volving donation of electron density into the Se 4*d* orbitals, or whether they are electrostatic dipole–dipole interactions. Similar short Se$\cdots$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)$\cdots$Cl(4′) interaction. Similarly, the Se(2)$\cdots$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}; $\mu(\text{Cu } K\alpha) = 148{\cdot}4$ 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 with the Patterson synthesis. It imposes molecular symmetry 2, and with it refinement proceeded smoothly to a final R of 0·067. All atoms were given anisotropic temperature factors, and statistical weights were used which analysis showed to be satisfactory. Analytical scattering factors were used (*International Tables for X-ray Crystallography*, 1974). Anomalous dispersion was included, but, although the crystals are chiral, calculation with the two hands showed only infinitesimal differences, no doubt because all but two atoms are related by a pseudosymmetry centre. A final difference synthesis showed no significant features, but the large temperature factors parallel to **a** for C(13) suggest that this atom may well occupy two slightly separated positions, and the abnormally long bond to C(12) is probably an artefact. Similarly, the CF_3 groups which were approximated by five partially occupied F atom positions may in reality show more complex disorder. The disorder, the pseudosymmetry and the crystal decomposition clearly limit the accuracy of the determination. Computing was carried out with X-RAY 72 (Stewart, Kruger, Ammon, Dickinson & Hall, 1972) on a CDC 7600 computer.

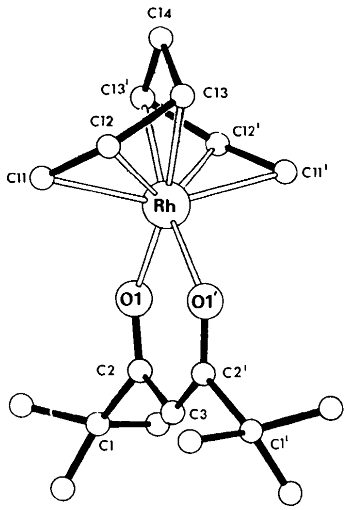

Fig. 1. The atomic numbering of the molecule. Only three of the F atoms are shown.

Fig. 1 shows the atomic numbering. Table 1 gives atomic coordinates and temperature factors, and Table 2 the bond lengths and angles.*

Discussion. This structure was studied to discover the fate of *trans*-divinylcyclopropane on coordination; a preliminary communication has been published (Alcock, Brown, Conneely & Stofko, 1975), and a full paper will examine the chemical implications (Brown,

Table 2. *Bond lengths* (Å) *and angles* (°) *with standard deviations in parentheses*

Rh—C(11)	2·26 (4)	C(11)—Rh—C(12)	36 (2)
Rh—C(12)	2·23 (3)	C(12)—Rh—C(13)	44 (2)
Rh—C(13)	2·09 (3)	C(13)—Rh—C(13′)	66 (1)
Rh—O(1)	2·15 (1)	O(1)—Rh—O(1′)	88 (1)
C(11)—C(12)	1·40 (7)	C(11)—C(12)—C(13)	117 (3)
C(12)—C(13)	1·63 (8)	C(12)—C(13)—C(14)	119 (4)
C(13)—C(14)	1·48 (3)	C(13)—C(14)—C(13′)	100 (2)
C(13)···C(13′)	2·25 (7)	C(2)—O(1)—Rh	122 (1)
O(1)—C(2)	1·23 (2)	O(1)—C(2)—C(3)	132 (2)
C(2)—C(3)	1·37 (2)	O(1)—C(2)—C(1)	110 (2)
C(2)—C(1)	1·68 (4)	C(1)—C(3)—C(1′)	123 (2)
C(1)—F	1·24 (av.)	C(2)—C(1)—F	111 (av.)

Table 1. *Atomic coordinates and temperature factors* (×10³) *with standard deviations in parentheses*

$$T=\exp[-2\pi^2(U_{11}h^2a^{*2}+U_{22}k^2b^{*2}+U_{33}l^2c^{*2}+2U_{12}hka^*b^*+2U_{13}hla^*c^*+2U_{23}klb^*c^*)].$$

