[μ-[1,1,2,2-Tetrakis(η^6-phenyl)-1,2-diphenyldisilane]-divanadium: Long Distance Exchange Interaction Mediated through a >SiPh–SiPh< Unit[1]

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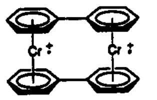
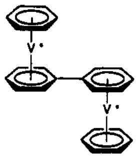
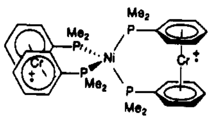
The favorable EPR properties of the bis(η^6-arene)metal(d^5) unit make it an ideal probe in the study of weak magnetic exchange. In this application the radicals bis(benzene)chromium ($1^{\bullet+}$) and bis(benzene)vanadium ($2^\bullet$) are complementary, because the isotropic hyperfine coupling constants $a(^1H, 1^{\bullet+}) = 0.35$ mT and $a(^{51}V, 2^\bullet) = 6.3$ mT cover different time scales.[2]



$3^{2\bullet2+}$



$4^{2\bullet}$

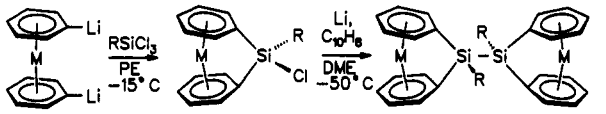


$5^{2\bullet2+}$

In a broadening of our efforts relating to intersandwich compounds of types 3[3] and 4,[4] we began with 5 to explore the effect of spacers on intermetallic communication. In compound 5, the two bis(arene)chromium units are fixed at a distance of 849 pm in an *orthogonal* orientation. The fluid solution EPR spectrum of the cation $5^{2\bullet2+}$ indicates spin localization in that hyperfine coupling to 10 equivalent protons is observed; the biradical character is, however, evident from the prominent $\Delta M_s = 2$ signal.[5] In our quest for suitable spacers which, at a comparable distance, could generate a *parallel* orientation, connectivity across a >SiR–SiR< bridging unit suggested itself in that the necessary precursor should be easily obtainable in analogy to the synthesis of $[(C_6H_5)_2Si(\eta^6\text{-}C_6H_5)_2]M$ (M = V, Cr).[6] In addition, the incorporation of a >SiR–SiR< bridge offers a possibility to study electronic long-distance interactions, mediated over two (formally) saturated Si atoms. Magnetic exchange interactions propagated by multiatom bridges[7] and the particular properties of oligoorganosilyl chains[8] continue to be the object of intense theoretical and experimental work.

Results and Discussion

The synthesis of the title compound 9 as well as the related complex 10 and its Cr analogue 11 is achieved through 1,1′-dilithiation and transformation into the respective sila[1]-metallocyclophanes, 6, 7, 8 followed by reductive coupling with lithium naphthalenide.



M= V	R= Phenyl	6	9
M= V	R= 4n−Propylphenyl	7	10
M= Cr	R= Phenyl	8	11

The extremely low solubility of the dinuclear species 9 and 11 made it necessary to introduce an *n*-propyl substituent at the para position of the noncoordinated phenyl ring. Only in this manner was it possible to obtain single crystals suitable for an X-ray structural analysis. As shown in Figure 1 complex 10 assumes an antiperiplanar configuration with nearly parallel bis(arene)vanadium units of very similar geometry (the following data comprise average values). The V–C distances vary from 216 pm for the "inner" to 226 pm for the "outer" carbon atoms, the rings themselves being practically planar (maximum deviation from the plane is 1.7(4) pm for the "inner" carbon atoms). The distance from the V atom to this plane is 168 pm. The angle of ring-tilt in the sandwich unit is 20.8°, which considerably exceeds that of the prototype $[(C_6H_5)_2Si(\eta^6\text{-}C_6H_5)_2]Cr$ 12 (14.4°[6]). This may be rationalized in terms of the greater atomic radius of V as compared to Cr. Consequently, η^6-arene ring slippage in 10 (7.8 pm) is more pronounced than that in 12 (5 pm). The C-Si-C angle at the sandwich unit is contracted to 94.5°. The Si–Si distance equals 237 pm, which is only slightly elongated in comparison to a normal Si–Si single bond. The V⋯V separation amounts to 760 pm. The molecule possesses no crystallographic symmetry, but displays the pseudosymmetry C_{2h} $(2/m)$. Apart from ordering defects in one of the *n*-propyl groups, the main deviation lies in a torsion along the Si–Si axis. This leads to dihedral angles of 15° between the phenyl rings at C11, C51 and C21, C41, respectively, which in ideal C_{2h} symmetry would be parallel.

