Methyl-H sites located from difference Fourier synthesis, mathematically optimized (tetrahedral geometry, C—H = 0·95 Å), and fixed. Final stage of refinement performed with 397 variables including all non-H positional and anisotropic thermal parameters, and one scale factor. Convergence yielded $R = 0{\cdot}040$, $wR = 0{\cdot}047$, $S = 1{\cdot}05$ and $(\Delta/\sigma)_{\max} = 0{\cdot}02$. When all (6084) reflections are included, $R = 0{\cdot}114$ and $wR = 0{\cdot}057$. Final difference synthesis produced $\Delta\rho_{\max} = 1{\cdot}06$ and $\Delta\rho_{\min} = -0{\cdot}96$ e Å^{-3}, both in the vicinity of the Os atom. Atomic scattering factors and anomalous-dispersion corrections from Cromer & Waber (1974). Positional and equivalent isotropic thermal parameters are listed in Table 1, selected interatomic distances and angles are given in Table 2.* Fig. 1 illustrates the molecular configuration, thermal motion and atom-numbering scheme. All computer programs from the *TEXSAN* crystal-structure-analysis package (Molecular Structure Corporation, 1985).

Related literature. This compound is one of a group of materials prepared in a study of the reactions of the hexabromoosmate ion with triphenylphosphine (Hinckley, Matusz, Kibala & Robinson, 1987; Hinckley, Ikuo & Robinson, 1988; Robinson, Hinckley & Ikuo, 1988).

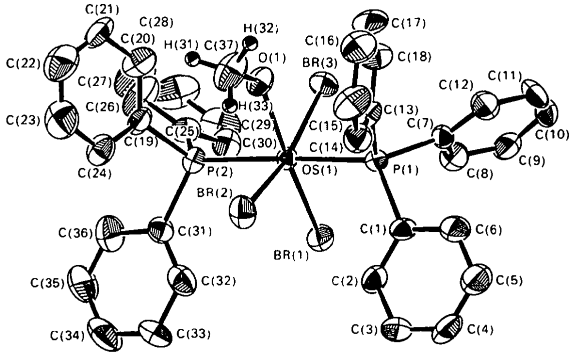

Fig. 1. Molecular configuration and atom-numbering scheme, thermal ellipsoids at the 50% probability level. Ring-H atoms omitted to improve clarity.

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* Lists of structure factors, H-atom coordinates, phenyl-ring bond distances and angles, intermolecular distances, torsion angles, anisotropic thermal parameters and selected least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52326 (51 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of $(Se_2N_2C—C_6H_4—CN_2Se_2)^{2+}.2SbF_6^-.3C_6H_5CN$

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Abstract. *P*-Di(1,2,3,5,-diselenadiazolium-4-yl)benzene hexafluoroantimonate benzonitrile solvate, $C_8H_4N_4Se_2^{2+}.2SbF_6^-.3C_7H_5N$, $M_r = 1252{\cdot}8$, triclinic, $P\bar{1}$, $a = 6{\cdot}829$ (2), $b = 13{\cdot}981$ (6), $c = 21{\cdot}320$ (9) Å, $\alpha = 72{\cdot}26$ (4), $\beta = 89{\cdot}03$ (3), $\gamma = 88{\cdot}78$ (3)°, $V = 1938$ (2) Å^3, $Z = 2$, $D_x = 2{\cdot}15$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 52{\cdot}1$ cm^{-1}, $F(000) = 1176$, $T = 293$ K, $R = 0{\cdot}049$ for 1975 reflections with $F_o^2 > 3\sigma(F_o^2)$. The unit cell contains four anions and six solvent molecules in general positions and two cations each located on crystallographic inversion centers. In the Se_2N_2C rings the Se—Se, Se—N, and

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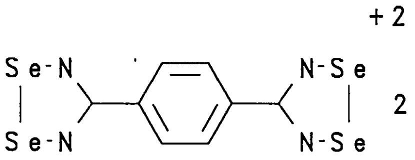
N—C distances average 2·259 (6), 1·72 (1), and 1·32 (4) Å, respectively, and the ring angles at Se, N and C average 91·1 (5), 117 (2) and 124 (2)°, respectively. In one cation the planar Se_2N_2C ring makes a dihedral angle of 24·4° with the plane of the central phenylene ring and in the other cation this angle is 10·1°.

