(Belli dell'Amico & Calderazzo, 1973). The shortest Au—Au distance in the present compound is 5·68 Å; so there is no evidence of Au—Au interactions as in (i-$C_3H_7NH_2$)$AuC{\equiv}CC_6H_5$ (3·27 and 3·72 Å) and (piperidine)AuCl [3·30 Å; Guy, Jones, Mays & Sheldrick (1977)].

We are grateful to the Science Research Council for providing the diffractometer, and for financial support to PDG and JJG. The calculations were performed with the Cambridge University IBM 370/165 computer and programs written by GMS, and Fig. 1 was drawn with *PLUTO* written by Dr W. D. S. Motherwell.

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Trifluoromethylselenium Trichloride

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Abstract. CF_3SeCl_3, orthorhombic, *Pbca*, $a = 18{\cdot}938$ (23), $b = 7{\cdot}399$ (20), $c = 18{\cdot}880$ (22) Å, $U = 2646$ Å^3, $Z = 16$, $D_x = 2{\cdot}55$ g cm^{-3}, μ(Mo $K\alpha$) = 66·08 cm^{-1}. The structure was solved by direct methods and refined to an R of 0·093 for 474 unique diffractometer data. It consists of discrete dimers $(CF_3SeCl_3)_2$, in which each Se is coordinated by a CF_3 group, two bridging Cl and two terminal Cl in an approximately square-pyramidal arrangement with axial CF_3. The two $SeCl_4$ planes make an angle of 154° with one another, and the dimer possesses approximate *mm* symmetry.

Introduction. Vapour-phase studies on the Group VI tetrahalides are consistent with structures based on a trigonal bipyramid with an equatorial position occupied by a lone pair, in accordance with the VSEPR hypothesis. Similar geometries are found in crystalline Ph_2SeX_2 (McCullough & Hamburger, 1941, 1942) and (p-tolyl)$_2$SeX_2 (McCullough & Marsh, 1950) (X = Cl, Br). On the other hand, the crystal structure of $TeCl_4$ (Buss & Krebs, 1971) contains isolated tetramers in which each Te is surrounded by a distorted octahedron of three terminal and three bridging Cl atoms. The vibrational spectrum of CF_3SeCl_3 also indicates extensive Cl-bridging in the solid, but an X-ray crystallographic study was necessary to determine the structure.

A sample of CF_3SeCl_3 was prepared by the action of excess Cl_2 on $(CF_3Se)_2$ and left in a sealed tube for about a year, during which time a few well formed crystals grew by sublimation. These were transferred to Lindemann glass capillary tubes in a dry bag because the compound is extremely sensitive to moisture. Intensities were determined with an automated Stoe two-circle diffractometer, Mo $K\alpha$ radiation, graphite monochromator and a crystal 0·17 × 0·57 × 0·23 mm (layers $h0$–$8l$). 933 reflexions were measured in an approximately constant count mode; after rejection of reflexions with a net count less than 2·5σ based on counting statistics, equivalent data were merged to give 474 unique reflexions. Lorentz, polarization and absorption corrections were applied. a and c were obtained by a least-squares fit to the diffractometer ω angle measurements of 120 $h0l$ reflexions; b was obtained from diffractometer μ measurements.

The structure was solved by multisolution sigma-2 sign expansion followed by successive difference syntheses. It was apparent that the F atoms of both crystallographically independent CF_3 groups were smeared out by the effects of thermal motion, and in the final full-matrix least-squares cycles it was necessary to constrain C—F to 1·33 Å and F—C—F to 108·5°. Anisotropic temperature factors were employed for the Se and F atoms; interlayer scale factors were fixed at the values to which they had refined during earlier cycles in which only isotropic temperature factors had been used, so the final values of U_{22} have little physical significance. Complex neutral-atom scattering factors were employed; the weighting scheme was $w = [\sigma^2(F) + 0{\cdot}0003F_o^2]^{-1}$. The refinement converged to $R' = \Sigma w^{1/2}\Delta/\Sigma w^{1/2}|F_o| = 0{\cdot}081$ and $R = 0{\cdot}093$. Positional and thermal parameters are given in Tables 1 and 2, and bond lengths and angles in Tables 3 and 4.*

Discussion. The structure (Fig. 1) consists of discrete dimers $(CF_3SeCl_3)_2$; each Se is coordinated by a CF_3 group, two bridging Cl and two terminal Cl atoms in an approximately square-pyramidal arrangement. The CF_3 groups occupy the axial positions of the square pyramids, and are *cis* to one another; the repulsive interactions between them are probably responsible for the deviation of the $Cl_2SeCl_2SeCl_2$ skeleton from planarity [the angle between the least-squares mean planes defined by Cl(1)Cl(2)Cl(4)Cl(5)Se(1) and Cl(1)Cl(3)Cl(4)Cl(6)Se(2) is 154°]. The lone-pair electrons presumably occupy the vacant positions in the 'ψ octahedron' around each Se atom, but there are also short intermolecular SE···Cl contacts in approximately these directions [Se(1)···Cl(4′) 3·19, Se(2)···Cl(3′) 3·41 Å]. It is debatable whether they represent significant covalent interactions, possibly in-

