

The molecule and the atomic labeling scheme are shown in Fig. 1. Final positional and equivalent isotropic thermal parameters are shown in Table 1;* some selected bond distances and angles are listed in Table 2.

Related literature. A review of similar complexes has been published by Davis & Kane-Maguire (1982). Structural work on related complexes has been reported by Graham, Akrigg & Sheldrick (1976, 1983, 1985) and by Graham & Fenn (1969, 1970).

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* Lists of structure factors, anisotropic thermal parameters, and complete intramolecular bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52161 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of (S₂N₂C—C₆H₄—CN₂S₂)²⁺·2SbF₆⁻·2C₆H₅CN

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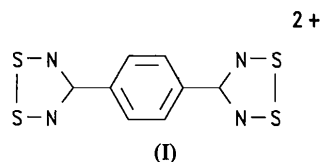
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Abstract. *p*-Di(1,2,3,5-dithiadiazolium-4-yl)benzene hexafluoroantimonate benzonitrile solvate, C₈H₄N₄S₄²⁺·2SbF₆⁻·2C₇H₅N, *M_r* = 962.1, monoclinic, *C*2/*c*, *a* = 11.578 (4), *b* = 22.122 (10), *c* = 13.672 (6) Å, β = 110.31 (3)°, *V* = 3284 (5) Å³, *Z* = 4, *D_x* = 1.95 g cm⁻³, λ(Mo *K*α) = 0.71073 Å, μ = 19.9 cm⁻¹, *F*(000) = 1848, *T* = 293 K, *R* = 0.046 for 1374 reflections with *F_o*² > 3σ(*F_o*²). The cation is located on a crystallographic center of symmetry and two independent SbF₆⁻ anions are each located on twofold rotation axes. One of the anions is disordered. In the cation the terminal S₂N₂C rings are twisted 15.8° with respect to the phenylene ring. The S—S distance is 2.009 (4) Å and the average S—N, and C—N distances are 1.577 (9) and 1.34 (1) Å, respectively.

Experimental. Title salt (I) prepared by metathesis of [(S₂N₂C)C₆H₄(CN₂S₂)]²⁺·2Cl⁻ with NOSbF₆ in

PhCN. The dichloride salt itself was prepared by the reaction of (Me₃Si)₂N(Me₃SiN)C—C₆H₄—C(NSiMe₃)N(SiMe₃)₂ (Boere, Oakley & Reed, 1987) with excess SCl₂. Data crystal obtained by slow cooling of a benzonitrile solution. Yellow air-sensitive crystal approximately 0.40 × 0.40 × 0.40 mm was embedded in wax in a glass capillary. Intensities measured with an Enraf-Nonius CAD-4 diffractometer using ω-2θ scans of 8° min⁻¹ in θ. Unit cell determined from least-squares analysis of angle data for 25 reflections with 17 < 2θ < 20°. Absorption correction based on ψ scans varied from



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Table 1. Fractional coordinates and isotropic or equivalent isotropic thermal parameters

The thermal parameters are of the form $B = 8\pi^2 U$ and $B_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$.

	x	y	z	B/B_{eq} (\AA^2)
Sr(1)	0.000	0.70241 (5)	0.250	4.58 (3)
Sr(2)	0.000	-0.00505 (5)	0.250	4.11 (2)
S(1)	0.1522 (3)	0.1561 (1)	-0.0314 (2)	5.52 (7)
S(2)	0.1564 (3)	0.1093 (1)	0.0958 (2)	5.48 (8)
F(1)	0.000	0.6187 (9)	0.250	11.4 (5)*
F(2)	0.000	0.7846 (9)	0.250	12.2 (6)*
F(3)	-0.0749 (8)	0.7015 (4)	0.1070 (6)	9.8 (2)*
F(4)	-0.158 (1)	0.7004 (6)	0.253 (1)	10.1 (3)*
F(5)	0.000	-0.0894 (4)	0.250	7.9 (3)
F(6)	0.000	0.0791 (4)	0.250	8.4 (3)
F(7)	-0.0399 (6)	-0.0062 (3)	0.1062 (4)	7.2 (2)
F(8)	-0.1645 (6)	-0.0051 (4)	0.2351 (6)	8.6 (2)
F(9)	0.105	0.643	0.230	12 (1)*
F(10)	0.074	0.770	0.230	10.1 (8)*
N(1)	0.2551 (8)	0.1176 (4)	-0.0533 (7)	4.9 (2)
N(2)	0.2589 (7)	0.0633 (4)	0.0921 (6)	4.9 (2)
N(3)	0.0111 (9)	0.2136 (4)	0.0756 (8)	7.6 (3)
C(1)	0.3040 (9)	0.0735 (5)	0.0150 (7)	4.2 (3)
C(2)	0.4030 (8)	0.0348 (4)	0.0063 (7)	3.6 (2)
C(3)	0.4714 (9)	-0.0005 (5)	0.0903 (7)	4.6 (3)
C(4)	0.565 (1)	-0.0362 (5)	0.0818 (8)	5.1 (3)
C(5)	0.8655 (9)	0.3014 (5)	0.0761 (8)	4.8 (3)
C(6)	0.821 (1)	0.3071 (6)	0.1563 (9)	6.1 (3)
C(7)	0.744 (1)	0.3548 (6)	0.1569 (9)	7.0 (3)
C(8)	0.711 (1)	0.3940 (5)	0.075 (1)	6.5 (3)
C(9)	0.754 (1)	0.3871 (5)	-0.005 (1)	7.2 (4)
C(10)	0.832 (1)	0.3413 (5)	-0.0047 (9)	6.0 (3)
C(11)	0.948 (1)	0.2510 (6)	0.0774 (8)	6.0 (3)

