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4-(4-Nitrobenzenesulfonamido)pyridinium chloride

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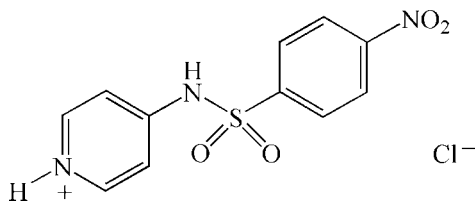
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Key indicators: single-crystal X-ray study; $T = 113$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.042; wR factor = 0.107; data-to-parameter ratio = 15.2.

In the title compound, $\text{C}_{11}\text{H}_{10}\text{N}_3\text{O}_4\text{S}^+\text{Cl}^-$, the benzene ring makes an angle of $89.2(1)^\circ$ with the pyridinium ring. The dihedral angle between the nitro group and the benzene ring is $15.7(1)^\circ$. The crystal structure is stabilized by $\text{N}-\text{H}\cdots\text{Cl}$ hydrogen bonds.

Related literature

For zwitterionic forms of *N*-arylbenzenesulfonamides, see: Li *et al.* (2007); Yu & Li (2007). For reference geometric data, see: Allen *et al.* (1987). Damiano *et al.* (2007) describe the use of pyridinium derivatives for the construction of supra-molecular architectures.



Experimental

Crystal data

$\text{C}_{11}\text{H}_{10}\text{N}_3\text{O}_4\text{S}^+\text{Cl}^-$
 $M_r = 315.73$
 Monoclinic, $C2/c$
 $a = 37.942(8)$ Å
 $b = 5.2446(10)$ Å
 $c = 13.713(3)$ Å
 $\beta = 107.77(3)^\circ$

$V = 2598.5(9)$ Å³
 $Z = 8$
 Mo $K\alpha$ radiation
 $\mu = 0.47$ mm⁻¹
 $T = 113(2)$ K
 $0.12 \times 0.10 \times 0.08$ mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
 Absorption correction: multi-scan (*CrystalClear*; Rigaku/MS, 2005)
 $T_{\min} = 0.932$, $T_{\max} = 0.963$

9693 measured reflections
 2865 independent reflections
 2330 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.043$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.107$
 $S = 1.06$
 2865 reflections
 189 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.39$ e Å⁻³
 $\Delta\rho_{\min} = -0.48$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1}\cdots\text{Cl1}^{\text{i}}$	0.93 (3)	2.12 (3)	3.039 (2)	171 (3)
$\text{N2}-\text{H2A}\cdots\text{Cl1}^{\text{ii}}$	0.89 (3)	2.18 (3)	3.066 (2)	173 (3)

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

Data collection: *CrystalClear* (Rigaku/MS, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2146).

References

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supplementary materials

Acta Cryst. (2008). E64, o2091 [doi:10.1107/S1600536808032054]

4-(4-Nitrobenzenesulfonamido)pyridinium chloride

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Comment

Organic pyridinium salts have been widely used in the construction of supramolecular architectures (Damiano *et al.*, 2007). As part of our ongoing studies of supramolecular chemistry involving the pyridinium rings (Li *et al.*, 2007), the structure of the title compound was determined by X-ray diffraction. In the cations of the title compound the short C—N distance [N2—C1 = 1.394 (3) Å] has a value between those of a typical C=N double and C—N single bond (1.47–1.50 Å and 1.34–1.38 Å, respectively; Allen *et al.*, 1987). This might be indicative of a slight conjugation of the sulphonamide π electrons N with those of the pyridinium ring. The benzene ring exhibits an angle of 89.2 (1)° with the pyridinium ring. The dihedral angle between the nitro group and the benzene ring is 164.3 (1)°. The crystal packing is stabilized by N—H \cdots Cl hydrogen bonds (Table 1). Fig. 2 showing supramolecular chains linked by N—H \cdots Cl hydrogen bonds.

