an approximately tetrahedral coordination with two hydrogen-bond acceptors (Cl^- ions) and two hydrogen-bond donors (ammonia molecules). The coordination about the chloride ions is rather irregular. Each has a hydrogen-bonded water molecule as closest neighbor, with distances 3.18 Å for Cl(1)–O(3) and 3.16 Å for Cl(2f)–O(4).

It is interesting to note that neither of the bridging NO_2 and NH_2 groups of the cation is involved in hydrogen bonding.

Thermal Motion.—The ellipsoids of thermal motion for the atoms of the cation are shown in Figure 2. The rms amplitudes along the principal directions range from 0.13 to 0.26 Å. The smallest and most isotropic movements are associated with the Co atoms. The atoms at the periphery of the cation have somewhat more pronounced movements than the atoms of the central ring. The largest thermal vibrations in the structure are displayed by the water molecules and chloride ions, with rms amplitudes up to 0.31 Å; their ellipsoids are described in Table VII. No attempt was made to correct the bond lengths for the effects of thermal vibration.

Acknowledgments.—This research was supported in part by the National Science Foundation under Grant No. GP-5768. We wish to thank Dr. Sten Samson for his assistance with the data collection. We also wish to acknowledge the help of Miss Lillian Casler in the preparation of the drawings.

CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY,
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The Crystal and Molecular Structure of Chlorobis(triphenylstibine)tetrakis(trifluoromethyl)rhodiacyclopentadiene–Dichloromethane Solvate, $RhCl(Sb(C_6H_5)_3)_2C_4(CF_3)_4 \cdot CH_2Cl_2$[1]

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Received December 8, 1969

The structure of chlorobis(triphenylstibine)tetrakis(trifluoromethyl)rhodiacyclopentadiene–dichloromethane solvate, $RhCl(Sb(C_6H_5)_3)_2C_4(CF_3)_4 \cdot CH_2Cl_2$, has been determined from three-dimensional X-ray data collected by counter methods. The final conventional and weighted R factors obtained from a block-diagonal least-squares refinement for 2537 reflections are both 0.044. The material crystallizes in the monoclinic system with space group $P2_1/c$ and a unit cell of dimensions $a = 13.250$ (1), $b = 25.496$ (2), $c = 16.852$ (1) Å; $\beta = 125.4$ (2)°. The calculated density of 1.805 g/cm^3 for four formula units agrees well with the experimental value, 1.80 (2) g/cm^3, determined by flotation. The crystal consists of discrete monomeric molecules interspersed with solvent molecules which do not interact significantly with the metal atoms. The coordination about rhodium is in the form of a slightly distorted trigonal bipyramid with Sb atoms in axial positions and a Cl atom and the 1 and 4 C atoms of the $C_4(CF_3)_4$ moiety occupying equatorial positions. The RhC_4 portion constitutes a five-membered ring which is planar. The chlorine atom and the carbon atoms of the trifluoromethyl groups are also very nearly in the same plane. The average Rh–C distance is 1.98 (1) Å, suggesting Rh–C π bonding. The C–C distances in the ring vary to some extent but are consistent with a considerable degree of delocalization over the four carbon atoms. Distortions from the idealized C_{2v} symmetry are attributed to packing requirements while all other dimensions in the molecule appear normal.

Introduction

In recent years, the search for efficient transition metal catalysts for the polymerization of unsaturated organic molecules has led to the production of a wide variety of novel organometallic complexes.[2,3] During studies on the triphenylstibine analog of the versatile hydrogenation catalyst chlorotris(triphenylphosphine)rhodium(I), the reaction with hexafluorobutyne-2 was attempted and a yellow crystalline compound of formula $RhCl(Sb(C_6H_5)_3)_2(C_8F_{12})$ was obtained.[4] Chemical and spectroscopic data for this complex were most consistent with the presence of a tetrakis(trifluoromethyl)rhodiacyclopentadiene moiety, but unequivocal proof was not possible. More recently, $IrCl(N_2)(P(C_6H_5)_3)_2$[5] and $Rh(C_6H_4)P(C_6H_5)_2(P(C_6H_5)_3)_2$[6] have been shown to undergo reactions with acetylenes containing electronegative substituents and similar metallocyclic products were postulated. In addition, $Rh(CO)Cl(P(C_6H_5)_3)_2$ was observed to trimerize these same acetylenes, but no intermediate metallocycles could be isolated.[7]

Although such metallocycles have been known for some time,[8] only a limited amount of structural data is

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(1) Supported by National Science Foundation Grant GP-8066.

(2) G. E. Coates, M. L. H. Green, and K. Wade, "Organometallic Compounds," Vol. 2, Methuen, London, 1968, Chapters 8 and 9.

(3) W. Hubel in "Organic Synthesis *via* Metal Carbonyls," Vol. 1, P. Pino and I. Wender, Ed., Wiley-Interscience, New York, N. Y., 1968, p 273.

(4) J. T. Mague and G. Wilkinson, *Inorg. Chem.*, **7**, 542 (1968).

(5) J. P. Collman, J. W. Kang, W. F. Little, and M. F. Sullivan, *ibid.*, **7**, 1298 (1968).

(6) W. Keim, *J. Organometal. Chem.*, **16**, 191 (1969).

(7) J. P. Collman and J. W. Kang, *J. Amer. Chem. Soc.*, **89**, 844 (1967).

(8) W. Hubel and E. H. Braye, *J. Inorg. Nucl. Chem.*, **10**, 250 (1959).

available[9-13] and that only on species containing at least two metal atoms. The structure of the present complex was thus of interest, not only to confirm the postulated structure but also to investigate the nature of the bonding in the heterocyclic ring in an instance where there would be no interaction of the ring with a second metal atom. A preliminary report of this structure determination has appeared.[14]

Unit Cell Data and Collection of Intensities

Yellow crystals of $RhCl(Sb(C_6H_5)_3)_2C_4(CF_3)_4 \cdot CH_2Cl_2$[4] were grown by the slow diffusion of petroleum ether (bp 30–60°) into a dichloromethane solution of the complex. Equiinclination Weissenberg photographs of the $0kl$–$2kl$ zones and precession photographs of the $h0l$ and $hk0$ zones (Ni-filtered Cu Kα radiation) established that the crystal belongs to the monoclinic system. The observed systematic absences ($h0l$ for $l = 2n + 1$ and $0k0$ for $k = 2n + 1$) uniquely determine the space group to be $P2_1/c$ ($C_{2h}{}^5$ no. 14[15]). The experimental density of 1.80 (2) g/cm^3 as measured by flotation in aqueous zinc iodide is in good agreement with the value of 1.805 g/cm^3 calculated on the basis of $Z = 4$. The molecules thus occupy general positions in this space group and no crystallographic symmetry need be imposed.

It was initially decided to employ copper radiation to collect the intensity data despite a large absorption coefficient ($\mu = 137.2$ cm^{-1}) in order to avoid overlap problems. However, the combined effects of crystal decomposition and an inadequate absorption correction for the rather large crystal ultimately prevented satisfactory refinement even though all the nonhydrogen atoms were easily located. The data were therefore recollected using molybdenum radiation and a smaller crystal. In the following discussion all references will be to operations performed using the latter data set with the exception of those involving the location of the nonhydrogen atoms which utilized the data set collected with copper radiation.

The final values for the cell dimensions were obtained by a least-squares analysis[16] of the setting angles of 39 high-order reflections which had been carefully centered on a Picker four-circle diffractometer using Zr-filtered, Mo Kα radiation (λ 0.7107 Å). These parameters are $a = 13.250$ (1), $b = 25.496$ (2), $c = 16.852$ (1) Å; $\beta = 125.4$ (2)°.

The crystal used for the collection of the final intensity data was a column of length 0.3 mm elongated along a. The ten prismatic faces of the crystal were identified by optical goniometry as all members of the forms $\{010\}$, $\{021\}$, and $\{011\}$. They ranged in width from 0.03 to 0.1 mm. The ends of the crystal were somewhat irregular and were assumed to be members of the form $\{100\}$ for the purposes of the absorption correction. The crystal was sealed in a nitrogen-filled, thin-walled glass capillary since preliminary observations showed a tendency toward decomposition presumably through loss of solvent of crystallization. It was mounted such that the crystallographic a axis was parallel to the ϕ axis of the goniostat. The data were collected at room temperature on a card-controlled, Picker four-circle diffractometer using Zr-filtered, Mo Kα radiation and a takeoff angle of 5.3°. The integrated intensities were measured with a scintillation counter employing a pulse-height analyzer set to admit about 90% of the Mo Kα pulse distribution. The moving-crystal, moving-counter scan technique[17] was employed with a 2θ scan of 2° centered about $2\theta_{\text{calcd}}$. The scan rate was 1°/min and background counts of 20 sec were made at each scan limit. Three standard reflections were monitored approximately every 4 hr throughout the course of the data collection and showed evidence for crystal decomposition. This decomposition did not result in more than a 10% diminution in the intensities of the standards and could be adequately treated by a linear correction based on the successive values found for the integrated intensities of the standards. Coincidence losses were found to affect only three reflections for which the counting rate exceeded 10,000 cps. These were remeasured at reduced power and scaled to the remaining data using the same standard reflections.

The unique quadrant having $h \geq 0$ and $(\sin \theta)/\lambda \leq 0.52$ was scanned to yield 4558 independent reflections. Inspection of the recorder trace showed that overlap affected the intensities of about 150 reflections, and since it did not prove feasible to eliminate this problem, these reflections were omitted. It was also apparent at this point that owing to the rather small size of the crystal there were relatively few useful data beyond $(\sin \theta)/\lambda = 0.45$. Therefore only the 3329 unique reflections within this smaller sphere were considered further. Raw intensities, I, were obtained from the diffractometer output with the formula $I = \text{CT} - 0.5(t_c/t_b)(B_1 + B_2)$ where CT is the total integrated count obtained in time t_c and B_1 and B_2 are the background counts, each obtained in time t_b. The standard deviation of the raw intensity, $\sigma(I)$, was estimated as $\sigma(I) = (\text{CT} + 0.25(t_c/t_b)^2(B_1 + B_2) + (pI)^2)^{1/2}$ with p[18] taken as 0.02. Using the criterion $I \leq 2\sigma(I)$, 934 reflections were rejected as statistically insignificant and were treated as unobserved. Each of these reflections was assigned a threshold value given by $I_{\text{th}} = 2\sigma(I)$. The reflections were then corrected for Lorentz,

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[9] A. A. Hock and O. S. Mills, *Acta Crystallogr.*, **14**, 139 (1961).

[10] R. P. Dodge, O. S. Mills, and V. Schomaker, *Proc. Chem. Soc.*, 380 (1963).

