

Unexpected Behavior of the Novel Organometallic Thioether $C_5H_5FeC_5H_4C\equiv CSC\equiv CSiMe_3$ toward $Ru_3(CO)_{12}$

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Summary: The new thioether $C_5H_5FeC_5H_4C\equiv CSC\equiv CSiMe_3$ affords the compound $[Ru_3(CO)_9(\mu_3-\eta^2-\eta^4-\eta^3-Me_3SiCC(C_2Fc)SC(Fc)CSC\equiv CSiMe_3)]$, among others, by reaction with $Ru_3(CO)_{12}$.

Recent research interest has been focused on the syntheses of polynuclear compounds containing metal centers which are connected to an unsaturated polycarbon chain, since these materials might show interesting electrical and optical properties.¹ Alkyne ligands of different chain lengths have been used, and since the first report of reaction of $Ru_3(CO)_{12}$ with the alkyne $HC\equiv CC_5H_4FeC_5H_5$ ² other polyacetylenic compounds containing the electroactive ferrocenyl (Fc) substituent have been used.³ In contrast with the amount of information on these types of ligands, the transition-metal chemistry of thioethers of types $RSC\equiv CR'$ and $RC\equiv CSC\equiv CR'$ ($R = R'$ or $R \neq R'$) has been poorly developed, although they may be useful as reagents to generate unsaturated polycarbon chains. The easy rupture of S–C bonds in organosulfur compounds and the possibility of elongating the carbon chain by C–C coupling reactions between acetylide ligands prompted us a few years ago to use bis(alkynyl) thioethers as starting materials in cluster chemistry. Thus, we reported⁴ the synthesis of the trinuclear compounds $[Ru_3(CO)_9(\mu_3-\eta^2-SC\equiv CR)(\mu_3-\eta^2-C\equiv CR)]$ ($R = R' = SiMe_3$; $R = SiMe_3$, $R' = Si^iPr_3$; $R = Si^iPr_3$, $R' = SiMe_3$; $R = Si^iPr_3$, $R' = H$; $R = H$, $R' = Si^iPr_3$) and $Ru_3(CO)_9(\mu_3-S)-$

$(\mu_3-\eta^2-C(SiMe_3)C(C\equiv CSiMe_3))$ as well as the tetranuclear clusters $[Ru_4(CO)_9(\mu-CO)_2(\mu_4-S)(\mu_4-\eta^2-C(R)C(C\equiv CR'))]$ ($R = R' = SiMe_3$; $R = H$, $R' = Si^iPr_3$) using the thioethers $RC\equiv CSC\equiv CR'$ ($R = R' = SiMe_3$; $R = SiMe_3$, $R' = Si^iPr_3$; $R = H$, $R' = Si^iPr_3$). Jeannin et al.⁵ also have studied the reactions of iron and ruthenium carbonyls with thioethers, $EtSC\equiv CR$ ($R = Ph$, Me), isolating compounds of different nuclearity as a result of either rupture of the S–C bond or C–C coupling with or without S–C cleavage. Following our research on the synthesis of cluster complexes bearing sulfur and vinyl and polyynyl ligands, we considered it of interest to prepare the organometallic end-capped thioether $C_5H_5FeC_5H_4C\equiv CSC\equiv CSiMe_3$, in which the ferrocenyl substituent acts as an electron donor, and to evaluate its reactivity with $Ru_3(CO)_{12}$. Also of potential interest was the electronic communication between the Fc group ($Fc = C_5H_5FeC_5H_4$) and the ruthenium atoms.

Synthesis of $C_5H_5FeC_5H_4C\equiv CSC\equiv CSiMe_3$ (**1**) was accomplished by addition of $SnCl_2$ to a mixture of $LiC\equiv CC_5H_4FeC_5H_5$ and $LiC\equiv CSiMe_3$ in diethyl ether, starting at -78 °C with subsequent warming to room temperature, following a modified literature procedure for organic thioethers.⁶ Compound **1** was purified by column chromatography on silica gel, affording an orange solid. ¹H NMR spectroscopy shows two triplets at 4.49 and 4.24 ppm and a singlet at 4.23 ppm, respectively, corresponding to the Fc group as well as a singlet at 0.20 ppm due to the $SiMe_3$ group. The absorptions at 2164 (m) and 2104 (s) cm^{-1} in the IR spectrum are assigned to the stretching frequencies $\nu_{C\equiv C}$. FAB mass spectrometry showed the molecular ion peak at m/z 338. The molecular structure (Figure 1) was determined by single-crystal X-ray diffraction, which revealed, as expected, an angular geometry at the sulfur atom.⁷

