

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

4-(4-Nitrobenzenesulfonamido)-pyridinium bromide

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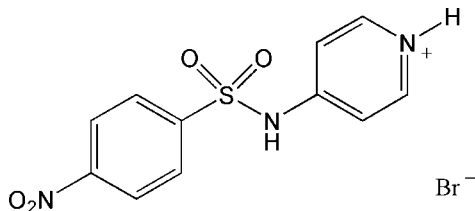
Received 19 October 2008; accepted 29 October 2008

Key indicators: single-crystal X-ray study; $T = 113$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.033; wR factor = 0.076; data-to-parameter ratio = 16.8.

In the title compound, $\text{C}_{11}\text{H}_{10}\text{N}_3\text{O}_4\text{S}^+\cdot\text{Br}^-$, the benzene ring makes an angle of $88.4(2)^\circ$ with the pyridinium ring. The dihedral angle between the nitro group and the benzene ring is $16.5(2)^\circ$. The ions in the crystal structure are linked by a combination of intermolecular $\text{N}-\text{H}\cdots\text{Br}$ and non-conventional $\text{C}-\text{H}\cdots\text{Br}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, forming a three-dimensional network.

Related literature

For zwitterionic forms of *N*-arylbenzenesulfonamides, see: Li *et al.* (2007); Yu & Li (2007). For bond-length data, see: Allen *et al.* (1987). For non-conventional hydrogen bonds, see: Desiraju & Steiner (2001). For the use of pyridinium derivatives in the construction of supramolecular architectures, see: Damiano *et al.* (2007).



Experimental

Crystal data

 $\text{C}_{11}\text{H}_{10}\text{N}_3\text{O}_4\text{S}^+\cdot\text{Br}^-$ $M_r = 360.19$

Monoclinic, $C2/c$
 $a = 38.242(8)$ Å
 $b = 5.2852(11)$ Å
 $c = 13.941(3)$ Å
 $\beta = 108.18(3)^\circ$
 $V = 2677.0(11)$ Å³

$Z = 8$
Mo $K\alpha$ radiation
 $\mu = 3.24$ mm⁻¹
 $T = 113(2)$ K
 $0.10 \times 0.04 \times 0.02$ mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MS, 2005)
 $T_{\min} = 0.710$, $T_{\max} = 0.938$

10460 measured reflections
3174 independent reflections
2635 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.050$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.076$
 $S = 1.05$
3174 reflections
189 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.68$ e Å⁻³
 $\Delta\rho_{\min} = -0.47$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1A}\cdots\text{Br1}^i$	0.89 (3)	2.30 (3)	3.195 (2)	173 (3)
$\text{N2}-\text{H2A}\cdots\text{Br1}$	0.84 (3)	2.38 (3)	3.225 (3)	174 (2)
$\text{C10}-\text{H10}\cdots\text{O3}^{ii}$	0.95	2.44	3.301 (3)	151
$\text{C5}-\text{H5}\cdots\text{Br1}^{iii}$	0.95	2.75	3.676 (3)	165

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

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: SI2122).

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supplementary materials

Acta Cryst. (2008). E64, o2308 [doi:10.1107/S1600536808035265]

4-(4-Nitrobenzenesulfonamido)pyridinium bromide

L. Zhao, Q.-F. Yu and Q. Wu

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), an X-ray structure analysis of the title compound has been performed. In the cations of the title compound the short C—N distance [N2—C3 = 1.387 (3) Å] has a value between those of a typical C=N double and C—N single bond (1.34–1.38 Å and 1.47–1.50 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 makes an angle of 88.4 (2) ° with the pyridinium ring. The dihedral angle between the nitro group and the benzene ring is 163.5 (2) °. The S atom has a tetrahedral geometry and the Br anion link the cationic molecule into chains along the *c* axis. The ions in the crystal structure are linked by a combination of intermolecular N—H \cdots Br and non-conventional C—H \cdots Br and C—H \cdots O hydrogen bonds (Table 1) to form a three-dimensional network (Desiraju & Steiner, 2001).

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 60.6%. A colorless single-crystal suitable for X-ray analysis was obtained by slow evaporation of an hydrobromic acid (5%) solution at room temperature over a period of a week. Analysis calculated for C₁₁H₁₀N₃O₄SBr: C 36.68, H 2.80, N 11.67%; found: C 36.70, H 2.52, N 11.98%.

