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Impact of Transition Metal Substituents on Polysilane Properties: Iron *versus* Ruthenium[#]

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Summary. The previously unknown ruthenio disilanes Rp–Si_2Me_4–C_6H_4X ($Rp = \eta^5$-$C_5H_5Ru(CO)_2$; $X = H$, Br, –CHO, CH=C(CN)$_2$) were synthesized from $ClSi_2Me_4C_6H_4X$ ($X = H$, Br) and Rp^- using conventional chemical methods. Trends in the UV/Vis absorption spectra indicate strong electronic coupling within the Rp–Si–Si–C_{aryl} fragment and, therefore, closely resemble the ones observed for the corresponding iron complexes. The four compounds however, were shown to be less sensitive towards UV irradiation. The crystal structure of Rp–Si_2Me_4–C_6H_4CH=C(CN)$_2$ was determined by X-ray diffraction and exhibits an *all-trans*-array of the Ru–Si–Si–C_{aryl} moiety, what is a basic requirement for optimal through-bond interaction.

Keywords. UV/Vis spectroscopy; Silicon compounds; Charge transfer; Ru–Si-bond.

Introduction

The chemistry of transition metal silicon compounds continues to generate considerable interest [1]. Special attention has been given to the influence of transition metal fragments on the properties, bonding, and reactivity of adjacent silicon atoms [2]. We have recently found strong evidence for considerable ground state electronic coupling within the Fe–Si–Si–C_{aryl} fragment in the donor/acceptor substituted disilane Fp–Si_2Me_4–C_6H_4CH=C(CN)$_2$ ($Fp = \eta^5$-$C_5H_5Fe(CO)_2$) [3]. Further studies of this class of compounds, however, are strongly impeded by the rapid photochemical degradation of the polysilane chain in the presence

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[#] Dedicated to Prof. *Ulrich Schubert* on the occasion of his 60th birthday of the *Fp* substituent (Eq. (1)), what has been investigated in detail by *Pannell et al.* [4].

$$FpSiMe_2SiPh_3 \xrightarrow{h\nu} \underset{8\%}{FpSiMe_2Ph} + \underset{85\%}{FpSiMePh_2} + \underset{7\%}{FpSiPh_3} \qquad (1)$$

The same authors, however, report, that the related η^5-$C_5H_5Ru(CO)_2$ (*Rp*) complexes are not photolabile with respect to deoligomerization into monosilyl metal derivatives [5]. Dipolar ruthenium oligosilanyl complexes, as a consequence, might be much better suited for detailed studies of properties like photoluminescence or second-order nonlinear optical activity than their iron counterparts.

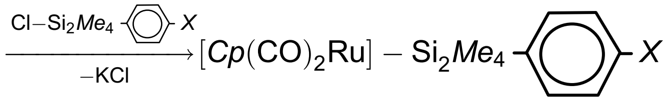
Herein, we describe the synthesis of the previously unknown ruthenium complexes Rp–Si_2Me_4–C_6H_4X (X = H, Br, CHO, CH=C(CN)$_2$). Trends in the spectroscopic parameters (UV/Vis, multinuclear NMR) and structural features are reported, compared to the properties of the corresponding iron compounds, and analyzed in terms of electronic interactions within the *Rp*–Si–Si–C_{aryl} bond system.

Results and Discussion

Synthesis

The ruthenium disilane complexes Rp–Si_2Me_4–C_6H_5 (**1**) and Rp–Si_2Me_4–C_6H_4Br (**2**) were made from $ClSiMe_2SiMe_2PhX$ (X = H, Br) and Rp^- as outlined in Eq. (2).

$$[Cp(CO)_2Ru]_2 \xrightarrow[-H_2,-Et_3B]{K[BEt_3H]} [Cp(CO)_2Ru]^-K^+$$

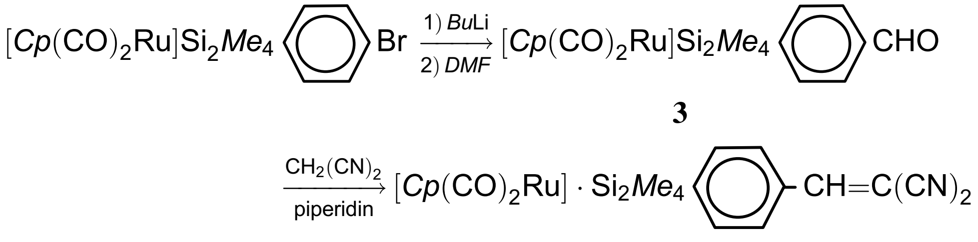


1: X = H, **2**: X = Br

(2)