The C(2)–S–C(3) angle is similar to those reported for the compounds *trans*-[Ph(Et₃P)₂PtC≡CSC≡CPT-

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(7) Crystal data of **1**: $C_{17}H_{18}SSiFe$, $M_r = 338.31$, monoclinic, $P2_1/c$, $a = 11.4422(16)$ Å, $b = 9.7857(12)$ Å, $c = 15.1962(19)$ Å, $\beta = 108.508(12)^\circ$, $V = 1613.5(4)$ Å³, $Z = 4$, yellow, $T = 180(2)$ K, $\mu = 1.125$ mm^{-1} , 16 051 reflections measured (5341 unique), final refinement converged at $R_w(F^2) = 0.0691$ with 185 parameters.

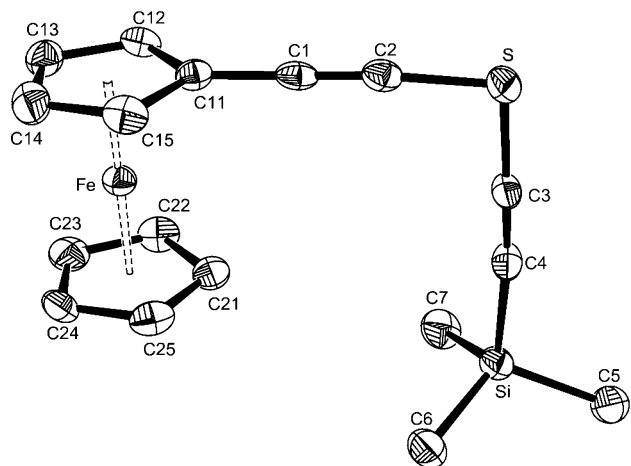


Figure 1. Molecular structure of **1**. Selected bond lengths (Å) and angles (deg): C(1)–C(2) = 1.185(4), C(3)–C(4) = 1.189(4); C(2)–S(1)–C(3) = 99.10(16). Ellipsoids are drawn at the 50% probability level.

(Et₃P)₂Ph] (105.0(3)°) and cis-[Pt(Me₂bipy){C≡CSC≡CSiPr₃}₂] (106.0(3)°).⁸ The ferrocenyl group shows a near-perfect eclipsed geometry with its rings, and the two C–C triple-bond distances are almost identical. Similar features have been observed in related structures of C₅H₅FeC₅H₄(C≡CC₆H₄)₂SMe⁹ and C₅H₅FeC₅H₄C≡C(C₆H₂S)₃C≡CC₅H₄FeC₅H₅.¹⁰ Of the two possible isomers of compound **1**, only the cis isomer was obtained, as determined by the X-ray diffraction data. The compound *trans*-[Ph(Et₃P)₂PtC≡CSC≡CPT(PEt₃)₂-Ph], obtained in the reaction of *trans*-[PtPh(Cl)(PEt₃)₂] and S(C≡CH)₂, is the only example reported of a related compound.⁸ The above reaction affording **1** also yielded a second product, the new symmetric thioether S(C≡CC₅H₄FeC₅H₅)₂ (**2**), in minor amounts. The ¹H NMR spectrum showed a singlet and two triplet resonances corresponding to the Fc group, and the FAB mass spectrum showed the molecular ion peak.¹¹ To compare the reactivity of compound **1** to that observed for known organic thioethers, we carried out its reaction with Ru₃(CO)₁₂ under the same conditions¹² (Scheme 1).

Surprisingly, after heating the reaction mixture in toluene at 65 °C, the new compound [Ru₃(CO)₉(μ₃-η²:η⁴:η³-Me₃SiCC(C₂Fc)SC(Fc)CSC≡CSiMe₃)] (**3**) was obtained after purification by column chromatography (46% yield). This complex is completely different from those reported earlier, from analogous reactions using organic thioethers. The analytical and spectroscopic data failed to identify the compound, but the crystal structure of **3** (Figure 2) was determined,¹³ confirming its structure.