[11] R. P. Dodge and V. Schomaker, *J. Organometal. Chem.*, **3**, 274 (1963).

[12] P. B. Hitchcock and R. Mason, *Chem. Commun.*, 242 (1967).

[13] Y. Degreve, J. Meunier-Piret, M. van Meersche, and P. Piret, *Acta Crystallogr.*, **23**, 119 (1967).

[14] J. T. Mague, *J. Amer. Chem. Soc.*, **91**, 3983 (1969).

[15] "International Tables for X-Ray Crystallography," Vol. 1, Kynoch Press, Birmingham, England, 1962.

[16] All computations were performed on an IBM 7044 computer at the Tulane University Computer Laboratory. Local programs were used except for those noted here: GSET-4, C. T. Prewitt (diffractometer settings); CELL, B. L. Trus (refinement of unit cell dimensions); ORION (data reduction), BLSA-H (modification of the Gantzel–Sparks–Trueblood UCALS-1 (ACA Program No. 317) for block-diagonal least-squares refinement and distance–angle calculation), C. J. Fritchie Jr.; ORABS, W. R. Busing (absorption corrections); ORTEP, C. K. Johnson (graphical illustrations).

[17] T. C. Furnas, "Single Crystal Orienter Instruction Manual," General Electric Co., Milwaukee, Wis., 1957.

[18] W. R. Busing and H. A. Levy, *J. Chem. Phys.*, **26**, 563 (1967).

polarization, and absorption effects. The linear absorption coefficient for the compound is 17.8 cm^{-1} for Mo Kα radiation which results in transmission factors ranging from 0.767 to 0.781.[16]

Solution and Refinement of the Structure

The structure was refined by a block-diagonal, least-squares process. Scattering factors for neutral Rh, Sb, Cl, C, and F were those tabulated by Cromer and Waber[19] while the values given by Stewart, *et al.*,[20] were used for hydrogen. The scattering factors for Rh, Sb, and Cl included both the real and imaginary parts of the correction for the effects of anomalous dispersion.[21] The function minimized was $\Sigma w\Delta^2$ where $\Delta = K|F_o| - G|F_c|$, $|F_o|$ and $|F_c|$ are, respectively, the observed and calculated structure factors, K and G are scale factors ($G = 1.0$), and $w = 1/\sigma^2(|F_o|)$. The standard deviation in $|F_o|$, $\sigma(|F_o|)$, was taken as $\sigma(|F_o|^2)/2|F_o|$. Unobserved reflections were included in the refinement if $|F_c|$ exceeded $|F_{th}|$.[22] In the final stages of refinement, 142 reflections met this criterion and of these, and only five had $|F_c| > 1.5|F_{th}|$. The conventional and weighted residuals, R_1 and R_2, are defined as $R_1 = \Sigma|\Delta|/\Sigma|F_o|$ and $R_2 = (\Sigma w\Delta^2/\Sigma wF_o^2)^{1/2}$.

A three-dimensional, unsharpened Patterson function was calculated (Cu data) which provided coordinates for the rhodium and antimony atoms. The remainder of the nonhydrogen atoms were subsequently located by the successive application of least-squares refinement of the positional parameters followed by a difference Fourier synthesis. It soon became apparent that a molecule of dichloromethane of solvation was also present in the crystal. With all of the nonhydrogen atoms in the calculation, refinement of positional and isotropic thermal parameters led to $R_1 = 0.131$. A difference Fourier map calculated at this point revealed pronounced anisotropy in the motions of the heavy atoms, the trifluoromethyl groups, and the solvent molecule. Refinement was therefore continued with the introduction of anisotropic thermal parameters of the form $\exp[-(B_{11}h^2 + B_{22}k^2 + B_{33}l^2 + B_{12}hk + B_{13}hl + B_{23}kl)]$ for these atoms. Several more cycles of refinement led to convergence at $R_1 = 0.101$. In addition to high standard deviations in the calculated bond lengths, the thermal ellipsoids for some of the fluorine atoms had assumed quite unrealistic dimensions. Difference Fourier syntheses calculated in the planes of the fluorine atoms of each trifluoromethyl group using structure factors which did not contain contributions from these atoms clearly showed a considerable degree of anisotropic motion for all the fluorine atoms. However it did not prove possible to account adequately for this in terms of any feasible librational, hindered rotational, or oscillatory motion of the CF_3 group as a whole.

At this point it was evident that no further information could be gained from the copper data, and a new set of intensities was collected as mentioned above. Using the molybdenum data, refinement was continued with the unrealistic fluorine thermal parameters being manually reset to more reasonable values on the assumption that, despite the considerable motion apparent from the difference maps, the refined thermal parameters were probably in error at this point due to the poor quality of the original data. After 12 cycles, R_1 had dropped to 0.059. Difference Fourier syntheses computed in the planes of the phenyl groups revealed reasonable positions for most of the hydrogen atoms (peak heights 0.4–0.6 e$^-$/Å^3) as well as some equally large features attributable to uncorrected thermal motion. It was therefore decided to include the contributions of the hydrogen atoms to the scattering as fixed quantities using their calculated positions (C–H = 1.0 Å). Isotropic temperature factors equal to 1.1 times that of the attached carbon atom were used and the positional and thermal parameters were recalculated and manually reset after each cycle. Following this R_1 and R_2 were 0.055 and 0.056 and after 14 more cycles converged at the common value of 0.044.

The use of the new data set did not significantly improve the appearance of the thermal ellipsoids of the fluorine atoms and we can only conclude that the ellipsoidal model is not an adequate description of the actual motion of these atoms. Again, attempts at treating the thermal motion in terms of a movement of the CF_3 group as a whole were unsuccessful. However as can be seen from Figures 1 and 2, the *relative* sizes of the derived ellipsoids parallel the number of intramolecular F---F contacts even though the actual magnitudes have little physical significance. A final difference map showed no features other than those attributable to

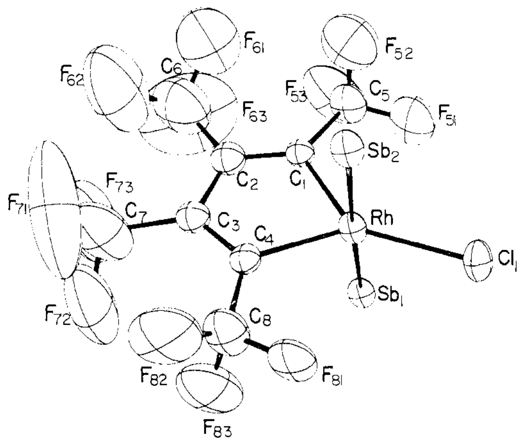

Figure 1.—A perspective view of the $RhCl(Sb(C_6H_5)_3)_2C_4(CF_3)_4$ molecule with the phenyl groups omitted for clarity.

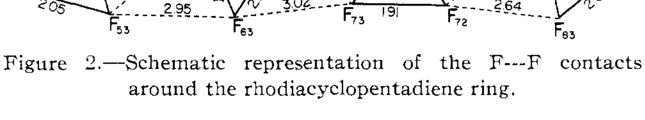

Figure 2.—Schematic representation of the F---F contacts around the rhodiacyclopentadiene ring.

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(19) D. T. Cromer and J. T. Waber, *Acta Crystallogr.*, **18**, 104 (1965).

(20) R. F. Stewart, E. R. Davidson, and W. T. Simpson, *J. Chem. Phys.*, **42**, 3175 (1965).

(21) D. T. Cromer, *Acta Crystallogr.*, **18**, 17 (1965).

(22) J. T. Mague, *Inorg. Chem.*, **8**, 1957 (1969).

uncorrected thermal motion in the vicinity of the phenyl and trifluoromethyl groups.

An analysis of the weights over ranges of $|F_o|$ and $(\sin \theta)/\lambda$ indicated that the absolute weights were too large, a fact also reflected in the final value of 1.71 obtained for the standard deviation of an observation of unit weight. In addition, the strong reflections were being somewhat downweighted but neither factor was judged to be serious enough to justify alteration of the weighting scheme. The final values of $|F_o|$ and F_c are presented in Table I. The positional and thermal parameters obtained from the last cycle of least-squares refinement are listed in Table II together with their associated standard deviations as estimated from the inverse least-squares matrix.

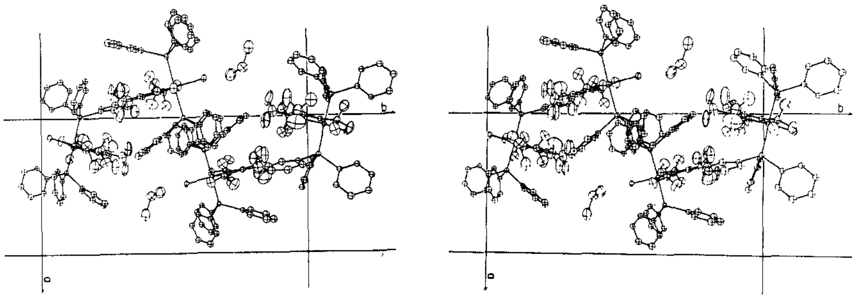

Figure 3.—Packing within the $RhCl(Sb(C_6H_5)_3)_2C_4(CF_3)_4 \cdot CH_2Cl_2$ crystal viewed perpendicular to [001].

Description of the Structure

The structure consists of discrete, monomeric molecules of the rhodiacyclopentadiene complex interspersed with molecules of dichloromethane. A perspective view of the molecule with the phenyl groups omitted for clarity is given in Figure 1 and the molecular packing in Figure 3. Tables III and IV list interatomic distances and interbond angles, respectively. From Figures 1 and 3, it is clear that the coordination about rhodium is that of a distorted trigonal bipyramid and that the solvent molecules do not interact significantly with the metal atoms as has been suggested for other five-coördinate rhodium complexes.[23] The closest approach of a solvent molecule to a metal atom, 5.86 Å, occurs between Cl_2 and the Rh at $(1 - x, {}^1/_2 - y, {}^1/_2 + z)$. No other intermolecular distances are tabulated as none is shorter than normal van der Waals contacts. The coordination about rhodium has very nearly C_{2v} symmetry with the antimony atoms of the two triphenylstibine groups occupying axial positions and a chlorine atom (Cl_1) and the 1 and 4 carbon atoms of the *cis*-1,2,3,4-tetratrifluoromethyl-1,3-butadienylene moiety occupying equatorial positions. The two independent Rh–Sb distances (Table III) are equal within experimental error. Although there appear to be no data available for a direct comparison, they agree well with the value of ~2.6 Å predicted from previously observed Rh–As distances[22] in similar compounds after correcting for the difference in covalent radius between the two elements (~0.2 Å[24]). Likewise, the Rh–Cl distance compares favorably with previously reported values.[22] The data in Table IV show that the Sb_1–Rh–Sb_2 unit is not quite linear. Although this deviation from linearity is significant, it appears to be simply the result of intramolecular packing. Both Sb_1 and Sb_2 are displaced toward Cl_1 and one phenyl group on each (rings 2 and 4) lies almost directly over the fluorocarbon portion. Moreover, for both of these phenyl groups, the line joining the two *ortho*-carbon atoms is roughly parallel to the mean plane of the rhodiacyclopentadiene ring. Whenever possible, the triphenyl pnictides seem to exhibit a staggered or "propeller-like" arrangement of the phenyl groups. Here, such a conformation would occasion either severe interaction between the phenyl and trifluoromethyl groups or a large bending of the Sb_1–Rh–Sb_2 unit. On the other hand, the rotation of one phenyl group so that the *ortho*-carbon line is exactly parallel to the plane of the ring would result in significant interaction between *ortho*-hydrogen atoms of this and the other two phenyl groups on the same antimony. Apparently, here, the conformation adopted is one which minimizes both types of contacts as well as the bending of the heavy-atom unit.