Experimental. Title compound (I) prepared by the metathesis of $[(Se_2N_2C)C_6H_4(CN_2Se_2)]^{2+}.2Cl^-$ with $NOSbF_6$ in PhCN (Del bel Belluz, Cordes, Kristof, Kristof, Liblong & Oakley, 1990). Data crystal obtained by slow cooling of a benzonitrile solution.



(I)

Pale yellow crystal approximately 0·10 × 0·22 × 0·46 mm embedded in paraffin wax in a capillary tube because of air sensitivity; the crystal gradually turned dark upon exposure to the X-ray beam. 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 $16 < 2\theta < 18°$. Absorption correction based on ψ scans varied from 0·76 to 1·00. Data collected to $(\sin\theta)/\lambda$ of 0·54 $Å^{-1}$, $0 \leq h \leq 7$, $-15 \leq k \leq 15$, $-23 \leq l \leq 23$. The intensities of three standard reflections (154, $1\bar{5}\bar{4}$, $2\bar{3}\bar{5}$) varied a few per cent during the 20·2 h of data collection; a non-linear correction was made which ranged from 0·98 to 1·04. 5571 reflections measured, 5060 unique (R_{int} = 0·023), 3085 reflections with $I < 3\sigma(I)$ where $\sigma^2(I) = \sigma^2_{cs}(I) + (0{\cdot}05I)^2$; $\sigma_{cs}(I)$ is standard deviation of I based on counting statistics. The large number of weak reflections was apparently due to the lack of long-range order in this rather soft solvent-laden crystal: less than 17% of the 2110 reflections with θ above 19° had $I > 3\sigma(I)$. Solved by direct methods using *MULTAN*11/82 (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 (C—H = 0·95 Å) with fixed isotropic B values of 1·2 times the B of the attached C atoms. Due to the paucity of data only the Sb, Se, F and cation-N atoms were refined anisotropically. The other non-H atoms were refined isotropically for a total of 327 parameters. R = 0·049, wR = 0·052, S = 1·3, where $I > 3\sigma(I)$ and non-Poisson $w^{-1} = [\sigma^2(I) + (0{\cdot}04I)^2]/4F^2$. Final $(\Delta/\sigma)_{max} < 0{\cdot}04$, $\Delta\rho_{max}$ = 0·62 (15) and $\Delta\rho_{min}$ = −0·27 (15) e $Å^{-3}$ 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. Fig. 1 shows the cation of Se(1) with the numbering

Table 1. *Fractional coordinates and isotropic or equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

