3-Carbamothioylpyridinium iodide

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In the crystal of the title salt, $C_6H_7N_2S^+ \cdot I^-$, inversion-related cations form an $R_2^2(8)$ dimer linked by a pair of N—H⋯S hydrogen bonds. Pairs of iodide anions are located between adjacent cation dimers and are linked to them by way of N—H⋯I hydrogen bonds. This results in zigzag chains propagating in [001] lying parallel to the bc plane.

Related literature

For graph-set theory, see: Bernstein *et al.* (1995).
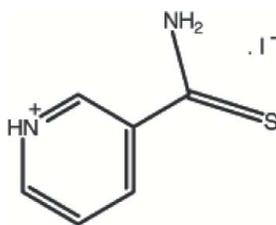
Experimental

Crystal data

$C_6H_7N_2S^+ \cdot I^-$

$M_r = 266.11$

Triclinic, $P\bar{1}$

$a = 4.4024$ (3) Å

$b = 8.1943$ (5) Å

$c = 12.6815$ (8) Å

$\alpha = 102.485$ (2)°

$\beta = 96.496$ (2)°

$\gamma = 102.288$ (2)°

$V = 430.31$ (5) Å^3

$Z = 2$

Mo $K\alpha$ radiation

$\mu = 3.89$ mm^{-1}

$T = 296$ K

$0.17 \times 0.15 \times 0.14$ mm

Data collection

Bruker Kappa APEXII CCD diffractometer

Absorption correction: none

8839 measured reflections

2087 independent reflections

1890 reflections with $I > 2\sigma(I)$

$R_{int} = 0.020$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.019$

$wR(F^2) = 0.047$

$S = 1.04$

2087 reflections

91 parameters

H-atom parameters constrained

$\Delta\rho_{max} = 0.65$ e Å^{-3}

$\Delta\rho_{min} = -0.43$ e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H$N1$⋯I1^i	0.86	2.62	3.444 (2)	161
N2—H2A⋯I1^{ii}	0.86	3.04	3.747 (3)	140
N2—H2B⋯S1^{iii}	0.86	2.58	3.420 (3)	164

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5088).

References

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supporting information

3-Carbamothioylpyridinium iodide

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S1. Comment

In the present study we attempted to prepare a palladium(II) iodide complex with thionicotinamide, but it is surprising to note that the resulting compound is a simple salt of pyridine. Here we report the crystal structure of the salt (I).

In the title compund (I), (Fig. 1), the bond lengths and angles are entirely as expected. In the crystal structure of (I), two crystallographically independent cations form a dimer through N—H⋯S hydrogen bonds. The two iodide anions are located between two adjacent dimers and forms N—H⋯I hydrogen bonds with two iodide anions from each dimer. Thus, the molecules linked in the form of zigzag in the layers parallel to the *bc* plane along the *b* axis (Fig. 2 and Fig. 3, Table 1).

S2. Experimental

The title compound was prepared by adding 2 equivalents of thionicotinamide in 15 ml methanol to a solution of $K_2[PdCl_4]$ (0.326 g) in 15 ml of water followed by addition of 2 equivalents of potassium iodide in water after half an hour stirring. The dark brown solution was the stirred for one hour. The resulting solution was filtrated and filtrate was kept at room temperature for crystallization. The brown product obtained from water-methanol mixture wasre-dissolved in methanol, which on slow evaporation yielded light brown crystals of (I).

