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3-(2-Chloroethyl)-2-methyl-4-oxo-6,7,8,9-tetrahydro-4*H*-pyrido[1,2-*a*]pyrimidin-1-ium chloride

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Key indicators

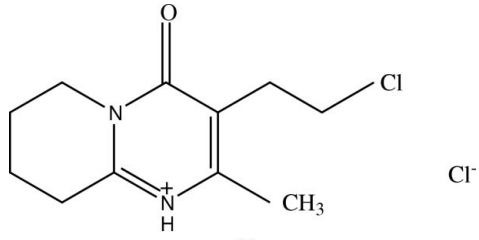
Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.053
wR factor = 0.131
Data-to-parameter ratio = 14.6

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

In the title compound, $C_{11}H_{16}ClN_2O^+\cdot Cl^-$, the chloroethyl side chain is in a synclinal conformation. The tetrahydropyridine ring adopts a half-chair conformation. The crystal structure is stabilized by intermolecular N—H···Cl hydrogen bonds.

Comment

Risperidone is an antipsychotic agent used in acute treatment and maintenance therapy in schizophrenia and related psychotic disorders (Kennis & Vandenberk, 1986; Umbricht & Kane, 1995). The crystal structure determination of the title compound, (I), was undertaken; (I) is an intermediate in the synthesis of the antipsychotic drug risperidone.



(I)

In all essential details, the molecular geometry in terms of bond distances and angles is in good agreement with related structures (Hermecz *et al.*, 1988; Peeters *et al.*, 1993; Jottier *et al.*, 1992; Blaton *et al.*, 1995; Ravikumar *et al.*, 2005). Atoms C3 and C4 of the tetrahydropyridine ring are disordered over two sites with occupancies of 0.710 (9) and 0.290 (9).

The C—N bond distances [mean value = 1.357 (4) Å] of the dihydropyrimidine are intermediate between the expected single- (1.47 Å) and double-bond (1.27 Å) distances. The sum of the angles around N1 and N2 are 360.9 and 359.9°, respectively, indicating sp^2 hybridization.

The dihydropyrimidine ring is planar and the methyl group and carbonyl O atom are coplanar with it. The chloroethyl side chain is in a synclinal (*-sc*) conformation. The chloroethyl side-chain (C9/C10/C11) plane is orthogonal to the dihydropyrimidine ring, with a dihedral angle of 79.3 (2)°.

The tetrahydropyridine ring adopts a half-chair conformation for both the major and minor components, with the displacement asymmetry parameter (Nardelli, 1983) ΔC_2(C1—N2) = 0.012 (2) and 0.057 (4), respectively. Atoms C3 and C4 of the major component are displaced by −0.340 (7) and 0.394 (7) Å, respectively, while the minor component atoms C31 and C41 are displaced by 0.444 (16) and −0.100 (18) Å, respectively, from the mean plane defined by the atoms C1/C2/C5/N2.

Intermolecular N—H···Cl hydrogen bonding is observed between atom N1 of the pyridopyrimidine ring and the chloride anion Cl2 (Table 2).

Experimental

To obtain crystals of (I) suitable for X-ray studies, the title compound (obtained from Jubilant Organosys Ltd, Nanjangud, Mysore, India) was dissolved in a mixture of methanol and water (80:20) and the solution was allowed to evaporate slowly.

Crystal data

$C_{11}H_{16}ClN_2O^+\cdot Cl^-$
$M_r = 263.16$
Orthorhombic, $P2_12_12_1$
$a = 6.609$ (5) Å
$b = 10.340$ (8) Å
$c = 18.473$ (14) Å
$V = 1262.5$ (17) Å^3
$Z = 4$
$D_x = 1.385$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.50$ mm^{-1}
$T = 273$ (2) K
Block, colorless
0.21 × 0.17 × 0.09 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: none
8301 measured reflections
2200 independent reflections
1980 reflections with $I > 2\sigma(I)$
$R_{int} = 0.056$
$\theta_{max} = 25.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.131$
$S = 1.10$
2200 reflections
151 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0819P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.68$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}
Absolute structure: Flack (1983), 890 Friedel pairs
Flack parameter: 0.06 (10)

Table 1
Selected geometric parameters (Å, °).

C1—N2	1.317 (3)	C6—N2	1.419 (4)
C1—N1	1.322 (4)	C8—N1	1.367 (4)
N2—C1—N1	119.6 (2)	C1—N2—C6	121.3 (2)
N1—C1—C2	117.9 (2)	C1—N2—C5	122.2 (2)
C1—N1—C8	123.7 (3)	C6—N2—C5	116.3 (2)
C7—C9—C10—Cl1	−61.4 (3)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···Cl2	0.84 (3)	2.18 (4)	3.017 (3)	176 (3)

The site-occupation factors of the disordered atoms (C3 and C4) were refined to 0.710 (9) and 0.290 (9). The geometries of the disordered atoms were restrained, where distances C2—C3, C2—C31, C3—C4, C31—C41, C4—C5 and C41—C5 were set to a target value 1.47 Å. For atoms C3/C31/C4/C41, a common U_{iso} parameter was refined. The H atom on the N atom [N—H = 0.84 (3) Å] was located in a difference density map and was refined freely with an isotropic displacement parameter. H atoms attached to C atoms were positioned geometrically and refined as riding atoms [methylene C—H = 0.97 Å, with $U_{iso}(H) = 1.2U_{eq}(C)$, and methyl C—H = 0.96 Å, with $U_{iso}(H) = 1.5U_{eq}(C)$]. The methyl groups were allowed to rotate but not to tip.

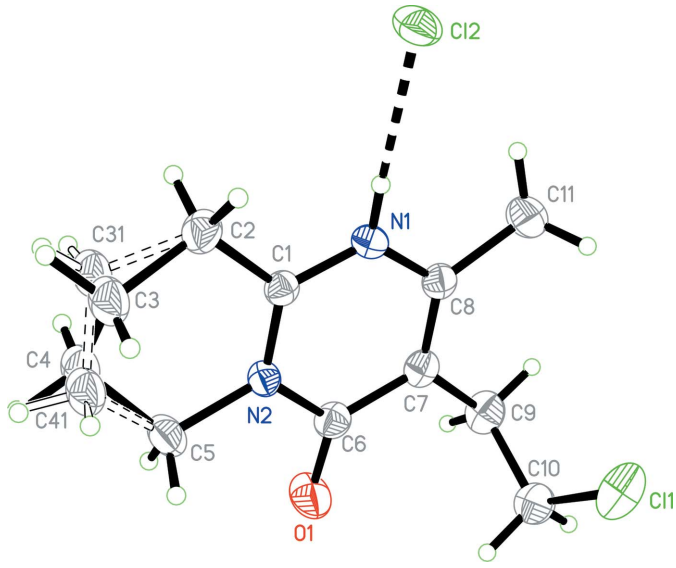


Figure 1
A view of the (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii. Hydrogen bonds are shown as dashed lines. The C atoms (C3 and C4) of the tetrahydropyridine are disordered over two sites, and the minor occupancy component is drawn with dashed open lines.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1990) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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