Although some preliminary studies have previously been made on triarylstibines and triarylstibine sulfides,[25] this appears to be the first instance of a complete structural determination involving one of these species. Unfortunately the constraints placed upon the orientations of the phenyl rings render inappropriate a detailed analysis of the geometry of the triphenylstibine molecule. Nevertheless, the angles about antimony (Table IV) are comparable to those found previously in $(C_6H_5)_3P$ (average value, 103.0 (2)°)[26] and $(p\text{-}CH_3C_6H_4)_3As$ (average value, 102 (2)°).[27] The six Sb–C distances are equal within experimental error, the average value being 2.113 (10) Å. This compares favorably with the value of 2.13 Å found for the Sn–C bond in $(p\text{-}CH_3C_6H_4)_4Sn$[28] and with the value of 1.96 (5) Å found for the As–C bond in $(p\text{-}CH_3C_6H_4)_3As$[27]

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[23] J. T. Mague and G. Wilkinson, *J. Chem. Soc., A*, 1736 (1966).

[24] L. Pauling, "The Nature of the Chemical Bond," 3rd ed, Cornell University Press, Ithaca, N. Y., 1960, Table 7-2.

[25] W. B. Pearson, Ed., "Structure Reports," N.V.A. Oosthoek's Vitgevers Mij Utrecht, 1953, Vol. 17, p 726.

[26] J. J. Daly, *J. Chem. Soc.*, 3799 (1964).

[27] J. Trotter, *Can. J. Chem.*, **41**, 14 (1963).

[28] I. G. Ismailzade and G. S. Zdanov, *Zh. Fiz. Khim.*, **27**, 550 (1953).

TABLE I

OBSERVED AND CALCULATED STRUCTURE AMPLITUDES (×10, IN ELECTRONS) FOR $RhCl(Sb(C_6H_5)_3)_2C_4(CF_3)_4 \cdot CH_2Cl_2$[a]