S3. Refinement

All H atoms were located geometrically and treated as riding with C—H = 0.93 Å and N—H = 0.86 Å with $U_{iso}(H) = 1.2U_{eq}(C, N)$.
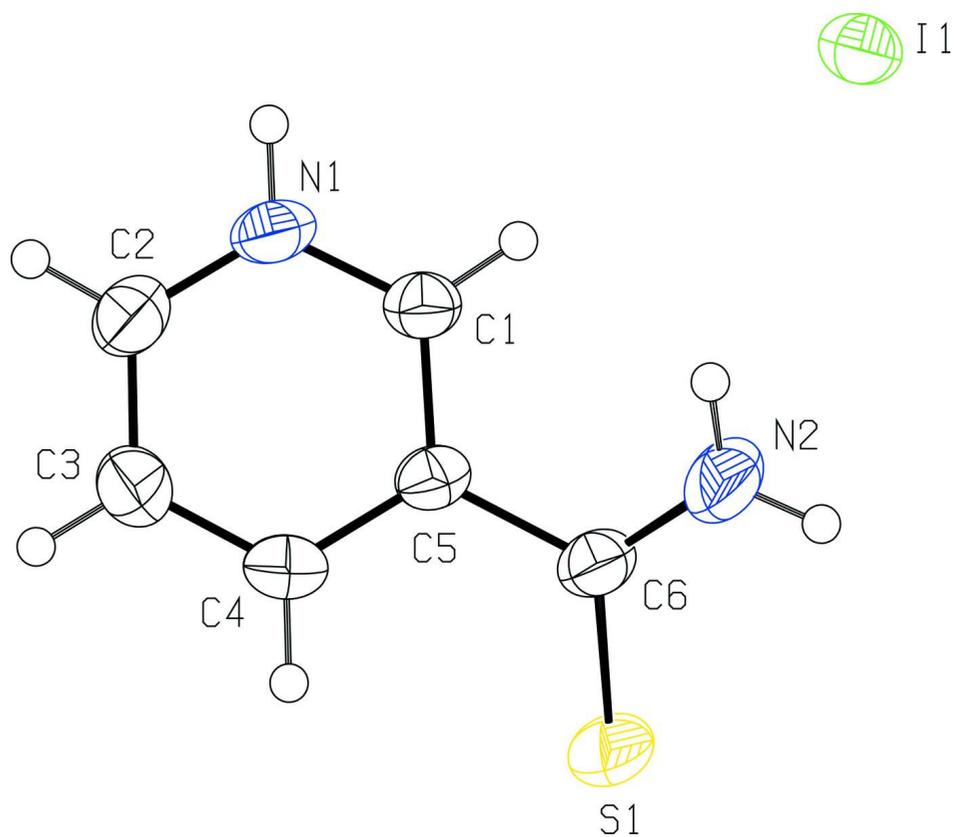
Figure 1

The molecular structure of (I) with displacement ellipsoids for the non-H atoms drawn at the 50% probability level.
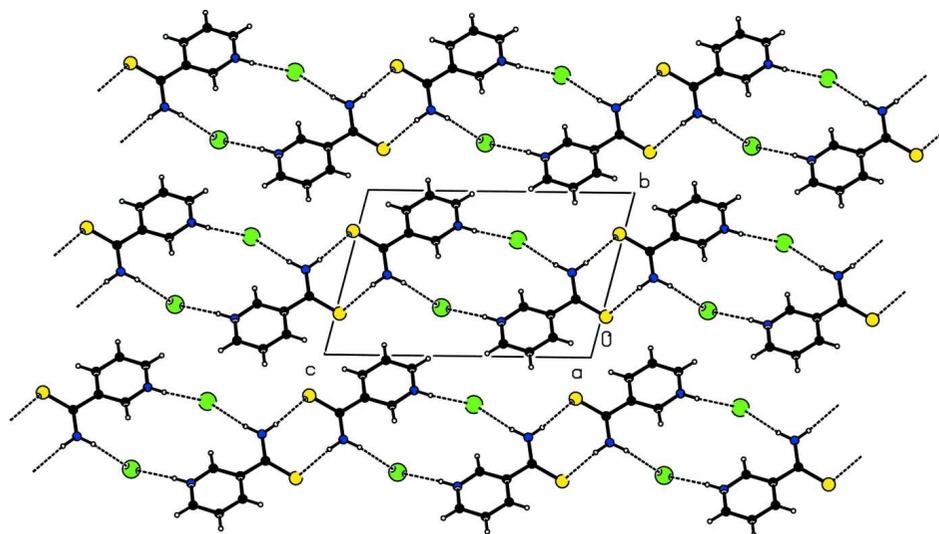
Figure 2