The same structure also exists in liquid solution since the ^1H NMR spectrum requires the presence of a mirror plane which contains the Si–Si bond and both Si⋯V vectors. From the two possibilities concordant with this condition, the synperiplanar conformation may be excluded because of the Pitzer strain present

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(1) Metal π Complexes of Benzene Derivatives. 42. Part 41: Elschenbroich, Ch.; Hoppe, S,; Metz, B. *Chem. Ber.* **1993**, *126*, 399.

(2) In the context of magnetic exchange the terms "slow", "fast", and "time scale" are used in a physically descriptive sense.

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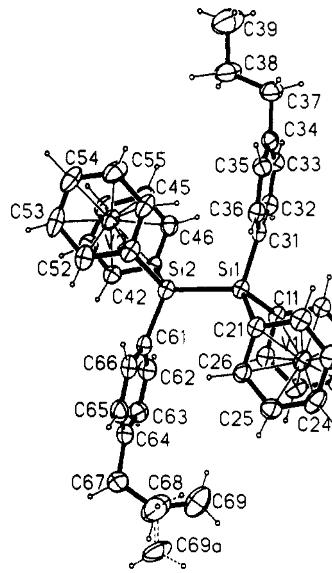

Figure 1. XP drawing[13] of a molecule of **10** in the crystal. Thermal ellipsoids at the 50% probability level, H atoms are given with arbitrary radius and an alternative position of the disordered *n*-propyl group is shown with dotted lines. Selected bond lengths (Å) and angles (deg): V1–C11 = 2.159(4), V1–C12 = 2.168(4), V1–C13 = 2.209(5), V1–C14 = 2.233(4), V1–C15 = 2.210(4), V1–C16 = 2.163(4), V1–C21 = 2.159(4), V1–C22 = 2.181(4), V1–C23 = 2.234(4), V1–C24 = 2.234(4), V1–C25 = 2.213(5), V1–C26 = 2.169(4), V2–C41 = 2.160(4), V2–C42 = 2.175(4), V2–C43 = 2.228(4), V2–C44 = 2.241(4), V2–C45 = 2.223(5), V2–C46 = 2.176(4), V2–C51 = 2.167(4), V2–C52 = 2.171(4), V2–C53 = 2.224(5), V2–C54 = 2.257(5), V2–C55 = 2.226(5), V2–C56 = 2.182(5), Si1–Si2 = 2.372(2), Si1–C11 = 1.883(4), Si1–C21 = 1.898(4), Si1–C31 = 1.860(4), Si2–C41 = 1.900(4), Si2–C51 = 1.887(4), Si2–C61 = 1.864(4) V1···V2 = 7.603(3), V1···Si1 = 2.932(2), V2···Si2 = 2.936(2), average C–C(sandwich) = 1.419, average C–C(free phenyl rings)···1.393; C11–Si1–C21 = 94.7(2), C11–Si1–C31 = 110.9(2), C21–Si1–C31 = 110.7(2), Si1–Si2-C41 = 116.8(1), Si1–Si2–C51 = 114.2(2), Si1–Si2–C61 = 110.3(1), C41–Si2–C51 = 94.3(2), C41–Si2–C61 = 108.8(2), C51–Si2–C61 = 111.5(2). Average (over *n* = 1, 2, 4, 5) of the angles C–C*nm*–C in the sandwich units: *m* = 1, 116.5°, *m* = 2, 6, 121.4°; *m* = 3, 5, 120.6°; *m* = 4, 119.6°.

