organic papers

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.047
wR factor = 0.133
Data-to-parameter ratio = 13.8

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

4-Chloropyridine-3-sulfonic acid

The title compound, $C_5H_4ClNO_3S$, was obtained by hydrolysis of 4-chloropyridine-3-sulfonamide in dilute hydrochloric acid. In the crystal structure, one-dimensional chains are formed *via* N—H···O hydrogen bonds. In addition, weak C—H···Cl hydrogen bonds link these chains into a two-dimensional network

Comment

The title compound, (I), is a key intermediate 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)
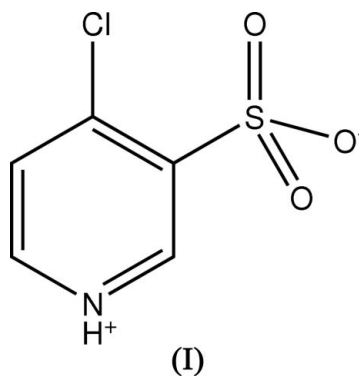
The molecular structure of (I) is shown in Fig. 1, selected torsion angles are given in Table 1 and details of the hydrogen-bonding geometry are given in Table 2 and Fig. 2. The structure of a related compound, ammonium 4-chloropyridine-3-sulfonate, is given in the preceding paper (Li *et al.*, 2006).

Experimental

4-Chloropyridine-3-sulfonamide (3.0 g) was dissolved in water (20 ml) and stirred for 15 min; hydrochloric acid (1 ml, 36%) was then added and the resulting solution stirred for 10 min. Colorless crystals of the title compound suitable for X-ray diffraction analysis were obtained after two weeks (m.p. 591–594 K).

Crystal data

$C_5H_4ClNO_3S$	D_x = 1.933 Mg m^{-3}
M_r = 193.60	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 2647
a = 7.1433 (7) Å	reflections
b = 6.8424 (7) Å	θ = 6.0–56.5°
c = 13.7397 (14) Å	μ = 0.83 mm^{-1}
β = 97.783 (2)°	T = 293 (2) K
V = 665.37 (12) Å^3	Prism, colorless
Z = 4	0.49 × 0.31 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer	1450 independent reflections
φ and ω scans	1343 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan	$R_{int} = 0.118$
(*SADABS*; Bruker, 2000)	$\theta_{max} = 27.0°$
$T_{min} = 0.674$, $T_{max} = 0.890$	$h = -9 \rightarrow 9$
3744 measured reflections	$k = -8 \rightarrow 8$
	$l = -17 \rightarrow 9$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0698P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.047$	$+ 0.0236P]$
$wR(F^2) = 0.133$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.10$	$(\Delta/\sigma)_{max} = 0.001$
1450 reflections	$\Delta\rho_{max} = 0.68$ e Å^{-3}
105 parameters	$\Delta\rho_{min} = -0.62$ e Å^{-3}
H atoms treated by a mixture of independent and constrained refinement	Extinction correction: *SHELXL97*
	Extinction coefficient: 0.097 (8)

Table 1
Selected torsion angles (°).

C5—N1—C1—C2	−2.3 (3)	S—C2—C3—Cl	2.2 (3)
N1—C1—C2—C3	−1.4 (3)	C2—C3—C4—C5	−2.5 (3)
N1—C1—C2—S	−179.05 (16)	Cl—C3—C4—C5	176.63 (18)
C1—C2—C3—C4	3.7 (3)	C1—N1—C5—C4	3.6 (4)
S—C2—C3—C4	−178.74 (16)	C3—C4—C5—N1	−1.2 (3)
C1—C2—C3—Cl	−175.36 (15)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C5—H5···Cl^i	0.93	2.74	3.639 (2)	162
N1—H1A···O1^i	0.94 (3)	1.88 (4)	2.775 (2)	159 (3)
N1—H1A···S^i	0.94 (3)	2.88 (3)	3.705 (2)	147 (2)

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

H1*A* was located in a Fourier difference map and then refined isotropically. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances of 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT*; 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: *PLATON* (Spek, 2003).

References

Bruker (2000). *SMART, SAINT, SADABS* and *SHELXTL*. Bruker AXS inc., Madison, Wisconsin, USA.

Che, D., Guntoori, B. R. & Duncan, S. C. (2005). US Patent Appl. Publ. US2005209460 (5 pp.).
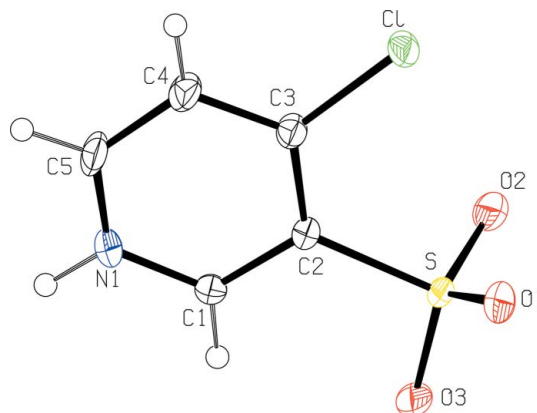
Figure 1
The molecular structure of (I), showing displacement ellipsoids drawn at the 40% probability level. H atoms are represented by circles of arbitrary size.
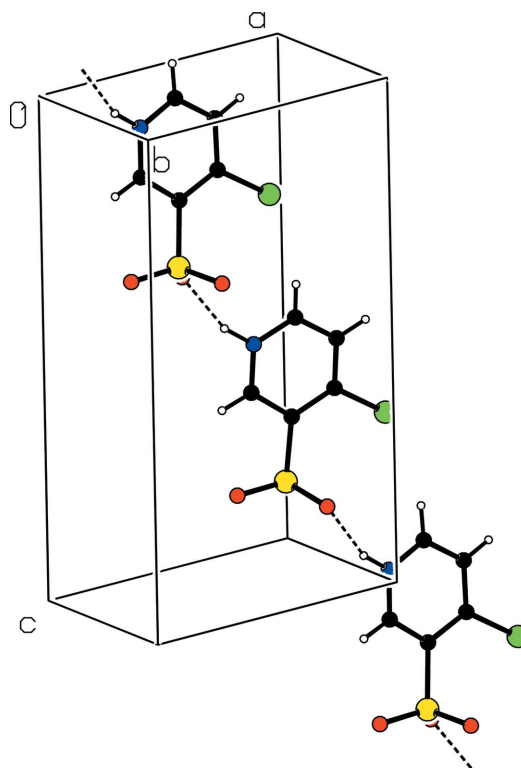

Figure 2
Partial packing plot (Spek, 2003), showing hydrogen bonds as dashed lines. Color codes: green Cl, yellow S, red O, blue N and black C.

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