L	FO	FC
0 0		
2	4618	-4654
4	1425	1439
6	1393	1436
8	1354	-1398
10	2314	2259
12	749	-794
0 1		
10	496	623
9	841	-852
8	1541	-1495
7	1629	1614
6	2293	2288
5	3602	-3604
4	3366	-3305
3	1055	1055
2	1210	1176
1	1326	-1272
0 2		
1	5209	-5343
2	341	-359
3	2110	2124
4	1038	-1072
5	798	-817
6	345	394
7	2364	-2248
8	960	-941
9	1910	1906
10	450	396
11	1534	-1465
0 3		
12	295	297
10	528	481
9	1228	1203
8	1340	-1371
7	1400	-1376
6	2487	2533
5	1414	1384
4	3447	-3545
2	2420	2450
1	517	547
0 4		
0	3282	-3175
1	1840	-1831
2	2149	2097
3	1611	1609
4	2592	-2520
7	851	-903
8	770	729
9	1460	1430
10	1530	-1426
11	594	-640
12	746	779
0 5		
10	834	-888
9	1161	1174
8	1442	1501
7	3036	-2990
5	3222	3205
4	1017	-1043
3	2234	-2172
2	545	-515
1	556	584
0 6		
0	2761	-2714
1	2170	2170
2	760	724
3	1312	-1341
4	489	583
5	503	522
8	1442	1445
9	763	-805
10	1375	-1375
11	672	741
0 7		
11	971	1020
10	707	-731
9	742	-744
8	1734	1753
6	1787	-1713
5	1330	1304
4	2385	2343
3	239	314
2	1104	-1111
1	397	-252
0 8		
0	1921	1847
1	1386	1379
2	1224	-1251
3	1562	-1580
4	1066	1092
5	637	617
6	1150	-1136
7	1235	1242
9	1231	-1190
11	939	998
0 9		
11	285	212
10	556	475
9	1041	-1058
8	549	-536
7	806	789
5	1083	-1125
4	415	354
3	1126	1091
2	672	-703
1	-186	218
0 10		
0	2409	2339
2	1418	-1403
3	1034	1023
4	478	516
5	1090	-1050
6	257	235
7	707	676
8	545	-605
9	408	-466
10	630	674
0 11		
10	450	542
9	739	789
8	908	-923
7	-239	-169
6	267	334
5	471	-523
4	556	-516
3	1006	1044
0 12		
0	1221	-1193
1	584	-561
3	-215	282
4	1646	-1622
6	735	750
8	1013	-992
0 13		
10	345	-475
9	915	905
8	700	663
7	456	-532
6	-239	-290
4	584	-556
3	295	362
1	341	186
0 14		
0	1090	1093
2	749	-720
4	443	442
5	700	757
7	689	-686
0 15		
8	476	495
6	-239	-255
5	594	-693
4	299	-354
2	327	341
0 16		
0	394	490
2	306	-323
4	637	733
5	-250	278
6	443	-485
7	514	-498
0 17		
7	331	305
6	433	511
5	-250	-306
4	654	-672
2	457	381
0 18		
0	703	-783
2	433	514
3	317	361
4	422	-279
6	429	-432
0 19		
6	306	361
5	482	512
4	496	-534
3	718	-724
2	524	621
0 20		
0	957	-959
2	510	603
1 0		
-12	1474	1472
-10	2427	-2400
-8	2839	2764
-6	2810	-2734
-4	1245	1211
-2	2751	2424
0	4098	-4125
2	1073	1041
4	2547	-2438
6	918	-893
1 1		
10	336	-383
8	278	323
5	1266	1287
4	591	577
3	1006	-1016
2	1386	-1304
1	317	-310
0	1857	-1837
-1	2519	2165
-2	1973	1824
-3	2212	-2078
-4	2395	-2319
-5	2828	2759
-6	1801	1791
-7	1351	-1310
-9	-229	243
-10	440	-394
-11	1090	1060
-12	865	890
1 2		
-12	461	468
-11	1759	-1732
-10	559	-542
-9	2438	2403
-8	436	500
-7	3057	-2995
-6	306	-383
-4	654	-660
-3	397	-368
-2	1214	1244
-1	2621	-2394
0	-141	226
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-4	1273	1278
-3	936	950
1	369	-336
2	855	872
3	651	712
4	848	-816
3 19		
3	809	820
2	-264	-273
1	1590	-1562
0	714	770
-1	1847	1883
-2	-285	-403
-3	1319	-1315
-4	-271	323
-5	412	616
-7	626	688
-8	394	-342
3 20		
-7	598	-625
-6	1699	-1727
-5	732	728
-4	1382	1446
-3	359	-543
2	675	-841
	376	280
7	972	860
4 20		
-2	394	397
-3	464	396
-4	693	-641
-6	703	719
4 19		
-8	408	394
-7	552	-633
-1	285	-413
4 18		
0	580	655
-4	552	-564
-5	492	553
-6	429	527
-7	320	-327
-8	521	-521
4 17		
-10	1284	-1250
-8	1252	1276
-6	732	-654
-3	911	964
-1	651	-702
4 16		
4	366	291
3	324	-374
2	429	-544
1	609	764
0	827	811
-1	-253	-300
-3	454	-494
-5	1312	1293
-7	1393	-1435
-8	415	-472
-9	686	721
-10	447	485
4 15		
-11	915	900
-10	647	-778
-9	1041	-1079
-7	992	1078
-6	777	666
-5	1010	-1077
-4	1667	-1660
-2	1449	1453
-1	373	355
0	795	-776
4	422	446
4 14		
5	-267	297
4	366	-385
3	651	-685
2	457	499
1	1572	1494
0	886	-848
-1	1326	-1287
-4	841	808
-6	1460	-1459
-7	1031	-1001
-8	957	933
-9	1259	1289
-11	594	-631
4 13		
-11	1206	1180
-10	580	707
-9	626	-619
-8	834	-807
-7	-250	-375
-6	1534	1601
-5	925	945
-4	1491	-1510
-3	1520	-1559
-1	1801	1833
1	352	-385
2	672	-683
5	777	760
6	267	-220
4 12		
6	387	413
5	352	-389
4	918	-944
2	1622	1606
0	1678	-1573
-1	960	-977
-2	732	737
-3	865	948
-5	1344	-1349
-6	820	-832
-7	306	360
-8	1801	1856
-9	303	-345
-10	1692	-1631
-12	760	836
4 11		
-12	1020	-999
-10	507	496
-9	489	484
-7	1389	-1345
-6	911	-942
-5	2121	2118
-4	1333	1368
-3	2153	-2124
-2	2087	-2040
-1	872	837
0	1294	1282
1	281	401
2	521	-655
3	834	-836
5	496	580
6	774	765
4 10		
7	-271	335
6	542	-528
5	1066	-1044
3	2395	2379
1	1790	-1859
0	999	-1010
-1	563	549
-2	988	962
-4	1031	-1004
-5	605	-586
-6	686	687
-7	1674	1677
-8	454	416
-9	1491	-1479
-11	1551	1611
-12	665	674
-13	528	-516
4 9		
-13	605	621
-12	897	-927
-11	682	-716
-10	352	316
-9	601	673
-8	837	877
-7	281	240
-6	1745	-1710
-5	457	-408
-4	2427	2374
-3	964	951
-2	1555	-1554
-1	2065	-2035
0	992	966
1	1804	1768
2	-237	273
3	549	-644
4	1379	-1277
6	1112	1136
7	478	523
4 8		
7	689	-710
6	1122	-1169
5	338	416
4	1576	1567
3	461	492
2	1963	-2029
1	1043	-997
0	1115	1203
-1	1365	1386
-2	528	-487
-3	1080	-1119
-4	732	-753
-5	524	549
-6	1607	1546
-8	1899	-1917
-10	1284	1262
-11	725	703
-12	957	-945
-13	714	-779
4 7		
-13	606	830
-12	436	470
-11	762	-775
-10	246	-333
-8	1010	931
-7	1541	1508
-5	2160	-2114
-4	313	-297
-3	2719	2700
-2	1287	1300
-1	1766	-1761
0	1291	-1298
1	798	815
2	1738	1788
3	981	1021
4	872	-855
5	1178	-1148
6	-257	290
7	893	881
4 6		
8	992	-928
7	1214	-1181
6	855	941
5	1034	1028
4	317	335
3	1354	-1327
2	693	-744
1	1182	1131
0	1168	1172
-1	1224	-1200
-2	1358	-1345
-3	668	-678
-4	609	618
-5	798	765
-6	345	271
-7	2392	-2381
-8	665	-697
-9	1235	1271
-10	1090	1079
-11	725	-717
-12	623	-694
4 5		
-14	376	-392
-13	292	-233
-12	791	801
-10	436	-460
-9	376	-388
-8	-211	251
-6	1534	1464
-5	862	-843
-4	2395	-2332
-3	359	-342
-2	2663	2627
-1	485	501
0	1600	-1549
1	1840	-1908
3	1516	1471
4	774	771
5	1277	-1282
6	711	-715
8	288	446
4 4		
8	1361	-1331
7	862	838
6	1766	1722
5	433	-439
4	869	-872
3	334	-421
2	915	981
1	1379	1406
0	253	320
-1	2765	-2742
-3	2016	2043
-4	1530	1502
-6	1217	-1208
-7	1351	-1276
-8	1372	1373
-9	946	965
-11	397	-479
-12	-257	287
4 3		
-13	503	-494
-12	559	553
-11	397	341
-10	331	-275
-9	369	-310
-8	791	-838
-7	535	-511
-6	492	1006
-5	1674	1661
-4	426	-406
-3	2765	-2722
-2	686	-658
-1	1762	1836
0	805	838
1	626	-622
2	2016	-2032
3	338	-326
4	1569	1554
6	711	-734
7	352	-272
4 2		
8	-271	330
7	2040	1994
6	503	-469
5	1495	-1526
3	1242	1250
2	478	484
1	303	296
0	1101	-1125
-1	1393	-1350
-2	1002	923
-3	1351	1265
-4	419	459
-6	1185	-1199
-8	858	904
-9	419	-425
-10	517	-514
-11	429	449
-13	-264	-382
4 1		
-13	-250	-290
-11	739	723
-9	236	239
-7	387	394
-6	267	-265
-5	1002	989
-4	1266	1237
-3	267	-328
-2	1418	-1428
-1	1199	-1209
0	1182	1174
1	2075	2121
2	1284	-1300
3	1878	-1849
4	1031	1066
5	932	898
4 0		
8	1836	1773
6	2012	-2049
4	932	897
0	1178	-1182
-2	2160	2211
-4	2589	-2666
-8	746	-735
-12	594	-546
-14	570	595
5 0		
-14	763	801
-12	1755	-1762
-10	1432	1398
-8	981	-969
-6	1949	-1865
-4	2592	2530
-2	3535	-3642
0	3704	3774
2	1660	-1741
4	559	-544
6	1446	1509
5 1		
7	703	719
6	964	954
5	1607	-1630
4	1301	-1240
3	2817	2802
2	1906	1930
1	2406	-2443
0	1066	-1076
-1	728	734
-2	253	285
-3	2128	2120
-4	2051	1983
-5	3187	-3186
-6	1361	-1343
-7	2522	2488
-8	1583	1535
-9	1280	-1276
-10	992	-964
-11	552	594
-12	507	509
5 2		
-14	306	295
-13	1396	-1392
-12	271	-252
-11	1245	1274
-10	507	521
-9	1583	-1528
-8	556	-562
-5	2730	2805
-3	4376	-4435
-2	753	-769
-1	3229	3298
0	1143	1112
1	2462	-2502
2	355	-289
3	447	437
4	-246	-262
5	915	901
6	443	418
7	1505	-1501
5 3		
7	820	-817
6	1509	1512
5	573	598
4	2019	-1961
3	703	-690
2	3057	3084
0	1284	-1313
-1	732	-763
-2	-200	-205
-3	707	661
-4	2311	2215
-5	387	430
-6	1980	-1898
-7	1178	-1182
-8	2628	2621
-9	1699	1723
-10	1734	-1734
-11	1221	-1150
-13	577	551
-14	753	762
5 4		
-14	1161	-1102
-13	591	-590
-12	1178	1155
-11	703	705
-10	1122	-1152
-9	1337	-1342
-7	976	948
-6	1175	1163
-4	2420	-2389
-3	1674	-1618
-2	2367	2342
-1	1266	1316
0	1966	-1977
1	1439	-1481
2	587	608
5	682	680
6	960	-959
7	749	-723
5 5		
7	971	-924
6	390	-442
5	1882	1866
3	1671	-1659
2	341	-398
1	2005	2014
-1	849	-853
-2	654	-633
-4	1189	1185
-5	2895	2842
-6	514	-565
-7	2603	-2521
-8	725	-666
-9	2276	2268
-10	700	673
-11	654	-625
-12	781	-837
-13	317	-370
-14	802	805
5 6		
-14	475	-440
-13	841	827
-12	1175	1144
-11	-246	-275
-10	1843	-1810
-8	1010	1044
-7	1291	1279
-6	447	490
-5	1379	-1359
-4	1083	-1064
-3	2069	2025
-2	1959	1977
-1	1396	-1364
0	1576	-1638
2	777	786
3	334	-401
4	-250	345
6	813	-785
5 7		
6	1073	-1141
4	1435	1422
2	1797	-1762
0	971	1008
-1	580	-568
-2	232	228
-3	798	-850
-4	1224	-1178
-5	426	437
-6	1632	1663
-7	401	-424
-8	2212	-2215
-10	1231	1182
-11	848	748
-12	327	-450
-13	1407	-1456
5 8		
-14	823	815
-13	672	711
-11	683	-692
-10	324	-316
-9	1555	1529
-8	1541	1530
-6	489	-479
-5	1136	-1117
-4	848	856
-3	1316	1357
-2	760	-740
-1	1646	-1628
0	988	1015
1	267	363
4	288	259
5	450	-549
6	303	213
5 9		
5	988	-936
4	285	336
3	1104	1063
2	331	-383
1	700	-756
-1	464	514
-2	556	606
-3	313	-321
-4	1020	-1026
-5	742	-717
-6	1449	1398
-7	1133	1136
-8	573	-561
-9	1161	-1186
-11	528	594
-12	1157	1155
-13	696	-728
5 10		
-13	373	-427
-12	805	-621
-11	559	-548
-10	331	387
-9	1389	1420
-8	355	-303
-7	1513	-1453
-6	324	-362
-5	355	457
-4	271	197
-3	830	-848
-2	1706	-1732
-1	672	638
0	1052	1020
1	352	-346
2	419	-445
3	292	210
6	443	417
5 11		
3	295	318
2	380	463
-3	489	485
-5	886	-938
-7	292	248
-8	791	813
-10	848	-870
-11	552	-528
-12	953	965
-13	739	776
5 12		
-10	939	982
-9	253	-104
-8	995	-967
-6	1301	1345
-4	707	-663
-3	510	-474
-1	461	525
4	352	-371
5 13		
-2	767	-778
-3	383	388
-4	236	226
-5	587	-528
-8	-243	-289
-9	436	365
-11	851	-857
-12	274	-298
5 14		
-9	714	-683
-8	292	-342
-7	1038	1005
-6	524	477
-5	623	-787
0	-253	354
5 15		
3	260	329
1	313	-268
-1	257	287
-2	267	-227
-4	383	506
-5	-243	-262
-8	591	-582
-9	408	-348
-10	809	833
-11	376	320
5 16		
-9	376	-354
-7	317	385
-6	295	-341
-4	538	572
-2	-253	-352
-1	644	655
0	303	297
1	412	-544
2	-264	-328
5 17		
-9	285	-385
-6	288	-364
-4	492	522
0	482	-551
2	549	586
5 18		
1	594	-625
0	422	-414
-1	739	770
5 19		
-7	303	-364
-5	640	561
-4	295	337
-2	-271	-318

after correction for the difference in covalent radius between arsenic and antimony. All six phenyl rings are planar within experimental error. The C–C distances within the rings range from 1.443 (21) (C_{24}–C_{25}) to 1.340 (19) Å (C_{23}–C_{24}) with the average value being 1.383 (16) Å. The root-mean-square deviation from the average is 0.023 Å indicating that the estimated standard deviations are reasonably accurate. The average C–C–C angle for the rings is 120.0 (11)° with the range being from 122.4 (12) (C_{41}–C_{42}–C_{43}) to

TABLE I (*Continued*)