in an eclipsed structure. In agreement with this structure, the η^6-C_6H_5 ring protons in the diamagnetic chromium complex **11** are fully anisochronic. A first-order ^{1}H NMR spectrum is observed, whereby the significant shift differences are attributable to the various proton positions relative to the anisotropic shielding cone on the noncoordinated benzene ring.

The EPR spectrum of binuclear **10** in liquid solution (Figure 2) features 15 hyperfine components which are separated by approximately half the coupling constant a(^{51}V) of mononuclear **6**. The qualitative conclusion $J \gg |a(^{51}\mathrm{V})|$ (J = electron exchange coupling constant) can be made more precise by spectral simulation[9] which yields the parameter $J = -0.48 \pm 0.02$ cm^{-1}, a value to be compared with $a(^{51}\mathrm{V}) = 0.63 \times 10^{-2}$ cm^{-1} (i.e. 5.63 mT) pertaining to the mononuclear unit **6**. The complicated EPR spectrum of **10** in rigid solution, which is governed by the anisotropies of the *g* and hyperfine tensors and zero-field splitting

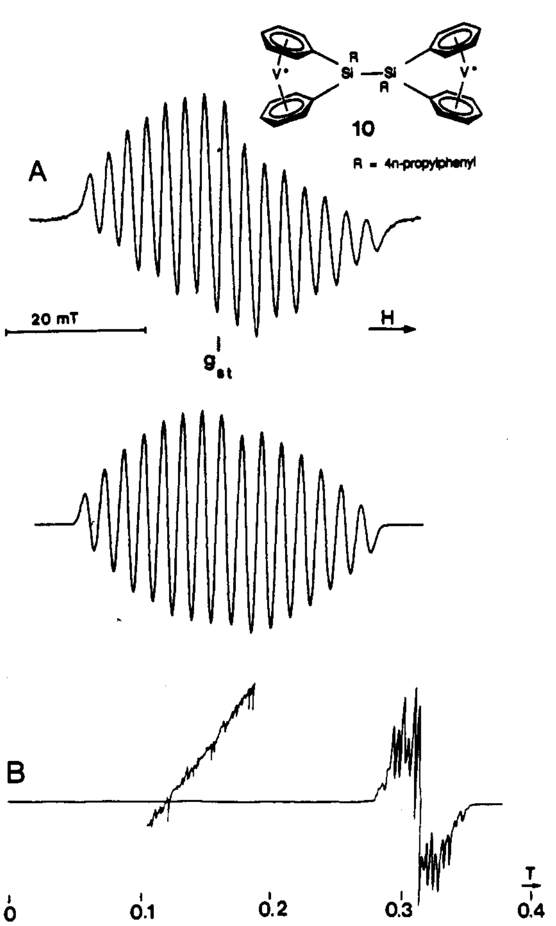

Figure 2. EPR spectra (X-band) of **10** in toluene at various temperatures: (A) at 300 K upper trace experimental, lower trace simulated[7] (parameters, see text); (B) at 140 K, half-field range amplified 10^3-fold, g_{st} = 2.0027.

has not yet yielded to analysis; a half-field signal ($\Delta M_s = 2$, $g \approx 4$) is not observed since the inherently low intensity[10] is spread over 15 hyperfine components. Furthermore, the macroscopic magnetic behavior of **10** also fails to reveal magnetic exchange coupling which is not surprising in view of the small magnitude of *J*. At best, the negative Curie–Weiss constant Θ, obtained via extrapolation of the magnetic susceptibility curve, suggests weak antiferromagnetism ($J < 0$). This example demonstrates how EPR spectroscopy complements the bulk measurement of magnetic susceptibility in the study of very weak *intramolecular* electron-electron spin-spin interactions ($J < 1$ cm^{-1}).[7e,f]