Because the reductive cleavage of Rp_2 with Na/Hg is known to give only low yields of the Rp^- anion [6], we followed the synthesis route published by *Pannell et al.* [5] for the synthesis of Ru complexes containing Si–Si bonds. In this paper the reduction of the *Rp* dimer is accomplished with potassium selectride and moderate yields of the *Rp*-silyl complexes were obtained. In the present study potassium superhydride KEt_3BH was used instead of potassium selectride in order to facilitate the removal of the formed borane. Additionally, the yield and the purity of the desired Ru-silyl complexes turned out to be significantly increased, when the Rp^- anion was isolated prior to its addition to the chlorosilane.

Starting from the bromophenyl derivative **2** the dipolar complex **4** bearing the dicyanovinyl acceptor group is easily accessible using a reaction sequence already applied successfully for the preparation of the corresponding iron derivative *Fp*–Si_2*Me*$_4$*Ph*CH=C(CN)$_2$ (**6**) (Eq. (3)). Moderately air-sensitive orange crystals of **4** were obtained in an overall yield of 45%.



3 (3)

4

All products were characterized by standard spectroscopic techniques (^{29}Si, ^{13}C, and ^{1}H NMR, IR, HRMS). The results (see Exp.) agree well with the proposed structures in all cases. ^{29}Si NMR chemical shift values are observed typical for compounds containing the *Rp*–Si–Si moiety with a low field shift of the resonance line of Si_α (Si atom directly bonded to the metal center) by ~20 ppm and ~5 ppm for Si_β as compared to *Ph*Si*Me*$_2$Si*Me*$_3$ [7]. Only weak electronic effects of the substituents attached to the aromatic ring are apparent in the ^{29}Si NMR spectra. Thus, compound **4** containing the electron withdrawing –CH=C(CN)$_2$ group, for instance, exhibits only a slight downfield shift for the Si_α resonance line of 0.6 ppm relative to **1**.

X-Ray Structure of 4

A drawing of the molecular structure of **4** with atom labeling is depicted in Fig. 1 together with selected bond lengths and angles. Compound **4** crystallizes in the

