Journal of Organometallic Chemistry, 191 (1980) 209—218
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STRUCTURE OF THE REACTION PRODUCT OF PENTAPHENYLGERMOLE AND DIIRON ENNEACARBONYL: μ-(1-(1,2,3,4-TETRAPHENYLBUTADIENYL)PHENYLGERMYLENE)-OCTACARBONYLDIIRON(*Fe—Fe*)

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(Received November 15th, 1979)

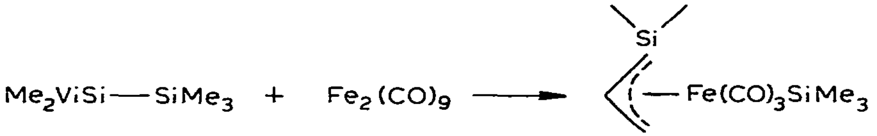
Summary

Pentaphenylgermole (1-germa-1,2,3,4,5-pentaphenylcyclopenta-2,4-diene) reacts with diiron enneacarbonyl in refluxing diethyl ether to produce an orange, crystalline complex. This complex was established by single crystal X-ray diffraction to be the result of a novel hydroferration of the Ge—C σ-bond of the germacyclopentadiene moiety. The structure is of the type, RR'Ge[Fe(CO)$_4$]$_2$, in which R is a phenyl and R' is the butadienyl fragment. The RR'Ge group bridges the Fe—Fe bond of the Fe$_2$(CO)$_8$ fragment. The coordination about each iron atom is distorted octahedral with one Fe, the Ge, and 4 CO's making up the coordination sphere. Three pairs of carbonyls are eclipsed and the structure shows severe distortions to relieve the non-bonded repulsions between these eclipsed carbonyl groups. The relevant crystal parameters are: a 11.466(3), b 11.228(4), c 16.178(5) Å, α 91.41(2), β 108.99(2), γ 101.34(2)°, V 1922(1) Å^3, $\rho = 1.46$, $Z = 2$, space group = $P\bar{1}$. The final R-values, based 3234 reflections with $I > 3\sigma(I)$ are $R = 0.079$ and $R_w = 0.095$ with all non-hydrogen atoms included with isotropic temperature factors except for the two iron and germanium atoms which are anisotropic. Some molecular parameters are (distances in pm): Ge—Fe^1, 240.8(2); Ge—Fe^2, 243.0(2); Fe^1—Fe^2, 278.5(3); Fe—CO, 174 ± 2; Ge—C, 198 ± 2, C—O, 117 ± 2; Fe^1-Ge—Fe^2, 70.30(8); Ge—Fe^1—Fe^2, 55.23(6); Ge—Fe^2—Fe^1, 54.47(6); and C—Ge—C, 105.5(5).

Introduction

Some years ago we were interested in stabilizing $p\pi$—$p\pi$ bonding between the heavier Group IV congeners and carbon by incorporating the heteroelement (E) in a cyclic, 6-π electron structure, e.g., a hetero-cyclopentadienide ion [1], or by coordination of the C—E π-system to transition metal species

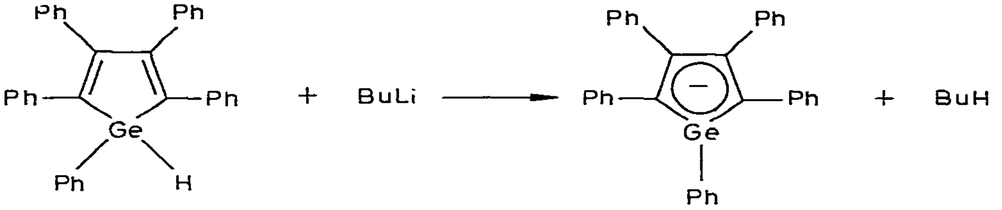
[1—6]. In our hands, the latter approach was unsuccessful but has apparently been brought to fruition by Sakurai et al. [7] who have reported the synthesis of the π-silaallyliron complex, I [7] *.



