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4-(2,3-Dichlorophenyl)-1-[3-oxo-3-(2-thienyl)-propyl]piperazin-1-ium chloride

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Key indicators

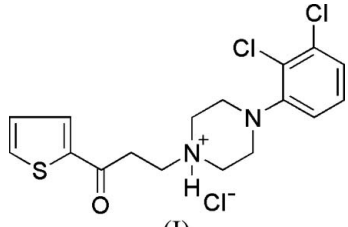
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
R factor = 0.058
wR factor = 0.139
Data-to-parameter ratio = 17.8

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

The title compound, $C_{17}H_{19}Cl_2N_2OS^+\cdot Cl^-$, was synthesized from 2-acetothiophene, 1-(2,3-dichlorophenyl)piperazine and paraformaldehyde. In the cation, the thienyl ring is coplanar with the oxopropyl group. The protonated piperazine ring exhibits a chair conformation. In the crystal packing the cations are connected by C—H···O and C—H···Cl hydrogen bonds.

Comment

3-(4-Arylpiperazin-1-yl)-1-arylpropane derivatives are a class of selective antidepressants with a low incidence of side effects. However, their therapeutic effect is achieved only after repeated administration (Oficialdegui *et al.*, 2000; Martinez *et al.*, 2001; Esparza *et al.*, 2001; Orus *et al.*, 2002).



(I)

The title compound, (I), was synthesized from 2-acetothiophene, 1-(2,3-dichlorophenyl)piperazine and paraformaldehyde. The molecular structure of (I) is illustrated in Fig. 1. The thiopene ring is coplanar with the oxopropyl group (O1/C5/C6/C7), the average deviation of contributing atoms from the least-squares plane being 0.0172 (2) Å. The protonated piperazine ring is in a normal chair conformation. In the crystal packing, the cations are linked by C—H···O and C—H···Cl hydrogen bonds (Fig. 2 and Table 1).

Experimental

A mixture of 2-acetothiophene (15 mmol), 1-(2,3-dichlorophenyl)-piperazine (15 mmol) and concentrated hydrochloric acid in absolute

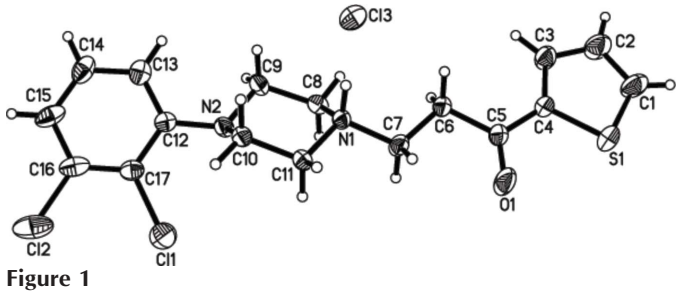


Figure 1
A view of (I), showing 30% probability displacement ellipsoids.

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ethanol (20 ml) was refluxed at 351 K. Paraformaldehyde (45 mmol) was added over a period of 20 min and the mixture was refluxed for 12 h. The solution was cooled to room temperature, and acetone (20 ml) was added with stirring. The resulting solid was filtered off, washed with diethyl ether and dried in a vacuum to give the title compound. Crystals suitable for X-ray analysis were grown by slow evaporation of an absolute methanol solution at room temperature over a period of 15 days.

Crystal data

$C_{17}H_{19}Cl_2N_2OS^+\cdot Cl^-$
$M_r = 405.75$
Orthorhombic, *Pbca*
$a = 15.400\ (2)$ Å
$b = 7.4361\ (11)$ Å
$c = 33.601\ (5)$ Å
$V = 3847.9\ (9)$ Å^3
$Z = 8$
$D_x = 1.401$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6615 reflections
$\theta = 2.4$–26.4°
$\mu = 0.59$ mm^{-1}
$T = 294\ (2)$ K
Block, colourless
$0.22 \times 0.18 \times 0.16$ mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
$T_{min} = 0.850$, $T_{max} = 0.910$
20192 measured reflections
3934 independent reflections
2963 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 26.4^\circ$
$h = -11 \rightarrow 19$
$k = -9 \rightarrow 8$
$l = -42 \rightarrow 33$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.139$
$S = 1.12$
3934 reflections
221 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.044P)^2 + 4.8252P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.35$ e Å^{-3}
$\Delta\rho_{min} = -0.31$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11B···O1^{i}	0.97	2.50	3.002 (4)	112
C9—H9B···Cl2^{ii}	0.97	2.75	3.606 (3)	147

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

The H atom attached to the N atom was located in a difference density map and refined isotropically. All other H atoms were positioned geometrically and refined as riding, with C—H = 0.93–0.97 Å and $U_{iso}(H) = 1.2\ U_{eq}(C)$.

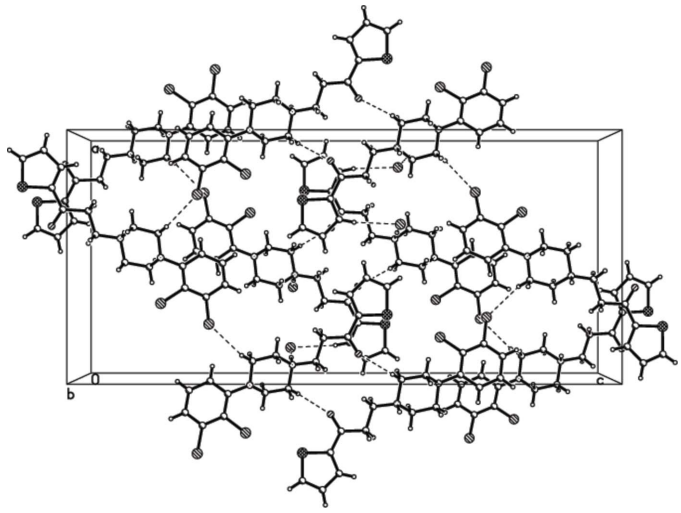

Figure 2
The crystal structure of (I), viewed along the *b* axis, showing the C—H···O and C—H···Cl hydrogen bonds (dashed lines). Cl^- anions have been omitted for clarity.

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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