* Atoms refined isotropically

Table 2. Some bond lengths (\AA) and bond angles ($^\circ$) with e.s.d.'s in parenthesesPrimed atoms at $1-x, -y, -z$.

S(1)	S(2)	2.009 (4)	C(1)	C(2)	1.468 (13)		
S(1)	N(1)	1.576 (9)	C(2)	C(3)	1.388 (13)		
S(2)	N(2)	1.577 (9)	C(2)	C(4')	1.378 (14)		
N(1)	C(1)	1.334 (12)	C(3)	C(4)	1.377 (14)		
N(2)	C(1)	1.348 (13)					
S(2)	S(1)	N(1)	95.3 (4)	N(2)	C(1)	C(2)	119.5 (9)
S(1)	S(2)	N(2)	95.6 (4)	C(1)	C(2)	C(3)	119.9 (9)
S(1)	N(1)	C(1)	115.3 (8)	C(1)	C(2)	C(4')	120.9 (9)
S(2)	N(2)	C(1)	114.6 (7)	C(3)	C(2)	C(4')	119.1 (9)
N(1)	C(1)	N(2)	119.0 (9)	C(2)	C(3)	C(4)	119.5 (9)
N(1)	C(1)	C(2)	122 (1)	C(2)	C(4')	C(3')	121.3 (9)

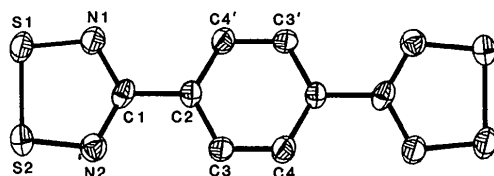


Fig. 1. ORTEP (Johnson, 1976) drawing and numbering scheme for the cation. The ellipsoids are drawn at the 30% probability surface and H atoms have been omitted.

0.87 to 1.00. Data collected to $(\sin\theta)/\lambda$ of 0.55 \AA^{-1} , $-12 < h < 12$, $0 < k < 23$, $0 < l < 14$. Two standard reflections ($\bar{3}72, 006$) varied 3.5% over 12.5 h of data collection; no correction was made. 2460 reflections measured, 2281 unique ($R_{\text{int}} = 0.015$), 907 reflections with $I < 3\sigma(I)$ where $\sigma^2(I) = \sigma_{\text{cs}}^2(I) + (0.08I)^2$; $\sigma_{\text{cs}}(I)$ is the standard deviation of I based on counting statistics. Two other data sets from different crystals had an even higher number of weak reflections. Solved by direct methods using *MULTAN11/82* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and Fourier methods. Full-matrix least squares minimized $\sum w(F_o - F_c)^2$. H atoms were constrained to idealized positions ($\text{C-H} = 0.95 \text{ \AA}$) with fixed isotropic B values of 1.2 times the B value of the attached C atoms. The F atoms of the disordered anion were constrained to positions indicated by difference maps with 70% occupancy for the F1-F2-F3-F4 octahedron and 30% for the F3-F9-F10 octahedron; only the isotropic B values were refined for these atoms. The occupancies used were the averages for the F-atom multiplicities obtained by including them in several cycles of the least-squares refinement. All other non-H atoms were refined anisotropically for a total of 197 parameters. $R = 0.046$, $wR = 0.061$, $\text{GOF} = 1.2$, where non-Poisson $w^{-1} = [\sigma^2(I) + (0.08I)^2]/4F^2$. Final $(\Delta/\sigma)_{\text{max}} < 0.1$, $\rho_{\text{max}} = 0.87 (7)$ and $\rho_{\text{min}} = -0.91 (7) \text{ e \AA}^{-3}$ on final difference map. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974) and programs used were those of Enraf-Nonius (1982) *SDP*.* Table 1 gives the atomic coordinates and Table 2 selected bond distances and angles of the cation; no unusual distances or angles were observed for the anions or for the solvent molecule. Fig. 1 shows the cation with the numbering scheme.