(1)

(I)

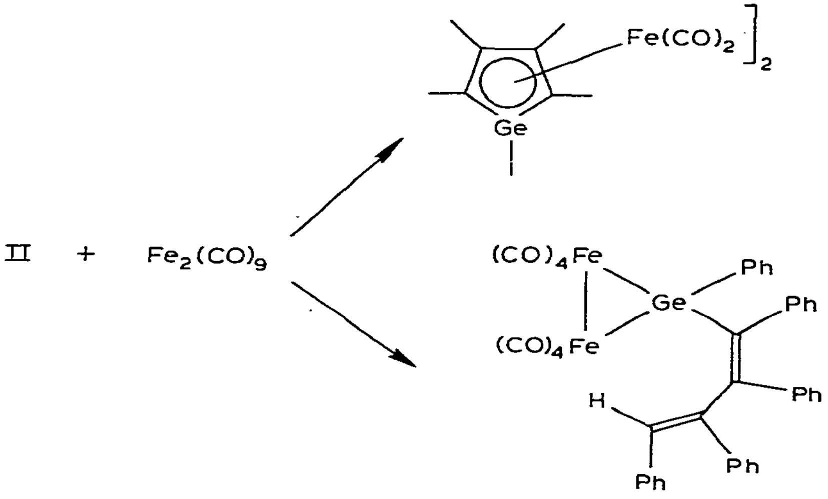
However, the anion of pentaphenylgermole (III) was deduced to be charged-delocalized, this deduction being based on the bright red color of III and the fact that pentaphenylgermole (II) is at least 10^6 times more acidic than triphenylgermane [1].



(2)

(II) (III)

Consequently, it was of some interest to determine if compound II would form π-germacyclopentadienyl metal complexes. Since cyclopentadiene reacts with metal carbonyls to give species of the type, (π-C_5H_5)$M(CO)_n$ or (π-C_5H_5)$_2M_2(CO)_m$, we allowed complex II to react with $Fe_2(CO)_9$, hoping to observe the reaction shown in (3a). Instead, the reaction has now been shown to proceed as in (3b) to produce a germanium bridged, iron dimer (IV) in which a Ge—C σ-bond of the germole ring has been "hydroferrated", thus transforming the germole ring into an acyclic tetraphenylbutadienyl ligand.



(3a)

(3b)

(IV)

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* Note added in proof: Radnia and McKennis [22] have shown the purported complexes I to be simple η^2-vinyliron tetracarbonyl complexes.

Experimental

Pentaphenylgermole [1] (1 g) and an equimolar amount of diiron enneacarbonyl were placed in a 100 ml Schnlenk tube under a N_2 atmosphere. Fifty ml of dry diethyl ether was added and the mixture refluxed for 1.5 h, after which time the insoluble $Fe_2(CO)_9$ had dissolved, giving a bright orange solution. The solution was concentrated and cooled to 0°C, affording a nearly quantitative yield of large, orange crystals. These were recrystallized from CH_2Cl_2/EtOH by first dissolving the solid in CH_2Cl_2, adding EtOH, and then slowly boiling off the methylene chloride. The pure products melts at 196—197°C (in air) with decomposition. Anal. Found: C, 59.23; H, 3.18; Fe, 13.16; Ge, 10.70 and 12.05. $C_{42}H_{26}Fe_2GeO_8$ calcd.: C, 59.85; H, 3.08; Fe, 13.25; Ge, 8.61%; MW, Found 818, calcd. 842.7 (analyses and MW by Galbraith Laboratories, Knoxville, Tennessee).

A suitable crystal was selected, mounted on a glass fiber, and then placed on a Syntex $P2_1$ diffractometer * (see Table 1 for relevant statistics). Initial counter data and axial oscillation photos showed the crystals to be triclinic. With $Z = 2$, the space group $P\bar{1}(C_i^1$, No. 2) was chosen. A Patterson map revealed the location of the germanium and two iron atoms. These heavy atom positions were refined once and the subsequent difference map revealed the positions of all the remaining non-hydrogen atoms. The structure converged with all atoms isotropic after two full matrix refinements. Two more cycles with the heavy atoms anisotropic gave the final structure. On the last cycle, the largest parameter shifts were comparable to the errors in the parameters. The total number of variables in the refinement was 228, giving a data/variable ratio = 14.2. The largest peak in the final difference map was 0.9 $e/Å^3$ and appeared about 0.8 Å from C13. No attempt was made to locate the hydrogen atoms.

