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1-[2-(4-Carboxyphenoxy)ethyl]piperidinium chloride

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.002 Å
R factor = 0.032
wR factor = 0.073
Data-to-parameter ratio = 19.2

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

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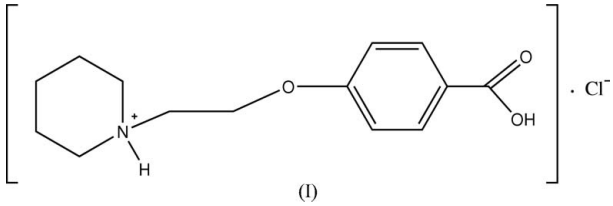
In the title structure, $C_{14}H_{20}NO_3^+\cdot Cl^-$, as expected, the pipiridine ring adopts a chair conformation and the carboxylic acid group and the benzene ring are essentially coplanar, forming a conjugated system. In the crystal structure, extended chains are formed through N—H···Cl and O—H···Cl hydrogen bonds.

Comment

The title compound, (I), is an intermediate product in the synthesis of raloxifene hydrochloride, a selective estrogen receptor modulator. It was prepared by the hydrolysis of methyl 4-(2-piperidinylethoxy)benzoate (Gong *et al.*, 2003) and was crystallized from ethanol. The title structure is shown in Fig. 1 and the torsion angles listed in Table 1 indicate that the carboxylic acid group and the benzene ring are essentially coplanar. The C1—C2 bond length is shorter than the normal single C—C bond (*ca* 1.54 Å), indicating that a conjugated system has been formed in this part of the molecule. In the crystal structure, extended chains are formed in the *c*-axis direction, through N—H···Cl and O—H···Cl hydrogen bonds (Table 2 and Fig. 2).



(I)

Experimental

The title compound was prepared according to the procedure of Gong *et al.* (2003). Suitable crystals were obtained by evaporation of an ethanol solution (m.p. 549–550 K).

Crystal data

$C_{14}H_{20}NO_3^+\cdot Cl^-$
M_r = 285.77
Orthorhombic, *Pbca*
a = 16.777 (4) Å
b = 7.4464 (15) Å
c = 23.192 (5) Å
V = 2897.3 (11) Å^3
Z = 8
D_x = 1.310 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 20969 reflections
θ = 3.0–27.5°
μ = 0.27 mm^{-1}
T = 296 (1) K
Block, colorless
0.30 × 0.20 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.918, T_{max} = 0.948
26060 measured reflections
3317 independent reflections
2190 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.036
θ_{max} = 27.5°
h = −21 → 21
k = −9 → 8
l = −30 → 30

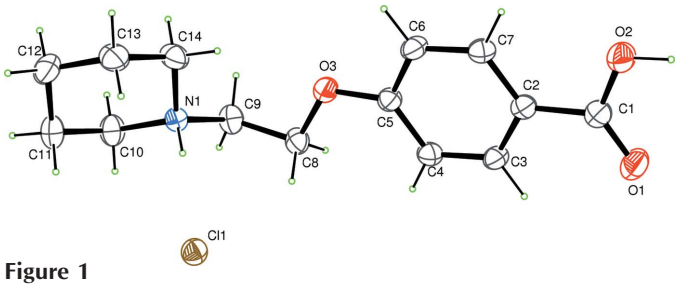


Figure 1
View of (I), showing 40% probability displacement ellipsoids. H atoms are drawn as spheres of arbitrary radii.

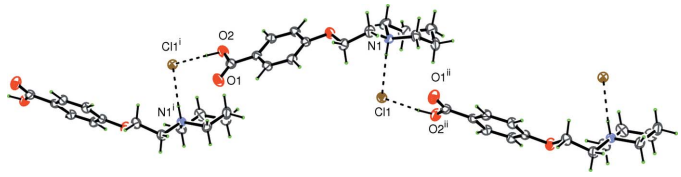


Figure 2
View of a hydrogen-bonded (dashed lines) chain. [Symmetry codes: (i) x, $\frac{1}{2}-y$, $\frac{1}{2}+z$; (ii) x, $\frac{1}{2}-y$, $-\frac{1}{2}+z$.]

Refinement

Refinement on F^2	$w = 1/[0.0001F_o^2 + \sigma(F_o^2)]/(4F_o^2)$
$R[F^2 > 2\sigma(F^2)] = 0.032$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.073$	$\Delta\rho_{max} = 0.22$ e Å^{-3}
$S = 1.00$	$\Delta\rho_{min} = -0.22$ e Å^{-3}
3317 reflections	Extinction correction: Larson (1970)
173 parameters	
H-atom parameters constrained	Extinction coefficient: 4.4 (3) × 10^2

Table 1
Selected geometric parameters (Å, °).

O2—C1	1.3323 (18)	C1—C2	1.4854 (19)
O3—C8	1.4184 (16)		
O1—C1—C2—C3	−4.0 (2)	O2—C1—C2—C3	177.40 (12)
O1—C1—C2—C7	174.10 (14)	O2—C1—C2—C7	−4.47 (18)

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H201···Cl1^{i}	0.93	2.11	3.0219 (9)	168
N1—H222···Cl1	0.86	2.27	3.1302 (11)	174

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

Atom H201 was located in a difference Fourier map. All other H atoms were placed in calculated positions, with N—H = 0.86 Å and C—H = 0.97 or 0.98 Å All H atoms were refined using a riding model, with U_{iso}(H) = 1.2U_{eq} of the carrier atom.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

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