Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-(4-Nitrobenzenesulfonamido)-pyridinium trichloroacetate

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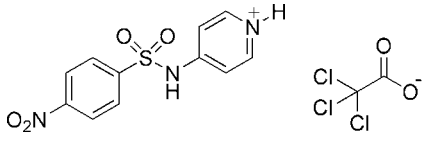
Received 7 November 2007; accepted 2 December 2007

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.004 Å; R factor = 0.039; wR factor = 0.094; data-to-parameter ratio = 14.6.

In the title compound, $C_{11}H_{10}N_3O_4S^+\cdot C_2Cl_3O_2^-$, the benzene ring forms an angle of 85.21 (13)° with the pyridinium ring. The nitro group is nearly coplanar with its attached benzene ring [dihedral angle = 3.68 (12)°]. In the crystal structure, strong N—H···O hydrogen bonds link the ion-pairs. The packing is further consolidated by weak C—H···O interations.

Related literature

For the synthesis and structure of the unprotonated amine, see: Yu & Li (2007). For reference geometrical data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{11}H_{10}N_3O_4S^+\cdot C_2Cl_3O_2^-$
M_r = 442.65
Monoclinic, $P2_1/n$
a = 5.9929 (11) Å
b = 17.790 (3) Å
c = 16.325 (3) Å
β = 98.377 (3)°
V = 1721.9 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.69 mm^{-1}
T = 294 (2) K
0.24 × 0.22 × 0.18 mm

Data collection

Bruker SMART 1K CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.852, T_{max} = 0.886
9742 measured reflections
3528 independent reflections
2427 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.094
S = 1.01
3528 reflections
241 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O6	0.87 (3)	1.93 (3)	2.803 (3)	172 (3)
N2—H2A···O6^{i}	0.79 (3)	2.00 (3)	2.785 (3)	171 (3)
C2—H2···O5^{i}	0.93	2.53	3.240 (3)	133
C3—H3···O1^{ii}	0.93	2.50	3.391 (4)	162
C8—H8···O5^{iii}	0.93	2.47	3.149 (3)	130
C10—H10···O3^{iv}	0.93	2.50	3.308 (4)	146
C5—H5···O2	0.93	2.33	2.978 (4)	126
C7—H7···O2	0.93	2.57	2.938 (3)	104

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

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

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2642).

References

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supplementary materials

Acta Cryst. (2008). E**64**, o254 [**doi:10.1107/S160053680706504X**]

4-(4-Nitrobenzenesulfonamido)pyridinium trichloroacetate

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Comment

The title compound, (I), comprises of a pyridinium cation and a trichloroacetate anion (Fig. 1). In the cation, the short C—N distance [N2—C1 = 1.390 (3) Å] occurs between typical C=N (1.34–1.38 Å) and C—N (1.47–1.50 Å) bond lengths (Allen *et al.*, 1987), indicative of significant double-bond character, despite of a strong electron-withdrawing sulfonyl group. The benzene ring forms an angle of 85.21 (13)° with the pyridinium ring. The nitro group is nearly coplanar and make an acute angle of 3.68 (12)° with the connected benzene ring.

The cation and anion are connected by a strong N—H···O hydrogen bond and weak C—H···O interactions (Table 1) complete the structure. Two short intramolecular C—H···O contacts also arise in the cation.

Experimental

4-Nitro-(*N*-pyridyl)benzenesulfonamide was prepared by the method of Yu & Li (2007). Colourless blocks of (I) were grown by natural evaporation from a trichloroacetic acid solution of the amide.

Refinement

The N-bound H atoms were located in a difference map and their positions were freely refined. The C-bound H atoms were positioned geometrically (C—H = 0.93 Å) and refined as riding atoms. The constraint Uĩso~(H) = 1.2 U~eq~(C and N) was applied.

Figures

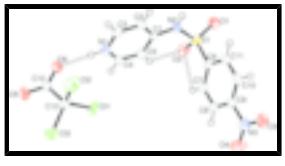


Fig. 1. A view of (I) with displacement ellipsoids drawn at the 50% probability level (arbitrary spheres for the H atoms). Hydrogen bonds are indicated by double-dashed lines.