Results

The asymmetric unit consists of one molecule of the iron-germanium complex. There are no unusual intermolecular contacts. Figure 1 shows a view of the molecule down the Fe^2—Fe^1 axis and shows the atom labelling scheme. Figure 2 is an ORTEP drawing of the inner coordination spheres of the iron atoms, the germanium, and the butadienyl fragment. Also shown in Figure 2 are the primary carbons of the phenyl groups bonded to germanium and the butadienyl fragment.

Table 2 gives the atomic positions in the unit cell and the temperature factors. Table 3 gives the derived bond distances and angles, and Table 4 ** describes some calculated, least-squares planes. Table 5 ** lists the observed and calculated structure factors.

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* A description of the programs and procedures in the crystallographic analysis have been given eleswhere [8].

** Supplementary material (Tables 4 and 5): see NAPS document 03622 for 13 pages of supplementary material. Order from NAPS, c/o Microfiche Publications, P.O. Box 3513, Grand Central Station, New York, New York 10017. Remit in advance for each NAPS accession number. Institutions and Organizations may use purchase orders when ordering, however, there is a $ 5.00 charge for this service. Make checks payable to "Microfiche Publications". Photocopies are $ 5.00. Microfiche are $ 3.00 each. Outside the United States and Canada, postage is $ 3.00 for a photocopy and $ 1.00 for a fiche.

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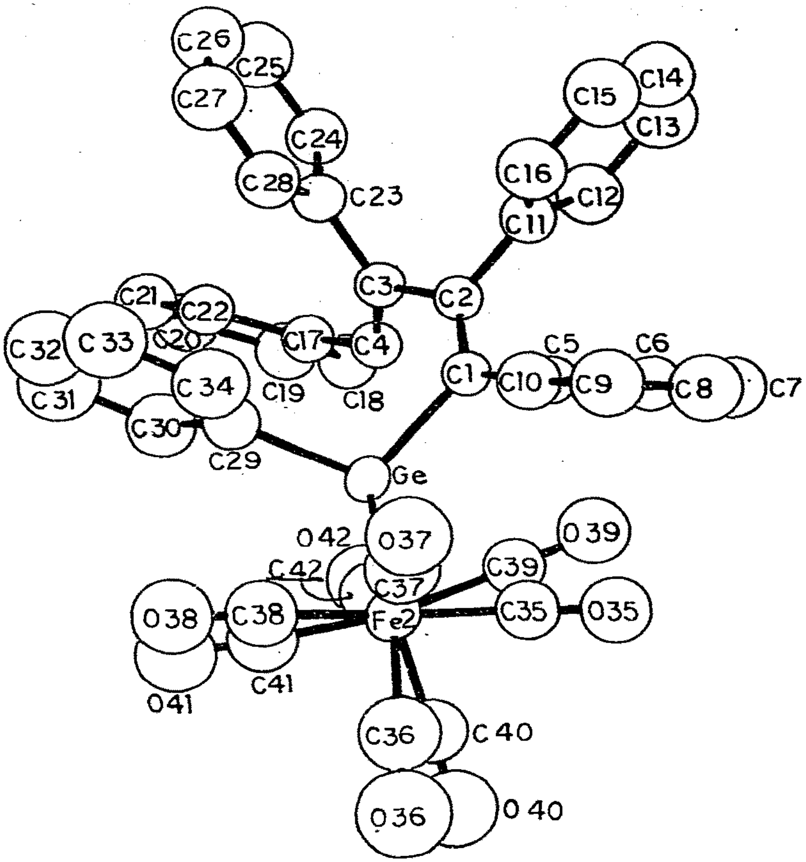


Fig. 1. A perspective down the Fe—Fe bond of μ-((Ph$_4$C$_4$H)PhGe))Fe$_2$(CO)$_8$.

