

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

4-(4-Nitrobenzenesulfonamido)-pyridinium nitrate

Liang-Bin Hu,^a Yu-Xiang Ma,^b Chang-Zhong Liu^c and Ji-Guo Yang^{d*}

^aSchool of Food Science, Henan Institute of Science and Technology, Xinxiang 453003, People's Republic of China, ^bCollege of Grain and Food, Henan University of Technology, Zhengzhou 450052, People's Republic of China, ^cSchool of Animal Science, Henan Institute of Science and Technology, Xinxiang 453003, People's Republic of China, and ^dCollege of Light Industry and Food Science, South China University of Technology, Guangzhou 510640, People's Republic of China
Correspondence e-mail: jiguoyang@yahoo.cn

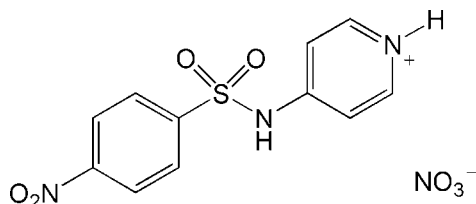
Received 11 November 2008; accepted 19 November 2008

Key indicators: single-crystal X-ray study; $T = 113$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.040; wR factor = 0.107; data-to-parameter ratio = 14.8.

A short C–N distance [1.394 (2) Å] in the title compound, $\text{C}_{11}\text{H}_{10}\text{N}_3\text{O}_4\text{S}^+\cdot\text{NO}_3^-$, is indicative of some conjugation of the sulfonamide π electrons with those of the pyridinium ring. The crystal structure is stabilized by N–H...O hydrogen bonds.

Related literature

For zwitterionic forms of *N*-arylbenzenesulfonamides, see: Li *et al.* (2007); Yu & Li (2007). For reference geometrical 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

 $\text{C}_{11}\text{H}_{10}\text{N}_3\text{O}_4\text{S}^+\cdot\text{NO}_3^-$ $M_r = 342.29$

Monoclinic, $C2/c$
 $a = 36.516$ (7) Å
 $b = 5.3742$ (11) Å
 $c = 13.964$ (3) Å
 $\beta = 99.54$ (3)°
 $V = 2702.5$ (10) Å³

$Z = 8$
Mo $K\alpha$ radiation
 $\mu = 0.29$ mm⁻¹
 $T = 113$ (2) K
 $0.20 \times 0.12 \times 0.04$ mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
 $T_{\min} = 0.94$, $T_{\max} = 0.99$

11804 measured reflections
3203 independent reflections
2648 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.037$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.107$
 $S = 1.09$
3203 reflections
216 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.30$ e Å⁻³
 $\Delta\rho_{\min} = -0.41$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N2}-\text{H2}\cdots\text{O5}$	0.79 (2)	1.92 (2)	2.7020 (19)	171 (2)
$\text{N1}-\text{H1}\cdots\text{O5}^i$	0.92 (2)	1.93 (2)	2.7764 (19)	151 (2)
$\text{N1}-\text{H1}\cdots\text{O6}^i$	0.92 (2)	2.18 (3)	2.979 (2)	144.6 (19)

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

Data collection: *CrystalClear* (Rigaku, 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: BG2225).

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Damiano, T., Morton, D. & Nelson, A. (2007). *Org. Biomol. Chem.* **5**, 2735–2752.
Li, J. S., Chen, L. G., Zhang, Y. Y., Xu, Y. J., Deng, Y. & Huang, P. M. (2007). *J. Chem. Res.* **6**, 350–352.
Rigaku (2005). *CrystalClear*. Rigaku/MSI Inc., The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
Yu, H.-J. & Li, J.-S. (2007). *Acta Cryst.* **E63**, o3399.

supplementary materials

Acta Cryst. (2008). E64, o2429 [doi:10.1107/S160053680803883X]

4-(4-Nitrobenzenesulfonamido)pyridinium nitrate

L.-B. Hu, Y.-X. Ma, C.-Z. Liu and J.-G. Yang

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 (2) 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 sulfonamide π electrons with those of the pyridinium ring. The dihedral angle between the benzene and the pyridinium rings is 81.3 (1)°, while the one between the nitro group and the benzene ring is 16.1 (1)°.

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 60.5%. A colorless single-crystal suitable for X-ray analysis was obtained by slow evaporation of an nitric acid (10%) solution at room temperature over a period of a week.

