Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

(*R*)-*N*-[(*R*)-1-(2-Hydroxy-5-methylphenyl)-2-phenylethyl]-2-methyl-1-phenylpropylammonium chloride

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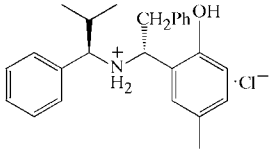
Received 11 May 2006
Accepted 27 June 2006
Online 29 July 2006

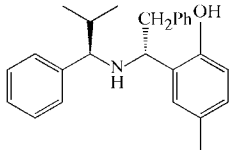
In the title compound, $C_{25}H_{30}NO^{+}\cdot Cl^{-}$, the molecules are linked by a combination of intermolecular N—H···Cl and O—H···Cl hydrogen bonds and intramolecular N—H···O hydrogen bonds. The absolute configuration of the new stereogenic centre (the C atom adjacent to the N atom on the phenol side) is determined to have an *R* configuration.

Comment

The synthesis of enantiopure amine alcohols with a variety of functionalities is an important subject of research because this class of compounds are widespread in natural products, show pharmacological activity and have recently found application in asymmetric synthesis as chiral bases, auxiliaries and ligands (Cimarelli *et al.*, 2002). Chiral aminophenols, which are similar to amino alcohols, are important building blocks in organic synthesis, and have attracted increasing attention in recent years owing to their effect in asymmetric synthesis and asymmetric induction (Cimarelli *et al.*, 2001; Palmieri, 1999, 2000; Xu *et al.*, 2002; Berrisford *et al.*, 1995; Cimarelli & Palmieri, 1998, 2000; Hayase *et al.*, 1997; Juaristi *et al.*, 1998; Kitamura *et al.*, 1986, 1989; Nakano *et al.*, 1997; Rijnberg *et al.*, 1997; Soai *et al.*, 1992; Sola *et al.*, 1998; Watanabe *et al.*, 1991).



(I)



(II)

As part of our continuing studies of chiral aminophenols, we have established the molecular structure of 4-methyl-2-[(1*R*)-1-{[(1*R*)-2-methyl-1-phenylpropyl]amino}phenylethyl]phenol, (II), which was initially prepared to test its asymmetric catalytic activity. Aminoalkylphenol (II) was prepared by conventional condensation of (*R*)-2-methyl-1-phenylpropan-1-amine with 1-(2-hydroxy-5-methylphenyl)-2-phenylethanone, followed by reduction using sodium borohydride in a tetrahydrofuran–ethanol (1:1 *v*/*v*) mixture. The isolated yield and the diastereoisomeric excess, d.e. (determined by chiral high-performance liquid chromatography), are 82.7 and 99.5%, respectively. Compound (*R*,*R*)-(II) was obtained as the main product.

In addition to the synthesis of the novel chiral aminoalkylphenol, (II), its hydrochloride, (I), was also synthesized. The occurrence of a Cl atom in compound (I) helps in establishing the absolute configuration of the compound and thus in deducing the absolute configuration of (II). Therefore, an X-ray study of the title compound, (I), was carried out and we present the results here.

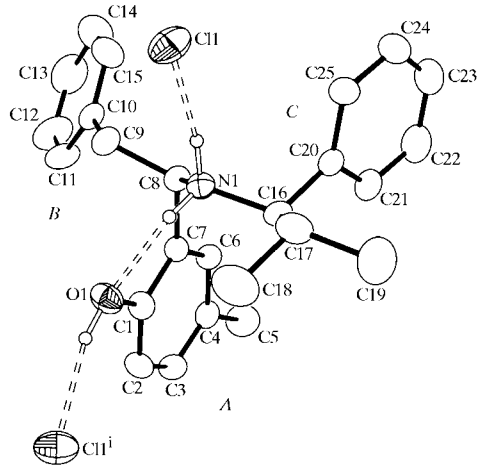


Figure 1
The asymmetric unit of (I), showing the atom- and ring-labelling schemes. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as spheres of arbitrary radii. Hydrogen bonds are indicated by double dashed lines. [Symmetry code: (i) $-x, y+\frac{1}{2}, -z+\frac{3}{2}$.]

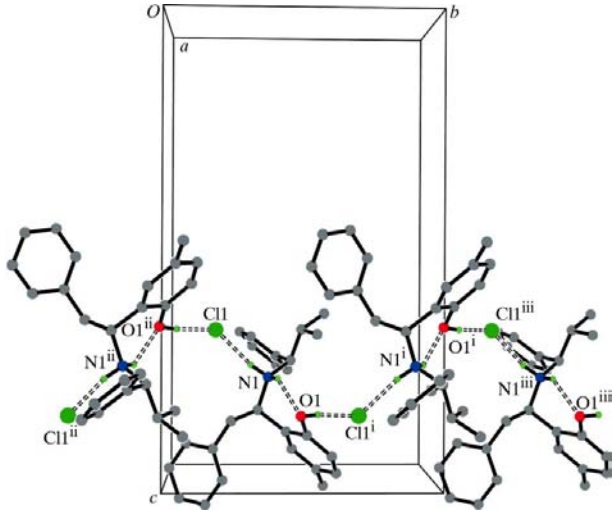


Figure 2
A packing diagram of (I), viewed down the *a* axis, showing the formation of helical chains through N—H···Cl and O—H···Cl hydrogen bonds (double dashed lines) along *b*. H atoms not involved in hydrogen bonding have been omitted. [Symmetry codes: (i) $-x, y+\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x, y-\frac{1}{2}, -z+\frac{3}{2}$; (iii) $x, y+1, z$.]

