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2-(2,4-Dichloro-5-fluorophenyl)-4-(2-hydroxyethyl)morpholin-4-ium chloride

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The title compound, $C_{12}H_{15}Cl_2FNO_2^+\cdot Cl^-$, has been synthesized from 2-(2,4-dichloro-5-fluorophenyl)-4-(2-hydroxyethyl)morpholin-2-ol *via* reduction by formic acid and acidification by hydrogen chloride. The morpholine unit adopts an almost ideal chair conformation. The crystal structure is consolidated by N—H···Cl and O—H···Cl intermolecular hydrogen bonding.

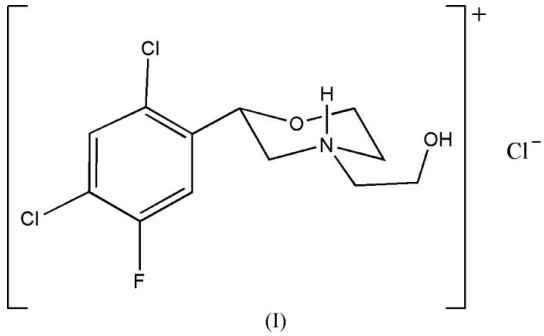
Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.035
wR factor = 0.091
Data-to-parameter ratio = 17.7

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

Comment

The 2-arylmorpholine unit has been identified as a central structural element of a number of biologically active compounds. For example, the biologically active compounds and drugs Phenmetrazine, Phendimetrazine (anorectic agents) and Oxaflozane (antidepressant) include a morpholine ring in their structure. Based on the synthesis of 2-arylmorpholin-2-ol derivatives (Hu *et al.*, 2004), we synthesized the title compound, (I).



(I)

The molecular structure of (I) is illustrated in Fig. 1. The morpholine ring system is in a chair conformation. The equatorial 2,4-dichloro-5-fluorophenyl and 4-(2-hydroxyethyl) groups are on the same side of the morpholine ring. The N atom of the morpholine ring and the O atom of the hydroxy group in the molecule act as a hydrogen-bond donors to the Cl^- anion (Table 1). The combination of both N—H···Cl and O—H···Cl hydrogen bonds generates a centroymmetric $R_4^2(14)$ (Bernstein *et al.*, 1995) aggregate of two cations and two anions (Fig. 2).

Experimental

2-Bromo-1-(2,4-dichloro-5-fluorophenyl)ethanone (0.01 mol) was added to a solution of diethanolamine (0.04 mol) in 10 ml *N*-methyl-2-pyrrolidone (NMP) (Perrine *et al.*, 2000), and stirred for 1 h at 333 K. Formic acid (88%, 0.04 mol) was then added and the mixture was refluxed for 11 h. After cooling to room temperature, the mixture was treated with 10% HCl solution, and extracted three times with

50 ml diethyl ether. The ether extracts were discarded and the aqueous phase was cooled and made basic with 30% NaOH solution. The mixture was extracted three times with 60 ml diethyl ether. The ether extracts were combined and dried over anhydrous Na_2SO_4. The mixture was filtered and the filtrate was stirred in an ice bath. Anhydrous HCl was passed through slowly, yielding colorless crystals of (I) (yield 56%).

Crystal data

$C_{12}H_{15}Cl_2FNO_2^+\cdot Cl^-$
M_r = 330.60
Monoclinic, $P2_1/c$
a = 14.1063 (8) Å
b = 7.2927 (4) Å
c = 13.8748 (8) Å
β = 98.834 (1)°
V = 1410.41 (14) Å^3
Z = 4
D_x = 1.557 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.66 mm^{-1}
T = 294 (2) K
Block, colorless
0.48 × 0.45 × 0.40 mm

Data collection

Bruker SMART 1000 CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.735, T_{max} = 0.771
11388 measured reflections
3064 independent reflections
2288 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 27.1°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.091
S = 1.05
3064 reflections
173 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0365P)^2 + 0.6923P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···Cl3	0.91	2.21	3.0886 (15)	163
O2—H2···Cl3^{i}	0.82	2.30	3.1178 (18)	173

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

H atoms were positioned geometrically, with C—H = 0.98 (methine), 0.97 (methylene) or 0.93 Å (aromatic), O—H = 0.82 and N—H = 0.91 Å, and refined in riding mode, with U_{iso}(H) = 1.2U_{eq}(C,N) or 1.5U_{eq}(O).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; 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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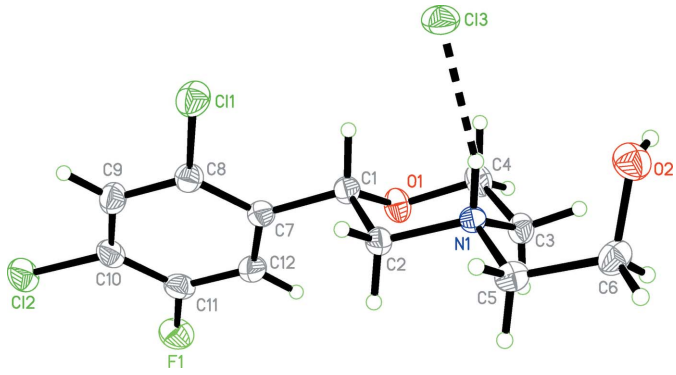

Figure 1
The molecular structure of (I), shown with 30% probability displacement ellipsoids (arbitrary spheres for H atoms). The dashed line indicates a hydrogen bond.

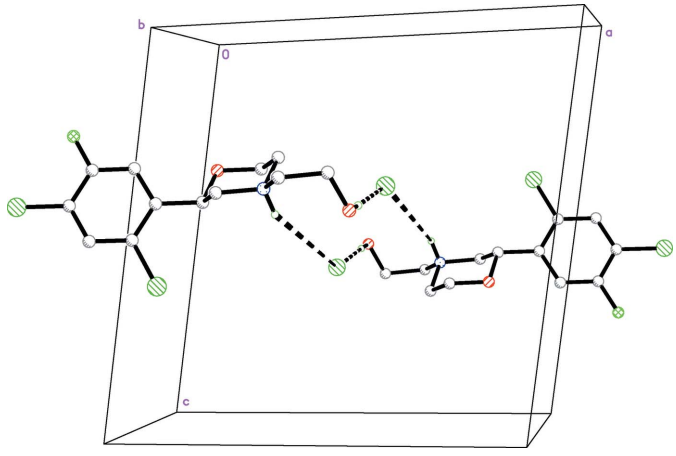

Figure 2
A packing diagram for (I). H atoms bonded to C atoms have been omitted for clarity. Dashed lines indicate hydrogen bonds.

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