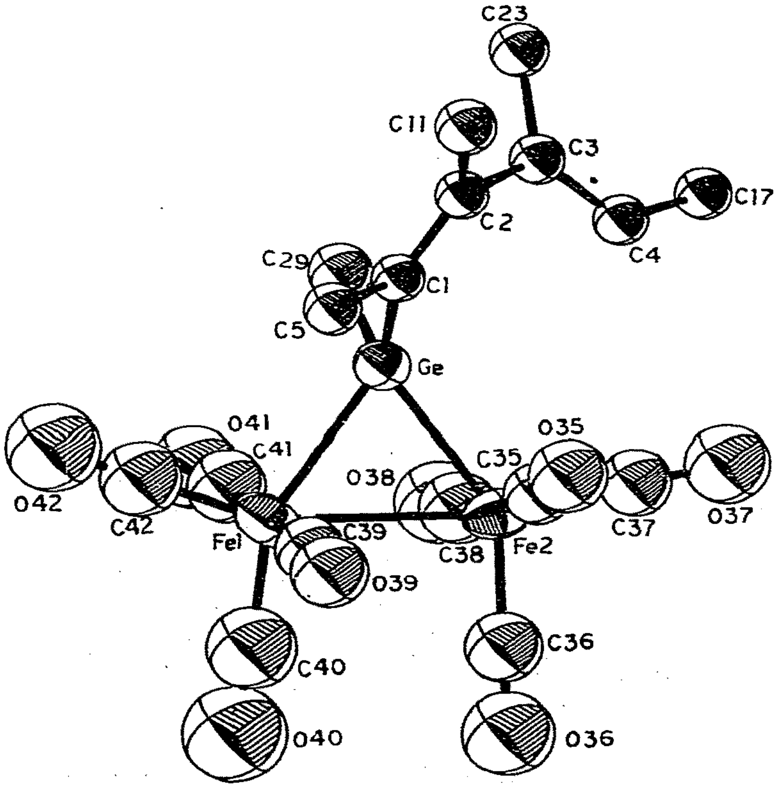


Fig. 2. The inner coordination sphere of μ-((Ph$_4$C$_4$H)PhGe)Fe$_2$(CO)$_8$.

TABLE 1

SUMMARY OF CRYSTAL AND DATA COLLECTION STATISTICS

a, b, c (Å)	11.466(3), 11.228(4), 16.178(5)
α, β, γ (°)	91.41(2), 108.99(2), 101.34(2)
V (Å^3), Z, $\rho_{calc.}$	1922(1), 2, 1.46 g/cm^3
Crystal dimensions (mm)	0.20, 0.17, 0.34
Radiation	Mo-K_α (monochromatized from graphite)
Take Off Angle	4°
μ (cm^{-1})	15.6 (Mo-K_α)
Transmission factors	0.77 (max.), 0.71 (min.)
Scan speed (°/min)	2—15 as a function of peak intensities
Scan range	K_{α_1} −0.8° to K_{α_2} +0.8°
Background/Scan time	0.8
Std. reflections [a]	221, 020, 002
2θ limit	45°
Reflections	5059 (total), 3234 (with $I > 3\sigma(I)$)
R_1, R_2	0.079, 0.095
$[\Sigma\omega(\lvert F_0\rvert - \lvert F_c\rvert)^2/(NO - NV)]^{1/2}$	2.86

[a] The intensities of the standard reflections did not change during data collection.

