organic compounds

Diisopropylammonium thiocyanate

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In the title molecular salt, $C_6H_{16}N^+ \cdot NCS^-$, the cation possesses approximate local twofold rotation symmetry. One of its NH atoms forms a hydrogen bond to a thiocyanate N atom and the other to a thiocyanate S atom. This results in [001] chains of alternating cations and anions.

Related literature

For background to molecular ferroelectrics, see: Fu *et al.* (2011).
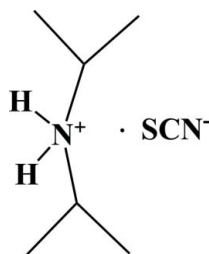
Experimental

Crystal data

$C_6H_{16}N^+ \cdot NCS^-$
$M_r = 160.28$
Orthorhombic, $Pbca$
$a = 11.785$ (2) Å
$b = 12.716$ (3) Å
$c = 13.288$ (3) Å
$V = 1991.3$ (7) Å^3
$Z = 8$
Mo $K\alpha$ radiation
$\mu = 0.27$ mm^{-1}
$T = 298$ K
$0.20 \times 0.05 \times 0.05$ mm

Data collection

Rigaku Mercury2 CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
$T_{min} = 0.90$, $T_{max} = 1.00$
19260 measured reflections
2286 independent reflections
1864 reflections with $I > 2\sigma(I)$
$R_{int} = 0.051$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.107$
$S = 1.12$
2286 reflections
96 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2D···N1^i	0.90	1.99	2.888 (2)	172
N2—H2E···S1	0.90	2.47	3.3490 (14)	166

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

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*.

References

Fu, D.-W., Zhang, W., Cai, H.-L., Ge, J.-Z., Zhang, Y. & Xiong, R.-G. (2011). *Adv. Mater.* **23**, 5658–5662.
Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

Diisopropylammonium thiocyanate

Jie Xu

Comment

Simple organic salts containig amino cations have attracted attention as materials that display ferroelectric-paraelectric phase transitions (Fu *et al.*, 2011). As part of our ongonig studies in this area, we now present the crystal structure of the title compound, di-isopropylammonium thiocyanate.

The asymmetric unit of the title compound contains one di-isopropylammonium cation and one SCN⁻ anion (Fig. 1). The amine N2 atom was protonated. The SCN⁻ anion is almost linear with the bond angle of 179.80 (2)°. The geometric parameters of the title compound are in the normal range.

In the crystal structure, all the ammonium H atoms are involved in intermolecular N—H···N and N—H···S H-bonding interactions with the N and S atoms of the SCN⁻ anion (with N···N and N···S distances of 2.888 (2)Å to 3.349 (2)Å, respectively). These hydrogen bonds link the ionic units into a one-dimentional chain along the *c*-axis (Table 1 and Fig.2).

Experimental

A mixture of di-isopropylamine (0.8 mmol), HCl (0.8 mmol) and KSCN (0.8 mmol) were dissolved in EtOH/distilled water (1:1 *v/v*) solvent. The solution was slowly evaporated in air affording colourless blocks.

Refinement

All H atoms attached to C atoms were fixed geometrically and treated as riding with C-H = 0.98 Å(C methine) and C-H = 0.96 Å(C methyl) with $U_{iso}(H) = 1.2U_{eq}$(C methine) and $U_{iso}(H) = 1.5U_{eq}$(C methyl). The positional parameters of the H atoms (N) were refined freely. And in the last stage of the refinement, they were restrained with the H—N = 0.90 (2)Å, and $U_{iso}(H)=1.2U_{eq}$(N).