Related literature. The structural parameters of the cation are to be compared with those observed in monofunctional 1,2,3,5-dithiadiazolium cations RCN_2S_2^+ , e.g. $R = \text{NMe}_2$ (Cordes, Goddard, Oakley & Westwood, 1989), Cl (Hofs, Mews, Clegg, Noltemeyer, Schmidt & Sheldrick, 1983), CF_3 (Hofs, Bats, Gleiter, Hartman, Mews, Eckert-Maksic, Oberhammer & Sheldrick, 1986) and CCl_3 (Andreasen, Hazell & Hazell, 1977).

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* Tables of H-atom coordinates, anisotropic temperature factors, distances and angles for the anions and the solvent, and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52157 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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trans-Carbonylodobis(triphenylphosphine)rhodium(I)

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Abstract. $[RhI(CO)\{P(C_6H_5)_3\}_2]$, $M_r = 782.41$, monoclinic, $P2_1/n$, $a = 9.823$ (2), $b = 15.340$ (2), $c = 21.980$ (3) Å, $\beta = 95.51$ (1)°, $V = 3296.7$ (8) Å³, $Z = 4$, $D_m = 1.50$ (1), $D_x = 1.58$ g cm⁻³, $\lambda(Mo K\alpha) = 0.71073$ Å, $\mu = 15.8$ cm⁻¹, $F(000) = 1552$, $T = 298$ K, $R = 0.060$ for 4278 absorption-corrected observed reflections. The coordination environment about the Rh atom is square planar with a maximum deviation [0.14 (14) Å] from planarity at the carbonyl C atom. Rh—C(1) = 1.81 (1), Rh—I = 2.683 (1), Rh—P(1) = 2.336 (2) and Rh—P(2) = 2.316 (2) Å.

Experimental. The oxidative addition of CH₃I to $[Rh(acac)(CO)(PPh_3)]$ gives an equilibrium mixture of $[Rh(acac)I(CH_3)(CO)(PPh_3)]$ and $[Rh(acac)I(COCH_3)(PPh_3)]$ which on reacting with PPh₃ in methanol yields a mixture of crystals consisting of ruby-red $[Rh(acac)I_2(PPh_3)_2]$ (Basson, Leipoldt, Potgieter, Roodt & van der Walt, 1986), orange-yellow $[Rh(acac)(COCH_3)(OCH_3)(PPh_3)_2]$ and light-yellow $[RhI(CO)(PPh_3)_2]$. Crystals of the title compound were selected from the mixture and recrystallized from methanol. Density determined by flotation in NaI solution. Crystal size 0.20 × 0.15 × 0.05 mm, Enraf-Nonius CAD-4F diffractometer, graphite monochromator, Mo K α radiation, $\omega/2\theta$ scan technique, variable scan width where $\Delta\omega = (0.5 + 0.34 \times \tan\theta)$ °, scan speed maximum 5.49° min⁻¹ in ω and minimum corresponding to 60 s per reflection, unit-cell parameters from least-squares refinement of 25

reflections with $7 < \theta < 17^\circ$, measuring range $3 < \theta < 25^\circ$, empirical absorption corrections (North, Phillips & Mathews, 1968) with minimum correction factor 0.94 and maximum 0.99, three standard reflections measured every 3600 s of X-ray exposure time varied by $\pm 0.1\%$, all possible reflections with $(\sin\theta)/\lambda < 0.60$ Å⁻¹ in the index ranges $0 < h < 11$, $0 < k < 18$, $-26 < l < 25$. 5997 unique reflections of which 4278 observed reflections with $I > 2.0\sigma(I)$ were used in all calculations (*XRAY72*, Stewart, Kruger, Ammon, Dickinson & Hall, 1972). The structure was solved by the heavy-atom method and subjected to anisotropic full-matrix least-squares refinement on F (380 variables). H atoms were not placed. Neutral-atom scattering factors (Cromer & Mann, 1968) and anomalous-dispersion corrections for Rh and I from *International Tables for X-ray Crystallography* (1962). Final $R = 0.060$ and $wR = 0.060$ with unit weights taken, $\Delta\rho_{\max} = 6.5$ and $\Delta\rho_{\min} = -4.5$ e Å⁻³ within 1 Å from iodine, $(\Delta/\sigma)_{\max} = 0.196$. Final atomic coordinates are given in Table 1* and main geometrical parameters in Table 2, according to the numbering scheme given in Fig. 1.

* Lists of anisotropic thermal parameters, structure factors and least-squares-plane deviations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52207 (32 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.