L	FO	FC
0	306	-389
	5	20
-2	570	511
-4	560	-485
	6	0
-14	1618	-1590
-12	2621	2498
-10	2564	-2455
-8	2543	2513
-6	559	-563
-4	1235	-1253
-2	2603	2717
2	1143	1137
	6	1
6	292	-425
5	763	702
3	827	-814
2	303	-351
1	598	622
0	415	-449
-1	883	873
-2	753	772
-3	1224	-1285
-4	1812	-1693
-5	2118	2117
-6	1607	1585
-7	1808	-1782
-8	1231	-1192
-9	1052	1065
-10	429	405
-12	359	387
-13	1175	-1164
-14	774	-792
-15	1323	1330
	6	2
-15	707	-741
-14	619	-605
-12	383	451
-11	2511	-2454
-10	630	-650
-9	2969	2983
-8	405	-417
-7	1280	-1241
-4	756	-739
-3	1259	1254
-2	545	494
-1	1815	-1909
0	1024	-1020
1	1467	1455
2	345	303
3	566	-595
6	630	-648
	6	3
6	503	-473
4	556	515
3	271	-236
2	932	-917
1	573	576
0	306	330
-1	538	-596
-2	1463	1421
-3	672	670
-4	2343	-2490
-5	749	-811
-6	3415	3453
-7	1017	1019
-8	2008	-1993
-9	383	-372
-10	746	767
-11	601	-586
-12	616	599
-13	711	672
-14	1572	-1553
	6	4
-14	974	960
-13	830	822
-12	1727	-1726
-11	953	-923
-10	2817	2852
-9	922	920
-8	2093	-2066
-7	243	271
-6	865	828
-5	939	-967
-3	1465	1446
-2	1491	-1513
-1	1607	-1516
0	1449	1453
1	1002	1009
2	1048	-1047
3	327	253
	6	5
5	516	-525
4	-257	324
3	422	417
2	1136	-1142
1	661	-629
0	1537	1497
-1	436	-452
-2	1717	-1730
-3	1386	1411
-4	1404	1482
-5	2723	-2670
-6	700	-726
-7	2624	2534
-8	475	-480
-9	1284	-1301
-11	324	-281
-12	619	-617
-13	1020	1054
	6	6
-14	675	654
-13	510	-573
-12	1097	-1115
-11	1562	1580
-10	1301	1273
-9	1421	-1403
-8	412	-488
-7	1147	1191
-4	1365	1310
-2	1667	-1669
0	1024	1102
1	869	-873
3	1442	1397
4	485	-537
5	848	-868
	6	7
4	524	-537
3	1119	1097
1	1305	-1287
-1	2033	2073
-2	1161	-1167
-3	1449	-1466
-4	2075	2069
-6	2177	-2160
-7	1217	1267
-8	925	942
-9	1161	-1136
-10	408	-411
-11	897	957
-12	552	-582
-14	823	857
	6	8
-14	644	-610
-13	1017	-1041
-11	1214	1187
-10	524	-502
-9	1273	-1228
-8	661	720
-6	1555	-1593
-5	967	968
-4	1604	1575
-3	1101	-1134
-2	869	-916
-1	897	856
0	507	-525
1	577	-479
2	1467	1503
4	1365	-1375
5	471	425
	6	9
4	633	-688
2	1509	1464
1	703	-649
0	1657	-1617
-1	1052	1077
-2	1882	1834
-3	1305	-1288
-4	637	-665
-5	510	536
-6	784	-750
-7	844	-844
-8	960	972
-9	-263	287
-10	637	-673
-11	327	322
-13	503	-516
	6	10
-12	376	420
-9	348	367
-8	591	510
-7	1031	-988
-6	348	314
-5	2139	2153
-3	1801	-1751
-2	1476	1505
0	753	-767
1	890	831
2	707	760
3	1340	-1385
	6	11
3	967	-921
2	433	483
1	1045	1027
0	1010	-995
-1	1133	-1159
-2	1157	1182
-4	619	-642
-5	295	270
-7	1010	-1013
-9	517	443
-11	559	-508
	6	12
-9	397	420
-8	865	-814
-7	401	-417
-6	1041	1053
-5	471	512
-4	1231	-1273
-3	253	206
-2	788	786
-1	348	-401
1	950	1008
2	742	-766
3	619	-579
	6	13
3	478	-454
2	820	-835
1	925	909
0	563	575
-1	1312	-1318
-3	851	764
-9	331	388
-12	-264	-265
	6	14
-10	303	-259
-9	503	-618
-7	781	793
-6	499	470
-5	619	-701
-4	257	-250
-3	908	894
-1	703	-678
0	380	364
2	552	-553
3	359	471
	6	15
2	809	-842
0	735	738
-2	707	-721
-3	373	-321
-4	295	357
-10	-267	287
	6	16
-10	507	-499
-9	260	44
-8	637	654
-6	313	-274
-5	303	-170
-3	-264	268
-2	612	-516
1	-264	-340
	6	17
1	331	-304
0	306	-261
-1	711	706
-2	433	413
-3	503	-521
-5	373	284
	6	18
-7	549	-568
-2	-264	-292
	6	19
-5	468	-442
	7	18
-3	324	384
-4	-292	-308
-6	651	637
-7	-271	-328
	7	17
-9	313	352
-6	1024	1027
-4	1358	-1373
-2	978	1023
	7	16
-1	591	-556
-2	703	713
-4	422	-461
-5	528	527
-7	1112	-1129
-8	338	343
-9	545	564
-10	380	-431
	7	15
-10	950	-949
-9	447	552
-8	869	850
-7	760	-695
-5	946	1024
-4	355	-294
-3	1168	-1270
-2	373	363
-1	725	671
0	538	-515
	7	14
1	524	-585
0	827	-854
-1	679	749
-3	883	-832
-4	348	446
-5	661	733
-6	1147	-1129
-7	345	-382
-8	1235	1224
-10	612	-631
-11	591	605
	7	13
-11	872	907
-10	714	-745
-9	1108	-1115
-8	739	857
-7	953	957
-6	840	-796
-4	556	570
-3	865	-856
-2	496	-464
-1	862	896
0	-281	334
1	830	-820
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1	1294	-1310
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-4	1271	1228
-5	306	-451
-6	1083	-1067
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-8	471	478
-9	665	-711
-10	461	370
-11	376	395
-12	1277	-1304
	7	11
-12	560	-591
-11	939	917
-10	1080	1110
-9	872	-875
-8	1330	-1351
-7	1333	1337
-6	499	565
-5	855	-844
-4	703	652
-2	1171	-1148
-1	394	400
0	1273	1230
1	288	-266
2	348	-450
	7	10
3	654	607
2	967	-940
1	266	-297
0	654	624
-1	1006	-1040
-2	770	-757
-3	1938	1868
-5	1748	-1733
-6	369	408
-7	1127	1125
-8	718	-673
-9	313	-228
-10	412	464
-11	763	-784
-12	644	-661
-13	911	887
	7	9
-13	841	844
-12	313	-368
-11	601	-644
-10	1083	1098
-9	918	882
-8	2216	-2208
-7	915	-873
-6	1692	1603
-4	612	-562
-3	1013	1067
-2	475	-446
-1	1076	-1011
0	830	808
1	549	571
2	823	-838
	7	8
0	257	-139
-1	613	-724
-2	1495	1422
-3	1214	1218
-4	1449	-1446
-6	1636	1585
-8	485	-515
-9	503	498
-11	626	-606
-12	879	870
-13	471	574
-14	679	-662
	7	7
-14	703	-686
-13	-281	319
-12	563	672
-11	1372	-1407
-10	630	-695
-9	1572	1587
-7	1671	-1652
-6	507	584
-5	-232	246
-4	380	-405
-3	264	269
-2	405	376
-1	1256	-1272
1	960	967
3	658	-678
	7	6
4	320	377
-1	665	656
-2	886	882
-3	974	-981
-4	943	-950
-5	932	987
-6	766	777
-7	1463	-1452
-8	239	288
-9	542	607
-10	1027	-1061
-12	978	955
-13	594	-648
-14	485	-501
	7	5
-14	285	-307
-13	616	-617
-12	1112	1088
-10	1513	-1482
-9	-232	242
-8	1333	1372
-7	918	-890
-6	700	-698
-5	960	993
-3	443	-452
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-1	-257	-341
0	665	-718
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4	521	-502
	7	4
2	440	-462
0	549	557
-1	749	766
-2	802	-761
-3	412	-372
-4	264	-212
-5	674	830
-6	798	-839
-7	544	-568
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-9	357	334
-10	756	-742
-11	1129	1073
-13	862	-879
-14	352	436
	7	3
-14	777	804
-13	841	-707
-12	390	-438
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-9	753	-797
-8	457	454
-7	327	336
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0	380	-409
1	341	-370
3	557	602
	7	2
3	637	-623
2	-250	271
1	749	794
-1	557	-564
-2	236	-309
-8	412	366
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-10	594	576
-11	900	914
-12	665	-687
-13	651	-618
-14	587	587
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-12	341	-404
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	8	0
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-2	545	-579
-3	946	944
-4	507	672
-5	802	-762
-8	310	-475
-9	753	715
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-11	1161	-1131
-12	422	-371
-13	619	624
	8	2
-15	303	-373
-14	-271	-374
-12	369	433
-11	454	429
-9	1368	-1433
-8	415	-420
-7	1217	1227
-6	-232	287
-5	995	-972
-4	-236	254
-3	711	737
-2	654	-662
0	412	376
1	355	-298
2	334	284
3	271	238
	8	3
3	-274	325
1	573	-549
0	466	537
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-2	739	-750
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-5	679	-727
-6	823	-826
-7	-239	425
-8	507	-437
-9	348	308
-12	456	-478
-14	383	443
	8	4
-14	457	-503
-13	285	267
-11	-253	-300
-10	-246	-347
-9	-239	-271
-6	243	-272
-4	556	570
-2	-250	-345
-1	327	349
1	457	-431
2	-267	335
	8	5
2	563	587
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-3	408	-391
-4	281	314
-6	285	-263
-8	489	-564
-10	317	405
-11	538	536
-13	559	-550
	8	6
-13	753	751
-10	-250	258
-9	334	372
-5	552	581
-3	443	-473
-1	612	631
0	440	-469
	8	7
1	394	414
	8	8
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-9	514	-473
-7	-246	292
-5	405	-401
-2	352	354
0	707	-691
2	436	505
	8	9
0	496	502
-1	433	337
-3	580	-627
-5	380	358
-8	524	492
-9	-257	-336
-13	310	-331
	8	10
-13	580	-576
-12	369	383
-11	380	322
-10	570	-592
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-6	471	-552
-4	419	483
-3	362	459
-1	791	-735
0	359	-429
1	700	718
	8	11
0	285	-354
-1	700	695
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-4	703	-736
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-9	303	-341
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-11	-271	357
-12	426	428
	8	12
-12	510	541
-11	584	-612
-10	369	-409
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-4	303	323
-3	682	-711
-2	872	-845
-1	-274	-377
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-1	566	-516
-2	1231	1215
-3	644	691
-4	-281	-107
-5	786	-798
-6	-267	275
-7	-267	309
-9	496	548
-10	313	-353
-11	489	-788
	8	14
-11	503	-522
-10	806	797
-9	521	507
-8	1189	-1232
-7	848	-820
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-5	630	621
-4	964	-978
-3	665	-699
-1	-285	337
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-4	612	679
-5	953	-930
-6	464	-490
-7	345	370
-9	623	658
-10	552	596
	8	16
-9	1252	-1243
-8	739	-833
-7	1477	1450
-6	932	933
-5	1059	-1022
-4	707	-768
-3	367	313
	8	17
-6	879	-883
	9	15
-7	-281	353
	9	14
-4	475	482
-5	299	-393
-6	925	-885
-7	-285	-356
-8	1038	1055
-9	436	503
	9	13
-10	890	876
-8	795	-845
-7	886	-880
-6	774	753
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-4	299	-288
-3	1034	-1010
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-4	584	-626
-5	668	-641
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-9	791	-811
-10	992	-1000
-11	461	534
	9	11
-12	514	-513
-11	310	-484
-9	1048	1021
-8	285	449
-7	953	-887
-6	598	-606
-5	811	804
-4	911	914
-3	538	-581
-2	960	-911
	9	10
-1	1182	1193
-2	623	634
-3	858	-800
-4	711	-590
-6	647	592
-7	612	564
-9	1411	-1408
-11	1351	1380
	9	9
-13	271	331
-11	570	-512
-10	1002	-966
-8	1326	1362
-7	496	457
-6	1238	-1235
-5	552	-603
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-2	422	-404
-1	689	-716
	9	8
0	995	1000
-1	640	636
-2	1122	-1071
-3	679	-681
-5	612	613
-6	647	662
-8	693	-941
-10	1245	1278
-11	524	540
-12	876	-941
-13	-278	-325
	9	7
-13	359	468
-12	-271	323
-10	397	-411
-9	753	-768
-7	1555	1598
-5	1291	-1247
-4	777	-858
-3	714	737
-2	967	923
0	415	-514
	9	6
0	415	517
-1	1010	-1065
-2	1168	-1164
-4	587	534
-5	317	323
-6	348	-275
-7	788	-693
-8	408	-419
-9	791	779
-10	739	779
-11	598	-667
-12	703	-714
	9	5
-14	-278	-313
-12	295	449
-11	457	468
-9	267	-175
-8	957	-943
-6	1558	1571
-4	1477	-1461
-3	616	-542
-2	770	807
-1	834	860
0	-274	302
	9	4
1	419	441
0	725	-732
-1	1108	-1035
-2	369	349
-3	630	741
-4	292	215
-5	303	-319
-6	408	-534
-7	355	-355
-8	542	592
-9	900	931
-11	408	-391
-12	-267	296
-14	-267	-274
	9	3
-14	334	-424
-13	267	-230
-11	-267	328
-10	362	-501
-7	419	-417
-6	429	382
-5	686	754
-3	879	-934
-2	711	-729
-1	359	340
0	851	913
	9	2
0	714	-667
-2	686	693
-3	538	479
-6	394	-394
-7	-246	283
-8	327	298
-10	426	-456
-11	524	451
	9	1
-14	274	-222
-13	390	-374
-10	299	-264
-6	264	-152
-5	-250	343
-4	-246	294
0	-267	308
1	531	557
	9	0
0	327	-315
-2	672	779
-4	253	-277
-8	338	351
-12	419	-418
-14	521	489
	10	0
-14	1133	1099
-12	1206	-1194
-10	626	604
-8	883	876
-6	1147	-1180
-4	1998	2085
-2	2100	-2144
	10	1
-1	641	900
-2	394	425
-4	281	254
-5	658	-732
-6	499	-547
-7	1112	1162
-8	929	867
-9	1147	-1046
-10	1133	-1100
-11	806	804
-12	542	585
-13	401	-481
-14	303	-281
	10	2
-14	341	250
-13	798	-855
-12	288	-350
-11	763	736
-10	352	334
-9	267	306
-7	1347	-1323
-5	1861	1895
-4	658	645
-3	2005	-2022
-2	858	-845
-1	1073	1124
	10	3
-1	612	-580
-2	324	464
-3	387	351
-4	507	454
-6	1266	-1157
-8	1505	1571
-9	834	839
-10	1516	-1474
-11	964	-982
-12	644	627
-13	426	508
	10	4
-13	577	-562
-12	929	933
-11	866	907
-9	433	-522
-8	1317	-1298
-6	1748	1679
-5	936	910
-4	1509	-1453
-3	1066	-1007
-2	1027	1034
	10	5
-2	468	-512
-4	464	480
-5	978	991
-6	464	-479
-7	1541	-1537
-9	1358	1379
-10	461	432
-11	1119	-1102
-12	890	-907
	10	6
-12	1094	1129
-11	-271	308
-10	967	-968
-9	732	-674
-7	1470	1414
-6	686	663
-5	1245	-1247
-4	971	-1009
-3	683	840
-2	648	695
	10	7
-2	630	-621
-3	390	-261
-5	813	684
-6	672	839
-7	837	-440
-8	1291	-1293
-9	665	609
-10	1034	1039
-11	478	451
-12	721	-725
	10	8
-12	-281	313
-11	739	-806
-10	918	-885
-9	869	863
-8	978	963
-6	922	-893
-5	697	-939
-4	387	464
-3	883	863
	10	9
-4	-274	-319
-6	654	681
-7	394	420
-8	728	-766
-11	946	903
-12	703	662
	10	10
-11	844	-849
-9	1273	1261
-7	886	-800
-6	303	-337
-5	-271	286
-4	429	468
	10	11
-5	577	-635
-6	362	293
-7	563	524
-10	-310	-448
	10	12
-8	598	-661
-6	415	494