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

	x	y	z	B/B_{eq}($Å^2$)
Sb(1)	0·7963 (2)	0·2602 (1)	0·23748 (8)	4·16 (4)
Sb(2)	0·6917 (2)	0·7588 (1)	0·22798 (8)	3·91 (4)
Se(1)	0·0766 (4)	0·9618 (2)	0·2590 (1)	4·42 (6)
Se(2)	0·3652 (4)	1·0308 (2)	0·2710 (1)	4·54 (6)
Se(3)	0·4097 (4)	0·4829 (2)	0·2551 (1)	4·79 (6)
Se(4)	0·1143 (4)	0·5420 (2)	0·2759 (1)	4·92 (7)
F(1)	0·819 (2)	0·3775 (9)	0·2566 (7)	8·2 (4)
F(2)	0·846 (3)	0·326 (1)	0·1509 (8)	10·9 (6)
F(3)	0·766 (2)	0·1392 (9)	0·2170 (8)	9·1 (5)
F(4)	0·748 (3)	0·193 (1)	0·3234 (9)	12·7 (7)
F(5)	0·533 (2)	0·280 (1)	0·2302 (8)	9·8 (5)
F(6)	1·059 (2)	0·234 (1)	0·2511 (9)	10·7 (5)
F(7)	0·723 (2)	0·6400 (8)	0·2034 (6)	6·0 (4)
F(8)	0·653 (2)	0·8276 (9)	0·1410 (7)	8·6 (5)
F(9)	0·673 (2)	0·8788 (8)	0·2500 (7)	7·1 (4)
F(10)	0·736 (2)	0·683 (1)	0·3168 (7)	9·2 (5)
F(11)	0·425 (2)	0·736 (1)	0·2389 (8)	8·7 (4)
F(12)	0·955 (2)	0·778 (1)	0·2148 (8)	8·7 (5)
N(1)	0·001 (2)	0·963 (1)	0·3363 (7)	3·7 (4)
N(2)	0·299 (2)	1·034 (1)	0·3483 (7)	3·6 (4)
N(3)	0·491 (2)	0·478 (1)	0·3326 (7)	4·1 (4)
N(4)	0·187 (2)	0·539 (1)	0·3540 (8)	4·4 (5)
N(5)	0·304 (3)	−0·002 (1)	0·137 (1)	6·5 (5)*
N(6)	0·820 (3)	0·482 (1)	0·861 (1)	6·4 (5)*
N(7)	0·336 (3)	0·789 (1)	0·397 (1)	7·4 (6)*
C(1)	0·124 (3)	0·995 (1)	0·372 (1)	3·7 (5)*
C(2)	0·061 (3)	1·001 (1)	0·4380 (9)	2·8 (4)*
C(3)	−0·135 (3)	1·001 (1)	0·4554 (9)	3·2 (5)*
C(4)	0·197 (3)	0·999 (1)	0·4863 (9)	3·2 (4)*
C(5)	0·371 (3)	0·509 (1)	0·3691 (9)	2·8 (4)*
C(6)	0·440 (3)	0·506 (1)	0·436 (1)	4·0 (5)*
C(7)	0·636 (3)	0·485 (1)	0·452 (1)	4·5 (5)*
C(8)	0·306 (3)	0·519 (1)	0·487 (1)	4·0 (5)*
C(9)	0·235 (3)	−0·081 (1)	0·052 (1)	4·3 (5)*
C(10)	0·177 (3)	−0·174 (2)	0·066 (1)	5·4 (6)*
C(11)	0·138 (4)	−0·218 (2)	0·016 (1)	7·3 (7)*
C(12)	0·154 (4)	−0·170 (2)	−0·047 (1)	7·2 (7)*
C(13)	0·210 (3)	−0·074 (2)	−0·063 (1)	5·9 (6)*
C(14)	0·253 (3)	−0·026 (2)	−0·011 (1)	5·7 (6)*
C(15)	0·271 (3)	0·967 (1)	0·096 (1)	4·9 (6)*
C(16)	0·744 (3)	0·574 (1)	0·946 (1)	4·7 (5)*
C(17)	0·749 (3)	0·517 (2)	1·011 (1)	5·3 (6)*
C(18)	0·703 (3)	0·566 (2)	1·060 (1)	6·1 (6)*
C(19)	0·653 (4)	0·665 (2)	1·036 (1)	6·8 (7)*
C(20)	0·637 (4)	0·729 (2)	0·970 (1)	8·0 (8)*
C(21)	0·686 (4)	0·673 (2)	0·926 (1)	7·1 (7)*
C(22)	0·779 (4)	0·522 (2)	0·899 (1)	6·6 (7)*
C(23)	0·392 (3)	0·239 (1)	0·518 (1)	4·4 (5)*
C(24)	0·425 (3)	0·237 (2)	0·455 (1)	5·2 (6)*
C(25)	0·282 (4)	0·250 (2)	0·411 (1)	6·3 (6)*
C(26)	0·099 (4)	0·268 (2)	0·422 (1)	7·8 (7)*
C(27)	0·055 (4)	0·274 (2)	0·481 (1)	7·0 (7)*
C(28)	0·196 (4)	0·262 (2)	0·533 (1)	6·3 (6)*
C(29)	0·463 (3)	0·780 (1)	0·432 (1)	4·7 (5)*

* Atoms refined isotropically.