At first sight exchange coupling over a distance of 760 pm is surprising considering the facts that in the mononuclear unit the unpaired electron is essentially located in a virtually nonbonding V(3d$_{z^2}$) orbital,[11] there is no V–Si bond and the spacer consists of two (formally) saturated silicon atoms which are connected "in series".[12] Apparently, the special position of the Si atoms relative to the tori of the V(3d$_{z^2}$) orbitals even at a distance of 293 pm generates sufficient overlapp of the type 3d$_{z^2}$(V)σ^*(Si–Si)3d$_{z^2}$(V) to account for the observed exchange coupling (compare: the sum of the covalent radii of V and Si amounts to 250 pm). This pathway is aided by the decreased magnitude of

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(9) The EPR spectrum was simulated using the general spin Hamiltonian program MAGRES: Keijzers, C. P.; Reijerse, E. J.; Stam, P.; Dumont, M. F. and Gribnau, M. C. M. *J. Chem. Soc., Faraday Trans. 1* **1987**, *83*, 3613. The spin Hamiltonian consisted of two electron spins $S = {}^1/_2$ and two vanadium nuclear spins $\mathrm{I} = {}^7/_2$ leading to a total multiplicity of 256 energy levels. Each of the two electron spins was coupled to a different vanadium nucleus ($A_{iso} = -52.9 \times 10^{-4}$ cm^{-1}). The two electron spins were coupled to each other through an exchange interaction ($J = -.048 \pm 0.02$ cm^{-1}). Optimal agreement between the simulated and the experimental traces is achieved, if $a(^{51}\mathrm{V})$ and *J* are provided with opposite signs. The choice of signs, given above, concords with the magnetic susceptibility curve for **10** (negative Θ_c and the conclusion, that the central metal isotropic hyperfine coupling constant in (η^6-arene)$_2$M(d^5) complexes bears a positive sign: Elschenbroich, Ch.; Möckel, R.; Zenneck, U.; Clack, D. W. *Ber. Bunsen-Ges. Phys. Chem.* **1979**, *83*, 1008.

(10) Eaton, S. S.; More, K. M.; Sawant, B. M.; Eaton, G. R. *J. Am. Chem. Soc.* **1983**, *105*, 6560.

(11) Concerning the electronic structure of (η^6-C_6H_6)$_2$V see: (a) Cloke, F. G. N.; Dix, A. N.; Green, J. C.; Perutz, R. N.; Seddon, E. A. *Organometallics* **1983**, *2*, 1150 (b) Elschenbroich, Ch.; Bilger, E.; Metz, B. *Organometallics* **1991**, *10*, 2823.

(12) For the spin–spin interaction Cr^{II}–Ni^{II} in intersandwich compounds which feature chromocene and nickelocen units separated by two *parallel*–SiR$_2$– spacers, the parameter $J = -1.95$ cm^{-1} has been derived: Bergerat, P.; Blümel, J.; Fritz, M.; Hiermeier, J.; Hudeczek, P.; Kahn, O.: Köhler, F. H. *Angew. Chem., Int. Ed. Engl.* **1982**, *31*, 1258.