[a] The data are separated into groups having the common values of *h* and *k* listed above each one. Unobserved reflections for which $|F_c| > |F_{th}|$ (see text) are indicated by a negative F_o.

116.8 (13)° (C_{42}–C_{43}–C_{44}). The root-mean-square deviation from the average is 1.0°.

The C–Cl distances in the dichloromethane molecule are equal and compare well with tabulated values.[29] The Cl_2–C_9–Cl_3 angle here is slightly smaller than has been reported previously[29] but the difference is probably not significant. The hydrogen atoms could not be unequivocally located, presumably as a result of large amplitudes of vibration.

The most interesting feature of the present molecule is the rhodiacyclopentadiene ring. As mentioned earlier, this is the first structural characterization of this type of compound which does not have a second metal atom associated with the ring. In the previous determinations, the M–C bonds in the MC_4 moiety were found to be equal as were the C–C bonds but it was not possible to determine whether this was an inherent feature of this moiety or was due to the postulated π bonding between the other metal atom(s) present and the ring.

The ring consisting of Rh, C_1, C_2, C_3, and C_4 is essentially planar with the distances (Å) of the various atoms from the weighted least-squares plane[30] (σ_{plane} = 0.02 Å) being Rh, 0.001 (1); C_1, 0.003 (11); C_2, −0.013 (12); C_3, 0.020 (12); C_4, −0.016 (11). Although the deviations from planarity appear statistically significant,[31] they are unlikely to be significant chemically and can be accounted for by packing considerations (*vide infra*). The distances (Å) of other atoms of interest from the mean plane of the ring are: Cl_1, 0.022 (3); C_5, 0.011 (13); C_6, 0.007 (18); C_7, −0.016 (17); C_8, −0.078 (14). It is thus apparent that this whole portion of the molecule is very nearly planar as would be expected.

The Rh–C_1 (2.000 (10) Å) and Rh–C_4 (1.964 (11) Å) distances are different to an extent which is only marginally significant (Δ/σ = 3.3) and there does not appear to be any compelling reason to consider it to be real. The average value of 1.98 (1) Å is essentially the same as that found for the Rh–C(carbonyl) distance in π-$C_5H_5Rh(CO)(C_2F_5)I$ (1.97 (3) Å)[32] even before correction for the difference in radius between sp² and sp carbon atoms (∼0.03 Å). It is also possibly shorter than the Rh–C(perfluoroethyl) distance in π-$C_5H_5Rh(CO)(C_2F_5)I$ (2.08 (3) Å) after correction for the difference in radius between sp² and sp³ carbon atoms (∼0.03 Å). Although this latter Rh–C bond is

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[29] "Tables of Interatomic Distances and Configuration in Molecules and Ions," Special Publication No. 11, The Chemical Society, London, 1958, p M108.

[30] The equation of the plane is: $-0.964X + 0.255Y - 0.082Z + 2.171 = 0$, where X, Y, and Z are orthogonal coordinates (in Å). The transformations from fractional monoclinic coordinates (x, y, z) are: $X = ax + cz\cos\beta$; $Y = by$; $Z = cz\sin\beta$.

[31] The results of a χ^2 test show that rigorous planarity is only significant at an 0.02 confidence level: G. H. Stout and L. H. Jensen, "X-Ray Structure Determination: A Practical Guide," Macmillan, New York, N. Y., 1968, Section 18-7.

[32] M. R. Churchill, *Inorg. Chem.*, **4**, 1734 (1965).

TABLE II

FINAL POSITIONAL AND THERMAL PARAMETERS[a] FOR $RhCl(Sb(C_6H_5)_3)_2C_4(CF_3)_4 \cdot CH_2Cl_2$

Atom[b]	x	y	z	B_{11}[c]	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Sb_1	0.21900 (6)	0.12769 (3)	0.28364 (5)	5.74 (6)	1.19 (2)	3.71 (4)	0.46 (5)	5.38 (6)	0.02 (4)
Rh	0.41720 (7)	0.10382 (4)	0.29561 (6)	5.64 (6)	1.04 (2)	3.53 (4)	0.20 (6)	5.14 (7)	−0.05 (4)
Sb_2	0.61453 (6)	0.07705 (3)	0.30867 (5)	5.82 (5)	1.17 (2)	3.90 (3)	0.18 (5)	5.86 (6)	0.11 (4)
Cl_1	0.4014 (2)	0.0174 (1)	0.3411 (2)	9.2 (2)	1.3 (1)	7.5 (2)	0.4 (2)	11.3 (2)	1.1 (2)
F_{51}	0.2486 (7)	0.0694 (3)	0.0960 (5)	25.6 (9)	3.6 (2)	6.6 (4)	−12.7 (6)	14.6 (8)	−5.3 (5)
F_{52}	0.3404 (7)	0.1011 (4)	0.0448 (5)	24.4 (7)	5.1 (2)	11.1 (4)	−9.5 (7)	26.5 (7)	−7.3 (5)
F_{53}	0.1820 (9)	0.1375 (4)	0.0160 (6)	22.4 (12)	5.3 (3)	7.4 (6)	12.0 (9)	−7.7 (14)	−4.1 (6)
F_{61}	0.3726 (8)	0.2052 (4)	0.0388 (5)	36.3 (11)	4.6 (3)	13.0 (4)	8.3 (9)	31.7 (9)	8.2 (6)
F_{62}	0.4542 (10)	0.2623 (4)	0.1266 (6)	69.0 (14)	3.0 (2)	23.7 (5)	0.1 (10)	67.1 (10)	4.1 (6)
F_{63}	0.2542 (9)	0.2491 (5)	0.0549 (7)	44.9 (10)	13.5 (4)	29.8 (7)	42.2 (10)	59.6 (12)	34.1 (8)
F_{71}	0.6094 (8)	0.2793 (4)	0.3307 (10)	37.9 (9)	2.8 (2)	66.3 (13)	−13.8 (8)	84.3 (14)	−16.3 (9)
F_{72}	0.5112 (11)	0.2834 (4)	0.3891 (7)	46.3 (16)	2.2 (2)	19.1 (8)	−9.2 (10)	32.7 (16)	−5.5 (7)
F_{73}	0.4174 (10)	0.3009 (4)	0.2533 (8)	48.4 (15)	1.5 (2)	23.7 (8)	4.7 (9)	94.4 (14)	2.7 (6)
F_{81}	0.5833 (6)	0.1326 (3)	0.4990 (4)	19.2 (8)	2.4 (2)	3.2 (3)	−1.0 (6)	6.4 (7)	−0.4 (4)
F_{82}	0.6755 (7)	0.1983 (4)	0.4987 (5)	16.8 (8)	6.8 (3)	6.4 (4)	−13.0 (8)	7.0 (9)	−2.9 (6)
F_{83}	0.5080 (7)	0.2047 (4)	0.4893 (5)	23.7 (8)	4.4 (2)	7.3 (4)	6.9 (7)	16.1 (7)	−1.4 (5)
C_5	0.2824 (10)	0.1141 (5)	0.0839 (8)	9.8 (11)	2.2 (3)	5.0 (6)	−2.0 (10)	6.7 (11)	−1.2 (7)
C_6	0.3542 (13)	0.2343 (8)	0.0995 (9)	30.3 (15)	5.1 (5)	12.8 (7)	−8.0 (12)	33.2 (13)	−6.6 (10)
C_7	0.5153 (15)	0.2710 (7)	0.3134 (10)	29.4 (19)	3.2 (4)	9.4 (9)	6.8 (15)	18.9 (17)	0.0 (10)
C_8	0.5670 (10)	0.1779 (5)	0.4567 (9)	11.9 (11)	2.2 (3)	6.4 (6)	0.4 (10)	10.4 (11)	−0.6 (8)
C_9	0.7450 (14)	0.3790 (8)	0.2562 (11)	17.7 (16)	5.3 (6)	10.1 (10)	1.9 (17)	14.4 (17)	−1.9 (12)
Cl_2	0.7935 (4)	0.3973 (3)	0.3743 (4)	30.8 (5)	5.8 (2)	21.2 (3)	−5.2 (5)	39.9 (5)	−6.1 (4)
Cl_3	0.8786 (5)	0.3578 (2)	0.2660 (4)	36.2 (5)	4.4 (2)	21.1 (3)	−0.2 (5)	41.6 (5)	−2.1 (4)