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* Tables of distances and angles in the anions and solvent molecules, anisotropic temperature factors, H-atom positions and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52433 (39 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Selected bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Se(1)	Se(2)	2·265 (3)	Se(2)	Se(1)	N(1)	90·6 (5)
Se(1)	N(1)	1·724 (15)	Se(1)	Se(2)	N(2)	91·5 (5)
Se(2)	N(2)	1·714 (15)	Se(4)	Se(3)	N(3)	91·0 (5)
Se(3)	Se(4)	2·253 (3)	Se(3)	Se(4)	N(4)	91·2 (5)
Se(3)	N(3)	1·733 (15)	Se(1)	N(1)	C(1)	118 (1)
Se(4)	N(1)	1·73 (2)	Se(2)	N(2)	C(1)	117 (1)
C(1)	N(1)	1·32 (2)	Se(3)	N(3)	C(5)	117 (1)
C(1)	N(2)	1·35 (2)	Se(4)	N(4)	C(5)	115 (1)
C(1)	C(2)	1·49 (2)	N(1)	C(1)	N(2)	122 (2)
C(3)	C(4′)	1·31 (2)	N(1)	C(1)	C(2)	120 (2)
C(2)	C(3)	1·38 (2)	N(2)	C(1)	C(2)	118 (2)
C(2)	C(4)	1·39 (2)	C(1)	C(2)	C(3)	121 (2)
C(5)	N(3)	1·28 (2)	C(1)	C(2)	C(4)	121 (2)
C(5)	N(4)	1·33 (2)	C(3)	C(2)	C(4)	118 (2)
C(5)	C(6)	1·49 (3)	C(2)	C(3)	C(4′)	123 (2)
C(6)	C(7)	1·39 (2)	C(2)	C(4)	C(3′)	119 (2)
C(6)	C(8)	1·47 (2)	N(3)	C(5)	N(4)	126 (2)
C(7)	C(8″)	1·35 (2)	N(2)	C(5)	C(6)	117 (2)
Sb	F	1·816–1·895	N(4)	C(5)	C(6)	117 (2)
		1·849 (av.)	C(5)	C(6)	C(7)	120 (2)
			C(5)	C(6)	C(8)	123 (2)
			C(7)	C(6)	C(8)	117 (2)
			C(6)	C(7)	C(8″)	119 (2)
			C(6)	C(8)	C(7″)	124 (2)

scheme; the other cation was numbered in a corresponding manner.

Related literature. The structural parameters of the cation in this crystal can be compared to those found in the closely related monofunctional $(PhCN_2Se_2)^+$ cation (Del bel Belluz, Cordes, Kristof, Kristof, Liblong & Oakley, 1990) and those of the corresponding sulfur-based dication $(S_2N_2C{-}C_6H_4{-}CN_2S_2)^{2+}$ (Liblong, Oakley & Cordes, 1990).

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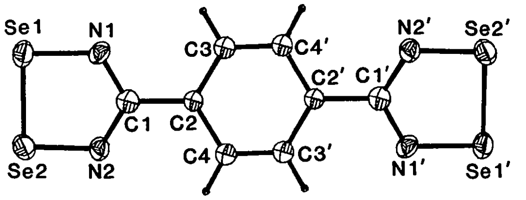

Fig. 1. *ORTEP* (Johnson, 1976) drawing and numbering scheme for the cation containing Se(1). The ellipsoids are drawn at the 30% probability surface and H atoms have been given arbitrary radii for figure clarity.

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Acta Cryst. (1990). C**46**, 701–703

Structure of Triphenyltin Adduct $C_{26}H_{28}O_2Sn$* Resulting from a Free-Radical Reaction

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Abstract. $C_{26}H_{28}O_2Sn$, $M_r = 491{\cdot}20$, monoclinic, $P2_1/c$, $a = 6{\cdot}557\,(2)$, $b = 36{\cdot}419\,(8)$, $c = 9{\cdot}964\,(4)$ Å, $\beta = 110{\cdot}23\,(3)°$, $V = 2232{\cdot}6$ Å^3, $Z = 4$, $D_x = 1{\cdot}462$ Mg m^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}54178$ Å, $\mu = 9{\cdot}45$ mm^{-1}, $F(000) = 1000$, $T = 220$ K, $R = 0{\cdot}055$, $wR = 0{\cdot}050$ for 2569 observed reflections. During the course of some triphenyltin hydride-mediated cyclizations of activated and unactivated dienes, it was found that a major product resulted from addition of the hydride to the terminal double bond followed by intramolecular cyclization. The structure was analyzed by X-rays to determine the relative configuration of the ring junction which could not be established unambiguously by ^{1}H NMR studies. The

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* (±)-*cis*-4-(Triphenylstannylmethyl)-2-oxabicyclo[3.3.0]octan-7-ol.

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