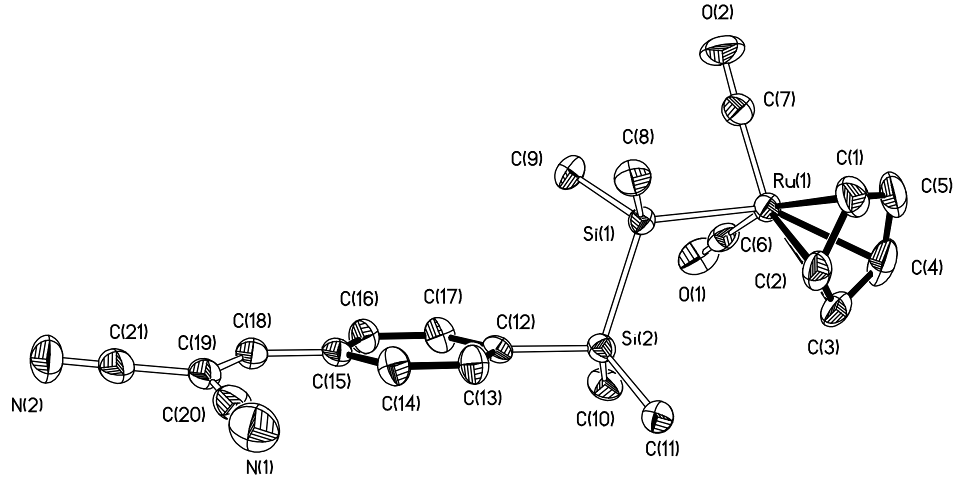


Fig. 1. The molecular structure and numbering of **4**; selected bond lengths [pm]: Si(1)–Si(2) 235.42(11), Ru(1)–Si(1) 241.39(9), Si(2)–C(12) 189.1(3), $Si–C_{methyl}$ (mean) 187.8; bond angles [°]: Ru(1)–Si(1)–Si(2) 112.83(4), Si(1)–Si(2)–C(12) 107.11 (9), $C_{methyl}–Si–C_{methyl}$ (mean) 106.93, $Si–Si–C_{methyl}$ (mean) 109.61, $C_{cp}–C_{cp}–C_{cp}$ (mean) 108.00, $C_{ph}–C_{ph}–C_{ph}$ (mean) 120.00; and dihedral angles [°]: Ru(1)–Si(1)–Si(2)–C(12) −177.17(9), Si(1)–Si(2)–C(12)–C(17) 84.4(2), C(16)–C(15)–C(18)–C(19) 174.7(3), C(15)–C(18)–C(19)–C(21) 177.0(3)

centrosymmetric point group $P2_1/n$ with four molecules in the unit cell. The Ru–Si-distance of 241.4 pm is close to the mean value of 244 pm found for Ru–silyl complexes [8]. Structural features closely resemble the ones observed for the iron complex **6** [3]. The nearly identical Si–Si-bond lengths of **4** and **6** are slightly larger than the common value of 234 pm for disilanes bearing small substituents. The geometry around the silicon atoms is approximately tetrahedral. The 2,2-(dicyanovinyl)phenyl group appears to be nearly perfectly planar (compare *e.g.* the dihedral angle of 174.7° for C(16)–C(15)–C(18)–C(19)). The Ru(1)–Si(1)–Si(2)–C(12) fragment exhibits an *all-trans*-array and a roughly perpendicular arrangement of the plane of the phenyl ring relative to the Si–Si bond with a torsional angle of 84.4° for Si(1)–Si(2)–C(12)–C(17). The molecular structures adopted by **4** and **6** in the solid state, therefore, provide an excellent basis for efficient through-bond interaction between the organometallic donor and the 2,2-(dicyanovinyl)phenyl acceptor group *via* the central Si–Si-bond, because optimal through-bond interaction of functional groups *via* an array of σ-bonds is only feasible, when the corresponding orbitals and the σ-bonds have an *all-trans* relationship [9].

UV/Vis Absorption Spectra

UV absorption data of compound **4** are presented in Table 1 and Fig. 2 together with the data obtained for the corresponding iron complex **6** and for the reference compounds **1** and $Me_3SiSiMe_2PhCH{=}C(CN)_2$ (**5**), which represent reasonable model systems to estimate the properties of the donor and the acceptor moiety in the absence of interaction.

The longest wavelength absorption band observed in the spectrum of **1** exhibits a 65 nm shift to higher energy as compared to $FpSiMe_2SiMe_2Ph$ [3]. Similar hypsochromic shifts are also observed comparing *Rp*- and *Fp*-alkyl complexes [10]. The spectral features apparent in the spectrum of the dipolar compound **4** are dominated by the presence of the $PhCH{=}C(CN)_2$ acceptor moiety and closely resemble the ones shown by the iron complex **6** [3]. The 320 nm absorption band of the isolated acceptor chromophore is shifted hypsochromically in the spectrum of **4** and **6** combined with an intensity decrease. At the long wavelength side of the spectrum

Table 1. UV absorption data of **1**, **4**, **5**, and **6**

Compound	$\frac{\lambda_{max}}{nm}$	$\frac{\varepsilon}{mol^{-1}\,dm^3\,cm}$
$RpSi_2Me_4Ph$ (**1**)	249	13500
	265 sh	11400
$RpSi_2Me_4PhCH{=}C(CN)_2$ (**4**)	301	20200
	360	17500
$Me_3SiSiMe_2PhCH{=}C(CN)_2$ (**5**)	333	26400
$FpSi_2Me_4PhCH{=}C(CN)_2$ (**6**)[a]	309	19900
	360 sh	12000

[a] Taken from Ref. [3]