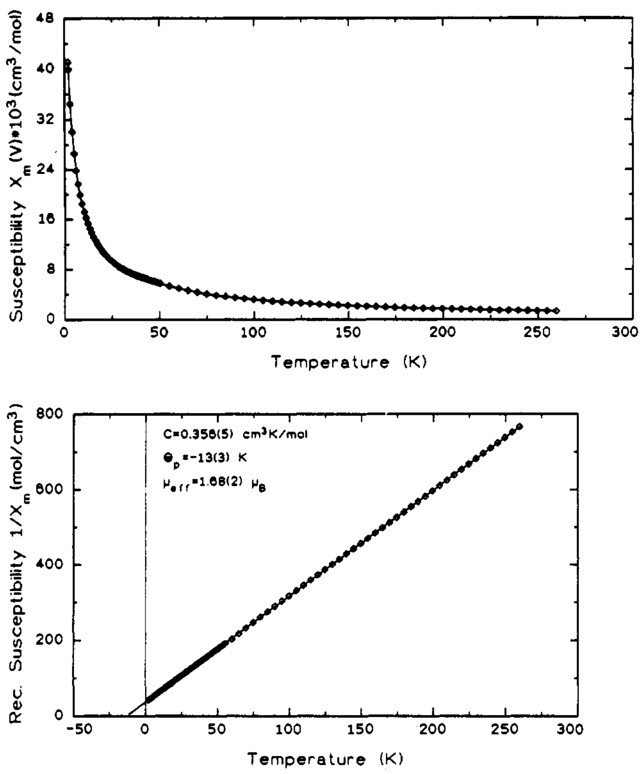

Figure 3. Temperature dependence of the magnetic susceptibility χ and of χ^{-1} for **10**.

the HOMO–LUMO gap for oligoorganosilanes, compared to alkanes.[13] The well-known ability of cyclic organosilanes to form electron delocalized anion and cation radicals is also pertinent in this context.[13]

Unequivocal experimental evidence for the proposed direct exchange route V···Si–Si···V was expected from a comparative EPR study of the isoelectronic biradical dication $\mathbf{11}^{2\bullet 2+}$ since in the latter case, due to the contracted nature of the cation centered $Cr^+(3d_{z^2})$ orbital, exchange coupling should be diminished. Unfortunately, as judged from cyclic voltammetry, the dication $\mathbf{11}^{2\bullet 2+}$ is inaccessible, oxidation of **11** being chemically irreversible even at low temperature. This also applies to the oxidation of **10** as well as to electrochemical reduction of **10** and **11**. While oxidation leads to opening of the (η^6-arene)C–Si linkage[6] reduction probably effects cleavage of the central Si–Si bond. However, in the biradical dication $\mathbf{5}^{2\bullet 2+}$ which is readily prepared,[5] two Cr^+ magnetic orbitals are located at a distance comparable to that in $\mathbf{11}^{2\bullet 2+}$. In the hyperfine splitting pattern of $\mathbf{5}^{2\bullet 2+}$ there is, vide supra, no indication of an exchange interaction. This supports the premise that an overlap of the $V(3d_{z^2})$ orbital with the bridging Si atom is essential for the exchange interaction in $\mathbf{9}^{2\bullet}$.

Experimental Section

All procedures including the preparation of spectroscopic samples were performed under an atmosphere of purified nitrogen using conventional Schlenk techniques. The solvents were thoroughly dried and freed from oxygen. The spectra were recorded employing the instruments Varian CH7A (EI MS) and 711 (HR MS and FD MS), Bruker AC-300 (^{1}H, ^{13}C NMR), Varian EE 12 (X-band EPR), and AMEL, 552, 556, and 563 (cyclic voltammetry). Voltammograms were obtained from solutions in DME/Bu^4NClO_4, using a glassy carbon working electrode, a platinum counter electrode and a SCE reference electrode.

(4-*n*-prop-C_6H_4)$SiCl_3$. To the Grignard reagent, prepared from 1-bromo-(4-*n*-propyl)benzene (25 g, 126 mmol) and magnesium turnings (3.11 g, 128 mmol) in diethyl ether (60 mL) was added dropwise tetrachlorosilane (21.41 g, 126 mmol). Usual workup and vacuum distillation at 50–60 °C/5 × 10^{-4} mbar afforded 13.11 g (39%) of (4-*n*-prop-C_6H_4)$SiCl_3$ as colorless liquid.