Computing details

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear* (Rigaku, 2005); data reduction: *CrystalClear* (Rigaku, 2005); 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* (Sheldrick, 2008).
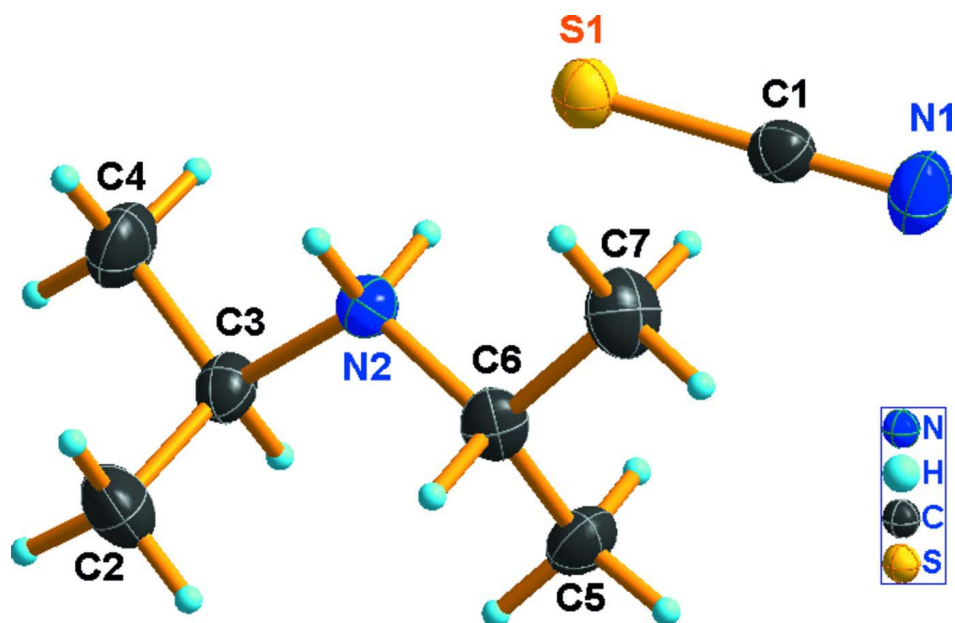
Figure 1

A view of the title compound with displacement ellipsoids drawn at the 30% probability level.
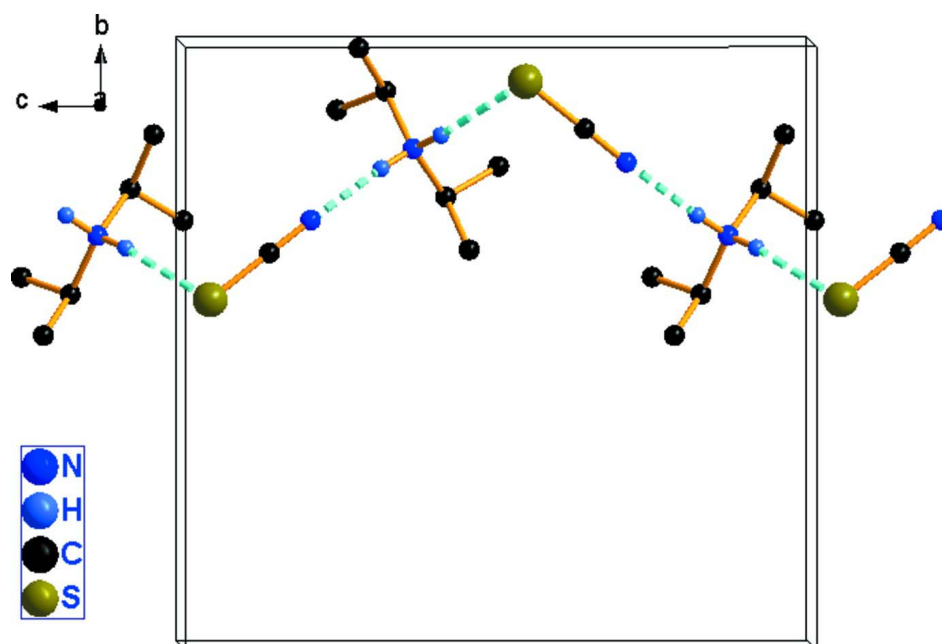

Figure 2

The crystal packing of the title compound showing the one-dimensionnal hydrogen bondings chain along the *c* axis (dashed line). H atoms not involved in hydrogen bonding (dashed line) have been omitted for clarity.

