Synthesis of dibenzocymantrene derivatives (η^5-9-$RC_{13}H_8$)$Mn(CO)_3$ (R = Ph and Bu^t). Crystal structure of (η^5-9-$PhC_{13}H_8$)$Mn(CO)_3$

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η^5-Fluorenyl complexes of manganese (η^5-9-$RC_{13}H_8$)$Mn(CO)_3$, where R = Ph (**1**) and Bu^t (**2**), have been prepared and characterized for the first time. The structure of complex **1** has been established by X-ray structural analysis.

Key words: fluorenyltricarbonylmanganese, haptotropic rearrangements; NMR and IR spectroscopy; X-ray structural analysis.

Interring haptotropic rearrangements of fluorenyl complexes of transition metals were studied extensively for 18-electron compounds.[1] Recently, these processes were studied also for 19-electron fluorenyl complexes of iron,[2] chromium,[3] and manganese.[4] A comparative study of interring haptotropic rearrangements for 18- and 19-electron fluorenyl complexes[1,5] makes it possible to obtain essential data on the effect of the electronic configuration of metal atoms on the reactivity of these complexes. These studies require adequate model compounds, specifically, fluorenyl complexes substituted at position 9, because the presence of substituents at this position may have a substantial effect on the redistribution of π-electron density in the fluorenyl ligand as well as on the thermodynamic and kinetic stability of the η^6- and η^5-forms both in the 18- and in 19-electron states. In this connection, this work is devoted to the synthesis and studies of the structures of manganese complexes (η^5-9-$RC_{13}H_8$)$Mn(CO)_3$ (R = Ph (**1**) and Bu^t (**2**)).

Results and Discussion

Three main methods of preparation of (η^5-fluorenyl)tricarbonylmanganese complexes are known: (1) the reaction of a fluorenide anion with manganese carbonyl halides, $Mn(CO)_5X$ [6] or $Mn(CO)_3(MeCN)_2X$ [7]; (2) the thermal reaction of hydrocarbons with $Mn_2(CO)_{10}$ [8]; (3) the interring η^6:η^5-rearrangement of η^6-fluorenyl zwitterions (η^6-$C_{13}H_8$)$Mn(CO)_3$.[9]

In this work, we used the first method for the synthesis of complex **1** and the third method for the synthesis of **2** (Scheme 1).

In deciding on a particular method of the synthesis of complex **1**, we are guided by the following reasons: if 9-phenylfluorene is used as a starting compound, an electrophilic particle, which is formed as a result of the reaction of manganese carbonyl halides with $AlCl_3$,[9] may, in principle, attack five- or six-membered rings of fluorene as well as the phenyl ring of the substituent at position 9. In the case of the 9-phenylfluorenide anion, a selective attack on the five-membered ring by electrophile is likely to be preferential. The reaction of lithium 9-fluorenide with $[Mn(CO)_4Br]_2$ in THF at room temperature followed by heating of the reaction products in hexane (1—1.5 h) afforded complex **1** in 47 % yield (see Scheme 1, *a* (*1*) + *b*). Note that unlike the procedure described previously,[6] changing from THF to hexane at the final stage of synthesis of **1** followed by boiling of the solution made it possible to enhance the yield of this complex to 47 % (whereas, the yield of **1** from THF was no more than 1—2 %). Deprotonation of hydrocarbon **3** with lithium bis(trimethylsilyl)amide and carboxylation of the formed carbanion with solid CO_2 were used for purifying complex **1** from excess and unreacted 9-phenylfluorene **3** (see Scheme 1, *a*, (*2*) + *c*).

We used an alternative approach (see Scheme 1, *d* + *e* + *f*), which involved preparation of [(η^6-9-Bu^t$C_{13}H_9$)$Mn(CO)_3$]PF_6 and its deprotonation followed by thermal η^6:η^5-rearrangement, for the synthesis of complex **2**. This method is analogous to that previously reported[9] for preparation of (η^5-$C_{13}H_9$)$Mn(CO)_3$ (see also Ref. 10). Previously, we reported the results of