Mass spectrum (70 ev) *m/z*: 522 ($[M]^+$, 76), 223 ($[M^+ - C_2H_5]^+$, 96), 209 ($[C_6H_4SiCl_3]^+$, 51), 133 ($[SiCl_3]^+$, 52), 119 ($[C_6H_4 - prop]^+$, 94), 91 ($[C_6H_4CH_3]^+$, 94). ^{1}H NMR ($CDCl_3$, 25 °C): δ 0.99 (t, CH_3), 1.70 (sextet, CH_2), 2.67 (t, CH_2), 7.32 (dd, 2 H_{ar}), 7.73 (dd, 2 H_{ar}). ^{13}C NMR ($CDCl_3$, 25 °C): δ 14.0 (CH_3), 24.5 (CH_2), 38.3 (CH_2), 129 (CH), 133.3 (CH).

[(η^6-C_6H_5)$_2$Si(Cl)(C_6H_5)]V (6). Bis(benzene)vanadium (**2**) (1.57 g, 7.6 mmol) in cyclohexane (90 mL) is metalated at 85 °C during 2 h with *n*-butyllithium (18.2 mmol, 1.6 molar solution in hexane) and *N,N,N',N'*-tetramethylethylenediamine (TMEDA 2.8 mL, 18.2 mmol). After the mixture is cooled to ambient temperature and the cyclohexane phase is decanted, petroleum ether (40/60, 100 mL) is added and at –15 °C phenyltrichlorosilane (1.77 g, 8.3 mmol) is introduced dropwise during 1 h. The reaction mixture is kept at room temperature for 12 h and filtered over silylated silica gel. Cooling to –20 °C yields **6** (0.85 g, 33%) as a greenish brown material which is purified by recrystallization from toluene. Mass spectrum (70 eV): *m/z* 345 ($[M]^+$, 100), 294 ($[M - V]^+$, 56), 217 ($[Ph_2SiCl]^+$, 63), 51 ($[V]^+$, 20). EPR (25 °C, toluene): $\langle g \rangle = 1.984$, $a(^{51}V) = 5.66$ mT. Anal. Calcd for $C_{18}H_{15}ClSiV$ (345.8): C, 62.54; H, 4.34. Found: C, 61.11; H, 3.88.[14]

[(η^6-C_6H_5)$_2$Si(Cl)(4-*n*-prop-C_6H_4)]V (7). Preparation in analogy to that of **6** from **2** (1.51 g, 7.3 mmol) in cyclohexane (90 ml), *n*-BuLi (17.0 mmol), 1.6 M solution in hexane), TMEDA (2.6 ml, 17.0 mmol) and (4-*n*-propylphenyl)trichlorosilane in 38% yield as a dark brown microcrystalline powder. Mass spectrum (70 eV) *m/z*: 387 ($[M^+]$, 100), 336 ($[M - V]^+$, 35), 259 ($[Ph_3Si]^+$, 41), 217 ($[Ph_2SiCl]^+$, 17), 196 ($[Ph_2SiCH_2]^+$, 36), MS (high resolution): calcd for $C_{21}H_{21}ClSiV$, 387.05411; found, 387.05569. EPR (25 °C, toluene): $\langle g \rangle = 1.985$, $a(^{51}V) = 5.63$ mT.