Complex **3** consists of the ruthenium units Ru₂(CO)₆ and Ru(CO)₃ joined to a polycarbon sulfur chain made up of two C₅H₅FeC₅H₄C≡CSC≡CSiMe₃ thioethers. There

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(11) Selected data for **2** are as follows. ¹H NMR (300 MHz, CDCl₃, 22 °C): δ 4.48 (t, 2H, C₅H₄, J = 1.9 Hz), 4.24 (t, 2H, C₅H₄, J = 1.9 Hz), 4.23 (s, 5H, C₅H₅). IR (KBr, cm⁻¹): ν_{C=C} 2182 (sh) and 2157 (m). MS (FAB): *m/z* 450.0 [M⁺]. Anal. Calcd for C₂₄H₁₈Fe₂S: C, 64.03; H, 4.03. Found: C, 63.59; H, 4.11.

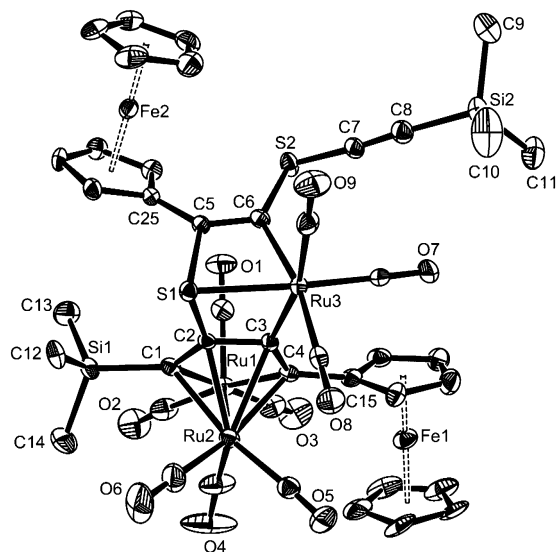


Figure 2. Molecular structure of **3**. Selected bond lengths (Å) and angles (deg): Ru(1)–Ru(2) = 2.7073(5), Ru(3)–S(1) = 2.4414(10), Ru(3)–C(6) = 2.102(4), Ru(3)–C(3) = 2.126(4), C(1)–C(2) = 1.403(5), C(2)–C(3) = 1.436(5), C(3)–C(4) = 1.406(5), C(5)–C(6) = 1.344(5), C(7)–C(8) = 1.208(6), C(2)–S(1) = 1.838(4), C(5)–S(1) = 1.822(4); C(2)–S(1)–C(5) = 92.86(16), C(6)–S(2)–C(7) = 102.73(18). Ellipsoids are drawn at the 50% probability level.

are two remarkable features in the formation of **3**. First, there is the formation of the ruthenole unit by a head-to-head C–C coupling between the SC≡CFC and C≡CSiMe₃ ligands, obtained by cleavage of only one S–C bond in one molecule of C₅H₅FeC₅H₄C≡CSC≡CSiMe₃. Jeannin and al.^{5c} have previously reported the synthesis of the three ferrole type isomers [Fe₂(CO)₆{C(SET)C(R)C(R)C(R)C(R)C(SET)}], [Fe₂(CO)₆{C(SET)C(R)C(SET)C(R)C(SET)C(R)}], and [Fe₂(CO)₆{C(R)C(SET)C(R)C(SET)C(R)C(SET)C(R)}], but without rupture of a S–C bond. In the last case the thioether behaves as an alkyne ligand. The second noticeable feature is the formation of a new S–C bond by coupling of the sulfur atom out of the ruthenole group with the C_β of the acetylenic group, carrying the Fc fragment, in the other thioether molecule. Taking into account that one of the sulfur atoms is also coordinated to the Ru(CO)₃