4-(4-Nitrobenzenesulfonamido)pyridinium trichloroacetate

Crystal data

$C_{11}H_{10}N_3O_4S^+\cdot C_2Cl_3O_2^-$	F_{000} = 896
M_r = 442.65	D_x = 1.707 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 2690 reflections

a = 5.9929 (11) Å	θ = 2.5–25.7º
b = 17.790 (3) Å	μ = 0.69 mm^{-1}
c = 16.325 (3) Å	T = 294 (2) K
β = 98.377 (3)º	Block, colourless
V = 1721.9 (6) Å^3	0.24 × 0.22 × 0.18 mm
Z = 4	

Data collection

Bruker SMART 1K CCD diffractometer	3528 independent reflections
Radiation source: fine-focus sealed tube	2427 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.038
T = 294(2) K	θ_{max} = 26.4º
ω scans	θ_{min} = 1.7º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -7 \rightarrow 7$
T_{min} = 0.852, T_{max} = 0.886	$k = -9 \rightarrow 22$
9742 measured reflections	$l = -20 \rightarrow 20$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.039	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.094	$w = 1/[\sigma^2(F_o^2) + (0.0353P)^2 + 0.9782P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.01	$(\Delta/\sigma)_{max}$ = 0.001
3528 reflections	$\Delta\rho_{max}$ = 0.37 e Å^{-3}
241 parameters	$\Delta\rho_{min}$ = −0.38 e Å^{-3}
Primary atom site location: structure-invariant direct methods	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 $F^2 > 2$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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.20681 (12)	0.30768 (4)	0.84918 (4)	0.03291 (18)
O1	0.2730 (4)	0.26231 (11)	0.92092 (11)	0.0449 (5)
O2	−0.0263 (3)	0.32167 (11)	0.82059 (13)	0.0448 (5)
O3	0.8738 (4)	0.60031 (12)	0.95523 (13)	0.0495 (5)
O4	0.5848 (4)	0.65976 (12)	0.89304 (15)	0.0575 (6)
N1	0.2323 (4)	0.30604 (14)	0.52313 (15)	0.0413 (6)
H1	0.215 (5)	0.3155 (17)	0.470 (2)	0.050*
N2	0.3239 (4)	0.26681 (13)	0.77572 (13)	0.0321 (5)
H2A	0.422 (5)	0.2386 (17)	0.7939 (18)	0.039*
N3	0.6823 (4)	0.60178 (14)	0.91655 (14)	0.0372 (6)
C1	0.2886 (4)	0.28291 (14)	0.69149 (15)	0.0270 (6)
C2	0.4432 (5)	0.25395 (16)	0.64290 (16)	0.0358 (7)
H2	0.5671	0.2263	0.6673	0.043*
C3	0.4103 (5)	0.26665 (16)	0.55900 (17)	0.0416 (7)
H3	0.5130	0.2476	0.5267	0.050*
C4	0.0846 (5)	0.33458 (17)	0.56820 (18)	0.0430 (7)
H4	−0.0383	0.3615	0.5418	0.052*
C5	0.1084 (5)	0.32541 (16)	0.65210 (17)	0.0386 (7)
H5	0.0059	0.3472	0.6826	0.046*
C6	0.3464 (4)	0.39542 (14)	0.86639 (15)	0.0284 (6)
C7	0.2334 (4)	0.46213 (15)	0.84301 (16)	0.0320 (6)
H7	0.0850	0.4610	0.8166	0.038*
C8	0.3429 (4)	0.53001 (15)	0.85921 (16)	0.0337 (6)
H8	0.2698	0.5751	0.8443	0.040*
C9	0.5640 (4)	0.52928 (15)	0.89817 (15)	0.0299 (6)
C10	0.6797 (5)	0.46378 (16)	0.92142 (17)	0.0347 (7)
H10	0.8285	0.4653	0.9474	0.042*
C11	0.5696 (4)	0.39591 (15)	0.90528 (16)	0.0334 (6)
H11	0.6438	0.3510	0.9202	0.040*
O5	0.1076 (4)	0.40196 (11)	0.23515 (11)	0.0457 (5)
O6	0.1360 (3)	0.33610 (11)	0.35309 (11)	0.0412 (5)
Cl1	0.23049 (13)	0.47867 (4)	0.44855 (4)	0.0447 (2)
Cl2	0.60598 (13)	0.41336 (5)	0.37668 (6)	0.0579 (3)
Cl3	0.34546 (14)	0.53626 (4)	0.29679 (5)	0.0486 (2)
C12	0.1730 (4)	0.39111 (15)	0.30852 (16)	0.0293 (6)
C13	0.3314 (4)	0.45299 (15)	0.35494 (16)	0.0305 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0421 (4)	0.0312 (4)	0.0291 (4)	−0.0018 (3)	0.0176 (3)	−0.0008 (3)
O1	0.0724 (15)	0.0374 (12)	0.0295 (10)	−0.0025 (10)	0.0229 (10)	0.0056 (9)
O2	0.0358 (11)	0.0469 (13)	0.0559 (13)	−0.0052 (10)	0.0205 (10)	−0.0086 (10)
O3	0.0457 (13)	0.0540 (14)	0.0462 (12)	−0.0078 (11)	−0.0020 (10)	0.0001 (11)