TABLE 2

FRACTIONAL CELL COORDINATES AND TEMPERATURE FACTORS

Atom	x	y	z
Fe	0.4222(1)	0.2008(1)	0.2374(1)
Fe1	0.3755(2)	0.3358(2)	0.1235(1)
Fe2	0.1955(2)	0.1866(2)	0.1783(1)
C1	0.494(1)	0.055(1)	0.226(1)
C2	0.617(1)	0.057(1)	0.255(1)
C3	0.721(1)	0.169(1)	0.293(1)
C4	0.731(1)	0.264(1)	0.245(1)
C5	0.402(1)	−0.061(1)	0.185(1)
C6	0.399(1)	−0.117(1)	0.106(1)
C7	0.312(1)	−0.231(1)	0.068(1)
C8	0.233(2)	−0.280(1)	0.112(1)
C9	0.233(2)	−0.229(2)	0.191(1)
C10	0.319(1)	−0.116(1)	0.226(1)
C11	0.668(1)	−0.056(1)	0.252(1)
C12	0.744(1)	−0.062(1)	0.201(1)
C13	0.788(2)	−0.181(2)	0.199(1)
C14	0.750(2)	−0.267(1)	0.244(1)
C15	0.682(2)	−0.265(2)	0.292(1)
C16	0.624(1)	−0.154(1)	0.298(1)
C17	0.830(1)	0.377(1)	0.271(1)
C18	0.880(1)	0.428(1)	0.207(1)
C19	0.981(1)	0.532(1)	0.232(1)
C20	1.027(1)	0.586(1)	0.316(1)
C21	0.977(1)	0.541(1)	0.379(1)
C22	0.878(1)	0.436(1)	0.357(1)
C23	0.815(1)	0.159(1)	0.382(1)
C24	0.944(1)	0.181(1)	0.393(1)
C25	1.030(1)	0.169(1)	0.480(1)
C26	0.985(2)	0.139(1)	0.544(1)
C27	0.859(2)	0.115(1)	0.535(1)
C28	0.767(1)	0.124(1)	0.449(1)
C29	0.498(1)	0.264(1)	0.363(1)
C30	0.581(1)	0.377(1)	0.388(1)
C31	0.635(2)	0.422(2)	0.483(1)

TABLE 2 (continued)

Atom	*x*	*y*	*z*
C32	0.596(2)	0.347(2)	0.534(1)
C33	0.521(2)	0.240(2)	0.518(1)
C34	0.460(2)	0.190(2)	0.422(1)
C35	0.178(1)	0.088(1)	0.086(1)
O35	0.157(1)	0.020(1)	0.025(1)
C36	0.068(2)	0.244(2)	0.116(1)
O36	−0.023(2)	0.279(1)	0.076(1)
C37	0.134(2)	0.068(2)	0.226(1)
O37	0.093(1)	−0.015(1)	0.260(1)
C38	0.218(1)	0.281(1)	0.272(1)
O38	0.226(1)	0.343(1)	0.335(1)
C39	0.402(1)	0.221(1)	0.062(1)
O39	0.426(1)	0.147(1)	0.023(1)
C40	0.255(2)	0.376(1)	0.037(1)
O40	0.176(1)	0.406(1)	−0.020(1)
C41	0.376(1)	0.446(2)	0.203(1)
O41	0.381(1)	0.523(1)	0.254(1)
C42	0.511(1)	0.432(1)	0.114(1)
O42	0.599(1)	0.490(1)	0.103(1)

Atom	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}	*B*
Ge	3.39(6)	4.28(7)	2.81(6)	0.87(5)	0.87(4)	−0.20(5)	3.46(3)
Fe1	5.13(10)	4.94(10)	3.24(8)	1.84(8)	0.77(7)	0.29(7)	4.33(5)
Fe2	3.34(9)	7.30(12)	4.38(9)	1.65(8)	0.96(7)	−0.65(8)	4.64(5)

