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N-Ethylethanaminium 3,5,6-trichloropyridin-2-olate

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In the title salt, $C_4H_{12}N^+\cdot C_5HCl_3NO^-$, the non-H atoms of the cation are essentially coplanar. Intermolecular N—H···O hydrogen bonds link two cations and two anions into a centrosymmetric cluster. The crystal packing is further stabilized by van der Waals forces.

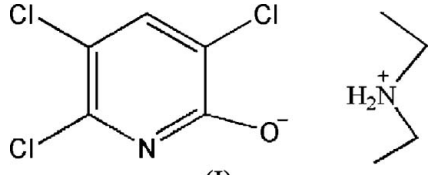
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.116
Data-to-parameter ratio = 21.6

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

Comment

The title compound, (I), was prepared as an intermediate for the synthesis of 3,5,6-trichloropyridin-2-ol-containing compounds, which exhibit potential bioactivity, such as chloropyrifos (Fakhraian *et al.*, 2004), chloropyrifos methyl (Baughman, 1989) and triclopyr (Fox *et al.*, 2002). The title compound was obtained by mixing sodium 3,5,6-trichloropyridin-2-olate with *N*-ethylethanaminium chloride and crystallized from diethyl ether.



(I)

In the *N*-ethylethanaminium cation of (I), the non-H atoms form a serrated structure with C6—C7—N2 and C7—N2—C8 angles of 111.5 (2) and 112.56 (19)° (Table 1), respectively. The 3,5,6-trichloropyridin-2-olate anion, with an O1—C1 distance of 1.2699 (17) Å, is essentially planar, with an O1—C1—C2—Cl1 torsion angle of 0.7 (2)°. In the crystal structure, intermolecular N—H···O hydrogen bonds (Table 2) link two cations and two anions into a centrosymmetric cluster (Fig. 1). The crystal packing is further stabilized by van der Waals forces.

Experimental

Sodium 3,5,6-trichloropyridin-2-olate (1.10 g, 5 mmol) was dissolved in distilled water (20 ml) at 373 K, the solution was cooled to room temperature, and then *N*-ethylethanaminium, which was generated from diethylamine (1.0 ml, 10 mmol) with HCl (36%, 1.0 ml), was added dropwise with stirring for 0.5 h. The solution was extracted with diethyl ether (2 × 15 ml) and dried over anhydrous magnesium sulfate. Suitable crystals were obtained from a diethyl ether solution (m.p. 399–400 K).

Crystal data

$C_4H_{12}N^+\cdot C_5HCl_3NO^-$
M_r = 271.57
Triclinic, $P\overline{1}$
a = 7.527 (6) Å
b = 9.196 (6) Å
c = 10.978 (7) Å
α = 106.36 (2)°
β = 105.32 (3)°
γ = 105.28 (3)°
V = 654.6 (8) Å^3
Z = 2
D_x = 1.378 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5373 reflections
θ = 3.0–27.5°
μ = 0.68 mm^{-1}
T = 298 (1) K
Chunk, colourless
0.38 × 0.36 × 0.28 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.761, T_{max} = 0.827
6442 measured reflections
2964 independent reflections
2106 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.023
θ_{max} = 27.5°
$h = -9 \rightarrow 9$
$k = -11 \rightarrow 10$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.116
S = 1.01
2964 reflections
137 parameters
H-atom parameters constrained
$w = 1/[0.0011F_o^2 + \sigma(F_o^2)]/(4F_o^2)$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}
Extinction correction: Larson (1970)
Extinction coefficient: 42 (12)

Table 1
Selected geometric parameters (Å, °).

Cl1—C2	1.728 (2)	N1—C5	1.3181 (19)
Cl3—C5	1.735 (2)	N2—C7	1.480 (3)
O1—C1	1.2699 (17)	N2—C8	1.467 (3)
N1—C1	1.347 (2)	C1—C2	1.421 (2)
C1—N1—C5	120.24 (15)	N1—C1—C2	118.02 (13)
C7—N2—C8	112.56 (19)	Cl1—C2—C1	118.02 (12)
O1—C1—N1	118.98 (15)	Cl1—C2—C3	121.17 (15)
O1—C1—C2	123.00 (18)	N2—C7—C6	111.5 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H201···O1	0.90	1.88	2.7022 (19)	152
N2—H202···O1^{i}	0.90	1.87	2.748 (2)	166

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

All H atoms were placed in calculated positions, with C—H = 0.93–0.97 Å and N—H = 0.90 Å, and refined as riding with U_{iso}(H) = 1.17–1.34U_{eq}(carrier atom).

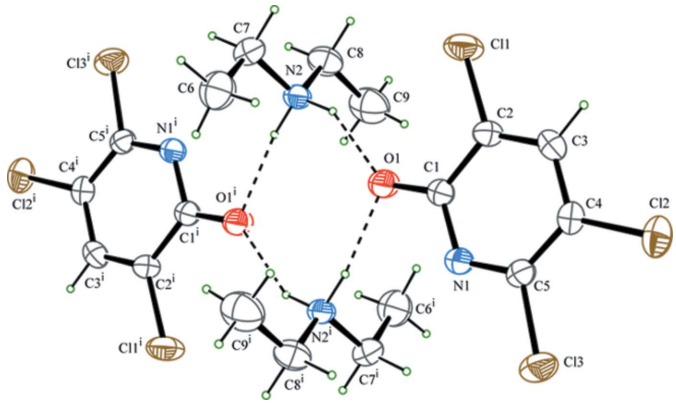


Figure 1
The centrosymmetric hydrogen-bonded (dashed lines) cluster in (I), showing the atom numbering scheme and 40% probability displacement ellipsoids [symmetry code: (i) $-x, -y, 1-z$].

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1999); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

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