[(η^6-C_6H_5)$_2$Si(Cl)(C_6H_5)]Cr (8). Preparation analogous to that of **6** from bis(benzene)chromium (**1**) (2.66 g, 12.8 mmol) in cyclohexane (150 mL), *n*-BuLi (31 mmol, 1.6 M solution in cyclohexane), TMEDA (4.7 mL, 30.7 mmol), and phenyltrichlorosilane (3.0 g, 14.1 mmol) gives **8** in 56% yield as yellow granules. Mass spectrum (70 eV): *m/z* 346 ($[M]^+$, 55), 294 ($[M - Cr]^+$, 60), 259 ($[Ph_3Si]^+$, 100), 217 ($[Ph_2SiCl]^+$, 73), 52 ($[Cr]^+$, 39). ^{1}H NMR (C_6D_6, 25 °C): δ 7.88 (d, H-32, 36), 7.16 – 7.00 (m, H-33, 34, 35), 4.60 (t, H-14, 24), 4.42 (t, H-13, 23), 4.30 (d, H-12, 22), 4.18 (t, H-15, 25), 3.71 (d, H-16, 26). ^{13}C NMR (C_6H_6, 25 °C): δ 135.6 (C-31), 134.5 (C-32, 36), 132.0 (C-34), 129.1 C-33, 35), 83.7, 82.5 (C-13, 23, 15, 25), 80.6 (C-14, 24), 76.2, 75.2 (C-12, 22, 16, 26), 39.7 (C-11, 21). Anal. Calcd for $C_{18}H_{15}ClCrSi$ (346.9): C, 62.36; H, 4.33. Found: C, 62.52; H 5.06.

μ-[(η^6-C_6H_5)$_2$(C_6H_5)SiSi(C_6H_5)(η^6-C_6H_5)$_2$]V_2 (9). To a cooled solution (–50 °C) of **6** (0.37 g, 1.07 mmol) in DME (50 mL) were added dropwise 10 mL of a solution of $Li^+C_{10}H_8^-$ (1.28 mmol) during 1 h. After the mixture was held for 2 h at –50 °C and warmed to ambient temperature, DME and naphthalene were removed in vacuo and the crude product was extracted with 100 mL of warm toluene. Filtration over silylated silica gel, reduction of volume, and cooling to –25 °C provides 50 mg (16% yield) of **9** as a brown powder. Mass spectrum (70 eV): *m/z* 620 ($[M]^+$, 23), 569 ($[M - V]^+$, 64), 518 ($[Ph_6Si_2]^+$, 22), 259 ($[Ph_3Si]^+$, 100). Anal. Calcd for $C_{36}H_{30}Si_2V_2$ (620.7): C, 69.69; H, 4.84. Found: C, 64.82; H, 4.95.[14]

μ-[(η^6-C_6H_5)$_2$(4-*n*-prop-C_6H_4)SiSi(4-*n*-prop-C_6H_4)(η^6-C_6H_5)$_2$]V_2 (10). Preparation in analogy to that of **9** from **7** (1.09 g, 2.8 mmol) in DME (50 mL) and $Li^+C_{10}H_8^-$ (3.4 mmol) in DME (100 mL) and crystallization from toluene/petroleum ether (1:4) led to 130 mg (6.6% of **10** as violet crystals. Mass spectrum (70 eV): *m/z* 704 ($[M]^+$, 8), 6.53 ($[M - V]^+$, 47), 602 ($[M - 2V]^+$, 17), 343 ($[Ph_2Si(C_6H_4 - prop)_2]^+$, 36), 301 ($[Ph_2Si(C_6H_4 - prop)]^+$, 100). MS (high resolution): calcd for $C_{42}H_{42}Si_2V_2$, 704.17038; found, 704.18908. Anal. Calcd: C, 71.57; h, 6.01. Found: C, 61.29; H 5.95.[14]

μ-[(η^6-C_6H_5)$_2$(C_6H_5)Si-Si(C_6H_5)(η^6-C_6H_5)$_2$]Cr_2 (11) was prepared analogously to **9** from **8** (0.59 g, 1.7 mmol) in DME (50 mL) and $Li^+C_{10}H_8^-$ (1.9 mmol) in DME (12 mL). Evaporation to dryness, extraction with toluene (100 mL) at 50 °C, and concentration yields 100 mg (19%) of **11** as purple red microcrystalline powder. Mass spectrum (70 eV): *m/z*

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(14) Notoriously low carbon values in the microanalyses of Si- and V-containing compounds in our experience is a common phenomenon.