O4	0.0529 (14)	0.0316 (12)	0.0862 (18)	0.0019 (11)	0.0046 (13)	0.0025 (12)
N1	0.0639 (18)	0.0385 (14)	0.0203 (11)	0.0062 (13)	0.0024 (12)	0.0031 (11)
N2	0.0416 (14)	0.0330 (14)	0.0230 (11)	0.0110 (11)	0.0088 (10)	0.0022 (10)
N3	0.0369 (14)	0.0409 (15)	0.0351 (13)	−0.0002 (12)	0.0096 (11)	−0.0020 (11)
C1	0.0348 (15)	0.0230 (13)	0.0241 (13)	−0.0013 (11)	0.0072 (11)	−0.0008 (11)
C2	0.0434 (17)	0.0366 (16)	0.0288 (15)	0.0147 (13)	0.0099 (12)	0.0046 (13)
C3	0.060 (2)	0.0372 (17)	0.0307 (16)	0.0131 (15)	0.0165 (15)	0.0015 (13)
C4	0.0457 (18)	0.0436 (18)	0.0369 (17)	0.0128 (15)	−0.0032 (14)	0.0050 (14)
C5	0.0423 (17)	0.0390 (17)	0.0360 (16)	0.0128 (14)	0.0108 (13)	0.0019 (13)
C6	0.0334 (15)	0.0285 (15)	0.0259 (13)	0.0022 (12)	0.0134 (11)	0.0004 (11)
C7	0.0288 (15)	0.0344 (16)	0.0329 (15)	0.0020 (12)	0.0045 (12)	−0.0002 (12)
C8	0.0333 (15)	0.0310 (16)	0.0374 (16)	0.0080 (12)	0.0069 (12)	0.0031 (12)
C9	0.0342 (15)	0.0316 (15)	0.0254 (13)	−0.0006 (12)	0.0089 (12)	0.0008 (11)
C10	0.0301 (15)	0.0429 (18)	0.0312 (15)	0.0028 (13)	0.0046 (12)	0.0045 (13)
C11	0.0339 (15)	0.0328 (16)	0.0344 (15)	0.0089 (13)	0.0085 (12)	0.0084 (12)
O5	0.0601 (14)	0.0476 (13)	0.0255 (10)	−0.0095 (10)	−0.0068 (9)	0.0008 (9)
O6	0.0569 (13)	0.0356 (11)	0.0289 (10)	−0.0117 (10)	−0.0014 (9)	0.0025 (9)
Cl1	0.0561 (5)	0.0464 (4)	0.0335 (4)	−0.0041 (4)	0.0126 (3)	−0.0148 (3)
Cl2	0.0328 (4)	0.0677 (6)	0.0683 (6)	0.0159 (4)	−0.0085 (4)	−0.0226 (5)
Cl3	0.0610 (5)	0.0360 (4)	0.0514 (5)	−0.0091 (4)	0.0168 (4)	0.0002 (3)
C12	0.0289 (14)	0.0295 (15)	0.0279 (14)	0.0011 (12)	−0.0009 (11)	−0.0054 (12)
C13	0.0279 (14)	0.0339 (15)	0.0291 (14)	0.0019 (12)	0.0020 (11)	−0.0071 (11)

