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(+)-*N*-[(*R*)-1-(2-Hydroxy-5-methylphenyl)propyl]-*N*-[(*R*)-2-methyl-1-phenylpropyl]ammonium chloride

Xiao-Feng Yang,[a]* Da-Qi Wang,[b] Guang-You Zhang[c] and Takuji Hirose[a]

[a]Department of Applied Chemistry, Faculty of Engineering, Saitama University, 225 Shimo-ohkubo, Sakura, Saitama 338-8570, Japan, [b]Department of Chemistry and Chemical Engineering, Liaocheng University, Liaocheng, Shandong 252059, People's Republic of China, and [c]Department of Chemistry and Chemical Engineering, Jinan University, Jinan, Shandong 250022, People's Republic of China

Correspondence e-mail: jn_yangxiaofeng@hotmail.com

Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.039
wR factor = 0.102
Data-to-parameter ratio = 9.5

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

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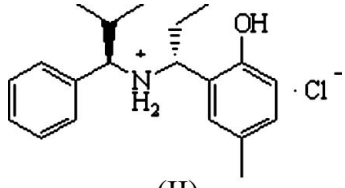
The title compound, $C_{20}H_{28}NO^{+}\cdot Cl^{-}$, was synthesized by a condensation reaction. The absolute configuration of the new stereogenic centre (the C atom between the N atom and the phenol ring) was determined as *R*. The crystal structure is stabilized through N—H···Cl and O—H···Cl hydrogen bonds and intramolecular N—H···O hydrogen bonding.

Comment

The search for new chiral ligands to be used in asymmetric catalysis is of great interest in the field of synthetic chemistry (Pu & Yu 2001; Cimarelli *et al.*, 2002). Carbon-carbon bond-forming reactions remain an active area of research (Le Goanvic *et al.*, 2002). A popular reaction is that of dialkylzinc with pro-chiral aldehydes (Le Goanvic *et al.*, 2002; Vilaplana *et al.*, 2002; Superchi *et al.*, 2002), since the chiral secondary alcohols that are formed are important substrates for drug synthesis. Amino alcohols are excellent ligands for this reaction, as shown by Noyori & Kitamura (1991). 1,3-Aminophenols are analogous to amino alcohols, although they were not used as chiral ligands in asymmetric catalysis until 1998 (Cardellicchio *et al.*, 1998, 1999), when they showed high enantioselectivity. Since then, interest in the synthesis of aminophenols and their derivatives, and in their application in asymmetric catalysis, has increased significantly (Lu *et al.*, 2002; Li *et al.*, 2003; Zhang *et al.*, 2003; Yang, Zhang, Zhang *et al.*, 2005). Following on from our previous work (Yang, Zhang, Liu *et al.*, 2005), we prepared a new aminoalkylphenol, namely 4-methyl-2-[(*R*)-1-((1*R*)-2-methyl-1-phenylpropylamino)-propyl]phenol, (I). In this paper, we report the structure of the title compound, (II), which is a salt of (I) with HCl.



(II)

The molecular structure of (II) is shown in Fig. 1. The absolute configuration of the new stereogenic centre C1 is *R*. The chain of atoms C3/C2/C1/N1/C11/C12/C14 shows an approximately all-*trans* conformation (Table 1). The dihedral angle between the C4–C9 and C15–C20 aromatic rings is 12.9 (2)°.

Atom N1 acts as a hydrogen-bond donor to phenol atom O1 and to the Cl1 anion (Fig. 1 and Table 2). There is also an O—H···Cl hydrogen bond, forming chains along the *b* axis (Fig. 2).

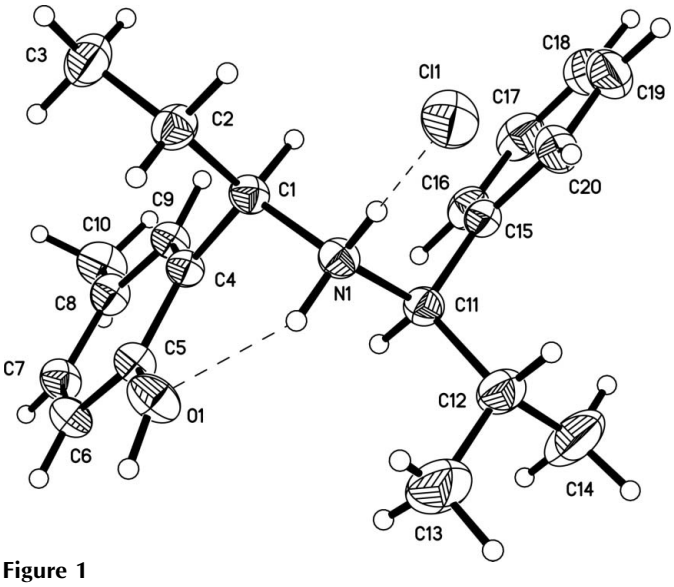


Figure 1
The asymmetric unit of (II), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Dashed lines indicate hydrogen bonds.