Packing diagram for (I) viewed down the *a* axis, showing the $R_2^2(8)$ dimer motif further linked by N—H···I hydrogen bonds between the adjacent dimers thorough the iodide anions to form an infinite chain in the [010] direction. Hydrogen atoms not involved in the showed interactions have been omitted for clarity.
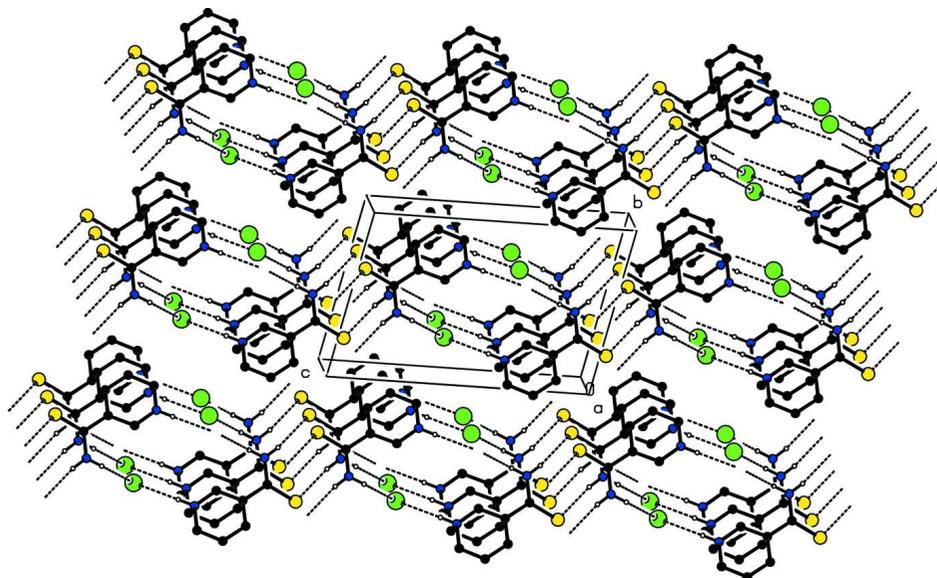
Figure 3

A view of the packing and hydrogen bonding of (I). Hydrogen atoms not involved in the showed interactions have been omitted for clarity.

3-Carbamothioylpyridinium iodide

Crystal data

$C_6H_7N_2S^+ \cdot I^-$	$Z = 2$
$M_r = 266.11$	$F(000) = 252$
Triclinic, $P\overline{1}$	$D_x = 2.054$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 4.4024$ (3) Å	Cell parameters from 6584 reflections
$b = 8.1943$ (5) Å	$\theta = 2.6–28.3°$
$c = 12.6815$ (8) Å	$\mu = 3.89$ mm^{-1}
$\alpha = 102.485$ (2)°	$T = 296$ K
$\beta = 96.496$ (2)°	Irregular chunk, light brown
$\gamma = 102.288$ (2)°	$0.17 \times 0.15 \times 0.14$ mm
$V = 430.31$ (5) Å^3	

Data collection

Bruker Kappa APEXII CCD diffractometer	1890 reflections with $I > 2\sigma(I)$
Radiation source: sealed tube	$R_{int} = 0.020$
Graphite monochromator	$\theta_{max} = 28.3°$, $\theta_{min} = 2.6°$
phi and ω scans	$h = -5 \rightarrow 5$
8839 measured reflections	$k = -9 \rightarrow 10$
2087 independent reflections	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	91 parameters
Least-squares matrix: full	0 restraints
$R[F^2 > 2\sigma(F^2)] = 0.019$	Primary atom site location: structure-invariant direct methods
$wR(F^2) = 0.047$	
$S = 1.04$	Secondary atom site location: difference Fourier map
2087 reflections	

