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4-Carboxypyridazin-1-ium chloride

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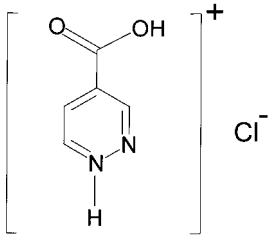
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.030; wR factor = 0.105; data-to-parameter ratio = 19.4.

The structure of the title compound, $C_5H_5N_2O_2^+\cdot Cl^-$, is composed of chloride anions and 4-carboxypyridazin-1-ium cations. Chloride anions bridge the cations *via* O—H···Cl and N—H···Cl hydrogen bonds to form ribbons. The latter, linked by van der Waals forces with lengths in the range 3.254 (2)–3.497 (2) Å, form coplanar layers. Very weak interactions operate also between adjacent layers, as indicated by their spacing of 3.339 (1) Å.

Related literature

For the crystal structure of pyridazine-3-carboxylic acid hydrochloride, see: Gryz *et al.* (2003). For a report of molecular layers in the structure of pyrazine-2-carboxylic acid, see: Takusagawa *et al.* (1974).



Experimental

Crystal data

$C_5H_5N_2O_2^+\cdot Cl^-$
M_r = 160.56
Monoclinic, $P2_1/n$
a = 6.8505 (14) Å
b = 6.5905 (13) Å
c = 14.561 (3) Å
β = 97.65 (3)°
V = 651.6 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.52 mm^{-1}
T = 293 (2) K
0.39 × 0.16 × 0.12 mm

Data collection

Kuma KM-4 four-circle diffractometer
Absorption correction: analytical (*CrysAlis RED*; Oxford Diffraction, 2008)
T_{min} = 0.942, T_{max} = 0.952
2062 measured reflections
1917 independent reflections
1318 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
3 standard reflections
every 200 reflections
intensity decay: 1.1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.104
S = 1.03
1917 reflections
99 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···Cl1	0.91 (3)	2.05 (3)	2.9464 (14)	169 (2)
N1—H1···Cl1^{i}	0.92 (3)	2.15 (3)	3.0373 (15)	160 (2)

Symmetry code: (i) $x+\frac{1}{2}, -y+\frac{3}{2}, z+\frac{1}{2}$.

Data collection: *KM-4 Software* (Kuma, 1996); cell refinement: *KM-4 Software*; data reduction: *DATAPROC* (Kuma, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RK2101).

References

Gryz, M., Starosta, W., Ptasiewicz-Bąk, H. & Leciejewicz, J. (2003). *J. Coord. Chem.* **56**, 1505–1511.
Kuma (1996). *KM-4 Software*. Kuma Diffraction Ltd, Wrocław, Poland.
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Takusagawa, F., Higuchi, T. & Shimada, A. (1974). *Bull. Chem. Soc. Jpn*, **47**, 1409–1414.

supplementary materials

Acta Cryst. (2008). E**64**, o1553 [**doi:10.1107/S1600536808022319**]

4-Carboxypyridazin-1-ium chloride

W. Starosta and J. Leciejewicz

Comment

The structure of the title compound $(C_5H_5N_2O_2)^+$ Cl^-, **I**, is built from chloride anions and heterocycle cations. Chloride anions bridge the cations *via* hydrogen bonds O2—H2···Cl1 2.05 (3)Å and N1—H1···$Cl1^{i}$ 2.15 (3)Å to form ribbons; symmetry code: (i) x+1/2, -y+3/2, z+1/2. The ribbons linked by van der Waals forces with lengths in the range from 3.254 (2) to 3.497 (2)Å make coplanar layers. The shortest distance between pyridazine rings belonging to adjacent layers is 3.339 (1)Å. The pyridazine ring are planar (r.m.s. 0.0060Å) and formes with the carboxylate group (C7/O1/O2) dihedral angle 27.7 (1)°. Bond lengths and bond angles within the cation agree well with those reported in the structure of pyridazine–3–carboxylic acid hydrochloride (Gryz *et al.*, 2003).

Experimental

Single crystals of **I** were obtained by recrystallization of pyridazine–4–carboxylic acid (ALDRICH) from warm 1*M* solution of hydrochloric acid. Attempts to recrystallize from water and alcohols yielded specimens unsuitable for collecting X–ray data.

Refinement

All H atoms bonded with C atoms were positioned geometrically and refined in riding model approximation with C—H = 0.93Å and $U_{iso}(H) = 1.2U_{eq}(C)$. The H atoms connected with N and O atoms were located in difference Fourier map and refined isotropically.