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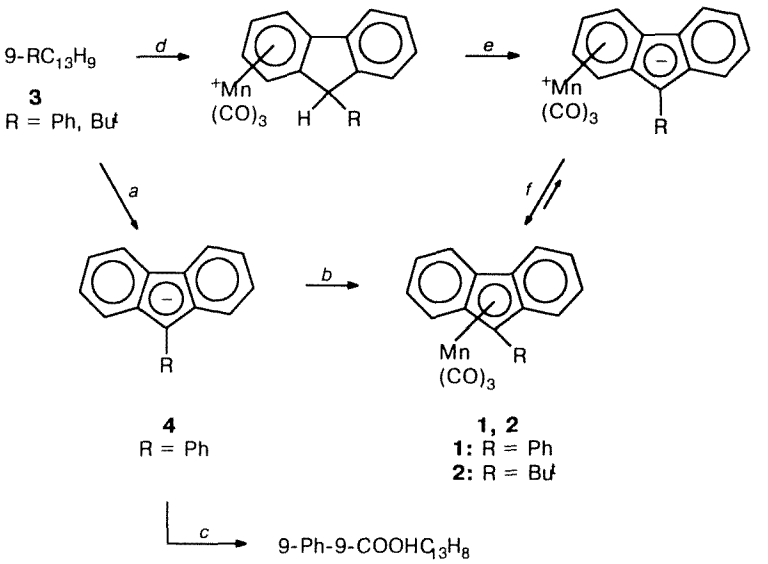
Scheme 1



Reagents and conditions: *a*, Bu^nLi (*1*), $(Me_3Si)_2NLi$ (*2*); *b*, $[Mn(CO)_4Br]_2$; *c*, CO_2 (*1*), H^+ (*2*); *d*, $Mn(CO)_5Br/AlCl_3$; *e*, $-H^+$; *f*, Δ (40—50 °C).

spectroscopic (IR, ^1H and ^{13}C NMR) and electrochemical studies of compound **2**.[11]

X-ray structural analysis unambiguously confirmed that the $Mn(CO)_3$ group in complex **1** is coordinated by the central ring of the fluorenyl system. The structure of complex **1** is shown in Fig. 1. The bond lengths and principal bond angles are given in Tables 1 and 2, respectively.

As far as we know, $(\eta^5\text{-}9\text{-}PhC_{13}H_8)Mn(CO)_3$ is the first structurally characterized fluorenyl π-complex of manganese, in which the five-membered ring acts as a π-ligand. Presently, the data on the structure of only one fluorenyl complex of manganese with the metal atom, which has a coordination of the η^6-type, are available.[12] Geometric parameters of the coordination unit of **1** are very close to the corresponding characteristics of cimantrene.[13] In molecule **1**, a slight (but, undoubtedly, exceeding an experimental error) distortion from planarity of the central ring is observed. This ring has an envelope conformation: the C(9) atom deviates from the mean plane (within 0.006 Å) through the C(1a), C(4a), C(5a), and C(8a) atoms by 0.054 Å toward the metal atom; the folding angle about the C(1a)···C(8a) line is 3.6°. This conformation of the ring correlates with pronounced differences in the Mn—C(fl) distances (fl is the fluorenyl ligand); one of these distances, Mn(1)—C(9) [2.154(4) Å], is substantially shorter than the remaining four distances (2.194—2.210 Å). As a whole, the Mn—C distances to the fluorenyl ligand are substantially longer than the Mn—C(Cp) bonds in cimantrene (2.133—2.142 Å).[13] Benzene rings fused to the five-membered ring (the wings of the fluorenyl ligand) as well as the Ph substituent at the C(9) atom deviate from the central ring in the direction opposite to that of the metal atom. The dihedral angles between the C(1)C(2)C(3)—C(4)C(4a)C(1a) and C(5)C(6)C(7)C(8)C(8a)C(5a) planes and the C(1a)C(4a)C(5a)C(8a) plane are 4.9° and 2.1°, respectively; the angle between the C(9)—C(10) bond and the C(1a)C(9)C(8a) plane is 9.6°. The most substantial distortion from the overall mirror symmetry of the molecule is associated with the rotation of the Ph ring C(10)C(11)C(12)C(13)C(14)C(15) about the C(9)—C(10) bond: the C(1a)—C(9)—C(10)—C(11) and C(8a)—C(9)—C(10)—C(11) torsion angles are 123.6° and