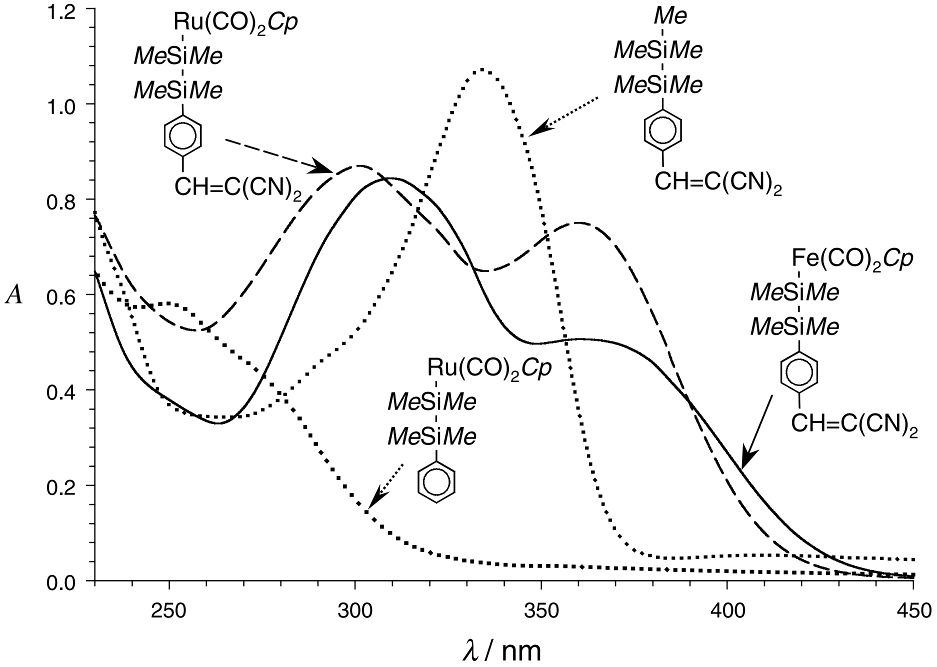


Fig. 2. UV absorption spectra of **4** and **6** compared to the corresponding donor and acceptor model systems (C_6H_{12} solution, $c = 4.3 \times 10^{-5}\,M$); the 320 nm absorption band of the isolated acceptor chromophore is shifted hypsochromically in the spectrum of **4** and **6**; at the long wavelength side of the spectra a new absorption band appears

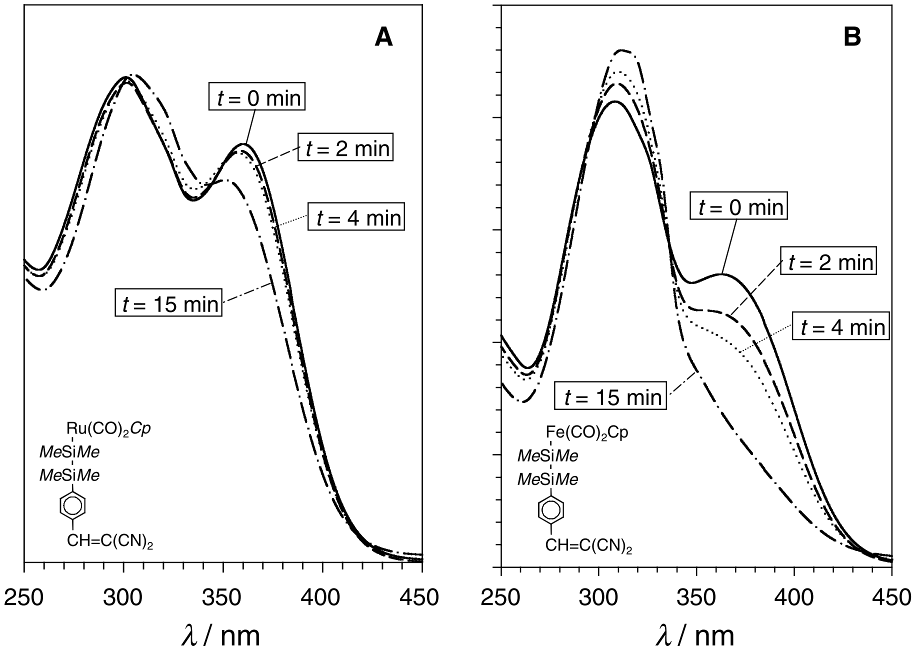


Fig. 3. Impact of irradiation (6 W 254 nm lamp) on the UV absorption spectra of **4** (Fig. 3A) and **6** (Fig. 3B) (C_6H_{12} solution, $c = 4.3 \times 10^{-5}\,M$); the intensity of the long wavelength band decreases faster in the iron compound