Table 1. *Atom coordinates* (× 10^4) *and isotropic temperature factors* (Å^2 × 10^3)

	x	y	z	U
Se(1)	3416 (3)	697 (7)	2412 (2)	
Se(2)	3423 (3)	−1031 (7)	4337 (3)	
Cl(1)	4281 (7)	697 (21)	3583 (7)	60 (5)
Cl(2)	3977 (8)	3002 (21)	1966 (8)	60 (5)
Cl(3)	2605 (8)	−2586 (25)	4950 (8)	76 (5)
Cl(4)	2806 (7)	−1962 (22)	3131 (8)	71 (6)
Cl(5)	2670 (7)	348 (23)	1577 (8)	65 (6)
Cl(6)	3959 (8)	−123 (22)	5303 (8)	76 (5)
C(1)	4107 (12)	−969 (35)	1937 (13)	95 (25)
F(1)	4753 (12)	−598 (35)	2172 (13)	
F(2)	4103 (12)	−786 (35)	1236 (13)	
F(3)	3957 (12)	−2678 (35)	2093 (13)	
C(2)	4036 (17)	−3305 (39)	4265 (18)	75 (22)
F(4)	3780 (17)	−4411 (39)	3773 (18)	
F(5)	4061 (17)	−4192 (39)	4878 (18)	
F(6)	4690 (17)	−2481 (39)	4083 (18)	

Table 3. *Bond lengths* (Å)

Symmetry transformation relating designated atoms to reference atoms at (x,y,z): (i) $\frac{1}{2} - x, -\frac{1}{2} + y, z$.

Cl(1)–Se(1)	2·75 (2)	Cl(1)–Se(2)	2·51 (2)
Cl(2)–Se(1)	2·18 (2)	Cl(3)–Se(2)	2·25 (2)
Cl(4)–Se(1)	2·65 (2)	Cl(4)–Se(2)	2·65 (2)
Cl(5)–Se(1)	2·13 (2)	Cl(6)–Se(2)	2·19 (2)
C(1)–Se(1)	2·01 (2)	C(2)–Se(2)	2·05 (2)
Cl(4)–Se(1^{i})	3·19	Cl(3)–Se(2^{i})	3·41

Table 4. *Bond angles* (°)

Cl(2)–Se(1)–Cl(1)	91·2 (6)	Cl(3)–Se(2)–Cl(1)	176·1 (5)
Cl(4)–Se(1)–Cl(1)	81·3 (6)	Cl(4)–Se(2)–Cl(1)	86·0 (6)
Cl(4)–Se(1)–Cl(2)	171·9 (6)	Cl(4)–Se(2)–Cl(3)	90·3 (6)
Cl(5)–Se(1)–Cl(1)	171·2 (6)	Cl(6)–Se(2)–Cl(1)	90·9 (6)
Cl(5)–Se(1)–Cl(2)	97·6 (7)	Cl(6)–Se(2)–Cl(3)	92·8 (7)
Cl(5)–Se(1)–Cl(4)	90·0 (6)	Cl(6)–Se(2)–Cl(4)	176·6 (6)
C(1)–Se(1)–Cl(1)	88·4 (10)	C(2)–Se(2)–Cl(1)	90·8 (11)
C(1)–Se(1)–Cl(2)	89·4 (10)	C(2)–Se(2)–Cl(3)	90·2 (12)
C(1)–Se(1)–Cl(4)	93·3 (10)	C(2)–Se(2)–Cl(4)	88·8 (12)
C(1)–Se(1)–Cl(5)	91·5 (10)	C(2)–Se(2)–Cl(6)	92·5 (12)
Se(2)–Cl(1)–Se(1)	94·0 (6)	Se(2)–Cl(4)–Se(1)	93·1 (6)
F(1)–C(1)–Se(1)	108·8	F(4)–C(2)–Se(2)	110·2
F(2)–C(1)–Se(1)	112·2	F(5)–C(2)–Se(2)	111·6
F(3)–C(1)–Se(1)	110·2	F(6)–C(2)–Se(2)	109·4

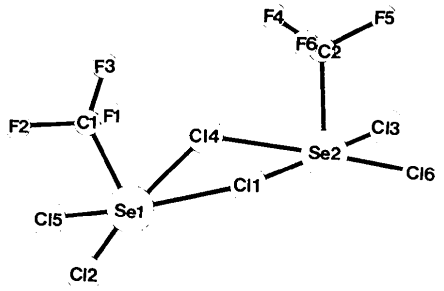

Fig. 1. The trifluoromethylselenium trichloride dimer.

Table 2. *Anisotropic temperature factors* (Å^2 × 10^3)

The temperature factor exponent takes the form: $-2\pi^2(U_{11}h^2a^{*2} + \cdots + 2U_{12}hka^*b^*)$.