Diisopropylammonium thiocyanate

Crystal data

$C_6H_{16}N^+ \cdot NCS^-$	$F(000) = 704$
$M_r = 160.28$	$D_x = 1.069$ Mg m^{-3}
Orthorhombic, *Pbca*	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ac 2ab	Cell parameters from 2286 reflections
$a = 11.785$ (2) Å	$\theta = 3.1–27.5°$
$b = 12.716$ (3) Å	$\mu = 0.27$ mm^{-1}
$c = 13.288$ (3) Å	$T = 298$ K
$V = 1991.3$ (7) Å^3	Block, colourless
$Z = 8$	$0.20 \times 0.05 \times 0.05$ mm

Data collection

Rigaku Mercury2 CCD diffractometer	19260 measured reflections
Radiation source: fine-focus sealed tube	2286 independent reflections
Graphite monochromator	1864 reflections with $I > 2\sigma(I)$
Detector resolution: 13.6612 pixels mm^{-1}	$R_{int} = 0.051$
CCD profile fitting scans	$\theta_{max} = 27.5°$, $\theta_{min} = 3.1°$
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)	$h = -15 \rightarrow 15$
	$k = -16 \rightarrow 16$
$T_{min} = 0.90$, $T_{max} = 1.00$	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.045$	$w = 1/[\sigma^2(F_o^2) + (0.0402P)^2 + 0.4971P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.107$	
$S = 1.12$	$(\Delta/\sigma)_{max} < 0.001$
2286 reflections	$\Delta\rho_{max} = 0.18$ e Å^{-3}
96 parameters	$\Delta\rho_{min} = -0.15$ e Å^{-3}
0 restraints	Extinction correction: *SHELXL97* (Sheldrick, 2008), Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	Extinction coefficient: 0.044 (3)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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}
N2	0.42899 (11)	0.17785 (9)	0.36893 (10)	0.0400 (3)
H2D	0.3915	0.2112	0.3193	0.048*
H2E	0.3769	0.1567	0.4141	0.048*

C3	0.48334 (15)	0.08132 (13)	0.32473 (13)	0.0476 (4)
H3A	0.5226	0.0434	0.3787	0.057*
C5	0.57312 (16)	0.20499 (14)	0.50250 (15)	0.0551 (5)
H5A	0.6217	0.1522	0.4741	0.083*
H5B	0.5224	0.1729	0.5500	0.083*
H5C	0.6184	0.2571	0.5361	0.083*
C6	0.50507 (15)	0.25665 (13)	0.41958 (13)	0.0471 (4)
H6A	0.5578	0.2850	0.3694	0.056*
C7	0.4316 (2)	0.34550 (15)	0.45802 (16)	0.0666 (6)
H7A	0.3883	0.3743	0.4033	0.100*
H7B	0.4788	0.3994	0.4864	0.100*
H7C	0.3809	0.3193	0.5087	0.100*
C4	0.3906 (2)	0.01151 (17)	0.28384 (17)	0.0727 (6)
H4A	0.4231	−0.0530	0.2598	0.109*
H4B	0.3527	0.0467	0.2294	0.109*
H4C	0.3369	−0.0036	0.3363	0.109*
C2	0.5690 (2)	0.1114 (2)	0.24603 (18)	0.0774 (7)
H2A	0.6292	0.1508	0.2768	0.116*
H2B	0.5330	0.1536	0.1954	0.116*
H2C	0.5996	0.0490	0.2157	0.116*
S1	0.26970 (4)	0.06903 (3)	0.54981 (3)	0.04839 (18)
N1	0.32617 (17)	0.20204 (15)	0.70857 (14)	0.0754 (6)
C1	0.30276 (14)	0.14704 (14)	0.64316 (13)	0.0459 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N2	0.0425 (7)	0.0407 (7)	0.0369 (7)	0.0020 (6)	−0.0008 (6)	0.0052 (6)
C3	0.0555 (10)	0.0441 (9)	0.0432 (9)	0.0098 (8)	−0.0096 (8)	−0.0048 (7)
C5	0.0523 (10)	0.0544 (11)	0.0586 (11)	−0.0096 (8)	−0.0106 (9)	−0.0078 (9)
C6	0.0553 (10)	0.0370 (8)	0.0489 (9)	−0.0081 (7)	0.0062 (8)	0.0020 (7)
C7	0.0863 (15)	0.0420 (10)	0.0714 (14)	0.0048 (10)	0.0026 (11)	−0.0043 (9)
C4	0.0806 (15)	0.0675 (13)	0.0700 (14)	−0.0024 (11)	−0.0161 (11)	−0.0230 (11)
C2	0.0745 (15)	0.0887 (16)	0.0690 (14)	0.0145 (12)	0.0166 (11)	−0.0150 (13)
S1	0.0527 (3)	0.0463 (3)	0.0462 (3)	−0.00456 (19)	0.00129 (19)	−0.00236 (19)
N1	0.0879 (14)	0.0808 (12)	0.0576 (11)	−0.0223 (11)	−0.0029 (9)	−0.0157 (9)
C1	0.0422 (9)	0.0503 (10)	0.0451 (9)	−0.0037 (7)	0.0023 (7)	0.0037 (8)