Geometric parameters (Å, °)

S1—O2	1.429 (2)	C4—H4	0.9300
S1—O1	1.430 (2)	C5—H5	0.9300
S1—N2	1.645 (2)	C6—C7	1.392 (3)
S1—C6	1.774 (3)	C6—C11	1.395 (4)
O3—N3	1.227 (3)	C7—C8	1.381 (4)
O4—N3	1.220 (3)	C7—H7	0.9300
N1—C4	1.331 (4)	C8—C9	1.384 (4)
N1—C3	1.338 (4)	C8—H8	0.9300
N1—H1	0.87 (3)	C9—C10	1.381 (4)
N2—C1	1.390 (3)	C10—C11	1.383 (4)
N2—H2A	0.79 (3)	C10—H10	0.9300
N3—C9	1.481 (3)	C11—H11	0.9300
C1—C5	1.396 (4)	O5—C12	1.220 (3)
C1—C2	1.402 (3)	O6—C12	1.258 (3)
C2—C3	1.374 (4)	Cl1—C13	1.783 (3)
C2—H2	0.9300	Cl2—C13	1.778 (3)
C3—H3	0.9300	Cl3—C13	1.768 (3)
C4—C5	1.366 (4)	C12—C13	1.574 (4)
O2—S1—O1	120.47 (13)	C1—C5—H5	120.4
O2—S1—N2	109.85 (12)	C7—C6—C11	121.0 (2)
O1—S1—N2	104.53 (12)	C7—C6—S1	120.5 (2)
O2—S1—C6	108.34 (12)	C11—C6—S1	118.5 (2)
O1—S1—C6	107.38 (12)	C8—C7—C6	119.6 (3)
N2—S1—C6	105.25 (12)	C8—C7—H7	120.2

C4—N1—C3	120.6 (2)	C6—C7—H7	120.2
C4—N1—H1	119 (2)	C7—C8—C9	118.4 (2)
C3—N1—H1	120 (2)	C7—C8—H8	120.8
C1—N2—S1	127.63 (19)	C9—C8—H8	120.8
C1—N2—H2A	120 (2)	C10—C9—C8	122.9 (3)
S1—N2—H2A	112 (2)	C10—C9—N3	118.2 (2)
O4—N3—O3	123.3 (3)	C8—C9—N3	118.9 (2)
O4—N3—C9	118.6 (2)	C9—C10—C11	118.5 (3)
O3—N3—C9	118.0 (2)	C9—C10—H10	120.7
N2—C1—C5	124.3 (2)	C11—C10—H10	120.7
N2—C1—C2	117.8 (2)	C10—C11—C6	119.5 (2)
C5—C1—C2	118.0 (2)	C10—C11—H11	120.3
C3—C2—C1	119.5 (3)	C6—C11—H11	120.3
C3—C2—H2	120.2	O5—C12—O6	129.2 (2)
C1—C2—H2	120.2	O5—C12—C13	116.5 (2)
N1—C3—C2	120.8 (3)	O6—C12—C13	114.2 (2)
N1—C3—H3	119.6	C12—C13—Cl3	113.58 (18)
C2—C3—H3	119.6	C12—C13—Cl2	107.13 (17)
N1—C4—C5	121.8 (3)	Cl3—C13—Cl2	108.93 (14)
N1—C4—H4	119.1	C12—C13—Cl1	109.61 (18)
C5—C4—H4	119.1	Cl3—C13—Cl1	107.11 (14)
C4—C5—C1	119.2 (3)	Cl2—C13—Cl1	110.50 (14)
C4—C5—H5	120.4		
O2—S1—N2—C1	40.4 (3)	S1—C6—C7—C8	178.0 (2)
O1—S1—N2—C1	171.0 (2)	C6—C7—C8—C9	0.3 (4)
C6—S1—N2—C1	−76.0 (3)	C7—C8—C9—C10	0.1 (4)
S1—N2—C1—C5	−15.4 (4)	C7—C8—C9—N3	−179.4 (2)
S1—N2—C1—C2	165.2 (2)	O4—N3—C9—C10	176.8 (2)
N2—C1—C2—C3	178.0 (3)	O3—N3—C9—C10	−3.5 (3)
C5—C1—C2—C3	−1.5 (4)	O4—N3—C9—C8	−3.6 (4)
C4—N1—C3—C2	0.8 (5)	O3—N3—C9—C8	176.0 (2)
C1—C2—C3—N1	−0.2 (4)	C8—C9—C10—C11	−0.2 (4)
C3—N1—C4—C5	0.4 (5)	N3—C9—C10—C11	179.3 (2)
N1—C4—C5—C1	−2.1 (5)	C9—C10—C11—C6	−0.1 (4)
N2—C1—C5—C4	−176.8 (3)	C7—C6—C11—C10	0.5 (4)
C2—C1—C5—C4	2.6 (4)	S1—C6—C11—C10	−178.2 (2)
O2—S1—C6—C7	−7.4 (2)	O5—C12—C13—Cl3	11.0 (3)
O1—S1—C6—C7	−138.9 (2)	O6—C12—C13—Cl3	−170.43 (19)
N2—S1—C6—C7	110.1 (2)	O5—C12—C13—Cl2	−109.3 (2)
O2—S1—C6—C11	171.35 (19)	O6—C12—C13—Cl2	69.2 (3)
O1—S1—C6—C11	39.8 (2)	O5—C12—C13—Cl1	130.8 (2)
N2—S1—C6—C11	−71.2 (2)	O6—C12—C13—Cl1	−50.7 (3)
C11—C6—C7—C8	−0.6 (4)		