Figures

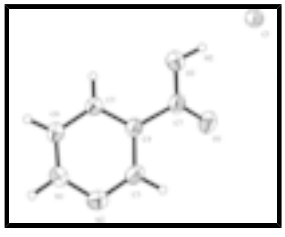

Fig. 1. A molecular structure of **I** with the atom labelling scheme. The displacement ellipsoids are drawn at 50% probability level. H atoms are presented as a small spheres of arbitrary radius.

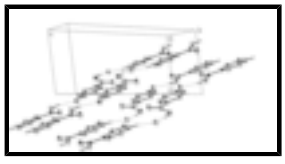

Fig. 2. The structure packing diagram of **I**.

4-Carboxypyridazin-1-ium chloride

Crystal data

$C_5H_5N_2O_2^+ \cdot Cl^-$	$F_{000} = 328$
$M_r = 160.56$	$D_x = 1.637$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 6.8505$ (14) Å	Cell parameters from 6 reflections
$b = 6.5905$ (13) Å	$\theta = 6$–15º
$c = 14.561$ (3) Å	$\mu = 0.52$ mm^{-1}
$\beta = 97.65$ (3)º	$T = 293$ (2) K
$V = 651.6$ (2) Å^3	Block, colourless
$Z = 4$	0.39 × 0.16 × 0.12 mm

Data collection

Kuma KM-4 four-circle diffractometer	$R_{int} = 0.024$
Radiation source: Fine–focus sealed tube	$\theta_{max} = 30.1$º
Monochromator: Graphite	$\theta_{min} = 2.8$º
$T = 293$(2) K	$h = -9 \rightarrow 0$
Profile data from ω/2θ scans	$k = 0 \rightarrow 9$
Absorption correction: Analytical (CrysAlis RED; Oxford Diffraction, 2008)	$l = -20 \rightarrow 20$
$T_{min} = 0.942$, $T_{max} = 0.952$	3 standard reflections
2062 measured reflections	every 200 reflections
1917 independent reflections	intensity decay: 1.2%
1318 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: Difmap
Least-squares matrix: Full	Hydrogen site location: Geom
$R[F^2 > 2\sigma(F^2)] = 0.030$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.104$	$w = 1/[\sigma^2(F_o^2) + (0.0577P)^2 + 0.1527P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} = 0.001$
1917 reflections	$\Delta\rho_{max} = 0.36$ e Å^{-3}
99 parameters	$\Delta\rho_{min} = -0.21$ e Å^{-3}
Primary atom site location: Direct	Extinction correction: None

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.

Refinement. Refinement of *F*^2^ against ALL reflections. The weighted *R*–factor *wR* and goodness of fit *S* are based on *F*^2^, conventional *R*–factors *R* are based on *F*, with *F* set to zero for negative *F*^2^. The threshold expression of *F*^2^ > σ(*F*^2^) is used only for calculating *R*–factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*–factors based on *F*^2^ are statistically about twice as large as those based on *F*, and *R*–factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.61082 (6)	0.18289 (6)	0.39362 (3)	0.03696 (13)
O1	0.8041 (2)	0.6663 (2)	0.46266 (8)	0.0485 (3)
C3	0.8870 (3)	0.9337 (3)	0.61752 (11)	0.0382 (4)
H3	0.8395	0.9943	0.5613	0.046*
C4	0.8811 (2)	0.7225 (2)	0.62339 (10)	0.0296 (3)
O2	0.7273 (2)	0.4276 (2)	0.56133 (8)	0.0414 (3)
C7	0.7988 (2)	0.6028 (3)	0.53974 (10)	0.0330 (3)
C5	0.9550 (2)	0.6321 (2)	0.70515 (11)	0.0337 (3)
H5	0.9548	0.4918	0.7120	0.040*
N2	0.9559 (2)	1.0516 (2)	0.68707 (10)	0.0414 (3)
C6	1.0306 (3)	0.7589 (3)	0.77771 (11)	0.0376 (4)
H6	1.0841	0.7045	0.8345	0.045*
N1	1.0257 (2)	0.9555 (2)	0.76522 (10)	0.0368 (3)
H1	1.063 (3)	1.042 (4)	0.8143 (17)	0.063 (7)*
H2	0.675 (4)	0.358 (4)	0.5097 (19)	0.069 (8)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0472 (2)	0.02641 (19)	0.0353 (2)	0.00189 (16)	−0.00212 (15)	−0.00355 (14)
O1	0.0698 (9)	0.0472 (8)	0.0264 (6)	0.0011 (7)	−0.0010 (5)	0.0038 (5)
C3	0.0507 (10)	0.0319 (8)	0.0309 (7)	0.0058 (7)	0.0017 (7)	0.0058 (6)
C4	0.0313 (7)	0.0310 (7)	0.0259 (6)	0.0027 (6)	0.0021 (5)	0.0015 (5)
O2	0.0567 (8)	0.0354 (6)	0.0303 (6)	−0.0046 (6)	−0.0010 (5)	−0.0028 (5)
C7	0.0371 (8)	0.0336 (8)	0.0267 (7)	0.0064 (6)	−0.0017 (6)	0.0005 (6)
C5	0.0406 (8)	0.0291 (7)	0.0295 (7)	0.0003 (6)	−0.0025 (6)	0.0030 (5)
N2	0.0554 (9)	0.0296 (7)	0.0386 (7)	0.0022 (7)	0.0041 (6)	0.0022 (5)
C6	0.0463 (9)	0.0349 (8)	0.0288 (7)	−0.0010 (7)	−0.0045 (6)	0.0028 (6)
N1	0.0443 (8)	0.0340 (7)	0.0315 (6)	−0.0034 (6)	0.0023 (5)	−0.0034 (5)