Table I. Experimental Data for the Crystal Structure Determination of **10**

formula; fw	$C_{42}H_{42}Si_2V_2$; 704.8	
abs (μ)	6.5 cm^{-1}, no corr	
space group	P1, Z = 2	
lattice consts (Mo Kα)	a = 11.834(2) Å	α = 69.30(2)°
	b = 12.087(3) Å	β = 75.22(2)°
	c = 13.085(4) Å	γ = 81.99(2)°
temperature	198 K	
density (d_c)	1.385 g cm^{-3}	
scan type	ω-scan	
scan width	$(0.8 + 0.35 \tan \theta)°$ and 25% on the left- and right-hand sides of a reflcn for bkgd determ	
measuring range	θ = 2–22.5°; $+h, \pm k, \pm l$	
no. of reflcns	4672, 4373 unique, 3577 > $3\sigma(F_o)$	
atomic scattering factors	for neutral atoms[16a]	
residuals	R = 0.042, R_w = 0.033 ($w = 1/\sigma 2(F_o)$)	
goodness of fit	2.37	
max param shift	0.001 × esd	
$\Delta\rho_{mas}$; $\Delta\rho_{min}$	+0.41; –0.29 e/Å^3	

622 ($[M]^+$, 3), 570 ($[M - Cr]^+$, 25), 518 ($[Ph_6Si_2]^+$, 17), 259 ($[Ph_3Si]^+$, 100), 52 ($[Cr]^+$, 20). ^1H NMR (C_6D_6, 25 °C): δ 8.11 (d, H-32, 36, 62, 66), 7.34 (t, H-33, 35, 63, 65), 7.25 (t, H-34, 64), 4.69 (d, H-16, 26, 46, 56), 4.62 (t, H-14, 24, 44, 54), 4.55 (t, H-15, 25, 45, 55), 4.45 (t, H-13, 23, 43, 53), 4.16 (d, H-12, 22, 42, 52); ^{13}C NMR (C_6H_6, 25 °C): δ 135.6 (C-32, 36, 62, 66), 133.8 (C-31, 61), 130.2 (C-34, 64), 129.1 (C-33, 35, 53, 65), 83.7, 79.8 (C-13, 23, 15, 25, 43, 53, 45, 55), 78.1 (C-14, 24, 44, 54), 77.0, 74.8 (C-12, 22, 16, 26, 42, 52, 46, 56), 35.2 (C-11, 21, 41, 51). Anal. Calcd for $C_{36}H_{30}Cr_2Si_2$ (622.8): C, 69.45; H, 4.82. Found: C, 68.63; H, 4.78.

X-ray Structure Determination. A violet single crystal of **10** ($C_{42}H_{42}Si_2V_2$) was investigated at 198 K on an Enraf-Nonius CAD4 diffractometer using graphite-monochromated Mo Kα radiation. The triclinic lattice constants were refined from 25 reflections (θ > 15°). They are collected, together with other crystallographic data, in Table I. The structure was solved by direct methods and difference Fourier maps and was refined by full-matrix least-squares methods.[15] Although all H atoms could be located, they were kept "riding" on idealized positions (C–H 0.96 Å) for a better reflections/parameter ratio (8.2). For all non-H atoms anisotropic temperature factors were used. One of the *n*-propyl groups proved to be disordered (*gauche* and *anti* forms). The refinement converged to R = 0.042 and R_w = 0.033. Supplemental Tables S1 and S2 give the complete bond lengths and angles.

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Supplementary Material Available: Tables of bond lengths (Table S1), bond angles (Table S2), best planes (Table S3), and atomic fractional coordinates and anisotropic temperature factors (Table S4) (8 pages). Ordering information is given on any current masthead page.

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(15) Sheldrick, G. M. SHELXTL-Plus. Release 4.0 for Siemens R3 Crystallographic Research Systems. Siemens Analytical X-RAY Instruments, Inc., Madison, WI, USA, 1989. Cromer, D. T.; Waber, J. T. *International Tables for X-ray Crystallography*; Vol. IV, Kynoch Press: Birmingham, England 1974.