Table II. Crystallographic Data for $(PPh_4)_2[Fe_2Cl_4(Cat)_2]\cdot(CH_3)_2CO$

mol wt	1206.5	Z	2
cryst system	triclinic	D_{calcd}, g cm^{-3}	1.276
space group	$P\bar{1}$	D_{exptl}, g cm^{-3}	1.25
a, Å	11.490 (2)	μ, cm^{-1}	7.27
b, Å	12.676 (3)	T_{max}, T_{min}	0.837, 0.736
c, Å	22.087 (4)	radiation (λ, Å)	Mo Kα (0.71073)
α, deg	88.32 (3)	temp, K	296–298
β, deg	77.55 (3)	R, R_w	0.055, 0.072
γ, deg	88.7 (3)	GOF	1.30
V, Å^3	3139 (1)		

Table III. Atomic Coordinates ($\times 10^4$) for the $[Fe_2Cl_4(Cat)_2]^{2-}$ Anion Containing Fe1

atom	x/a	y/b	z/b
Fe1	4165 (1)	1063 (1)	−59 (1)
Cl11	5029 (1)	2638 (1)	−26 (1)
Cl12	3290 (1)	1051 (1)	−882 (1)
O11	4334 (3)	−269 (2)	450 (1)
O12	2815 (3)	1224 (3)	618 (2)
C11	3486 (4)	−372 (4)	977 (2)
C12	2656 (4)	471 (5)	1057 (2)
C13	1724 (5)	451 (7)	1588 (3)
C14	1659 (6)	−361 (8)	2022 (3)
C15	2498 (7)	−1167 (6)	1934 (3)
C16	3415 (5)	−1183 (5)	1411 (3)

solution was reduced to approximately 25 mL and cooled to 0 °C. The dark blue precipitate was separated from the solution by filtration and recrystallized from acetone. Crystals of $(PPh_4)_2[Fe_2Cl_4(Cat)_2]$ obtained by this procedure as acetone solvates were suitable for crystallographic analysis.

Physical Measurements. Electronic spectra were recorded on a Perkin-Elmer Lambda 9 spectrophotometer. Cyclic voltammograms were obtained with a Cypress CYSY-1 computer-controlled electroanalysis system in CH_2Cl_2 solutions. A Ag/Ag$^+$ reference electrode was used, $(NBu_4)(PF_6)$ was used as the supporting electrolyte, and the Fc/Fc$^+$ couple was used as an internal standard.

Structure Determination of $(PPh_4)_2[Fe_2Cl_4(Cat)_2]\cdot(CH_3)_2CO$. Crystals of $(PPh_4)_2[Fe_2Cl_4(Cat)_2]$ were obtained by slow evaporation of an acetone solution of the complex as large dark blue parallelopipeds. Axial photographs indicated only triclinic symmetry, and the centered settings of 25 reflections in the 2θ range between 21 and 32° gave the unit cell dimensions listed in Table II. Data were collected by θ–2θ scans within the angular range 3.0–50.0°. The locations of the iron, chlorine, and phosphorus atoms and the phenyl rings of the two independent PPh$_4^+$ cations were determined by direct methods. A structure factor calculation carried out with these atoms revealed the positions of the atom of the catecholate ligand bonded to Fe1 and the two half-acetone solvate molecules. Refinement of the structure with all atoms except hydrogens and the catecholate ligand of Fe2 was carried out. Thermal parameters of Fe2 and the chlorine atoms bonded to Fe2, Cl21, and Cl22 were unusually large, and the pattern of peaks on a difference Fourier map revealed the disordered catecholate ligand. Two sets of catecholate atoms with occupancies of 0.27 and 0.73 were found to be approximately coplanar, indicating 2-fold disorder about an axis containing Fe2, the inversion center at $[^1/_2, ^1/_2, ^1/_2]$, and Fe2′ generated by the inversion center. Two fractional anions were constructed from the atom parameters of the ordered anion containing Fe1 and located about the inversion center at $[^1/_2, 0, 0]$. These atoms were included in the refinement with the catecholate phenyl ring of the fractional anion with occupancy 0.27 constrained with C–C lengths of 1.392 Å. Final cycles of refinement gave discrepancy indices of $R = 0.055$ and $R_w = 0.072$. Final positional and derived isotropic thermal parameters for the anion containing Fe1 are given in Table III. Tables containing full listings of atom positions, anisotropic thermal parameters, and hydrogen atom locations are available as supplementary material.

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Supplementary Material Available: Tables giving crystal data and details of the structure determination, atom coordinates and isotropic thermal parameters, anisotropic thermal parameters, hydrogen atom locations, and bond distances and angles for $(PPh_4)_2[Fe_2Cl_4(Cat)_2]\cdot(CH_3)_2CO$ (16 pages); a table of observed and calculated structure factors (28 pages). Ordering information is given on any current masthead page.

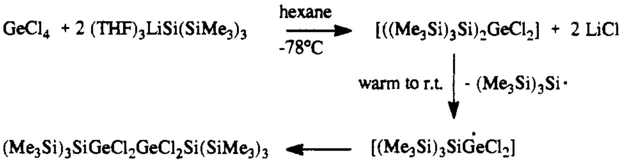
Additions and Corrections

1991, Volume 30

S. P. Mallela and R. A. Geanangel*: Preparation and Structural Characterization of New Derivatives of Digermane Bearing Tris(trimethylsilyl)silyl Substituents.

Page 1480. In the last sentence of the abstract, the formula should be $[(Me_3Si)_3SiGeR_2]_2$.

Page 1481. Scheme I should appear as follows:



—R. A. Geanangel