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

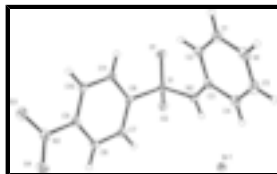


Fig. 1. View of one molecule of the title compound showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 35% probability level (arbitrary spheres for the H atoms).

4-(4-Nitrobenzenesulfonamido)pyridinium bromide

Crystal data

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

$M_r = 360.19$

Monoclinic, $C2/c$

Hall symbol: $-C\ 2yc$

$a = 38.242\ (8)\ \text{\AA}$

$b = 5.2852\ (11)\ \text{\AA}$

$c = 13.941\ (3)\ \text{\AA}$

$\beta = 108.18\ (3)^\circ$

$V = 2677.0\ (11)\ \text{\AA}^3$

$Z = 8$

$F_{000} = 1440$

$D_x = 1.787\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation

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

Cell parameters from 3479 reflections

$\theta = 2.2\text{--}27.9^\circ$

$\mu = 3.24\ \text{mm}^{-1}$

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

Needle, colorless

$0.10 \times 0.04 \times 0.02\ \text{mm}$

Data collection

Rigaku Saturn CCD area-detector
diffractometer

Radiation source: rotating anode

Monochromator: confocal

Detector resolution: $7.31\ \text{pixels mm}^{-1}$

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

ω and φ scans

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

$T_{\min} = 0.710$, $T_{\max} = 0.938$

10460 measured reflections

3174 independent reflections

2635 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.050$

$\theta_{\max} = 27.9^\circ$

$\theta_{\min} = 2.2^\circ$

$h = -45 \rightarrow 50$

$k = -6 \rightarrow 4$

$l = -18 \rightarrow 18$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.033$

$wR(F^2) = 0.076$

$S = 1.05$

3174 reflections

189 parameters

Primary atom site location: structure-invariant direct
methods

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

$$w = 1/[\sigma^2(F_o^2) + (0.0332P)^2]$$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.68\ \text{e \AA}^{-3}$

$\Delta\rho_{\min} = -0.47\ \text{e \AA}^{-3}$

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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.038458 (6)	-0.03831 (5)	0.156635 (19)	0.02228 (9)
S1	0.134013 (15)	-0.12886 (11)	0.40908 (5)	0.01823 (14)
O1	0.14776 (4)	-0.1752 (3)	0.51529 (13)	0.0248 (4)
O2	0.12760 (4)	-0.3323 (3)	0.33820 (13)	0.0240 (4)
O3	0.25567 (5)	0.7285 (4)	0.36991 (13)	0.0304 (4)
O4	0.21939 (5)	0.7239 (3)	0.21551 (13)	0.0277 (4)
N1	0.05972 (6)	0.5912 (4)	0.53090 (18)	0.0279 (5)
N2	0.09433 (5)	0.0162 (4)	0.38299 (17)	0.0191 (4)
N3	0.22900 (5)	0.6463 (4)	0.30319 (16)	0.0207 (4)
C1	0.09132 (7)	0.4746 (5)	0.5789 (2)	0.0271 (6)
H1	0.1046	0.5230	0.6460	0.033*
C2	0.10479 (6)	0.2877 (5)	0.53330 (18)	0.0227 (5)
H2	0.1277	0.2101	0.5672	0.027*
C3	0.08437 (6)	0.2113 (5)	0.43571 (18)	0.0196 (5)
C4	0.05109 (6)	0.3348 (5)	0.38843 (19)	0.0261 (6)
H4	0.0365	0.2863	0.3224	0.031*
C5	0.03964 (7)	0.5254 (5)	0.4376 (2)	0.0304 (6)
H5	0.0173	0.6115	0.4053	0.036*
C6	0.16392 (6)	0.0923 (4)	0.37923 (18)	0.0165 (5)
C7	0.16066 (6)	0.1306 (5)	0.27787 (18)	0.0192 (5)
H7	0.1436	0.0342	0.2268	0.023*
C8	0.18247 (6)	0.3099 (5)	0.25237 (17)	0.0186 (5)
H8	0.1808	0.3389	0.1838	0.022*
C9	0.20687 (6)	0.4465 (4)	0.32927 (18)	0.0164 (5)
C10	0.21101 (6)	0.4056 (5)	0.43050 (18)	0.0188 (5)
H10	0.2285	0.4995	0.4815	0.023*
C11	0.18912 (6)	0.2252 (5)	0.45572 (17)	0.0193 (5)
H11	0.1914	0.1931	0.5244	0.023*
H1A	0.0529 (8)	0.723 (6)	0.561 (2)	0.040 (8)*
H2A	0.0799 (8)	-0.010 (5)	0.324 (2)	0.018 (7)*

