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Ammonium 4-chloropyridine-3-sulfonate

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The title compound, $NH_4^+\cdot C_5H_3ClNO_3S^-$, was prepared by the hydrolysis of 4-chloropyridine-3-sulfonamide. In the crystal structure, a three-dimensional network is formed *via* N—H···O [H···O = 1.97 (3)–2.41 (2) Å] and N—H···N [H···N = 2.13 (3) Å] hydrogen bonds.

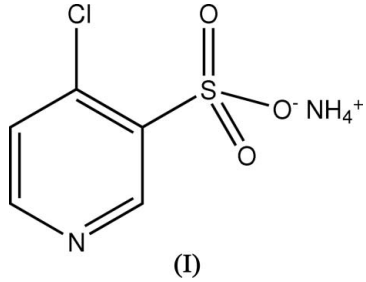
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.030
wR factor = 0.084
Data-to-parameter ratio = 13.3

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

Comment

The title compound, (I), is a key intermediate of in the synthesis of torsemide (Che *et al.*, 2005), a loop diuretic, which has been found to be effective in the treatment of edema associated with chronic renal failure.



(I)

The molecular structure of (I) is shown in Fig. 1, selected torsion angles are given in Table 1 and details of the hydrogen-bonding geometry are given in Table 2 and Fig. 2.

Experimental

4-Chloropyridine-3-sulfonamide (3.0 g) was dissolved in water (20 ml) and stirred for 15 min. Colorless crystals of the title compound suitable for X-ray diffraction analysis were obtained after two weeks (m.p. 499–501 K).

Crystal data

$NH_4^+\cdot C_5H_3ClNO_3S^-$
M_r = 210.64
Monoclinic, $P2_1/c$
a = 8.3501 (6) Å
b = 7.6684 (6) Å
c = 13.3162 (10) Å
β = 97.6170 (10)°
V = 845.14 (11) Å^3
Z = 4
D_x = 1.655 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2851 reflections
θ = 4.9–56.4°
μ = 0.67 mm^{-1}
T = 293 (2) K
Prism, colorless
0.50 × 0.48 × 0.24 mm

Data collection

Bruker SMART CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.719, T_{max} = 0.850
4804 measured reflections
1835 independent reflections
1628 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 27.0°
$h = -10 \rightarrow 10$
$k = -9 \rightarrow 9$
$l = -16 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.084$
$S = 1.07$
1835 reflections
138 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0476P)^2 + 0.1484P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.042 (3)

Table 1
Selected torsion angles (°).

C5—N1—C1—C2	0.6 (3)	S—C2—C3—Cl	1.5 (2)
N1—C1—C2—C3	−0.9 (3)	C2—C3—C4—C5	−0.3 (3)
N1—C1—C2—S	178.43 (13)	Cl—C3—C4—C5	179.66 (14)
C1—C2—C3—C4	0.7 (2)	C1—N1—C5—C4	−0.1 (3)
S—C2—C3—C4	−178.61 (13)	C3—C4—C5—N1	0.0 (3)
C1—C2—C3—Cl	−179.25 (12)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O2	0.78 (3)	2.16 (3)	2.894 (2)	159 (2)
N2—H3···O2^{i}	0.85 (2)	2.18 (2)	2.929 (2)	148 (2)
N2—H3···O3^{ii}	0.85 (2)	2.41 (2)	2.916 (2)	118.9 (19)
N2—H6···N1^{iii}	0.86 (3)	2.13 (3)	2.944 (2)	156 (2)
N2—H7···O1^{iv}	0.91 (3)	1.97 (3)	2.877 (2)	173 (2)

Symmetry codes: (i) $-x+2, y+\frac{1}{2}, -z+\frac{3}{2}$; (ii) $x, y+1, z$; (iii) $-x+1, -y+1, -z+1$; (iv) $-x+2, -y+1, -z+1$.

All H atoms were refined independently with isotropic displacement parameters [C—H = 0.90 (2)—0.937 (19) Å and N—H = 0.78 (3)–0.91 (3) Å].

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

References

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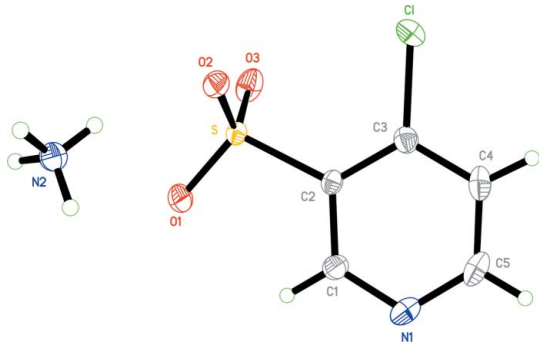

Figure 1
The structure of (I), showing displacement ellipsoids drawn at the 40% probability level. H atoms are represented by circles of arbitrary size.

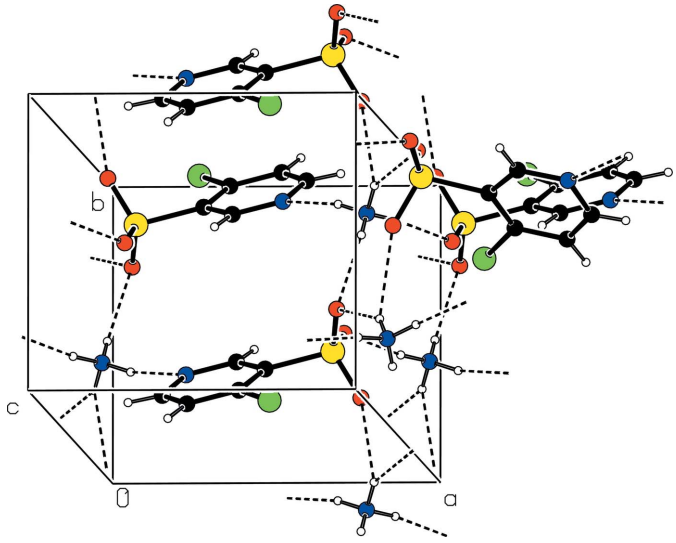

Figure 2
Partial packing plot (Spek, 2003), showing hydrogen bonds as dashed lines. Color codes: green Cl, yellow S, red O, blue N and black C.

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