Atom	*B*	Atom	*B*
C1	3.4(2)	C24	5.3(3)
C2	3.3(2)	C25	7.4(4)
C3	3.6(2)	C26	7.6(4)
C4	3.6(2)	C27	7.8(4)
C5	4.0(2)	C28	5.8(3)
C6	5.2(3)	C29	4.8(3)
C7	6.4(3)	C30	6.6(4)
C8	7.8(4)	C31	9.7(5)
C9	7.9(4)	C32	9.6(5)
C10	5.8(3)	C33	10.0(5)
C11	4.4(3)	C34	8.5(4)
C12	6.2(3)	C35	5.8(3)
C13	8.0(4)	O35	7.2(2)
C14	7.5(4)	C36	9.8(5)
C15	8.3(4)	O36	13.3(5)
C16	6.9(4)	C37	8.2(4)
C17	3.9(2)	O37	10.7(3)
C18	4.9(3)	C38	7.4(4)
C19	5.6(3)	O38	9.5(3)
C20	5.8(3)	C39	5.5(3)
C21	5.5(3)	O39	7.5(2)
C22	4.5(3)	C40	7.2(4)
C23	4.2(2)	O40	10.2(3)
		C41	7.3(4)
		O41	9.5(3)
		C42	6.3(3)
		O42	8.8(3)

TABLE 3

INTERATOMIC DISTANCES AND BOND ANGLES IN $Ph(Ph_4C_4H)GeFe_2(CO)_8$

Distances (pm)		Bond angles (deg)	
Ge—Fe1	240.8(2)	Fe1—Ge—Ge2	70.30(8)
Ge—Fe2	243.0(2)	Ge—Fe1—Fe2	55.23(6)
Fe1—Fe2	278.5(3)	Ge—Fe2—Fe1	54.47(6)
Ge—C29	197.(1)	C1—Ge—C29	105.5(5)
Ge—C1	200(1)	C39—Fe1—C40	96.1(6)
Fe1—C39	173(1)	C39—Fe1—C41	168.3(7)
Fe1—C40	176(2)	C39—Fe1—C42	87.9(6)
Fe1—C41	176(2)	C40—Fe1—C41	95.4(7)
Fe1—C42	176(2)	C40—Fe1—C42	101.4(7)
Fe2—C35	177(1)	C41—Fe1—C42	88.5(7)
Fe2—C36	173(2)	C37—Fe2—C35	91.3(7)
Fe2—C37	170(2)	C37—Fe2—C36	105.8(8)
Fe2—C38	174(2)	C37—Fe2—C38	87.7(8)
C1—C2	134(1)	C36—Fe2—C35	89.9(8)
C2—C3	151(1)	C36—Fe2—C38	92.5(8)
C3—C4	135(1)	C38—Fe2—C35	177.6(8)
C1—C5	148(1)	C2—C1—C5	119.7(9)
C2—C11	150(1)	C2—C1—Ge	123.4(8)
C3—C23	150(1)	C5—C1—Ge	116.8(7)
C4—C17	147(1)	C1—C2—C11	122.4(9)
C—C(Ph) [a, b]	140 ± 6	C1—C2—C3	125.2(9)
C35)O35	116(1)	C11—C2—C3	112.4(9)
C36—O36	119(2)	C4—C3—C23	124.9(9)
C37—O37	119(2)	C4—C3—C2	120.0(9)
C38—O38	119(2)	C3—C4—C17	126.2(9)
C39—O39	117(1)	C—C—C (Ph) [a, b]	120 ± 4
C40—O40	117(2)	Fe2—C35—O35	175(1)
C41—O41	116(2)	Fe2—C36—O36	176(2)
C42—O42	115(1)	Fe2—C37—O37	179(2)
C—O [a]	117 ± 2	Fe2—C38—O38	176(1)
		Fe1—C39—O39	177(1)
Bond angles (deg)		Fe1—C40—O40	178(1)
C29—Ge—Fe1	121.4(1)	Fe1—C41—O41	176(1)
C29—Ge—Fe2	113.4(1)	Fe1—C42—O42	176(1)
C1—Ge—Fe2	120.3(1)	Fe—C—O [a]	177 ± 1
C39—Fe1—Ge	82.8(1)	C37—Fe2—Ge	104.1(1)
C39—Fe1—Fe2	96.4(1)	C37—Fe2—Fe1	157.4(1)
C40—Fe1—Ge	144.9(1)	C36—Fe2—Ge	150.0(1)
C40—Fe1—Fe2	90.3(1)	C36—Fe2—Fe1	96.1(1)
C41—Fe1—Ge	88.2(1)	C38—Fe2—Ge	85.3(1)
C41—Fe1—Fe2	84.8(1)	C38—Fe2—Fe1	96.3(1)
C42—Fe1—Ge	113.6(1)	C35—Fe2—Ge	92.6(1)
C42—Fe1—Fe2	167.0(1)	C35—Fe2—Fe1	83.6(1)