Experimental

A solution of 4-nitrobenzenesulfonyl chloride (2.2 g, 10 mmol) in CH₂Cl₂ (10 ml) was added dropwise to a suspension of 4-aminopyridine (0.9 g, 10 mmol) in CH₂Cl₂ (10 ml) at room temperature with stirring. The reaction mixture was stirred overnight. The yellow solid obtained was washed with warm water to obtain the title compound in a yield of 61.6%. A colorless single-crystal suitable for X-ray analysis was obtained by slow evaporation of an hydrochloric acid (10%) solution at room temperature over a period of a week. Analysis calculated for C₁₁H₁₀N₃O₄SCl: C 41.84, H 3.19, N 13.31%; found: C 41.95, H 3.52, N 13.69%.

Refinement

The N-bound H atoms were located in a difference map and their coordinates were refined with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$. The C-bound H atoms were positioned geometrically (C—H = 0.95 Å) and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

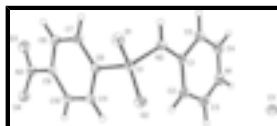


Fig. 1. View of one molecule of the title compound showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 35% probability level (arbitrary spheres for the H atoms).

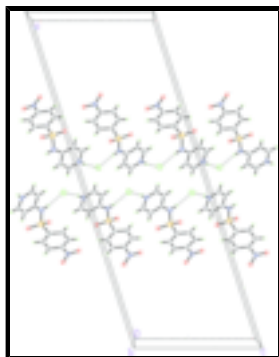


Fig. 2. The packing of title compound, view down the *b* axis, showing supramolecular chains linked by N—H...Cl hydrogen bonds which are indicated by dashed lines.

4-(4-Nitrobenzenesulfonamido)pyridinium chloride

Crystal data

$C_{11}H_{10}N_3O_4S^+ \cdot Cl^-$

$M_r = 315.73$

Monoclinic, $C2/c$

$a = 37.942(8) \text{ \AA}$

$b = 5.2446(10) \text{ \AA}$

$c = 13.713(3) \text{ \AA}$

$\beta = 107.77(3)^\circ$

$V = 2598.5(9) \text{ \AA}^3$

$Z = 8$

$F_{000} = 1296$

$D_x = 1.614 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 3179 reflections

$\theta = 2.6\text{--}27.1^\circ$

$\mu = 0.47 \text{ mm}^{-1}$

$T = 113(2) \text{ K}$

Block, colourless

$0.12 \times 0.10 \times 0.08 \text{ mm}$

Data collection

Rigaku Saturn CCD area-detector diffractometer

Radiation source: Rotating anode

Monochromator: confocal

Detector resolution: $7.21 \text{ pixels mm}^{-1}$

$T = 113(2) \text{ K}$

ω and φ scans

Absorption correction: multi-scan (CrystalClear; Rigaku/MSO, 2005)

