cut needle, colorless  
 $0.30 \times 0.20 \times 0.10$  mm

### Data collection

Bruker SMART 1000 diffractometer  
 $\omega$  scans  
 Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)  
 $T_{\min} = 0.555$ ,  $T_{\max} = 0.808$   
 6633 measured reflections

3211 independent reflections  
 1809 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.100$   
 $\theta_{\max} = 25.0^\circ$   
 $h = -7 \rightarrow 5$   
 $k = -14 \rightarrow 14$   
 $l = -26 \rightarrow 29$

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.063$   
 $wR(F^2) = 0.122$   
 $S = 1.04$   
 3211 reflections  
 186 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.034P)^2 + 2.308P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.60$  e Å $^{-3}$   
 $\Delta\rho_{\min} = -0.27$  e Å $^{-3}$   
 Absolute structure: Flack (1983);  
 1305 Friedel pairs  
 Flack parameter = 0.05 (3)

**Table 1**

Selected geometric parameters (Å, °).

C1–N1	1.511 (8)	C4–C5	1.516 (12)
C1–C2	1.533 (9)	C5–N1	1.514 (9)
C2–C3	1.540 (11)	C8–O1	1.377 (10)
C3–C4	1.506 (11)		
O1–C8–C7	117.9 (8)	C1–N1–C5	112.2 (6)
O1–C8–C9	122.0 (8)	C1–N1–C6	109.6 (6)
C7–C8–C9	120.1 (9)	C5–N1–C6	110.7 (7)

**Table 2**

Hydrogen-bonding geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1–H1C $\cdots$ Br1 $^i$	0.91	2.43	3.332 (8)	170
O1–H1 $\cdots$ Br1 $^i$	0.82	2.42	3.235 (9)	174

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

Although most H atoms were visible in difference maps, all H atoms were placed in geometrically calculated positions (0.97 Å for methylene C–H, 0.93 Å for aromatic C–H, 0.96 Å for methyl C–H, 0.91 Å for N–H and 0.82 Å for O–H). They were included in the final refinement in the riding-model approximation, with displacement parameters  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier atom})$  for N–H, aromatic

C—H and methylene C—H, and  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{carrier atom})$  for O—H and methyl C—H.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXL97*.

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