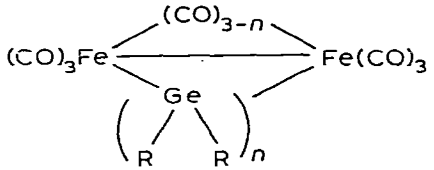
[a] Average values. Standard deviations of the averages were calculated from the formula, $\sigma = [\Sigma(x_n - \bar{x})^2/n(n-1)]^{1/2}$. [b] The ranges of the C—C bond distances and C—C—C bond angles in the phenyl groups are 130—152 pm and 115—132°.

Discussion

The reaction of pentaphenylgermole with $Fe_2(CO)_9$ results in the transfer of the hydrogen originally on the germanium to one of the α-carbons of the germole ring with concommitant cleavage of the Ge—C σ-bond. The resulting RR′Ge fragment then bridges the Fe—Fe bond of a $(CO)_4Fe—Fe(CO)_4$ moiety.

While such recombinations of ligands on germanium may occur when, e.g., R_3GeH, reacts to give R_2Ge complexes [9], the nature of the cleaved organic fragment has not been established previously. In our case, the cyclic structure of the germole retains the cleavage fragment, allowing its identification. The mechanism of the transfer of hydrogen from germanium to carbon has not been established, however.

Organogermanes form an interesting series of complexes with the iron and cobalt carbonyls [3,9,10] in which the R_2Ge group replaces a bridging carbonyl in the parent $M_2(CO)_n$ ($M = Fe$, $n = 9$; $M = Co$, $n = 8$). Structure V is an example of this type of complex. A second type of dimeric iron germanium complex is typified by structure VI in which there is no formal metal—metal bond [11]. The structure of IV reported here is apparently the first structurally characterized complex of type VII although the related phenyl derivative (VII, R = Ph) has been reported as one of the products of the reaction of Ph_2GeH_2 with $Fe_2(CO)_9$ [12], and several related tin complexes are known and structurally characterized [13,14].



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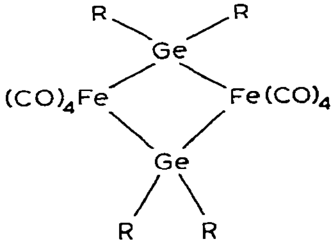

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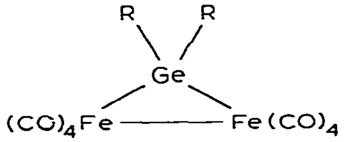

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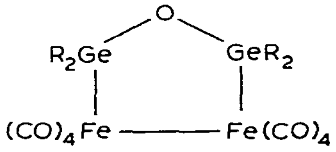

(VIII)

Somewhat related structures VIII are formed from the interaction of tetraalkyldigermoxanes with $Fe_2(CO)_9$ or $Fe(CO)_5$ upon photolysis [15,16].

The mixing of the orbitals of bridging R_2E ($E = Ge$, Sn) groups with the orbitals used to form metal—metal bonds has been discussed previously, and the conclusion was drawn that the bridging groups actually contribute a net bonding interaction (in spite of apparent strain!) to the metal—metal bond [17,18]. A comparison of the structure of IV with several other types of Fe—Ge complexes supports this earlier conclusion. Thus, the Fe—Fe bond increases in length in the series: $(CO)_3Fe(CO)_3Fe(CO)_3$ (252.3 ppm [19]) < $(CO)_3Fe(\mu\text{-}CO)(\mu\text{-}GePh_2)_2Fe(CO)_3$ (266.6 pm) < $(CO)_3Fe(\mu\text{-}GeMe_2)_3Fe(CO)_3$ (275.0 pm [20]) < IV (278.5 pm) < VIII (287.6 pm). The bridging carbonyls are more effective in strengthening the Fe—Fe bond than bridging R_2Ge groups, and in VIII, the geometry precludes extensive interaction of the bridging groups which lead to stabilization of the metal—metal bond (see ref. 19 for a pictorial representation of the orbital mixing).