	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Rh	0	65·2 (1)	250	9·1 (1)	3·44 (7)	5·0 (1)	0	−0·7 (7)	0
C(11)	254 (4)	30 (2)	229 (5)	11 (2)	8 (2)	7 (5)	4 (2)	3 (3)	4 (3)
C(12)	202 (4)	0 (1)	341 (3)	11 (2)	5 (1)	7 (2)	4 (2)	−2 (2)	1 (2)
C(13)	38 (9)	−54 (1)	346 (2)	33 (9)	4 (1)	9 (2)	7 (3)	−5 (4)	3 (1)
C(14)	0	−119 (1)	250	16 (3)	4 (1)	4 (1)	0	1 (11)	0
O(1)	23 (6)	170 (1)	381 (1)	9 (3)	4 (1)	6 (1)	1 (1)	0 (2)	0 (1)
C(1)	−24 (9)	314 (2)	476 (3)	9 (5)	5 (1)	9 (2)	−1 (3)	−4 (5)	0 (1)
C(2)	21 (6)	252 (1)	356 (2)	5 (3)	4 (1)	8 (1)	−1 (1)	−2 (2)	−1 (1)
C(3)	0	297 (2)	250	10 (2)	6 (1)	6 (2)	0	6 (4)	0
F(1)†	−83 (9)	378 (4)	455 (6)	20 (10)	6 (4)	10 (4)	4 (5)	−8 (5)	−5 (3)
F(2)†	−57 (8)	280 (2)	560 (4)	19 (10)	5 (2)	2 (2)	3 (2)	−2 (3)	1 (1)
F(3)†	−151 (11)	318 (7)	496 (9)	12 (5)	14 (6)	15 (8)	5 (4)	−7 (5)	−9 (6)
F(4)†	128 (13)	291 (8)	541 (9)	17 (8)	25 (9)	11 (7)	10 (7)	−8 (6)	−13 (6)
F(5)†	−6 (15)	403 (4)	459 (6)	20 (8)	3 (2)	6 (2)	−4 (4)	−5 (5)	−3 (1)

† 0·5 occupancy.

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31981 (4 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

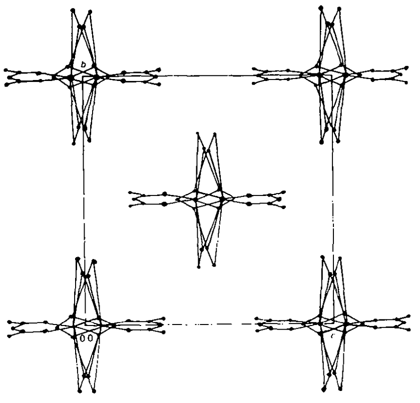
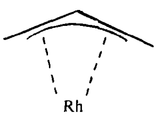
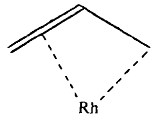


Fig. 2. A packing diagram viewed down **a**. F atoms are omitted.

Golding & Stofko, 1976). Structurally, the major effect is the opening of the cyclopropane ring, *i.e.* conversion of a bis(vinyl) to a bis(allyl) hydrocarbon; the C(13)–C(13′) distance is 2·25 Å. The resulting complex is similar to the bis(η-allyl)rhodium chloride dimer (McPartlin & Mason, 1967), but the presence of the bridgehead atom, C(14), produces a significant difference. In an ideal η-allyl, all three atoms are sp^2 hybridized, the p orbitals point towards the metal, and all five H atoms are to a first approximation in the plane of the three C atoms.* However, C(14), replacing one terminal H atom, is 0·80 Å out of the plane of C(11)–C(13). The p orbital on C(11)–C(12) can then only be weakly conjugated with that on C(13) [dihedral angle of 60° between C(11)–C(13) and C(13)–C(14)–C(13′)]; the bonding must largely go over from (*A*) to (*B*).



(*A*)



(*B*)

In keeping with this, the Rh–C(13) length, 2·09 Å, is considerably shorter than Rh–C(11) and Rh–C(12) at 2·26 and 2·23 Å. In contrast, if there is a two-carbon bridge, as in $C_{10}H_{16}Ru(PF_3)Cl_2$ (Hitchcock, Nixon & Sinclair, 1975), the atoms corresponding to C(14) are virtually in the allyl planes (by visual inspection of the published figure); the individual Rh–C lengths have not been published, but would be expected to be more uniform.

The packing (Fig. 2) is regular and dominated by F–F interactions.

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* In theory, this planarity could be affected by rehybridization from sp^2 to sp^3 if partly localized M–C σ-bonds are formed, while experimentally, in 2-methyl derivatives, the methyl group may be out of plane either towards or away from the metal atom (Marsh, Howard & Woodward, 1973).

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3-Pyridinesulphonic Acid

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Abstract. $C_5H_5NO_3S$, an antimetabolite of niacin, orthorhombic, *Pbca*; $a = 11{\cdot}449$ (2), $b = 14{\cdot}975$ (3), $c = 7{\cdot}186$ (1) Å, $V = 1232$ Å^3; $Z = 8$, $D_c = 1{\cdot}718$, $D_o = 1{\cdot}72$ g cm^{-3}; $M_r = 159{\cdot}2$. The structure was solved by heavy-atom and Fourier methods and refined by full-matrix least squares to $R = 11{\cdot}5\%$ for 910 in-

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* Contribution No. 439.