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.02226 (14)	0.02479 (15)	0.01857 (15)	-0.00186 (10)	0.00461 (11)	-0.00007 (10)
S1	0.0162 (3)	0.0167 (3)	0.0210 (3)	0.0022 (2)	0.0046 (2)	0.0039 (2)
O1	0.0228 (8)	0.0270 (10)	0.0216 (10)	0.0012 (8)	0.0025 (7)	0.0111 (8)
O2	0.0219 (8)	0.0179 (9)	0.0323 (10)	0.0006 (7)	0.0085 (8)	-0.0030 (8)
O3	0.0290 (9)	0.0333 (11)	0.0296 (11)	-0.0136 (8)	0.0099 (8)	-0.0083 (9)
O4	0.0351 (10)	0.0260 (10)	0.0230 (10)	-0.0022 (8)	0.0105 (8)	0.0055 (8)
N1	0.0323 (12)	0.0243 (12)	0.0317 (14)	-0.0043 (10)	0.0170 (11)	-0.0065 (10)
N2	0.0139 (10)	0.0224 (11)	0.0182 (12)	0.0011 (8)	0.0010 (9)	-0.0021 (9)
N3	0.0230 (10)	0.0178 (10)	0.0248 (12)	0.0004 (9)	0.0124 (9)	-0.0026 (9)
C1	0.0280 (14)	0.0316 (15)	0.0234 (15)	-0.0087 (11)	0.0104 (12)	-0.0043 (12)
C2	0.0209 (12)	0.0261 (13)	0.0209 (13)	-0.0024 (10)	0.0063 (11)	0.0011 (11)
C3	0.0186 (11)	0.0182 (12)	0.0250 (13)	-0.0050 (10)	0.0109 (10)	-0.0001 (10)
C4	0.0203 (12)	0.0310 (15)	0.0256 (15)	0.0033 (11)	0.0052 (11)	-0.0009 (12)
C5	0.0246 (13)	0.0292 (15)	0.0398 (17)	0.0059 (11)	0.0136 (13)	0.0012 (13)
C6	0.0137 (10)	0.0182 (12)	0.0163 (12)	0.0017 (9)	0.0029 (9)	0.0013 (9)
C7	0.0204 (11)	0.0185 (12)	0.0162 (13)	0.0008 (10)	0.0022 (10)	-0.0038 (10)
C8	0.0226 (11)	0.0208 (12)	0.0129 (11)	0.0003 (10)	0.0063 (10)	-0.0005 (10)
C9	0.0172 (11)	0.0146 (11)	0.0194 (13)	0.0029 (9)	0.0087 (10)	0.0011 (10)
C10	0.0160 (11)	0.0232 (12)	0.0149 (12)	0.0011 (9)	0.0015 (10)	-0.0018 (10)
C11	0.0175 (11)	0.0246 (13)	0.0146 (12)	0.0042 (10)	0.0032 (9)	0.0037 (10)

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

S1—O2	1.4288 (18)	C2—H2	0.9500
S1—O1	1.4292 (18)	C3—C4	1.399 (3)
S1—N2	1.637 (2)	C4—C5	1.366 (4)
S1—C6	1.773 (2)	C4—H4	0.9500
O3—N3	1.226 (3)	C5—H5	0.9500
O4—N3	1.232 (3)	C6—C11	1.385 (3)
N1—C5	1.333 (4)	C6—C7	1.394 (3)
N1—C1	1.335 (4)	C7—C8	1.380 (3)
N1—H1A	0.89 (3)	C7—H7	0.9500
N2—C3	1.387 (3)	C8—C9	1.385 (3)
N2—H2A	0.84 (3)	C8—H8	0.9500
N3—C9	1.468 (3)	C9—C10	1.387 (3)
C1—C2	1.359 (3)	C10—C11	1.385 (3)
C1—H1	0.9500	C10—H10	0.9500
C2—C3	1.400 (3)	C11—H11	0.9500
O2—S1—O1	121.05 (11)	C5—C4—C3	119.6 (3)
O2—S1—N2	104.58 (11)	C5—C4—H4	120.2
O1—S1—N2	109.04 (11)	C3—C4—H4	120.2
O2—S1—C6	108.52 (10)	N1—C5—C4	120.3 (2)
O1—S1—C6	107.48 (11)	N1—C5—H5	119.8
N2—S1—C6	105.09 (11)	C4—C5—H5	119.8