a new absorption band appears centered at 360 nm. Both features strongly suggest considerable ground state electronic interaction between the metal center and the (2,2-dicyanoethenyl)phenyl chromophore *via* the Si–Si-bond. In view of the fact, that intramolecular D/A-interactions were also detected in donor-bridge-acceptor compounds containing saturated hydrocarbon bridges [11] and of the electron donor/acceptor character of the substituents it seems logical to assign the 360 nm band appearing in the absorption spectra of **4** and **6** to a so-called σ-coupled transition with considerable charge-transfer character [12].

Although *Rp* disilanyl complexes were reported to be stable upon UV irradiation [5], we observed significant photobleaching, when diluted hydrocarbon solutions of **1** and **4** are irradiated with a 6 W 254 nm lamp in a quartz tube. Figure 3 shows the impact of UV irradiation on the absorption spectra of **4** and its iron counterpart **6**. In both cases the intensity of the longest wavelength absorption band rapidly decreases. The only observed difference is the enhanced photochemical reactivity of the iron complex **6**, which is completely decomposed after irradiation times as short as 15 minutes (compare Fig. 3). This finding is consistent with literature studies on (η^5-C_5H_5)M(CO)$_2$CH$_3$ (M = Fe, Ru) complexes, which are both reported to undergo dissociative loss of CO as the primary photoprocess following near UV excitation with higher quantum yields for M = Fe [10]. The loss of a CO ligand is also suggested to occur as the initial step in the course of the photochemical deoligomerization of *Fp* substituted oligosilanes [1b].

Conclusions

The *all-trans* relationship along the M–Si–Si–C_{aryl} fragment of **4** and **6** in the solid state provides the basis for optimal through-bond interaction between the organometallic donor and the 2,2-(dicyanovinyl)phenyl acceptor *via* the central Si–Si-bond. UV absorption data also support this assumption for solutions and indicate remarkable intramolecular electronic coupling within the molecular framework of **4** and **6**. The observed photolability of the ruthenium complexes addressed in this paper, however, prevents further studies of their photophysical properties just as like as in the iron case.

Experimental

All reactions and other manipulations were carried out using standard *Schlenk* techniques under an inert atmosphere of dry N_2. All solvents were dried and distilled under N_2 prior to use. Malononitrile was used as purchased without further purification. *N*,*N*-Dimethylformamide was allowed to stand on 4 Å molecular sieve and distilled from CaH_2. [(η^5-C_5H_5)Ru(CO)$_2$]$_2$ [13], Cl*Me*$_2$SiSi*Me*$_2$*Ph*Br [14], Cl*Me*$_2$SiSi*Me*$_2$*Ph*, and Cl*Me*$_2$SiSi*Me*$_2$Cl [15] were synthesized as previously reported. UV/Vis absorption spectra were recorded on a Perkin-Elmer Lambda 35 spectrometer in cyclohexane solution. NMR spectra were recorded on a Bruker 300-MSL spectrometer; ^{1}H NMR: 300.13 MHz; ^{13}C NMR: 75.47 MHz; ^{29}Si NMR: 59.62 MHz. HRMS spectra were run on a Kratos Profile mass spectrometer equipped with a solids probe inlet.

1-[Dicarbonyl(η^5-cyclopentadienyl)ruthenio]-2-phenyltetramethyldisilane (**1**, $C_{17}H_{22}O_2RuSi_2$)
To 0.77 g [(η^5-C_5H_5)Ru(CO)$_2$]$_2$ (1.73 mmol) dissolved in 50 cm^3 *THF* 6.9 cm^3 1.0 *M* K[B*Et*$_3$H] in *THF* were slowly added. After the evolution of H_2 had ceased, the solution was stirred overnight at 40°C.