Table 1. Bond lengths (*d*) in the structure of **1**

Bond	*d*/Å	Bond	*d*/Å
Mn(1)—C(1a)	2.194 (4)	C(4)—C(4a)	1.409 (7)
Mn(1)—C(4a)	2.210 (4)	C(4a)—C(5a)	1.441 (6)
Mn(1)—C(5a)	2.206 (4)	C(5)—C(5a)	1.410 (6)
Mn(1)—C(8a)	2.194 (4)	C(5)—C(6)	1.366 (7)
Mn(1)—C(9)	2.154 (4)	C(5a)—C(8a)	1.440 (6)
Mn(1)—C(16)	1.803 (4)	C(6)—C(7)	1.417 (7)
Mn(1)—C(17)	1.777 (5)	C(7)—C(8)	1.356 (6)
Mn(1)—C(18)	1.815 (5)	C(8)—C(8a)	1.431 (6)
O(1)—C(16)	1.142 (5)	C(8a)—C(9)	1.451 (6)
O(2)—C(17)	1.161 (6)	C(9)—C(10)	1.487 (6)
O(3)—C(18)	1.145 (7)	C(10)—C(11)	1.395 (6)
C(1)—C(1a)	1.426 (6)	C(10)—C(15)	1.388 (6)
C(1)—C(2)	1.373 (7)	C(11)—C(12)	1.380 (7)
C(1a)—C(4a)	1.459 (6)	C(12)—C(13)	1.393 (7)
C(1a)—C(9)	1.449 (6)	C(13)—C(14)	1.378 (7)
C(2)—C(3)	1.415 (8)	C(14)—C(15)	1.391 (7)
C(3)—C(4)	1.364 (8)		

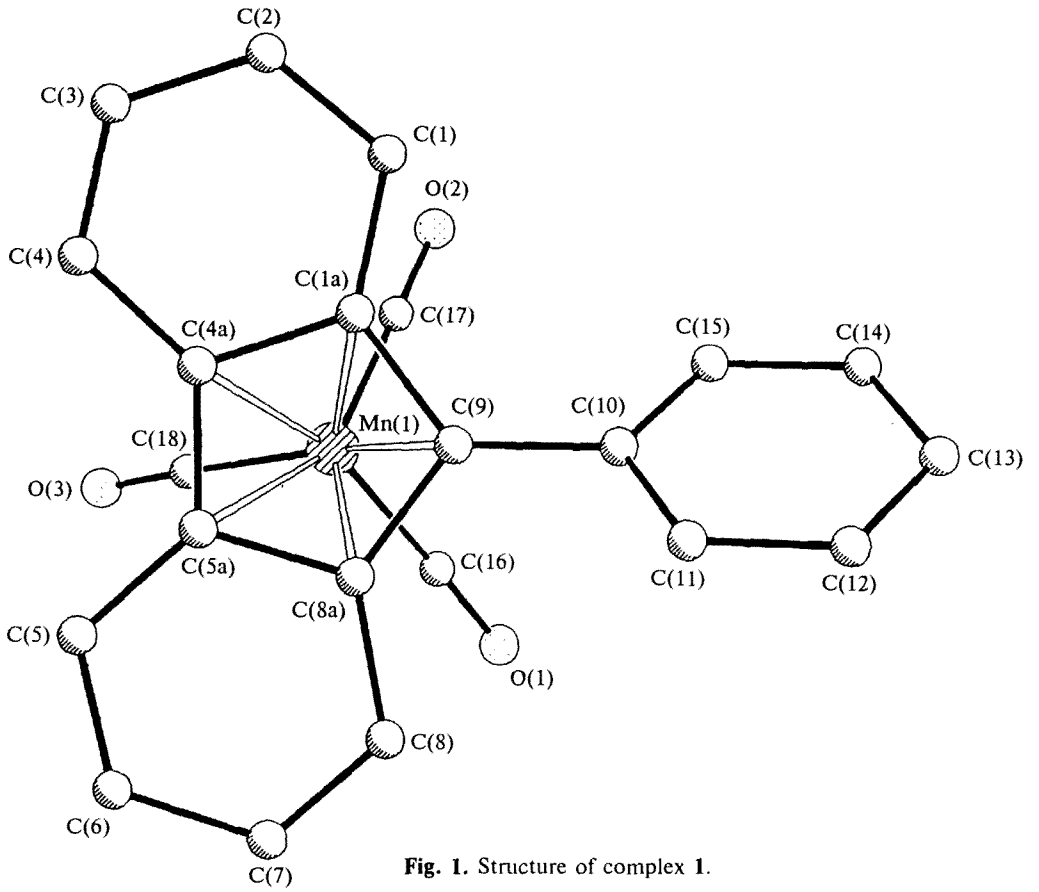


Fig. 1. Structure of complex **1**.