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Se(1)	43 (3)	40 (3)	33 (4)	0 (3)	1 (3)	1 (5)
Se(2)	59 (4)	43 (3)	37 (4)	−4 (3)	−2 (3)	−6 (5)
F(1)	83 (26)	172 (43)	120 (32)	−47 (32)	−34 (22)	57 (37)
F(2)	192 (48)	158 (47)	198 (49)	−74 (44)	71 (41)	96 (44)
F(3)	227 (50)	55 (27)	245 (52)	42 (30)	167 (43)	64 (37)
F(4)	453 (98)	121 (47)	201 (54)	−73 (43)	−159 (61)	148 (60)
F(5)	337 (81)	197 (71)	381 (98)	108 (70)	64 (74)	221 (64)
F(6)	200 (57)	153 (52)	405 (84)	53 (58)	219 (63)	77 (49)

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31980 (4 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

volving donation of electron density into the Se 4*d* orbitals, or whether they are electrostatic dipole–dipole interactions. Similar short Se···Cl contacts are found in $(C_9H_8NO)SeOCl_3$ (Cordes, 1967) and py_2SeOCl_2 (Lindqvist & Nahringbauer, 1959). The Se–C distances (mean 2·03 Å) are consistent with the values obtained by electron diffraction in $(CF_3)_2Se$[1·978 (9) Å] and $(CF_3Se)_2$ [2·018 (20) Å] by Marsden & Sheldrick (1971*a*,*b*). The mean Se–Cl(terminal) distance of 2·19 (2) Å is close to the sum of Pauling covalent radii, but significantly shorter than the axial Se–Cl bonds in $(p\text{-tolyl})_2SeCl_2$ [mean 2·38(2) Å] and the terminal Se–Cl bonds in py_2SeOCl_2 and $(C_9H_8NO)SeOCl_3$ [mean 2·39 (2) and 2·25 (2) Å respectively]. Similarly, the Se–Cl(bridging) bonds (mean 2·64 Å) are shorter than those in the other five-coordinate Se species, although it should be noted that the bridging Cl in $(C_9H_8NO)SeOCl_3$ is also involved in hydrogen bonding. One of the Se–Cl–Se bridges is symmetrical but the other is not; this is probably because of the influence of the Se(1)···Cl(4′) interaction. Similarly, the Se(2)···Cl(3′) interaction probably accounts for the slight lengthening of the Se(2)–Cl(3) bond.

We are grateful to the Science Research Council for providing the diffractometer, and for financial support to CJM and RT. The calculations were performed on the Cambridge University IBM 370/165 computer with programs written by GMS, and Fig. 1 was drawn with *PLUTO* written by Dr W. D. S. Motherwell.

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(±)-(*Z*,*Z*)-(1–3-η:5–7-η-Heptadienediyl)rhodium(I) Hexafluoroacetylacetonate

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Abstract. $C_{12}H_{11}F_6O_2Rh$, orthorhombic, $C222_1$; $a = 8{\cdot}599$ (1), $b = 14{\cdot}684$ (3), $c = 11{\cdot}264$ (2) Å, $V = 1422{\cdot}3$ Å^3 at 18 °C, $Z = 4$, $D_x = 1{\cdot}89$ g cm^{-3}; μ(Cu $K\alpha$) = 148·4 cm^{-1}. The ring of the parent *trans*-divinylcyclopropane has completely opened (C–C, 2·25 Å), and it coordinates to Rh as two allyl groups (Rh–C, between 2·09 and 2·26 Å). The complex has twofold symmetry, and Rh is also coordinated by two O atoms of the hexafluoroacetylacetonate (Rh–O, 2·15 Å).

Introduction. The title compound was prepared by the reaction of *trans*-divinylcyclopropane with bis(ethylene)rhodium(I) hexafluoroacetylacetonate and recrystallized from pentane (Brown, Golding & Stofko, 1976). A crystal was mounted in a capillary because it tended to sublime, and data were collected rapidly (over 10 h) on a Syntex $P2_1$ diffractometer with graphite-monochromatized Cu $K\alpha$ radiation to a $2\theta_{\max.}$ of 130° at scan rates between 1·5 and 29·3° min^{-1}, depending on the intensity of a 2 s prescan. There was significant loss of intensity of three standard reflexions and the data were renormalized. 521 reflexions were considered observed, $I/\sigma(I) \geq 3{\cdot}0$, and corrected for Lorentz, polarization and absorption effects.

Apparent systematic absences: hkl, $h + k = 2n + 1$ and $h0l$, $l = 2n + 1$, indicate space groups $Cmcm$, $Cmc2_1$ or $C2cm$ (= $Ama2$ rotated). With the Rh position, from a Patterson synthesis, in the special position 0, y, $\frac{1}{4}$, $Cmcm$ (which was provisionally assumed) requires mm site symmetry. With this, light atoms were located and $R = 0{\cdot}13$ was reached, but the hydrocarbon appeared to be nine-membered, with four atoms off the mirror planes. This was implausible, and $Cmc2_1$ or $C2cm$ were no more satisfactory (requiring m symmetry). It seemed likely, therefore, that the apparent glide-plane absences had arisen by accident because all but two of the atoms nearly conformed to it; if it is relaxed, $C222_1$, $C222$, $Cmm2$ and $Cmmm$ are possible, of which only the first has a special position consistent