After verifying the formation of the $[(\eta^5\text{-}C_5H_5)Ru(CO)_2]^-$-anion by IR ($\bar{\nu}_{CO}$ at 1896 and 1811 cm^{-1}) the solution was concentrated, $[(\eta^5\text{-}C_5H_5)Ru(CO)_2]K$ was precipitated by addition of *n*-pentane and isolated by centrifugation and washing with *n*-pentane until the overlaying solution appeared to be colorless. The resulting light-brown powder was then dissolved in 100 cm^3 *THF* and slowly added at −78°C to a solution of 0.6 g $ClSi\mathit{Me}_2Si\mathit{Me}_2\mathit{Ph}$ (2.62 mmol) in 100 cm^3 *THF*. Subsequently the reaction mixture was stirred at room temperature overnight before the solvents were stripped off in vacuum. After addition of *n*-pentane the solution was filtered, concentrated, and subjected to column chromatography on a silica gel column. Elution with *n*-heptane yielded an orange band, which, upon collection and removal of the solvent gave 0.46 g (42%) of the title complex as a colorless oil. IR (*THF*): $\bar{\nu} = 2006$, 1948 (CO) cm^{-1}; ^{29}Si NMR (C_6D_6, ext. *TMS*): $\delta = 2.0$ ($\mathit{Rp}\text{–}\mathit{Si}(CH_3)_2$), −16.5 ($C_6H_5\text{–}\mathit{Si}(CH_3)_2$) ppm; ^{1}H NMR ($C_6D_6$, ext. *TMS*): $\delta = 7.50\text{–}7.16$ (m, 5H, $C_6\mathit{H}_5$), 4.45 (s, 5H, $C_5\mathit{H}_5$), 0.60 (s, 6H, $C_6H_5\text{–}Si(C\mathit{H}_3)_2$), 0.45 (s, 6H, $\mathit{Rp}\text{–}Si(C\mathit{H}_3)_2$) ppm; ^{13}C NMR ($C_6D_6$, ext. *TMS*): $\delta = 202.1$ (CO), 141.5, 134.0, 128.5, 128.0 (C_6H_5), 86.5 (C_5H_5), 3.8 ($\mathit{Rp}\text{–}Si(\mathit{C}H_3)_2$), −2.9 ($C_6H_5\text{–}Si(\mathit{C}H_3)_2$) ppm; HRMS ($C_{17}H_{22}O_2Si_2Ru$, $[M^+]$): calc. 416.02085, found 416.02057.

1-[Dicarbonyl(η^5-cyclopentadienyl)ruthenio]-2-(p-bromophenyl)-tetramethyldisilane
(**2**, $C_{17}H_{21}BrO_2RuSi_2$)

The procedure followed was that used for **1** with 1.56 g $[(\eta^5\text{-}C_5H_5)Ru(CO)_2]_2$ (3.51 mmol), 16.0 cm^3 1.0 *M* $K[B\mathit{Et}_3H]$ in *THF* (16.0 mmol), and 1.9 g (5.76 mmol) $ClSi\mathit{Me}_2Si\mathit{Me}_2C_6H_4Br$. Yield 1.56 g (45%) of an orange oil; IR (*THF*): $\bar{\nu} = 2008$, 1949 (CO) cm^{-1}; ^{29}Si NMR (C_6D_6, ext. *TMS*): $\delta = 1.3$ ($\mathit{Rp}\text{–}\mathit{Si}(CH_3)_2$), −16.8 ($p\text{-}BrC_6H_4\text{–}\mathit{Si}(CH_3)_2$) ppm; ^{1}H NMR ($C_6D_6$, ext. *TMS*): $\delta = 7.37$, 7.35, 7.16, 7.14 (AA′BB′, 4H, $C_6\mathit{H}_4$), 4.40 (s, 5H, $C_5\mathit{H}_5$), 0.53 (s, 6H, $p\text{-}BrC_6H_4\text{–}Si(C\mathit{H}_3)_2$), 0.34 (s, 6H, $\mathit{Rp}\text{–}Si(C\mathit{H}_3)_2$) ppm; ^{13}C NMR ($C_6D_6$, ext. *TMS*): $\delta = 202.0$ (CO), 140.4, 135.6, 131.2, 123.3 (C_6H_4), 86.5 (C_5H_5), 3.8 ($\mathit{Rp}\text{–}Si(\mathit{C}H_3)_2$), −3.0 ($p\text{-}BrC_6H_4\text{–}Si(\mathit{C}H_3)_2$) ppm; HRMS ($C_{17}H_{21}O_2Si_2RuBr$, $[M^+]$): calc. 493.92774, found 493.93030.