Atom	x	y	z	B, Å²	Atom	x	y	z	B, Å²
C_1	0.3591 (8)	0.1454 (4)	0.1753 (6)	3.0 (3)	C_{62}	0.8864 (9)	0.0587 (5)	0.4814 (7)	4.1 (3)
C_2	0.3947 (9)	0.1953 (5)	0.1889 (7)	4.3 (3)	C_{63}	0.9831 (10)	0.0434 (5)	0.5750 (8)	4.9 (3)
C_3	0.4643 (9)	0.2131 (5)	0.2850 (7)	3.8 (3)	C_{64}	0.9589 (9)	0.0253 (5)	0.6401 (7)	4.5 (3)
C_4	0.4888 (8)	0.1734 (4)	0.3487 (6)	3.1 (3)	C_{65}	0.8388 (9)	0.0228 (5)	0.6110 (7)	4.4 (3)
C_{11}	0.2379 (8)	0.1207 (5)	0.4171 (7)	3.3 (3)	C_{66}	0.7408 (9)	0.0379 (5)	0.5158 (7)	3.6 (3)
C_{12}	0.3414 (9)	0.0968 (5)	0.4975 (7)	4.2 (3)	H_{12}[d]	0.4109	0.0845	0.4949	4.7
C_{13}	0.3475 (10)	0.0902 (5)	0.5830 (8)	5.0 (3)	H_{13}	0.4204	0.0716	0.6406	5.5
C_{14}	0.2550 (10)	0.1088 (5)	0.5879 (8)	5.0 (3)	H_{14}	0.2626	0.1053	0.6503	5.5
C_{15}	0.1540 (10)	0.1316 (5)	0.5101 (8)	4.8 (3)	H_{15}	0.0852	0.1436	0.5141	5.2
C_{16}	0.1435 (9)	0.1390 (5)	0.4221 (7)	4.4 (3)	H_{16}	0.0693	0.1571	0.3648	4.8
C_{21}	0.1449 (9)	0.2041 (5)	0.2361 (7)	3.8 (3)	H_{22}	0.2536	0.2378	0.3732	6.8
C_{22}	0.1868 (11)	0.2444 (6)	0.3023 (8)	6.2 (4)	H_{23}	0.1661	0.3239	0.3194	8.3
C_{23}	0.1366 (12)	0.2947 (6)	0.2710 (10)	7.6 (4)	H_{24}	0.0113	0.3399	0.1511	8.2
C_{24}	0.0466 (12)	0.3088 (6)	0.1735 (10)	7.5 (4)	H_{25}	−0.0610	0.2721	0.0371	8.6
C_{25}	0.0054 (12)	0.2649 (6)	0.1079 (10)	7.8 (4)	H_{26}	0.0277	0.1838	0.0902	6.1
C_{26}	0.0565 (10)	0.2128 (6)	0.1389 (8)	5.6 (4)	H_{32}	0.1488	0.0218	0.1675	4.3
C_{31}	0.0591 (9)	0.0813 (5)	0.1877 (7)	3.6 (3)	H_{33}	−0.0340	−0.0309	0.0652	4.5
C_{32}	0.0669 (9)	0.0340 (5)	0.1511 (7)	3.9 (3)	H_{34}	−0.2270	−0.0023	0.0259	5.4
C_{33}	−0.0399 (9)	0.0033 (5)	0.0913 (7)	4.1 (3)	H_{35}	−0.2420	0.0789	0.0875	6.4
C_{34}	−0.1510 (10)	0.0197 (5)	0.0687 (8)	5.0 (3)	H_{36}	−0.0606	0.1319	0.1908	5.0
C_{35}	−0.1596 (11)	0.0669 (6)	0.1046 (8)	5.9 (4)	H_{42}	0.8035	0.1676	0.4039	5.6
C_{36}	−0.0541 (10)	0.0979 (5)	0.1646 (8)	4.6 (3)	H_{43}	0.8898	0.2354	0.3583	6.8
C_{41}	0.6918 (8)	0.1342 (4)	0.2688 (7)	3.3 (3)	H_{44}	0.8110	0.2422	0.1900	6.3
C_{42}	0.7757 (10)	0.1698 (5)	0.3346 (8)	5.1 (3)	H_{45}	0.6707	0.1813	0.0758	5.9
C_{43}	0.8255 (11)	0.2103 (6)	0.3085 (8)	6.2 (4)	H_{46}	0.5866	0.1141	0.1202	5.6
C_{44}	0.7809 (11)	0.2133 (6)	0.2115 (8)	5.8 (4)	H_{52}	0.7676	0.0252	0.2437	4.6
C_{45}	0.6983 (10)	0.1786 (5)	0.1450 (8)	5.4 (4)	H_{53}	0.7496	−0.0500	0.1500	5.7
C_{46}	0.6502 (10)	0.1390 (5)	0.1709 (8)	5.1 (3)	H_{54}	0.5819	−0.1088	0.0857	5.8
C_{51}	0.5973 (9)	0.0122 (5)	0.2236 (7)	3.6 (3)	H_{55}	0.4188	−0.0901	0.1036	5.7
C_{52}	0.6935 (9)	0.0016 (5	0.2134 (7)	4.2 (3)	H_{56}	0.4318	−0.0155	0.1925	3.9
C_{53}	0.6835 (10)	−0.0430 (5)	0.1600 (8)	4.9 (3)	H_{62}	0.9048	0.0717	0.4351	4.5
C_{54}	0.5860 (10)	−0.0767 (5)	0.1216 (8)	5.2 (3)	H_{63}	1.0709	0.0455	0.5959	5.3
C_{55}	0.4919 (10)	−0.0660 (5)	0.1328 (8)	5.2 (3)	H_{64}	1.0289	0.0141	0.7077	4.9
C_{56}	0.4992 (9)	−0.0223 (5)	0.1840 (7)	3.6 (3)	H_{65}	0.8206	0.0101	0.6576	4.8
C_{61}	0.7652 (8)	0.0561 (4)	0.4516 (6)	3.1 (3)	H_{66}	0.6529	0.0354	0.4944	3.9

[a] The estimated standard deviation in the least significant figure(s) is given in parentheses following the value of each parameter. None is given for the hydrogen atoms as these were not refined. [b] The numbering system corresponds to that shown in Figure 1. The dichloromethane molecule consists of the atoms C_9, Cl_2, and Cl_3. The subscripts attached to the remaining carbon atoms give the number of the phenyl ring of which it is a part (first digit) followed by the number of the atom in the ring. Rings 1–3 are attached to Sb_1 while rings 4–6 are attached to Sb_2. The atoms in each ring are numbered sequentially around the ring with atom 1 being the one bound to antimony (*i.e.*, C_{21} is the carbon atom of ring 2 which is attached to Sb_1). The phenyl hydrogens are numbered to correspond to the carbon to which they are attached. [c] Anisotropic thermal parameters ($\times 10^3$). [d] Hydrogen atom positions as calculated on the assumption of a C–H distance of 1.0 Å. No esd's are given as these parameters were not refined.

TABLE III

INTERATOMIC DISTANCES (Å)[a]

Rh–Sb_1	2.586 (1)	C_6–F_{62}	1.329 (20)
Rh–Sb_2	2.584 (1)	C_6–F_{63}	1.138 (18)
Rh–Cl_1	2.381 (3)	C_7–F_{71}	1.122 (18)
Rh–C_1	2.000 (10)	C_7–F_{72}	1.346 (17)
C_1–C_2	1.330 (16)	C_7–F_{73}	1.324 (19)
C_2–C_3	1.388 (14)	C_8–F_{81}	1.307 (15)
C_3–C_4	1.367 (15)	C_8–F_{82}	1.283 (14)
Rh–C_4	1.964 (11)	C_8–F_{83}	1.372 (14)
C_1–C_5	1.486 (15)	Sb_1–C_{11}[b]	2.120 (9)
C_2–C_6	1.611 (19)	Sb_1–C_{21}	2.116 (12)
C_3–C_7	1.577 (22)	Sb_1–C_{31}	2.118 (10)
C_4–C_8	1.481 (14)	Sb_2–C_{41}	2.106 (10)
C_5–F_{51}	1.284 (15)	Sb_2–C_{51}	2.111 (11)
C_5–F_{52}	1.314 (13)	Sb_2–C_{61}	2.106 (9)
C_5–F_{53}	1.285 (15)	C_9–Cl_2	1.757 (16)
C_6–F_{61}	1.396 (18)	C_9–Cl_3	1.765 (16)

[a] Estimated standard deviation in least significant figure(s) given in parentheses after each value. [b] See Table II, footnote *b*, for explanation of numbering system for this and subsequent entries.