(12) Reaction of **1** with Ru₃(CO)₁₂: C₅H₅FeC₅H₄C≡CSC≡CSiMe₃ (0.30 g, 0.89 mmol) was added at 65 °C to a solution of Ru₃(CO)₁₂ (0.20 g, 0.31 mmol) in toluene (30 mL). The mixture was stirred for 3 h. The solvent was removed and the solid residue purified by column chromatography on silica gel. Elution with hexane gave **5** in trace amounts. A second band, eluted with hexane–toluene (10:1), gave the orange compound **4** (0.04 g, 0.05 mmol, 15%). Elution with hexane–toluene (5:1) gave the major band of compound **3**, isolated as a red solid (0.18 g, 0.14 mmol, 46%). Single crystals of **3** were obtained from hexane–ether at 4 °C and from hexane at –20 °C for **4**. Spectral and analytical data for **3** are as follows. ¹H NMR (CDCl₃, 300 MHz, 22 °C): δ 4.50 (m, 2H, C₅H₄), 4.37 (m, 2H, C₅H₄), 4.22 (m, 7H, C₅H₄ and C₅H₅), 4.18 (m, 7H, C₅H₄ and C₅H₅), 0.13 (s, 9H, SiMe₃), 0.03 (s, 9H, SiMe₃). IR (hexane, cm⁻¹): ν_{CO} 2097 (m), 2071 (s), 2036 (vs), 2002 (s), 1982 (m). MS (FAB): *m/z* 1232 [M⁺]. Anal. Calcd for C₄₃H₃₆O₉S₂Si₂-Fe₂Ru₃: C, 41.92; H, 2.95. Found: C, 42.41; H, 3.17. Data for **4** are as follows. ¹H NMR (CDCl₃, 300 MHz, 22 °C): δ 4.43 (t, 2H, C₅H₄, J = 1.9 Hz), 4.23 (s, 5H, C₅H₅), 4.21 (t, 2H, C₅H₄, J = 1.9 Hz), 0.62 (s, 9H, SiMe₃). IR (hexane, cm⁻¹): ν_{CO} 2093 (w), 2072 (vs), 2050 (vs), 2016 (vs), 1988 (w). MS (FAB): *m/z* 894.6 [M⁺ + H]. Anal. Calcd for C₂₆H₁₈O₉-SSiFeRu₃: C, 34.94; H, 2.03. Found: C, 35.11; H, 2.21. Data for **5** are as follows. ¹H NMR (CDCl₃, 300 MHz, 22 °C): δ 4.82 (t, 2H, C₅H₄, J = 1.8 Hz), 4.30 (t, 2H, C₅H₄, J = 1.8 Hz), 4.27 (s, 5H, C₅H₅), 0.24 (s, 9H, SiMe₃). IR (hexane, cm⁻¹): ν_{CO} 2084 (m), 2056 (vs), 2014 (vs), 1994 (m). MS (FAB): *m/z* 709.7 [M⁺ + H].

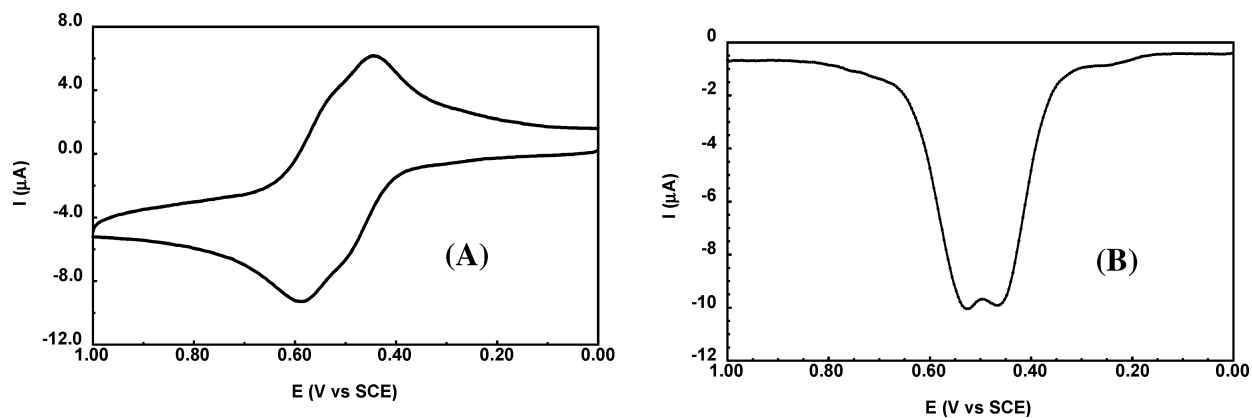
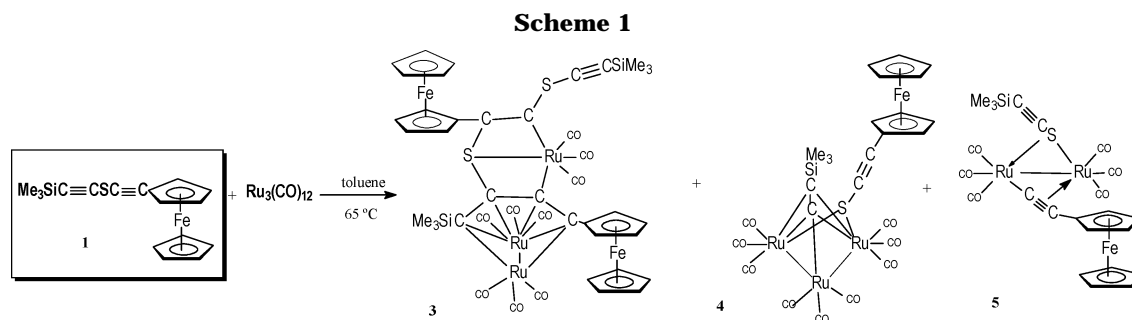