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 = 0.93 Å) and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

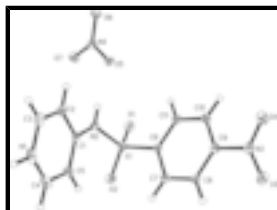


Fig. 1. View of one molecule of the title compound showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 35% probability level (arbitrary spheres for the H atoms).

4-(4-Nitrobenzenesulfonamido)pyridinium nitrate

Crystal data

C₁₁H₁₀N₃O₄S⁺·NO₃⁻

$M_r = 342.29$

$F_{000} = 1408$

$D_x = 1.683 \text{ Mg m}^{-3}$

supplementary materials

Monoclinic, $C2/c$

Hall symbol: $-C\ 2yc$

$a = 36.516\ (7)\ \text{\AA}$

$b = 5.3742\ (11)\ \text{\AA}$

$c = 13.964\ (3)\ \text{\AA}$

$\beta = 99.54\ (3)^\circ$

$V = 2702.5\ (10)\ \text{\AA}^3$

$Z = 8$

Mo $K\alpha$ radiation

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

Cell parameters from 4207 reflections

$\theta = 2.3\text{--}27.9^\circ$

$\mu = 0.29\ \text{mm}^{-1}$

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

Block, colorless

$0.20 \times 0.12 \times 0.04\ \text{mm}$

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, 2005)

$T_{\min} = 0.94$, $T_{\max} = 0.99$

11804 measured reflections

3203 independent reflections

2648 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.037$

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

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

$h = -47 \rightarrow 46$

$k = -7 \rightarrow 7$

$l = -14 \rightarrow 18$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.040$

$wR(F^2) = 0.107$

$S = 1.10$

3203 reflections

216 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

$$w = 1/[\sigma^2(F_o^2) + (0.0612P)^2 + 0.5371P]$$

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

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

$\Delta\rho_{\max} = 0.30\ \text{e \AA}^{-3}$

$\Delta\rho_{\min} = -0.41\ \text{e \AA}^{-3}$

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)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.133115 (11)	0.62515 (7)	0.45086 (3)	0.01800 (13)
O1	0.12702 (3)	0.8195 (2)	0.51613 (8)	0.0246 (3)
O2	0.14580 (3)	0.6801 (2)	0.36175 (8)	0.0256 (3)
O3	0.25592 (4)	-0.2298 (3)	0.63494 (9)	0.0323 (3)
O4	0.22026 (4)	-0.2269 (2)	0.74432 (8)	0.0285 (3)
N1	0.06215 (5)	-0.1019 (3)	0.23971 (11)	0.0287 (4)
N2	0.09333 (4)	0.4790 (2)	0.42774 (10)	0.0167 (3)
N3	0.22946 (4)	-0.1490 (3)	0.66941 (10)	0.0211 (3)
C1	0.08430 (4)	0.2815 (3)	0.36372 (10)	0.0158 (3)
C2	0.05600 (4)	0.1211 (3)	0.38117 (11)	0.0186 (3)
H2A	0.0441	0.1453	0.4361	0.022*
C3	0.04565 (5)	-0.0704 (3)	0.31855 (12)	0.0259 (4)
H3	0.0268	-0.1822	0.3304	0.031*
C4	0.08911 (5)	0.0488 (3)	0.22111 (12)	0.0280 (4)
H4	0.1000	0.0222	0.1648	0.034*
C5	0.10119 (5)	0.2409 (3)	0.28211 (11)	0.0223 (4)
H5	0.1208	0.3456	0.2694	0.027*
C6	0.16389 (4)	0.4066 (3)	0.51584 (11)	0.0170 (3)
C7	0.18870 (5)	0.2755 (3)	0.46944 (11)	0.0199 (3)
H7	0.1905	0.3098	0.4037	0.024*
C8	0.21072 (5)	0.0943 (3)	0.52069 (11)	0.0203 (3)
H8	0.2279	0.0017	0.4909	0.024*
C9	0.20707 (4)	0.0509 (3)	0.61656 (11)	0.0178 (3)
C10	0.18289 (5)	0.1823 (3)	0.66407 (11)	0.0196 (3)
H10	0.1813	0.1492	0.7301	0.023*
C11	0.16114 (5)	0.3626 (3)	0.61267 (11)	0.0195 (3)
H11	0.1443	0.4568	0.6432	0.023*
O5	0.06125 (3)	0.4370 (2)	0.58801 (8)	0.0229 (3)
O6	0.01603 (3)	0.5377 (2)	0.66295 (8)	0.0261 (3)
O7	0.02485 (4)	0.7446 (2)	0.53506 (8)	0.0284 (3)
N4	0.03315 (4)	0.5777 (2)	0.59463 (9)	0.0187 (3)
H1	0.0541 (7)	-0.225 (5)	0.1953 (16)	0.050 (7)*
H2	0.0832 (6)	0.483 (4)	0.4735 (16)	0.035 (6)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0163 (2)	0.0161 (2)	0.0213 (2)	-0.00180 (15)	0.00217 (14)	0.00219 (14)
O1	0.0227 (6)	0.0169 (6)	0.0324 (6)	-0.0003 (5)	-0.0007 (5)	-0.0045 (5)
O2	0.0219 (6)	0.0286 (7)	0.0270 (6)	-0.0035 (5)	0.0062 (5)	0.0110 (5)
O3	0.0306 (7)	0.0354 (7)	0.0299 (7)	0.0162 (6)	0.0023 (5)	-0.0040 (6)
O4	0.0333 (7)	0.0257 (7)	0.0257 (6)	0.0001 (6)	0.0029 (5)	0.0056 (5)