$T_{\min} = 0.932$, $T_{\max} = 0.963$

9693 measured reflections

2865 independent reflections

2330 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.043$

$\theta_{\max} = 27.1^\circ$

$\theta_{\min} = 2.3^\circ$

$h = -35 \rightarrow 48$

$k = -6 \rightarrow 6$

$l = -17 \rightarrow 13$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.042$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$wR(F^2) = 0.107$	$w = 1/[\sigma^2(F_o^2) + (0.0506P)^2 + 1.967P]$
$S = 1.06$	where $P = (F_o^2 + 2F_c^2)/3$
2865 reflections	$(\Delta/\sigma)_{\max} = 0.001$
189 parameters	$\Delta\rho_{\max} = 0.39 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\min} = -0.48 \text{ e } \text{\AA}^{-3}$
	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.130148 (12)	0.37275 (10)	0.40268 (4)	0.02104 (16)
O1	0.12396 (4)	0.1690 (3)	0.32996 (12)	0.0268 (4)
O2	0.14305 (4)	0.3226 (3)	0.51052 (12)	0.0271 (4)
O3	0.21998 (4)	1.2270 (3)	0.21517 (12)	0.0320 (4)
O4	0.25562 (4)	1.2246 (3)	0.37259 (12)	0.0334 (4)
N1	0.05555 (5)	1.1001 (4)	0.52726 (16)	0.0297 (5)
N2	0.09049 (4)	0.5214 (4)	0.37499 (15)	0.0215 (4)
N3	0.22877 (5)	1.1462 (3)	0.30326 (14)	0.0246 (4)
C1	0.08046 (5)	0.7177 (4)	0.42979 (16)	0.0217 (4)
C2	0.10010 (6)	0.7864 (4)	0.52953 (17)	0.0253 (5)
H2	0.1227	0.7032	0.5645	0.030*
C3	0.08643 (6)	0.9760 (4)	0.57664 (18)	0.0284 (5)
H3	0.0992	1.0196	0.6456	0.034*
C4	0.03605 (6)	1.0410 (5)	0.43021 (19)	0.0323 (5)
H4	0.0141	1.1327	0.3966	0.039*
C5	0.04777 (5)	0.8496 (4)	0.38013 (18)	0.0277 (5)
H5	0.0338	0.8060	0.3120	0.033*
C6	0.16110 (5)	0.5922 (4)	0.37472 (16)	0.0190 (4)
C7	0.15830 (5)	0.6369 (4)	0.27247 (16)	0.0221 (5)
H7	0.1409	0.5446	0.2196	0.027*
C8	0.18104 (5)	0.8169 (4)	0.24842 (16)	0.0225 (4)
H8	0.1797	0.8499	0.1793	0.027*
C9	0.20570 (5)	0.9469 (4)	0.32797 (16)	0.0196 (4)
C10	0.20940 (5)	0.9012 (4)	0.43010 (16)	0.0229 (5)
H10	0.2272	0.9914	0.4827	0.028*

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C11	0.18659 (5)	0.7207 (4)	0.45380 (16)	0.0223 (4)
H11	0.1884	0.6856	0.5231	0.027*
C11	0.038070 (13)	0.52540 (11)	0.65583 (4)	0.02598 (16)
H1	0.0477 (8)	1.233 (6)	0.561 (2)	0.058 (9)*
H2A	0.0771 (8)	0.504 (6)	0.310 (2)	0.059 (9)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0197 (2)	0.0190 (3)	0.0238 (3)	0.00150 (18)	0.00570 (19)	0.0027 (2)
O1	0.0272 (7)	0.0204 (8)	0.0333 (9)	0.0011 (6)	0.0101 (6)	-0.0028 (7)
O2	0.0281 (7)	0.0289 (9)	0.0233 (9)	0.0018 (6)	0.0062 (6)	0.0100 (7)
O3	0.0388 (8)	0.0284 (9)	0.0310 (10)	-0.0013 (7)	0.0137 (7)	0.0059 (8)
O4	0.0313 (8)	0.0372 (10)	0.0321 (10)	-0.0125 (7)	0.0103 (7)	-0.0109 (8)
N1	0.0333 (10)	0.0246 (11)	0.0368 (12)	-0.0034 (8)	0.0190 (9)	-0.0056 (9)
N2	0.0189 (8)	0.0234 (10)	0.0207 (10)	0.0006 (7)	0.0038 (7)	-0.0008 (8)
N3	0.0261 (8)	0.0216 (10)	0.0287 (11)	-0.0001 (7)	0.0124 (7)	-0.0033 (8)
C1	0.0194 (9)	0.0231 (11)	0.0246 (11)	-0.0038 (8)	0.0097 (8)	0.0007 (9)
C2	0.0272 (10)	0.0249 (12)	0.0236 (12)	-0.0005 (8)	0.0074 (8)	0.0023 (10)
C3	0.0326 (11)	0.0301 (13)	0.0249 (12)	-0.0068 (9)	0.0126 (9)	-0.0016 (10)
C4	0.0261 (10)	0.0329 (14)	0.0392 (15)	0.0050 (9)	0.0117 (10)	-0.0031 (11)
C5	0.0209 (9)	0.0322 (13)	0.0281 (13)	0.0016 (8)	0.0046 (8)	-0.0034 (10)
C6	0.0185 (8)	0.0186 (11)	0.0195 (11)	0.0033 (7)	0.0055 (7)	0.0012 (8)
C7	0.0227 (9)	0.0222 (12)	0.0205 (11)	0.0000 (8)	0.0050 (8)	-0.0036 (9)
C8	0.0260 (9)	0.0239 (12)	0.0183 (11)	0.0013 (8)	0.0081 (8)	-0.0001 (9)
C9	0.0181 (8)	0.0205 (11)	0.0213 (11)	0.0009 (7)	0.0078 (7)	-0.0003 (9)
C10	0.0193 (9)	0.0277 (12)	0.0197 (11)	0.0008 (8)	0.0030 (8)	-0.0008 (9)
C11	0.0206 (9)	0.0275 (12)	0.0176 (11)	0.0011 (8)	0.0041 (7)	0.0013 (9)
Cl1	0.0256 (3)	0.0283 (3)	0.0228 (3)	0.00156 (19)	0.0056 (2)	-0.0007 (2)