Geometric parameters (Å, °)

O1—C7	1.203 (2)	O2—H2	0.91 (3)

C3—N2	1.313 (2)	C5—C6	1.392 (2)
C3—C4	1.395 (2)	C5—H5	0.9300
C3—H3	0.9300	N2—N1	1.334 (2)
C4—C5	1.367 (2)	C6—N1	1.308 (2)
C4—C7	1.497 (2)	C6—H6	0.9300
O2—C7	1.309 (2)	N1—H1	0.92 (3)
N2—C3—C4	123.60 (15)	C4—C5—C6	117.17 (16)
N2—C3—H3	118.2	C4—C5—H5	121.4
C4—C3—H3	118.2	C6—C5—H5	121.4
C5—C4—C3	118.55 (15)	C3—N2—N1	115.32 (14)
C5—C4—C7	122.31 (15)	N1—C6—C5	119.27 (15)
C3—C4—C7	119.12 (14)	N1—C6—H6	120.4
C7—O2—H2	111.4 (17)	C5—C6—H6	120.4
O1—C7—O2	126.14 (16)	C6—N1—N2	126.06 (15)
O1—C7—C4	121.39 (16)	C6—N1—H1	120.2 (16)
O2—C7—C4	112.47 (13)	N2—N1—H1	113.5 (16)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···Cl1	0.91 (3)	2.05 (3)	2.9464 (14)	169 (2)
N1—H1···Cl1^{i}	0.92 (3)	2.15 (3)	3.0373 (15)	160 (2)

Symmetry codes: (i) *x*+1/2, −*y*+3/2, *z*+1/2.

Fig. 1

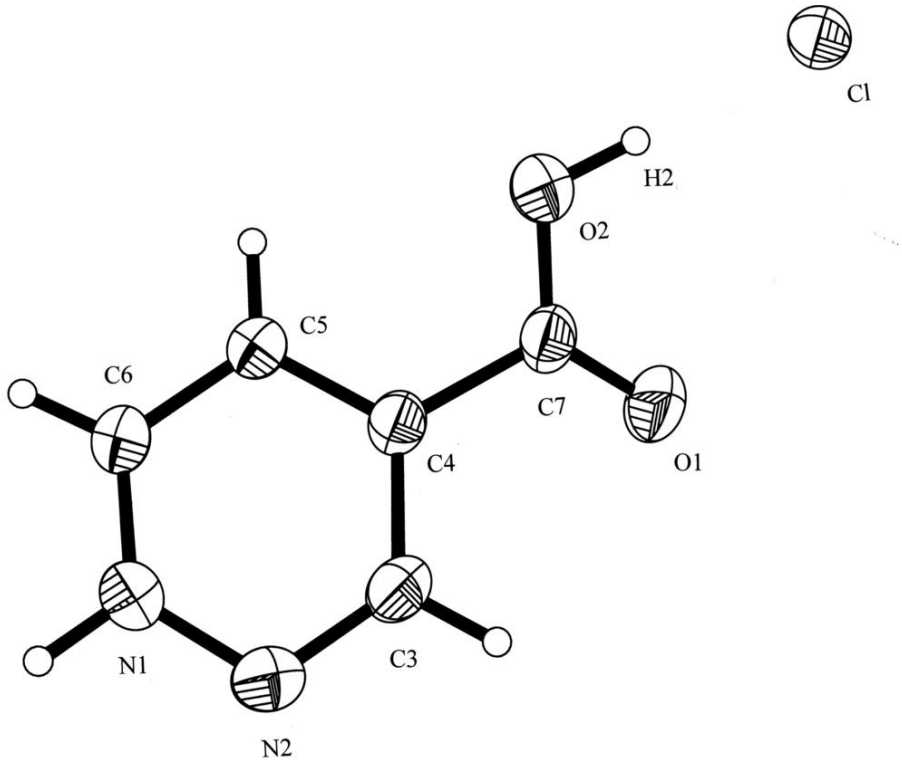

Fig. 2