supplementary materials

N1	0.0367 (10)	0.0211 (8)	0.0229 (7)	0.0070 (7)	-0.0106 (6)	-0.0066 (6)
N2	0.0157 (7)	0.0185 (7)	0.0168 (6)	-0.0016 (5)	0.0049 (5)	-0.0014 (5)
N3	0.0216 (8)	0.0185 (7)	0.0216 (7)	0.0016 (6)	-0.0012 (5)	-0.0035 (5)
C1	0.0171 (7)	0.0150 (7)	0.0142 (7)	0.0036 (6)	-0.0009 (5)	0.0020 (6)
C2	0.0167 (8)	0.0189 (8)	0.0192 (7)	-0.0012 (6)	0.0000 (6)	0.0033 (6)
C3	0.0232 (9)	0.0214 (9)	0.0296 (9)	-0.0017 (7)	-0.0062 (7)	0.0016 (7)
C4	0.0365 (11)	0.0289 (9)	0.0174 (8)	0.0103 (8)	0.0013 (7)	-0.0014 (7)
C5	0.0253 (9)	0.0247 (9)	0.0174 (7)	0.0052 (7)	0.0052 (6)	0.0030 (6)
C6	0.0149 (8)	0.0173 (8)	0.0185 (7)	-0.0030 (6)	0.0015 (5)	-0.0006 (6)
C7	0.0179 (8)	0.0243 (9)	0.0183 (7)	-0.0018 (7)	0.0055 (6)	0.0005 (6)
C8	0.0169 (8)	0.0237 (9)	0.0212 (8)	0.0016 (7)	0.0058 (6)	-0.0039 (6)
C9	0.0155 (8)	0.0177 (8)	0.0193 (7)	-0.0017 (6)	0.0004 (6)	-0.0023 (6)
C10	0.0210 (8)	0.0220 (8)	0.0158 (7)	-0.0015 (7)	0.0032 (6)	-0.0023 (6)
C11	0.0192 (8)	0.0212 (9)	0.0185 (7)	0.0004 (6)	0.0048 (6)	-0.0046 (6)
O5	0.0238 (6)	0.0257 (6)	0.0203 (6)	0.0055 (5)	0.0068 (4)	0.0010 (5)
O6	0.0281 (7)	0.0321 (7)	0.0211 (6)	0.0029 (5)	0.0125 (5)	0.0057 (5)
O7	0.0311 (7)	0.0268 (7)	0.0281 (6)	0.0048 (6)	0.0070 (5)	0.0132 (5)
N4	0.0208 (7)	0.0192 (7)	0.0165 (6)	-0.0017 (6)	0.0040 (5)	0.0008 (5)

Geometric parameters (Å, °)