Figure 3. Cyclic voltammogram (scan rate 100 mV s^{-1}) (A) and DPV (B) of **3** in $\text{CH}_2\text{Cl}_2/\text{TBAH}$, at a glassy-carbon-disk electrode.



fragment, the resulting organosulfur ligand acts in the molecule as a 10-electron donor. It is known that the rupture of one S–C bond in the compounds $\text{RSC}\equiv\text{CR}'$ affords SR ($\text{R} = \text{alkyl, alkynyl}$) and $\text{C}\equiv\text{CR}'$ ligands, whose oxidative addition to group 8 metal carbonyls generate $[\text{M}_2(\text{CO})_6(\mu\text{-CCR}')(\mu\text{-SR})]$. Also, there has been reported^{5c} the reaction between $\text{Fe}_2(\text{CO})_9$ and two $\text{EtSC}\equiv\text{CMe}$ molecules to generate $[\text{Fe}_2(\text{CO})_6(\mu\text{-SEt})(\mu\text{-S(Et)C}\{\text{C}(\text{Me})(\text{C}\equiv\text{CMe})\})]$. Its formation implies the rupture of one S–C bond of the first thioether molecule to yield the compound $[\text{Fe}_2(\text{CO})_6(\mu\text{-CCMe})(\mu\text{-SEt})]$, which adds a second uncleaved $\text{EtSC}\equiv\text{CMe}$ molecule by forming a new C–C bond. To our knowledge compound **3** is the first example of the unprecedented conversion of two thioether ligands into a single dithioether moiety.

In addition to **3**, the minor compound **4** and traces of compound **5** were obtained in the reaction.¹² ^1H NMR, IR, and FAB data of **4** are consistent with a cluster of formula $[\text{Ru}_3(\text{CO})_9(\mu, \eta^2\text{-SC}\equiv\text{CFc})(\mu, \eta^2\text{-C}\equiv\text{CSiMe}_3)]$, whose structure was confirmed by single-crystal X-ray diffraction.¹⁴ Full details can be found in the Supporting Information. Cluster **4** shows an open triangular framework containing the $\text{SC}\equiv\text{CFc}$ and $\text{C}\equiv\text{CSiMe}_3$ ligands, which were added oxidatively to the $\text{Ru}_3(\text{CO})_9$ unit. Similar structures have been found in the clusters obtained in the reactions we carried out earlier, between

$\text{Ru}_3(\text{CO})_{12}$ and the symmetric and asymmetric thioethers containing SiMe_3 , Si^iPr_3 , and H substituents.⁴ In the formation of compound **4**, the thioether $\text{C}_5\text{H}_5\text{FeC}_5\text{H}_4\text{C}\equiv\text{CSC}\equiv\text{CSiMe}_3$ behaves in the same way as for other alkynes. In fact, Koridze and co-workers² have reported the cluster $[\text{Ru}_3(\text{CO})_n(\mu\text{-H})(\mu\text{-CCFc})]$ ($n = 8, 9$) obtained in the reaction between $\text{Ru}_3(\text{CO})_{12}$ and $\text{HC}\equiv\text{CFc}$ in refluxing hexane, but a complex with a closed-triangular geometry is formed instead, according to the donor ability of these ligands. The oily compound $[\text{Ru}_2(\text{CO})_6(\mu\text{-SC}\equiv\text{CSiMe}_3)(\mu\text{-C}\equiv\text{CFc})]$ (**5**) was characterized¹² by analytical and spectroscopic data in comparison to those for related complexes.^{4a} We have observed that the amount of **3** and **4** obtained in this reaction is strongly dependent on the temperature. Thus, at 50°C compound **4** was obtained as the main complex.