1-[Dicarbonyl(η^5-cyclopentadienyl)ruthenio]-2-(p-formylphenyl)-tetramethyldisilane
(**3**, $C_{18}H_{22}O_3RuSi_2$)

To 30 cm^3 of a cooled (−78°C) *THF* solution of 1.06 g **2** (2.14 mmol) 0.95 cm^3 2.5 *M* *n*-*Bu*Li (2.35 mmol) in cyclohexane were slowly added with a gas-tight syringe. The mixture was stirred for another 5 min followed by addition of 0.65 cm^3 dry *DMF* (8.43 mmol). The reaction mixture was then stirred overnight at room temperature and aqueously worked up with saturated NH_4Cl solution. After extraction with diethylether the combined organic layers were dried (Na_2SO_4) and concentrated. Column chromatography (silica gel) with toluene as the mobile phase yielded 0.54 g (57%) of **3** as a yellow oil. IR (*THF*): $\bar{\nu} = 2008$, 1949 (CO), 1705 (CHO) cm^{-1}; ^{29}Si NMR (C_6D_6, ext. *TMS*): $\delta = 1.4$ ($\mathit{Rp}\text{–}\mathit{Si}(CH_3)_2$), −15.7 ($p\text{-}OHCC_6H_4\text{–}\mathit{Si}(CH_3)_2$) ppm; ^{1}H NMR ($C_6D_6$, ext. *TMS*): $\delta = 9.72$ (s, 1H, $C\mathit{H}O$), 7.62, 7.59, 7.45, 7.43 (AA′BB′, 4H, $C_6\mathit{H}_4$), 4.40 (s, 5H, $C_5\mathit{H}_5$), 0.53 (s, 6H, $p\text{-}OHCC_6H_4\text{–}Si(C\mathit{H}_3)_2$), 0.37 (s, 6H, $\mathit{Rp}\text{–}Si(C\mathit{H}_3)_2$) ppm; ^{13}C NMR ($C_6D_6$, ext. *TMS*): $\delta = 201.9$ (CO), 191.4 (HO**C**), 150.2, 136.7, 134.3, 128.6 (C_6H_4), 86.5 (C_5H_5), 3.8 ($\mathit{Rp}\text{–}Si(\mathit{C}H_3)_2$), −3.1 ($p\text{-}OHCC_6H_4\text{–}Si(\mathit{C}H_3)_2$) ppm; HRMS ($C_{18}H_{22}O_3Si_2Ru$, $[M^+]$): calc. 444.01830, found 444.01551.

1-[Dicarbonyl(η^5-cyclopentadienyl)ruthenio]-2-[p-(2,2-dicyanoethenyl)phenyl]
tetramethyldisilane (**4**, $C_{21}H_{22}N_2O_2RuSi_2$)

To a solution of 0.44 g **3** (1 mmol) in 15 cm^3 *THF* 70.8 mg solid malonodinitrile (1.07 mmol) and one drop piperidine were added. In order to guarantee total conversion the mixture was allowed to stir overnight at room temperature (DC monitoring recommended). After removal of the solvent under reduced pressure and purification of the resulting oily residue by column chromatography (silica gel) with toluene as the mobile phase 0.38 g (78%) of the title compound were recovered as an orange oil, which could be crystallized from a mixture of toluene and *n*-pentane at −30°C. Mp 100–102°C; Anal: calc. for $C_{21}H_{22}N_2FeO_2Si_2$ C 56.50 H 4.97%, found C 56.81 H 4.92%; IR (*THF*): $\bar{\nu} = 2229$ (CN), $\bar{\nu} = 2008$, 1950 (CO) cm^{-1}; ^{29}Si NMR (C_6D_6, ext. *TMS*): $\delta = 1.4$ ($\mathit{Rp}\text{–}\mathit{Si}(CH_3)_2$), −15.2