TABLE IV

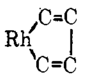
INTERBOND ANGLES (DEG)[a]

Sb_1–Rh–Sb_2	178.25 (4)	C_2–C_6–F_{63}	116.4 (3)
Sb_1–Rh–C_4	91.2 (3)	F_{61}–C_6–C_{62}	88.3 (12)
Sb_1–Rh–C_1	91.5 (3)	F_{61}–C_6–F_{63}	109.2 (16)
Sb_1–Rh–Cl_1	88.08 (7)	F_{62}–C_6–F_{63}	126.1 (18)
Sb_2–Rh–Cl_1	90.18 (7)	C_3–C_7–F_{71}	118.0 (13)
Sb_2–Rh–C_4	90.3 (3)	C_3–C_7–F_{72}	105.2 (11)
Sb_2–Rh–C_1	89.8 (3)	C_3–C_7–F_{73}	104.7 (12)
Cl_1–Rh–C_1	139.7 (3)	F_{71}–C_7–F_{72}	111.3 (15)
Cl_1–Rh–C_4	143.1 (3)	F_{71}–C_7–F_{73}	122.1 (16)
C_1–Rh–C_4	77.2 (4)	F_{72}–C_7–F_{73}	91.3 (12)
Rh–C_1–C_2	116.4 (7)	C_4–C_8–F_{81}	112.0 (9)
Rh–C_1–C_5	112.9 (7)	C_4–C_8–F_{82}	117.4 (11)
C_5–C_1–C_2	130.7 (9)	C_4–C_8–F_{83}	112.2 (9)
C_1–C_2–C_3	116.1 (9)	F_{81}–C_8–F_{82}	106.5 (10)
C_1–C_2–C_6	122.5 (11)	F_{81}–C_8–F_{83}	100.0 (8)
C_6–C_2–C_3	121.4 (12)	F_{82}–C_8–F_{83}	107.0 (9)
C_2–C_3–C_4	111.4 (10)	Rh–Sb_1–C_{11}[b]	114.5 (3)
C_2–C_3–C_7	122.2 (10)	Rh–Sb_1–C_{21}	118.1 (3)
C_7–C_3–C_4	126.2 (9)	Rh–Sb_1–C_{31}	116.4 (3)
C_3–C_4–Rh	118.8 (7)	C_{11}–Sb_1–C_{21}	102.6 (7)
C_3–C_4–C_8	125.6 (10)	C_{11}–Sb_1–C_{31}	102.0 (6)
C_8–C_4–Rh	115.6 (7)	C_{21}–Sb_1–C_{31}	100.9 (7)
C_1–C_5–F_{51}	113.8 (10)	Rh–Sb_2–C_{41}	116.6 (3)
C_1–C_5–F_{52}	114.2 (9)	Rh–Sb_2–C_{51}	116.6 (2)
C_1–C_5–F_{53}	113.3 (10)	Rh–Sb_2–C_{61}	113.5 (3)
F_{51}–C_5–F_{52}	102.5 (8)	C_{41}–Sb_2–C_{51}	101.8 (6)
F_{51}–C_5–F_{53}	106.1 (9)	C_{41}–Sb_2–C_{61}	103.2 (7)
F_{52}–C_5–F_{53}	106.0 (11)	C_{51}–Sb_2–C_{61}	103.4 (6)
C_2–C_6–F_{61}	104.6 (12)	Cl_2–C_9–Cl_3	107.1 (8)
C_2–C_6–F_{62}	106.8 (13)		

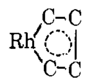
[a] The estimated standard deviation of the least significant figure(s) is given in parentheses. [b] See Table II, footnote *b*, for explanation of the numbering system for this and subsequent entries.

formally a simple σ bond involving an sp^3 carbon atom, it has been suggested that the observed bond length is shorter than would be expected on this basis. Support for a degree of rehybridization of the carbon atom to accommodate Rh–C multiple bonding is found in the pattern of angles about this carbon atom. However it was also pointed out that the shortening of the bond could be due to orbital contraction on the metal, and in the absence of a reliable value for the radius of Rh(III) in these situations no firm conclusion as to the amount of π character (if any) in the bond could be made.[32] Essentially the same conclusion was reached by Churchill and Mason in a subsequent discussion of transition metal–perfluoroalkyl bonds.[33] In the present case, on the other hand, there is a π system present in the fluorocarbon portion of the molecule which is ideally oriented for dπ–pπ overlap and the short Rh–C distances are certainly in accord with this. Further support for Rh–C multiple bonding is based on a comparison of the carbonyl stretching frequencies observed for $Rh(CO)Cl(Sb(C_6H_5)_3)_2C_4(CF_3)_4$[4] and the similar complexes $RhCl_2(CO)(P(C_6H_5)_3)_2R$ (R = C_6H_5, C_2H_5, CH_3).[34] In the latter series, $\nu_{C\equiv O}$ is 2072 cm^{-1} for the phenyl complex whereas for the methyl and ethyl analogs it occurs at 2062 and 2060 cm^{-1}, respectively. Thus, the complex with the organic group capable of π-back-bonding shows the higher carbonyl stretching frequency. For the carbonyl derivative of the fluorocarbon complex, $\nu_{C\equiv O}$ is 2096 cm^{-1} indicating an even greater competition by the organic group for metal dπ electrons. The same conclusion is also reached when the ^{19}F nmr spectra of the metallocycle and its carbonyl derivative[4] are compared. In the former a single complex resonance is observed for the two trifluoromethyl groups closest to the rhodium while in the latter, these groups, having become nonequivalent, give rise to two complex resonances which are both at lower fields than in the parent complex. Again, π interaction between the metal and the remainder of the ring is consistent with these observations.

An inspection of the C–C bond lengths in the remainder of the ring (Table III) shows that a considerable degree of π delocalization occurs over the four-carbon portion. The C_1–C_2 and C_2–C_3 distances are significantly different (Δ/σ = 3.8) whereas the distances C_1–C_2 and C_3–C_4 as well as C_2–C_3 and C_3–C_4 are equivalent at our level of accuracy. There does not appear to be any reasonable chemical explanation for the asymmetry of the ring and it may arise simply from packing considerations. (One explanation which could eliminate both this problem and that of the difference in the Rh–C distances is that C_1 is slightly misplaced but there is no evidence from either difference maps or thermal parameters that this is the case.) We conclude, therefore, that the C–C distances in the ring are substantially equal, and in view of our earlier remarks concerning the metal→ring π donation, the actual electronic structure is probably somewhere between the extremes



and



A similar conclusion has been reached in discussions of the electronic structures of a variety of metal com-

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(33) M. R. Churchill and R. Mason, *Adv. Organometal. Chem.*, **5**, 93 (1967).

(34) M. C. Baird, J. T. Mague, J. A. Osborn, and G. Wilkinson, *J. Chem. Soc., A*, 1347 (1967).

plexes of "butadiene-like" moieties.[33] Certainly, the C_2–C_3 distance is significantly shorter than the corresponding C–C distance in butadiene (1.483 (10) Å)[35] and cyclopentadiene (1.46 (4) Å)[36] indicating that the true description is closer to the latter extreme.

An examination of the C(ring)–C(trifluoromethyl) distances shows that C_1–C_5 and C_4–C_8 are equivalent with an average length of 1.484 (15) Å. Likewise C_2–C_6 and C_3–C_7 are equivalent, but their average length, 1.594 (21) Å, is significantly longer. The primary reason for this difference is most likely to be the considerably higher uncertainty in the positions of C_6 and C_7 as compared to C_5 and C_8 (Table II). Similar difficulty in accurately locating the carbon atoms of trifluoromethyl groups has been experienced previously with the result that the reported distances involving these atoms vary widely.[37–39] In the compound π-$C_5H_5RhC_6(CF_3)_6$, the C(ring)–C(trifluoromethyl) distances vary from 1.482 (25) to 1.550 (31) Å and within the limits of error of that determination[37] are the same as those found in the present case.

Figure 2 presents a schematic representation of the F---F contacts in the molecule. Within the trifluoromethyl groups containing C_5 and C_8 these are all $\sim$2.1 Å, which is less than a normal van der Waals contact but comparable to values found in other cases.[33,38,39] Within the other two trifluoromethyl groups, the range is considerably greater with some being as low as 1.9 Å. However this is mainly a result of large uncertainties of position due to the extremely large amplitudes of vibration for the fluorine atoms in these two groups. The F---F contacts between adjacent groups are, on the average, somewhat shorter than observed previously in similar compounds,[37–39] but none is substantially shorter than normal van der Waals contacts. As mentioned earlier, one can correlate the relative sizes of the derived thermal ellipsoids for the fluorine atoms (Figure 1) with these contacts. Thus F_{63} and F_{71} are seen to be the most unrestricted and in fact are those with the largest thermal ellipsoids. Further, the deviations of the atoms C_1–C_8 from the mean plane of the metallocycle can be regarded as the result of the minimization of F---F contacts between trifluoromethyl groups.

The ^{19}F spectrum of the complex which has been referred to above shows two broad and complex resonances at −9.8 and −12.7 ppm (benzotrifluoride, internal standard). The one at lower field has been assigned to the pair of trifluoromethyl groups adjacent to the metal (on C_1 and C_4) while the other is due to the pair on the outer end of the ring. The present study shows that the two groups in each pair are in fact very nearly equivalent. The broad, complex appearance of the signals suggests that the "interlocking" of the four CF_3 groups around the ring persists in solution thus rendering the fluorines within each group nonequivalent. By contrast the complex $[Rh(vdiars)_2(C_4F_6)]BF_4$[40] (vdiars = *cis*-$(C_6H_5)_2AsCH{=}CHAs(C_6H_5)_2$) which contains one molecule of coordinated hexafluorobut-2-yne and which could be expected not to contain similarly hindered trifluoromethyl groups shows a single sharp signal for these fluorine atoms in its ^{19}F nmr spectrum.

Acknowledgment.—We are especially grateful to Dr. Charles J. Fritchie, Jr., of this department for the use of the diffractometer and for much helpful advice and discussion.

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(35) See ref 29, p M164.

(36) See ref 29, p M180.

(37) M. R. Churchill and R. Mason, *Proc. Roy. Soc., Ser. A*, **292**, 71 (1966).

(38) M. Gerloch and R. Mason, *ibid., Ser. A*, **279**, 170 (1964).

(39) N. A. Bailey and R. Mason, *Acta Crystallogr.*, **21**, 652 (1966).

(40) J. T. Mague and J. P. Mitchener, *Chem. Commun.*, 911 (1968).