C5—N1—C1	121.6 (2)	C11—C6—C7	121.8 (2)
C5—N1—H1A	120.0 (19)	C11—C6—S1	119.90 (17)
C1—N1—H1A	118.3 (19)	C7—C6—S1	118.27 (18)
C3—N2—S1	128.28 (19)	C8—C7—C6	119.4 (2)
C3—N2—H2A	115.5 (17)	C8—C7—H7	120.3
S1—N2—H2A	114.9 (18)	C6—C7—H7	120.3
O3—N3—O4	123.7 (2)	C7—C8—C9	118.3 (2)
O3—N3—C9	118.3 (2)	C7—C8—H8	120.9
O4—N3—C9	117.9 (2)	C9—C8—H8	120.9
N1—C1—C2	121.1 (3)	C8—C9—C10	122.8 (2)
N1—C1—H1	119.4	C8—C9—N3	119.0 (2)
C2—C1—H1	119.4	C10—C9—N3	118.2 (2)
C1—C2—C3	119.1 (2)	C11—C10—C9	118.7 (2)
C1—C2—H2	120.5	C11—C10—H10	120.7
C3—C2—H2	120.5	C9—C10—H10	120.7
N2—C3—C4	117.2 (2)	C10—C11—C6	119.0 (2)
N2—C3—C2	124.6 (2)	C10—C11—H11	120.5
C4—C3—C2	118.2 (2)	C6—C11—H11	120.5
O2—S1—N2—C3	172.9 (2)	O1—S1—C6—C7	167.55 (17)
O1—S1—N2—C3	42.1 (2)	N2—S1—C6—C7	-76.4 (2)
C6—S1—N2—C3	-72.9 (2)	C11—C6—C7—C8	-1.6 (3)
C5—N1—C1—C2	1.7 (4)	S1—C6—C7—C8	177.10 (17)
N1—C1—C2—C3	-2.3 (4)	C6—C7—C8—C9	-0.2 (3)
S1—N2—C3—C4	168.56 (19)	C7—C8—C9—C10	1.9 (3)
S1—N2—C3—C2	-13.2 (3)	C7—C8—C9—N3	-177.11 (19)
C1—C2—C3—N2	-177.1 (2)	O3—N3—C9—C8	-164.7 (2)
C1—C2—C3—C4	1.1 (3)	O4—N3—C9—C8	16.1 (3)
N2—C3—C4—C5	178.9 (2)	O3—N3—C9—C10	16.2 (3)
C2—C3—C4—C5	0.5 (4)	O4—N3—C9—C10	-162.90 (19)
C1—N1—C5—C4	0.0 (4)	C8—C9—C10—C11	-1.8 (3)
C3—C4—C5—N1	-1.1 (4)	N3—C9—C10—C11	177.23 (19)
O2—S1—C6—C11	-146.30 (18)	C9—C10—C11—C6	0.0 (3)
O1—S1—C6—C11	-13.8 (2)	C7—C6—C11—C10	1.7 (3)
N2—S1—C6—C11	102.3 (2)	S1—C6—C11—C10	-176.96 (17)
O2—S1—C6—C7	35.0 (2)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1A \cdots Br1 ⁱ	0.89 (3)	2.30 (3)	3.195 (2)	173 (3)
N2—H2A \cdots Br1	0.84 (3)	2.38 (3)	3.225 (3)	174 (2)
C10—H10 \cdots O3 ⁱⁱ	0.95	2.44	3.301 (3)	151
C5—H5 \cdots Br1 ⁱⁱⁱ	0.95	2.75	3.676 (3)	165

Symmetry codes: (i) $x, -y+1, z+1/2$; (ii) $-x+1/2, -y+3/2, -z+1$; (iii) $-x, y+1, -z+1/2$.

Fig. 1