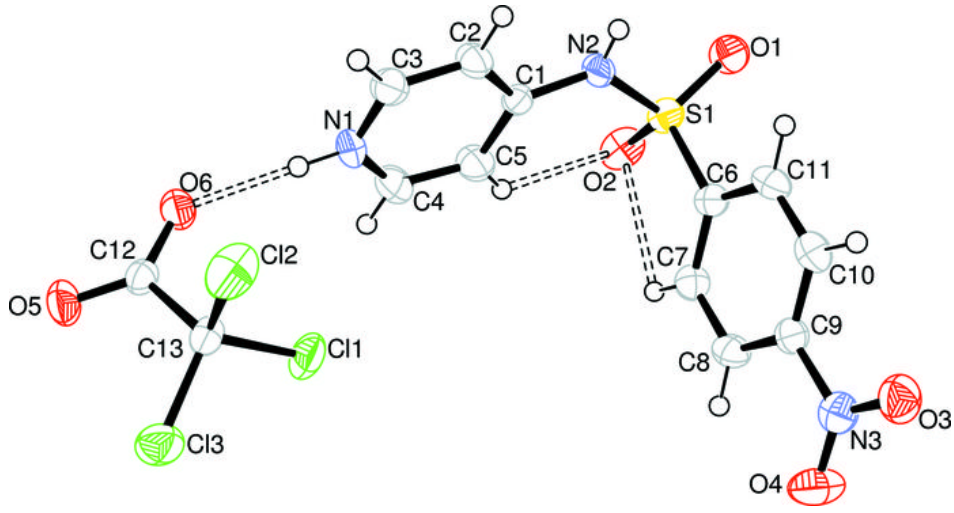
Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O6	0.87 (3)	1.93 (3)	2.803 (3)	172 (3)
N2—H2A···O6^{i}	0.79 (3)	2.00 (3)	2.785 (3)	171 (3)

C2—H2···O5^{i}	0.93	2.53	3.240 (3)	133
C3—H3···O1^{ii}	0.93	2.50	3.391 (4)	162
C8—H8···O5^{iii}	0.93	2.47	3.149 (3)	130
C10—H10···O3^{iv}	0.93	2.50	3.308 (4)	146
C5—H5···O2	0.93	2.33	2.978 (4)	126
C7—H7···O2	0.93	2.57	2.938 (3)	104

Symmetry codes: (i) *x*+1/2, −*y*+1/2, *z*+1/2; (ii) *x*+1/2, −*y*+1/2, *z*−1/2; (iii) −*x*, −*y*+1, −*z*+1; (iv) −*x*+2, −*y*+1, −*z*+2.

Fig. 1


C2
C3
N2
O1
C1
S1
N1
C5
O2
C6
C11
O6
C4
C12
Cl2
C7
C10
O5
C9
C13
Cl1
C8
O3
N3
Cl3
O4