Geometric parameters (Å, º)

N2—C6	1.504 (2)	C6—H6A	0.9800
N2—C3	1.504 (2)	C7—H7A	0.9600
N2—H2D	0.9000	C7—H7B	0.9600
N2—H2E	0.9000	C7—H7C	0.9600
C3—C2	1.503 (3)	C4—H4A	0.9600
C3—C4	1.509 (3)	C4—H4B	0.9600
C3—H3A	0.9800	C4—H4C	0.9600
C5—C6	1.513 (2)	C2—H2A	0.9600
C5—H5A	0.9600	C2—H2B	0.9600
C5—H5B	0.9600	C2—H2C	0.9600

C5—H5C	0.9600	S1—C1	1.6354 (19)
C6—C7	1.512 (3)	N1—C1	1.149 (2)
C6—N2—C3	117.69 (13)	C7—C6—H6A	108.6
C6—N2—H2D	107.9	C5—C6—H6A	108.6
C3—N2—H2D	107.9	C6—C7—H7A	109.5
C6—N2—H2E	107.9	C6—C7—H7B	109.5
C3—N2—H2E	107.9	H7A—C7—H7B	109.5
H2D—N2—H2E	107.2	C6—C7—H7C	109.5
C2—C3—N2	110.49 (15)	H7A—C7—H7C	109.5
C2—C3—C4	112.70 (17)	H7B—C7—H7C	109.5
N2—C3—C4	108.19 (15)	C3—C4—H4A	109.5
C2—C3—H3A	108.5	C3—C4—H4B	109.5
N2—C3—H3A	108.5	H4A—C4—H4B	109.5
C4—C3—H3A	108.5	C3—C4—H4C	109.5
C6—C5—H5A	109.5	H4A—C4—H4C	109.5
C6—C5—H5B	109.5	H4B—C4—H4C	109.5
H5A—C5—H5B	109.5	C3—C2—H2A	109.5
C6—C5—H5C	109.5	C3—C2—H2B	109.5
H5A—C5—H5C	109.5	H2A—C2—H2B	109.5
H5B—C5—H5C	109.5	C3—C2—H2C	109.5
N2—C6—C7	107.91 (15)	H2A—C2—H2C	109.5
N2—C6—C5	110.65 (13)	H2B—C2—H2C	109.5
C7—C6—C5	112.46 (16)	N1—C1—S1	179.8 (2)
N2—C6—H6A	108.6		
C6—N2—C3—C2	−59.45 (19)	C3—N2—C6—C7	−179.73 (14)
C6—N2—C3—C4	176.74 (15)	C3—N2—C6—C5	−56.31 (19)

Hydrogen-bond geometry (Å, º)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N2—H2D⋯N1[i]	0.90	1.99	2.888 (2)	172
N2—H2E⋯S1	0.90	2.47	3.3490 (14)	166

Symmetry code: (i) *x*, −*y*+1/2, *z*−1/2.