Geometric parameters (\AA , $^\circ$)

S1—O1	1.4311 (16)	C2—H2	0.9500
S1—O2	1.4331 (16)	C3—H3	0.9500
S1—N2	1.6332 (17)	C4—C5	1.365 (3)
S1—C6	1.768 (2)	C4—H4	0.9500
O3—N3	1.226 (2)	C5—H5	0.9500
O4—N3	1.233 (2)	C6—C11	1.388 (3)
N1—C3	1.330 (3)	C6—C7	1.393 (3)
N1—C4	1.347 (3)	C7—C8	1.385 (3)
N1—H1	0.93 (3)	C7—H7	0.9500
N2—C1	1.394 (3)	C8—C9	1.381 (3)
N2—H2A	0.89 (3)	C8—H8	0.9500
N3—C9	1.468 (3)	C9—C10	1.385 (3)
C1—C2	1.391 (3)	C10—C11	1.387 (3)
C1—C5	1.402 (3)	C10—H10	0.9500
C2—C3	1.371 (3)	C11—H11	0.9500
O1—S1—O2	120.92 (10)	N1—C4—C5	120.1 (2)

O1—S1—N2	104.52 (10)	N1—C4—H4	120.0
O2—S1—N2	109.13 (10)	C5—C4—H4	120.0
O1—S1—C6	108.27 (10)	C4—C5—C1	119.6 (2)
O2—S1—C6	107.63 (10)	C4—C5—H5	120.2
N2—S1—C6	105.35 (9)	C1—C5—H5	120.2
C3—N1—C4	121.6 (2)	C11—C6—C7	121.73 (19)
C3—N1—H1	118.5 (18)	C11—C6—S1	119.82 (16)
C4—N1—H1	119.9 (18)	C7—C6—S1	118.42 (15)
C1—N2—S1	127.65 (15)	C8—C7—C6	119.50 (19)
C1—N2—H2A	117 (2)	C8—C7—H7	120.3
S1—N2—H2A	112.8 (19)	C6—C7—H7	120.3
O3—N3—O4	123.83 (19)	C9—C8—C7	118.0 (2)
O3—N3—C9	118.12 (18)	C9—C8—H8	121.0
O4—N3—C9	118.04 (18)	C7—C8—H8	121.0
C2—C1—N2	124.68 (19)	C8—C9—C10	123.26 (19)
C2—C1—C5	118.6 (2)	C8—C9—N3	118.51 (19)
N2—C1—C5	116.69 (19)	C10—C9—N3	118.22 (18)