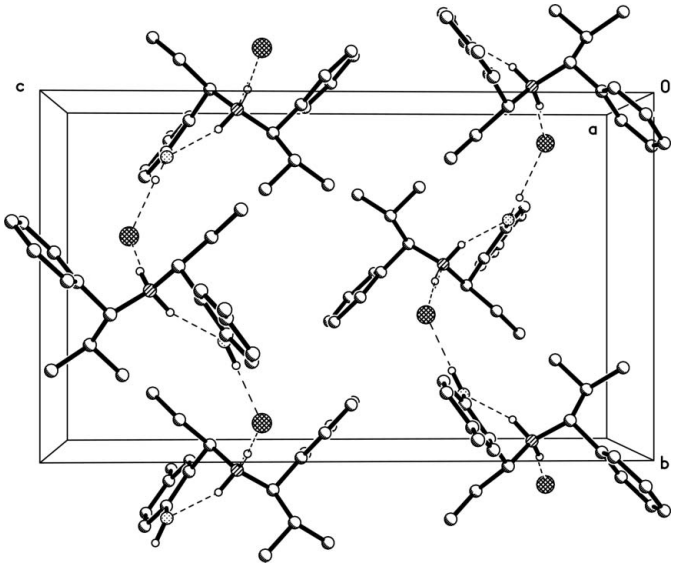


Figure 2
A view of the packing in (II). H atoms have been omitted, except for those involved in hydrogen bonds (dashed lines).

Experimental

(*R*)-2-methyl-1-phenylpropylamine (3 mmol; Yang, Zhang, Liu *et al.*, 2005) and 1-(2-hydroxy-5-methylphenyl)-propan-1-one (3 mmol; purchased from J & K Chemical Ltd) were dissolved in methanol (20 ml) and reacted at room temperature for 12 h. The solvent was removed and $NaBH_4$ (15 mmol) was added to the solution in a tetrahydrofuran–ethanol (1:1 *v*/*v*, 20 ml) mixture. The reaction was allowed to stand and was monitored by thin-layer silica-gel chromatography. The reaction was stopped when complete conversion of the starting material was detected by thin-layer silica-gel chromatography. 6 *M* HCl was then added dropwise to the reaction mixture until hydrogen production ceased and the mixture was then neutralized with Na_2CO_3. The aqueous solution was extracted with $CHCl_3$. The organic layer was dried with anhydrous sodium sulfate and filtered. The solvent was removed under reduced pressure. Further purification was carried out by thin-layer silica-gel chromatography (first eluted with chloroform–methanol, 60:1, *v*/*v*; second eluted with chloroform) to give the chiral compound, (I) {canary-yellow oil, $[a]_D^{15}$ = 52.2° (*c* = 0.5, $CHCl_3$)}. The chemical yield of (*R*,*R*)-(I) and the diastereoisomeric excess {measured by chiral high-performance liquid chromatography [Chiralcel OD-H, propan-2-ol–hexane (5:95, *v*/*v*) of the corresponding amide derivatives of racemic and chiral (I)]} are 76.2% and 96.0%, respectively.

Compound (I) and concentrated HCl were reacted at room temperature and a colourless solid was precipitated. The solvent was removed and the solid residue was recrystallized from an ethanol–hexane (7:3, *v*/*v*) mixture to yield compound (II) (m.p. 461–462 K).

Crystal data

$C_{20}H_{28}NO^+\cdot Cl^-$
M_r = 333.88
Orthorhombic, $P2_12_12_1$
a = 8.725 (2) Å
b = 11.641 (3) Å
c = 19.313 (5) Å
V = 1961.7 (9) Å^3
Z = 4
D_x = 1.131 Mg m^{-3}
Mo *K*α radiation
μ = 0.20 mm^{-1}
T = 298 (2) K
Block, colourless
0.33 × 0.21 × 0.13 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.937, T_{max} = 0.975
10299 measured reflections
1975 independent reflections
1503 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.102
S = 1.05
1975 reflections
208 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0453P)^2 + 0.4485P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Absolute structure: Flack (1983), with 1469 Friedel pairs
Flack parameter: −0.07 (9)

Table 1
Selected torsion angles (°).

C11—N1—C1—C2	−169.4 (3)	C1—N1—C11—C12	−168.3 (3)
N1—C1—C2—C3	177.8 (3)	N1—C11—C12—C14	176.3 (4)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···Cl1^{i}	0.90	2.27	3.144 (3)	163
N1—H1*B*···O1	0.90	2.06	2.737 (4)	131
O1—H1···Cl1^{ii}	0.82	2.24	3.032 (3)	161

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

All H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C—H = 0.93–0.98 Å, N—H = 0.90 Å and O—H = 0.82 Å, and with U_{iso}(H) = 1.2U_{eq}(C,N), or 1.5U_{eq}(C,O) for methyl and hydroxy groups.

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

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