The FeGeFe angle opens up in the above series as the Fe—Fe bond elongates as would be expected on simple geometrical grounds, the values being 66.8°, 70.0°, and 70.30° respectively for the first three germanium bridged iron complexes in the series. In compound VI the FeGeFe angle is 104.5°, reflecting the lack of an Fe—Fe bond. The Fe—Ge distances show considerable variation in the structures reported, but no trends are apparent. Thus, the Fe—Ge distances in $(CO)_3Fe(\mu\text{-}GePh_2)_2(\mu\text{-}CO)Fe(CO)_3$ range from 240.2 to 244.0 (242.5 ± 2.0 average), 247.5 in compound VIII, 239.8 in $(CO)_3Fe(\mu\text{-}GeMe_2)_3Fe(CO)_3$, 240.8 and 243.0 in IV, and 249.2 in VI (R = Et). The values found here for IV are thus in the middle of the reported range of Fe—Ge distances.

Complexes of the type VII and VIII are extremely crowded due to the eclipsing of the carbonyl groups across the Fe—Ge bond. The effect of this crowding is especially evident in Figures 1 and 2 which show how the $Fe(CO)_4$ fragments twist relative to each other in order to relieve the non-bonded repulsions between the eclipsed carbonyl groups (see also the discussion in ref. 19).

The effect of steric crowding is also seen in the tetraphenylbutadienyl fragment. Let plane 1 be defined by the atoms C2, C3, C4, C17, and C23, and plane 2 by atoms C5, Ge, Cl, C2, C3, and C4. The phenyl groups, (C17—C22) and (C23—C28) are depressed out of the mean plane 1, probably by repulsions from the phenyl (C29—C34) attached to the germanium (see Fig. 1). Also, the dihedral angle between planes 1 and 2 is 58.2°. This dihedral angle represents the degree of twist around the central C—C σ-bond of the butadiene, and this twisting can also be ascribed to repulsions from the phenyl on germanium and to Ph—Ph repulsions across the C2—C3 bond. Each phenyl bonded to the butadiene group is also twisted with respect to the planes of the double bonds. The phenyls attached to C1 and C2 are twisted the most (62.1° and 65.4° with respect to plane 2). The phenyls associated with C17 and C23 are twisted 42.0° and 51.6° with respect to plane 1.

It is interesting to compare the structure of the butadiene fragment determined here with that of *E,E*-tetraphenylbutadiene reported by Karle and Dragonette (K-D) [21]. In the K-D structure, the molecule assumes an *s-trans* configuration and the double bonds are strictly coplanar. The central phenyls have a dihedral angle of 75° with respect to the plane of the molecule, while the end phenyls are more nearly coplanar with dihedral angles of 34°. The average C—Ph distance in the K-D structure is 150 pm, compared to an average of 149 ± 1.5 for comparable C—Ph bonds in the butadiene group in this structure. The central C—C σ-bond and the C=C π-bonds in the K-D structure have lengths 149 and 136 pm, respectively. The comparable bond lengths in the structure determined here are 151 (C2—C3), and 134 (C1—C2) and 135 (C3—C4), respectively. Thus, in this structure, the central σ-bond is somewhat longer, and the double bonds somewhat shorter, than those in the K-D structure. These results are consistent with the loss of conjugation across the central C—C bond in the *s-gauche* conformation found here compared to the *s-trans* conformation in the K-D structure.

In summary, pentaphenylgermole reacts with $Fe_2(CO)_9$ to give a completely unexpected product, the structure of which is consistent with previously proposed bonding models for bridging R_2E-groups. The failure to form a derivative of an η^5-germanacyclopentadiene can be due either to a possible thermodynamic instability of such a structure or to kinetic control of the reaction path.

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