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0227P)^2 + 0.219P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.65$ e Å^{-3}
$\Delta\rho_{min} = -0.43$ e Å^{-3}

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R-factors wR and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -R-factor-obs *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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.1191 (2)	0.24949 (10)	−0.01595 (5)	0.0620 (3)
N1	0.3984 (5)	0.2110 (3)	0.37108 (16)	0.0423 (6)
N2	0.2489 (8)	0.5132 (3)	0.1550 (2)	0.0708 (12)
C1	0.3138 (6)	0.3026 (3)	0.30244 (18)	0.0377 (7)
C2	0.5245 (6)	0.0778 (3)	0.3413 (2)	0.0449 (8)
C3	0.5737 (7)	0.0300 (3)	0.2358 (2)	0.0479 (8)
C4	0.4852 (6)	0.1197 (3)	0.1621 (2)	0.0438 (8)
C5	0.3541 (5)	0.2583 (3)	0.19426 (17)	0.0345 (6)
C6	0.2468 (6)	0.3524 (3)	0.11482 (19)	0.0395 (7)
I1	−0.08685 (4)	0.70787 (2)	0.39126 (1)	0.0426 (1)
H1	0.22840	0.39550	0.32730	0.0450*
HN1	0.36980	0.23970	0.43790	0.0510*
H2	0.57890	0.01770	0.39200	0.0540*
H2A	0.31010	0.55920	0.22390	0.0850*
H2B	0.18910	0.57340	0.11260	0.0850*
H3	0.66530	−0.06150	0.21410	0.0570*
H4	0.51400	0.08680	0.08980	0.0530*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.1014 (6)	0.0573 (4)	0.0263 (3)	0.0304 (4)	−0.0032 (3)	0.0045 (3)
N1	0.0534 (12)	0.0464 (12)	0.0256 (9)	0.0090 (10)	0.0078 (9)	0.0083 (9)
N2	0.131 (3)	0.0457 (14)	0.0331 (12)	0.0312 (15)	−0.0095 (14)	0.0070 (10)
C1	0.0430 (12)	0.0388 (12)	0.0296 (11)	0.0094 (10)	0.0060 (9)	0.0055 (9)
C2	0.0541 (15)	0.0409 (13)	0.0377 (13)	0.0076 (11)	0.0003 (11)	0.0132 (11)
C3	0.0572 (15)	0.0428 (14)	0.0441 (14)	0.0204 (12)	0.0045 (12)	0.0057 (11)
C4	0.0539 (14)	0.0471 (14)	0.0291 (11)	0.0151 (12)	0.0083 (10)	0.0033 (10)
C5	0.0377 (11)	0.0371 (11)	0.0261 (10)	0.0063 (9)	0.0030 (8)	0.0063 (9)
C6	0.0470 (13)	0.0426 (13)	0.0283 (11)	0.0095 (11)	0.0046 (9)	0.0098 (9)
I1	0.0421 (1)	0.0492 (1)	0.0351 (1)	0.0154 (1)	0.0065 (1)	0.0034 (1)

Geometric parameters (Å, º)