Since the cluster compounds behave as electron reservoirs and the ferrocenyl group has been used as an electron donor, we considered it of interest to study the electrochemical properties of compounds **1**, **3**, and **4**.¹⁵ The cyclic voltammogram (CV) of compound **1** shows one reversible redox wave, with a formal potential value of 0.63 V vs SCE , which is clearly assigned to the one-electron oxidation of the ferrocenyl unit. This potential value is considerably more positive than the corresponding process for $(\eta^5\text{-C}_5\text{H}_5)_2\text{Fe}$ (0.45 V in CH_2Cl_2). This is due to the electron-withdrawing effect of the alkyne substituent when bound directly to the cyclopentadienyl ring, making the oxidation of the ferrocene

(13) Crystal data of **3**: $\text{C}_{43}\text{H}_{36}\text{O}_9\text{S}_2\text{Si}_2\text{Fe}_2\text{Ru}_3$, $M_r = 1231.93$, triclinic, $P1$, $a = 11.8715(9) \text{ \AA}$, $b = 12.5020(8) \text{ \AA}$, $c = 18.2701(14) \text{ \AA}$, $\alpha = 86.887(6)^\circ$, $\beta = 83.710(6)^\circ$, $\gamma = 66.344(6)^\circ$, $V = 2468.6(3) \text{ \AA}^3$, $Z = 2$, $T = 180(2) \text{ K}$, $\mu = 1.652 \text{ mm}^{-1}$, 13 925 reflections measured (7017 unique), final refinement converged at $R_w(F^2) = 0.0832$ with 601 parameters.

(14) Selected bond lengths (\AA) and angles (deg) for compound **4**: $\text{Ru}(1)\text{--Ru}(3) = 2.830(2)$, $\text{Ru}(1)\text{--Ru}(2) = 2.8333(19)$, $\text{Ru}(3)\text{--S}(1) = 2.445(4)$, $\text{Ru}(2)\text{--S}(1) = 2.431(4)$, $\text{C}(1)\text{--C}(2) = 1.33(2)$, $\text{C}(3)\text{--C}(4) = 1.22(3)$, $\text{Ru}(1)\text{--C}(1) = 1.933(18)$, $\text{Ru}(2)\text{--C}(1) = 2.240(17)$, $\text{Ru}(3)\text{--C}(1) = 2.284(16)$, $\text{Ru}(2)\text{--C}(2) = 2.371(16)$, $\text{Ru}(3)\text{--C}(2) = 2.323(17)$; $\text{Ru}(3)\text{--Ru}(1)\text{--Ru}(2) = 72.49(5)$, $\text{S}(1)\text{--C}(3)\text{--C}(4) = 175.3(17)$.

(15) Electrochemical measurements: cyclic voltammogram (CV) and differential pulse voltammogram (DPV) experiments were performed on a BAS CV-50W potentiostat, using a conventional three-electrode cell (glassy carbon, Pt, SCE) and operating under an atmosphere of prepurified nitrogen. Solutions typically were 1.0 mM in the redox-active species, and the supporting electrolyte (used in a concentration of 0.10 M) was tetra-*n*-butylammonium hexafluorophosphate (TBAH, Aldrich).

unit more difficult than that of the unsubstituted metallocene. In cluster **4** also only a one-electron redox process has been found, but the value of 0.57 V indicates that formation of the complex makes the oxidation easier.

The CV of compound **3**, which contains two ferrocenyl units, exhibits two reversible one-electron oxidation processes (Figure 3A). Likewise, differential pulse voltammetric (DPV) measurements for **3** show two very close oxidation peaks at $E^\circ = 0.55$ and 0.49 V vs SCE (Figure 3B). The difference in the redox potentials ($\Delta E^\circ = 0.06$ V) might be due to simple inequivalence of the two ferrocenyl groups and perhaps also to the existence of interactions between the two ferrocenyl units.^{3a,16}

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Supporting Information Available: Tables giving X-ray crystallographic data for **1**, **3**, and **4**; these data are also available as CIF files. This material is available free of charge via the Internet at <http://pubs.acs.org>. This material has also been deposited with the Cambridge Crystallographic Data Centre (reference numbers CCDC 245018, 245019, and 245020).

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