S1—O1	1.4277 (12)	C4—C5	1.364 (2)
S1—O2	1.4286 (12)	C4—H4	0.9500
S1—N2	1.6358 (14)	C5—H5	0.9500
S1—C6	1.7687 (17)	C6—C7	1.389 (2)
O3—N3	1.2281 (18)	C6—C11	1.393 (2)
O4—N3	1.2243 (17)	C7—C8	1.385 (2)
N1—C4	1.333 (3)	C7—H7	0.9500
N1—C3	1.350 (2)	C8—C9	1.387 (2)
N1—H1	0.92 (2)	C8—H8	0.9500
N2—C1	1.392 (2)	C9—C10	1.383 (2)
N2—H2	0.79 (2)	C10—C11	1.377 (2)
N3—C9	1.472 (2)	C10—H10	0.9500
C1—C2	1.398 (2)	C11—H11	0.9500
C1—C5	1.400 (2)	O5—N4	1.2902 (17)
C2—C3	1.363 (2)	O6—N4	1.2427 (16)
C2—H2A	0.9500	O7—N4	1.2270 (17)
C3—H3	0.9500		
O1—S1—O2	120.81 (7)	C5—C4—H4	119.6
O1—S1—N2	104.46 (7)	C4—C5—C1	119.05 (16)
O2—S1—N2	109.44 (8)	C4—C5—H5	120.5
O1—S1—C6	108.14 (7)	C1—C5—H5	120.5
O2—S1—C6	108.30 (8)	C7—C6—C11	121.62 (15)
N2—S1—C6	104.51 (7)	C7—C6—S1	120.35 (12)
C4—N1—C3	121.75 (15)	C11—C6—S1	117.96 (12)
C4—N1—H1	118.0 (14)	C8—C7—C6	118.90 (14)
C3—N1—H1	120.2 (14)	C8—C7—H7	120.5
C1—N2—S1	126.95 (11)	C6—C7—H7	120.5
C1—N2—H2	116.5 (16)	C7—C8—C9	118.47 (15)

S1—N2—H2	110.4 (16)	C7—C8—H8	120.8
O4—N3—O3	123.99 (14)	C9—C8—H8	120.8
O4—N3—C9	118.02 (14)	C10—C9—C8	123.25 (15)
O3—N3—C9	117.99 (13)	C10—C9—N3	118.54 (14)
N2—C1—C2	117.50 (13)	C8—C9—N3	118.19 (14)
N2—C1—C5	123.52 (15)	C11—C10—C9	117.92 (14)
C2—C1—C5	118.95 (14)	C11—C10—H10	121.0
C3—C2—C1	119.22 (15)	C9—C10—H10	121.0
C3—C2—H2A	120.4	C10—C11—C6	119.82 (15)
C1—C2—H2A	120.4	C10—C11—H11	120.1
N1—C3—C2	120.27 (17)	C6—C11—H11	120.1
N1—C3—H3	119.9	O7—N4—O6	123.22 (14)
C2—C3—H3	119.9	O7—N4—O5	119.33 (13)
N1—C4—C5	120.74 (16)	O6—N4—O5	117.45 (13)
N1—C4—H4	119.6		
O1—S1—N2—C1	176.50 (13)	O2—S1—C6—C11	167.74 (12)
O2—S1—N2—C1	45.81 (15)	N2—S1—C6—C11	-75.67 (14)
C6—S1—N2—C1	-69.99 (14)	C11—C6—C7—C8	1.1 (2)
S1—N2—C1—C2	154.93 (12)	S1—C6—C7—C8	-175.84 (12)
S1—N2—C1—C5	-27.1 (2)	C6—C7—C8—C9	0.0 (2)
N2—C1—C2—C3	178.18 (14)	C7—C8—C9—C10	-1.0 (2)
C5—C1—C2—C3	0.1 (2)	C7—C8—C9—N3	177.55 (15)
C4—N1—C3—C2	0.9 (3)	O4—N3—C9—C10	15.6 (2)
C1—C2—C3—N1	-1.1 (2)	O3—N3—C9—C10	-165.41 (15)
C3—N1—C4—C5	0.4 (3)	O4—N3—C9—C8	-163.03 (15)
N1—C4—C5—C1	-1.4 (3)	O3—N3—C9—C8	16.0 (2)
N2—C1—C5—C4	-176.80 (15)	C8—C9—C10—C11	0.8 (2)
C2—C1—C5—C4	1.2 (2)	N3—C9—C10—C11	-177.68 (14)
O1—S1—C6—C7	-147.78 (13)	C9—C10—C11—C6	0.3 (2)
O2—S1—C6—C7	-15.24 (16)	C7—C6—C11—C10	-1.2 (2)
N2—S1—C6—C7	101.35 (14)	S1—C6—C11—C10	175.76 (13)
O1—S1—C6—C11	35.21 (14)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2 \cdots O5	0.79 (2)	1.92 (2)	2.7020 (19)	171 (2)
N1—H1 \cdots O5 ⁱ	0.92 (2)	1.93 (2)	2.7764 (19)	151 (2)
N1—H1 \cdots O6 ⁱ	0.92 (2)	2.18 (3)	2.979 (2)	144.6 (19)

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

Fig. 1