S1—C6	1.661 (2)	C2—C3	1.366 (4)
N1—C1	1.337 (3)	C3—C4	1.379 (4)
N1—C2	1.330 (3)	C4—C5	1.386 (3)
N2—C6	1.304 (4)	C5—C6	1.489 (3)
N1—HN1	0.8600	C1—H1	0.9300
N2—H2B	0.8600	C2—H2	0.9300
N2—H2A	0.8600	C3—H3	0.9300
C1—C5	1.383 (3)	C4—H4	0.9300
I1···C1[i]	3.639 (3)	C1···C3[i]	3.433 (4)
I1···C2[ii]	3.818 (3)	C2···I1[x]	3.818 (3)
I1···N2	3.694 (3)	C2···I1[iv]	3.793 (3)
I1···N1[iii]	3.444 (2)	C3···C1[ix]	3.433 (4)
I1···C2[iv]	3.794 (3)	C4···S1[vii]	3.564 (3)
I1···H1	3.1600	C1···H2A	2.5200
I1···H2[v]	3.1900	H1···H2A	2.0800
I1···H2A[i]	3.0400	H1···I1	3.1600
I1···H2A	3.1100	H1···N2	2.5800
I1···H2[iv]	3.3800	HN1···I1[iii]	2.6200
I1···HN1[iii]	2.6200	H2···I1[xi]	3.1900
S1···N2[vi]	3.420 (3)	H2···I1[iv]	3.3800
S1···C4[vii]	3.564 (3)	H2A···H1	2.0800
S1···H3[viii]	3.0100	H2A···I1	3.1100
S1···H2B[vi]	2.5800	H2A···I1[ix]	3.0400
S1···H4	2.8000	H2A···C1	2.5200
N1···I1[iii]	3.444 (2)	H2B···S1[vi]	2.5800
N2···S1[vi]	3.420 (3)	H3···S1[viii]	3.0100
N2···I1	3.694 (3)	H4···S1	2.8000
N2···H1	2.5800	H4···H4[viii]	2.3800
C1···I1[ix]	3.639 (3)		
C1—N1—C2	123.4 (2)	C4—C5—C6	121.6 (2)
C1—N1—HN1	118.00	S1—C6—C5	119.92 (18)
C2—N1—HN1	118.00	N2—C6—C5	116.3 (2)
C6—N2—H2A	120.00	S1—C6—N2	123.7 (2)
H2A—N2—H2B	120.00	N1—C1—H1	120.00
C6—N2—H2B	120.00	C5—C1—H1	120.00
N1—C1—C5	119.6 (2)	N1—C2—H2	120.00
N1—C2—C3	119.5 (2)	C3—C2—H2	120.00
C2—C3—C4	118.9 (2)	C2—C3—H3	121.00
C3—C4—C5	121.0 (2)	C4—C3—H3	121.00
C1—C5—C4	117.7 (2)	C3—C4—H4	120.00
C1—C5—C6	120.6 (2)	C5—C4—H4	119.00
C2—N1—C1—C5	0.7 (4)	C3—C4—C5—C1	−0.4 (4)
C1—N1—C2—C3	0.3 (4)	C3—C4—C5—C6	−177.8 (2)

N1—C1—C5—C4	−0.6 (4)	C1—C5—C6—S1	−147.9 (2)
N1—C1—C5—C6	176.8 (2)	C1—C5—C6—N2	29.5 (4)
N1—C2—C3—C4	−1.2 (4)	C4—C5—C6—S1	29.4 (3)
C2—C3—C4—C5	1.3 (4)	C4—C5—C6—N2	−153.1 (3)

Symmetry codes: (i) *x*−1, *y*, *z*; (ii) *x*, *y*+1, *z*; (iii) −*x*, −*y*+1, −*z*+1; (iv) −*x*+1, −*y*+1, −*z*+1; (v) *x*−1, *y*+1, *z*; (vi) −*x*, −*y*+1, −*z*; (vii) −*x*, −*y*, −*z*; (viii) −*x*+1, −*y*, −*z*; (ix) *x*+1, *y*, *z*; (x) *x*, *y*−1, *z*; (xi) *x*+1, *y*−1, *z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H*N*1···I1ⁱⁱⁱ	0.86	2.62	3.444 (2)	161
N2—H2*A*···I1ⁱˣ	0.86	3.04	3.747 (3)	140
N2—H2*B*···S1ᵛⁱ	0.86	2.58	3.420 (3)	164

Symmetry codes: (iii) −*x*, −*y*+1, −*z*+1; (vi) −*x*, −*y*+1, −*z*; (ix) *x*+1, *y*, *z*.