(p-(NC)$_2$C=CHC$_6$H$_4$–*Si*(CH$_3$)$_2$) ppm; ^{1}H NMR (C$_6$D$_6$, ext. *TMS*): $\delta = 7.33$, 7.30, 7.29, 7.26 (AA′BB′, 4H, C$_6$*H*$_4$), 6.51 (s, 1H, C*H*=C(CN)$_2$), 4.42 (s, 5H, C$_5$*H*$_5$), 0.49 (s, 6H, p-(NC)$_2$C=CHC$_6$H$_4$–Si(C*H*$_3$)$_2$), 0.35 (s, 6H, *Rp*–Si(C*H*$_3$)$_2$) ppm; ^{13}C NMR (C$_6$D$_6$, ext. *TMS*): $\delta = 201.7$ (CO), 158.7 (*C*H=C(CN)$_2$), 151.3, 134.4, 130.7, 129.3 (*C*$_6$H$_4$), 114.0, 113.1 (CN), 86.5 (*C*$_5$H$_5$), 82.5 (CH=*C*(CN)$_2$), 3.8 (*Rp*–Si(*C*H$_3$)$_2$), −3.2 (p-(NC)$_2$C=CHC$_6$H$_4$–Si(*C*H$_3$)$_2$) ppm; HRMS (C$_{21}$H$_{22}$O$_2$N$_2$Si$_2$Ru, [M$^+$]): calc. 492.02637, found 492.02682.

X-Ray Crystallography

Suitable crystals of **4** were grown from diethylether solutions at −30°C. For X-ray structure analysis the crystals were mounted onto the tip of glass fibers, and data collection was performed with a BRUKER-AXS SMART APEX CCD diffractometer using graphite-monochromated Mo Kα radiation (0.71073 Å). Crystal data and the details of the structure determinations are given in Table 2. The data were reduced to F_o^2 and corrected for absorption effects with SADABS [16]. The structures were solved by direct methods and refined by full-matrix least-squares on F^2 method (SHELXL97) [17]. If not noted otherwise all non-hydrogen atoms were refined with anisotropic displacement parameters.

Table 2. Crystallographic data for compound **4**

Empirical formula	C$_{21}$H$_{22}$RuN$_2$O$_2$Si$_2$
Formula weight	491.66
Collection temperature/K	173(2)
Crystal system	Monoclinic
Crystal size/mm^3	$0.35 \times 0.25 \times 0.10$
Space group	$P2_1/n$
a/Å	7.1659(14)
b/Å	29.856(6)
c/Å	10.856(2)
α/°	90
β/°	98.00(3)
γ/°	90
Volume/Å^3	2299.9(8)
Z	4
Absorption coefficient/mm^{-1}	0.803
Density calc./g · cm^{-3}	1.420
F(000)	1000
θ range/°	1.36–25.00
Limiting indices	$-8 < h < 8$ $-35 < k < 35$ $-12 < l < 12$
Reflections collected	16147
Independent reflections	4058 [R(int) = 0.0495]
Completeness to $\theta = 25.00°$	99.9%
Max. and min. transmission	0.9240 and 0.7663
Data/restraints/parameters	4058/0/257
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0332$ $wR_2 = 0.0816$
R indices [all data]	$R_1 = 0.0417$ $wR_2 = 0.0850$
Goodness-of-fit on F^2	1.018
Largest difference peak to hole/e · Å^{-3}	0.547 and −0.286

All hydrogen atoms were located in calculated positions to correspond to standard bond lengths and angles. All diagrams were drawn with 30% probability thermal ellipsoids and all hydrogen atoms were omitted for clarity.

Supplementary Material

Crystallographic data for the structural analyses have been deposited with the Cambridge Crystallographic Data Center, No. CCDC 289614, for compound **4**. Copies of this information may be obtained free of charge from: The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (fax: +44 (1223) 336-033, e-mail: deposit@ccdc.cam.ac.uk, or http://www.ccdc.cam.ac.uk).

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