C3—C2—C1	119.0 (2)	C9—C10—C11	118.50 (19)
C3—C2—H2	120.5	C9—C10—H10	120.8
C1—C2—H2	120.5	C11—C10—H10	120.8
N1—C3—C2	121.1 (2)	C10—C11—C6	118.97 (19)
N1—C3—H3	119.5	C10—C11—H11	120.5
C2—C3—H3	119.5	C6—C11—H11	120.5
O1—S1—N2—C1	172.50 (18)	O2—S1—C6—C7	168.84 (15)
O2—S1—N2—C1	41.8 (2)	N2—S1—C6—C7	-74.81 (18)
C6—S1—N2—C1	-73.5 (2)	C11—C6—C7—C8	-1.0 (3)
S1—N2—C1—C2	-14.1 (3)	S1—C6—C7—C8	176.94 (15)
S1—N2—C1—C5	167.25 (16)	C6—C7—C8—C9	-0.4 (3)
N2—C1—C2—C3	-177.14 (19)	C7—C8—C9—C10	1.8 (3)
C5—C1—C2—C3	1.5 (3)	C7—C8—C9—N3	-177.26 (17)
C4—N1—C3—C2	1.5 (3)	O3—N3—C9—C8	15.3 (3)
C1—C2—C3—N1	-2.4 (3)	O4—N3—C9—C8	-165.44 (18)
C3—N1—C4—C5	0.1 (3)	O3—N3—C9—C10	-163.75 (18)
N1—C4—C5—C1	-1.0 (4)	O4—N3—C9—C10	15.5 (3)
C2—C1—C5—C4	0.1 (3)	C8—C9—C10—C11	-1.7 (3)
N2—C1—C5—C4	178.9 (2)	N3—C9—C10—C11	177.29 (17)
O1—S1—C6—C11	-145.42 (16)	C9—C10—C11—C6	0.3 (3)
O2—S1—C6—C11	-13.15 (19)	C7—C6—C11—C10	1.1 (3)
N2—S1—C6—C11	103.20 (18)	S1—C6—C11—C10	-176.88 (15)
O1—S1—C6—C7	36.57 (18)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1 \cdots C11 ⁱ	0.93 (3)	2.12 (3)	3.039 (2)	171 (3)
N2—H2A \cdots C11 ⁱⁱ	0.89 (3)	2.18 (3)	3.066 (2)	173 (3)

Symmetry codes: (i) $x, y+1, z$; (ii) $x, -y+1, z-1/2$.

Fig. 1

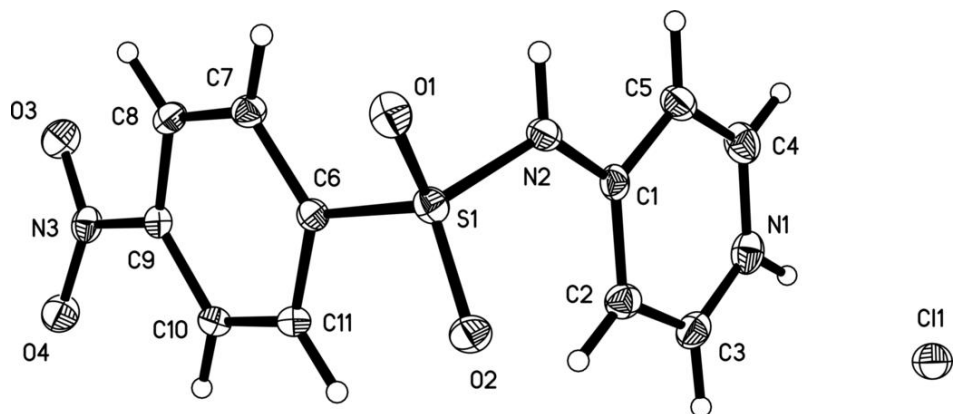


Fig. 2