  DOI: 10.1107/S0108270106024802

Selected bond lengths and angles of (I), including those of the new stereogenic carbon centre (C8), are reported in Table 1. As shown in Fig. 1, the absolute configuration of (I) is *R*,*R*, so it can be deduced that the absolute configuration of (II) is also *R*,*R*.

An extensive network of hydrogen bonds, listed in Table 2, appears to be a key factor in the stabilization of this structure. The molecules of compound (I) (Fig. 1) are linked by a combination of intermolecular N—H···Cl and O—H···Cl hydrogen bonds and intramolecular N—H···O hydrogen bonds (Fig. 1). An interesting feature of the structure is that the intermolecular N—H···Cl and O—H···Cl hydrogen-bonding interactions result in the formation of helical chains running parallel to the *b* axis (Fig. 2). Atom N1 acts as a hydrogen-bond donor to phenol atom O1, with N···O = 2.711 (3) Å, which indicates a comparatively strong intramolecular hydrogen bond (Table 2). No aromatic π–π stacking interactions are present in the structure of (I).

The terminal benzene rings are neither parallel nor normal to each other [interplanar angles: 49.17 (12)° for *A*/*B*, 62.74 (12)° for *B*/*C* and 15.39 (15)° for *A*/*C*; rings are as defined in Fig. 1].

Experimental

The title compound was prepared according to the procedure of Yang *et al.* (2005). (*R*)-2-Methyl-1-phenylpropan-1-amine (0.9 mmol) and 1-(2-hydroxy-5-methylphenyl)-2-phenylethanone (0.9 mmol) were dissolved in methanol (10 ml) and reacted at room temperature for 48 h. After removal of the solvent, $NaBH_4$ (4.5 mmol) was added to the solution in tetrahydrofuran–ethanol (20 ml, 1:1 *v*/*v*) and stirred at 273 K until the solution became colourless. The solvent was then removed under reduced pressure. Water (10 ml) was added to the residue and 1 *N* HCl was added dropwise until hydrogen production ceased, and the mixture was then neutralized with aqueous Na_2CO_3. The mixture was extracted with $CHCl_3$ and the organic layer was dried over anhydrous sodium sulfate. The solvent was then removed under reduced pressure. Further purification was carried out by thin-layer silica-gel chromatography (chloroform) to give chiral (II) [colourless solid; yield 86.3%; $[\alpha]_D^{22}$ = 84.1 ($c_{0.5}$, $CHCl_3$)]. Compound (II) (0.5 mmol) and concentrated HCl (1 ml) were reacted in methanol (5 ml) at room temperature. The solvent was removed and the solid residue was recrystallized from a mixed solvent of ethyl acetate and ethanol (95:1 *v*/*v*) to yield compound (I) (m.p. 475–476 K).

Crystal data

$C_{25}H_{30}NO^+\cdot Cl^-$
M_r = 395.95
Orthorhombic, $P2_12_12_1$
a = 9.964 (2) Å
b = 11.565 (2) Å
c = 19.882 (4) Å
V = 2291.1 (8) Å^3
Z = 4
D_x = 1.148 Mg m^{-3}
Mo *K*α radiation
μ = 0.18 mm^{-1}
T = 298 (2) K
Block, colourless
0.21 × 0.15 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.962, T_{max} = 0.981
11656 measured reflections
4040 independent reflections
2725 reflections with $I > 2\sigma(I)$
R_{int} = 0.057
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.101
S = 0.98
4040 reflections
257 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0373P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}
Absolute structure: Flack (1983), with 1736 Friedel pairs
Flack parameter: −0.07 (8)

Table 1
Selected geometric parameters (Å, °).

C7—C8	1.515 (3)	C16—N1	1.502 (3)
C8—N1	1.513 (3)	C16—C20	1.510 (4)
C8—C9	1.535 (4)	C16—C17	1.533 (4)
N1—C8—C7	109.5 (2)	N1—C16—C20	111.0 (2)
N1—C8—C9	107.5 (2)	N1—C16—C17	108.8 (2)
C7—C8—C9	114.2 (2)	C20—C16—C17	113.9 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···Cl1^{i}	0.82	2.17	2.978 (2)	169
N1—H1*B*···O1	0.90	2.03	2.711 (3)	131
N1—H1*A*···Cl1	0.90	2.22	3.083 (2)	162

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

All H atoms were included in calculated positions and treated as riding on their parent atoms, with N—H = 0.90 Å, O—H = 0.82 Å, aromatic C—H = 0.93 Å, methyl C—H = 0.96 Å, methylene C—H = 0.97 Å and methine C—H = 0.98 Å, and with U_{iso}(H) = 1.2U_{eq}(C,N,O) or 1.5U_{eq}(methyl C).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

The authors are grateful to the Natural Science Foundation of Shandong Province, China (grant No. G0231), and the Foundation of the Education Ministry of China for Returned Students (grant No. G0220) for financial support. X-ray data were collected at Shandong Normal University, China.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: HJ3012). Services for accessing these data are described at the back of the journal.

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