Table 2. Principal bond angles (ω) in the structure of **1**

Angle	ω/deg	Angle	ω/deg	Angle	ω/deg
C(1a)—Mn(1)—C(4a)	38.7(2)	C(16)—Mn(1)—C(18)	94.0(2)	C(6)—C(7)—C(8)	122.1(4)
C(1a)—Mn(1)—C(5a)	64.7(1)	C(17)—Mn(1)—C(18)	92.0(2)	C(7)—C(8)—C(8a)	119.2(4)
C(4a)—Mn(1)—C(5a)	38.1(2)	C(1a)—C(1)—C(2)	117.8(5)	Mn(1)—C(8a)—C(5a)	71.3(2)
C(1a)—Mn(1)—C(8a)	64.1(1)	Mn(1)—C(1a)—C(1)	126.9(3)	Mn(1)—C(8a)—C(8)	124.7(3)
C(4a)—Mn(1)—C(8a)	63.5(2)	Mn(1)—C(1a)—C(4a)	71.3(2)	C(5a)—C(8a)—C(8)	117.9(4)
C(5a)—Mn(1)—C(8a)	38.2(2)	C(1)—C(1a)—C(4a)	120.4(4)	Mn(1)—C(8a)—C(9)	69.0(2)
C(1a)—Mn(1)—C(9)	38.9(2)	Mn(1)—C(1a)—C(9)	69.1(2)	C(5a)—C(8a)—C(9)	109.6(3)
C(4a)—Mn(1)—C(9)	65.2(2)	C(1)—C(1a)—C(9)	131.6(4)	C(8)—C(8a)—C(9)	132.5(4)
C(5a)—Mn(1)—C(9)	65.6(2)	C(4a)—C(1a)—C(9)	108.0(3)	Mn(1)—C(9)—C(1a)	72.0(2)
C(8a)—Mn(1)—C(9)	39.0(2)	C(1)—C(2)—C(3)	122.2(5)	Mn(1)—C(9)—C(8a)	72.0(2)
C(1a)—Mn(1)—C(16)	135.2(2)	C(2)—C(3)—C(4)	121.0(5)	C(1a)—C(9)—C(8a)	106.7(3)
C(4a)—Mn(1)—C(16)	154.2(2)	C(3)—C(4)—C(4a)	120.4(5)	Mn(1)—C(9)—C(10)	130.8(3)
C(5a)—Mn(1)—C(16)	118.0(2)	Mn(1)—C(4a)—C(1a)	70.0(2)	C(1a)—C(9)—C(10)	126.6(3)
C(8a)—Mn(1)—C(16)	91.0(2)	Mn(1)—C(4a)—C(4)	127.6(3)	C(8a)—C(9)—C(10)	125.5(4)
C(9)—Mn(1)—C(16)	98.3(2)	C(1a)—C(4a)—C(4)	118.2(4)	C(9)—C(10)—C(11)	118.8(4)
C(1a)—Mn(1)—C(17)	91.0(2)	Mn(1)—C(4a)—C(5a)	70.8(2)	C(9)—C(10)—C(15)	123.2(4)
C(4a)—Mn(1)—C(17)	112.0(2)	C(1a)—C(4a)—C(5a)	108.4(4)	C(11)—C(10)—C(15)	118.0(4)
C(5a)—Mn(1)—C(17)	150.1(2)	C(4)—C(4a)—C(5a)	133.2(4)	C(10)—C(11)—C(12)	121.3(4)
C(8a)—Mn(1)—C(17)	145.6(2)	C(5a)—C(5)—C(6)	118.3(4)	C(11)—C(12)—C(13)	120.1(4)
C(9)—Mn(1)—C(17)	106.9(2)	Mn(1)—C(5a)—C(4a)	71.1(2)	C(12)—C(13)—C(14)	119.1(5)
C(16)—Mn(1)—C(17)	91.3(2)	Mn(1)—C(5a)—C(5)	125.3(3)	C(13)—C(14)—C(15)	120.5(4)
C(1a)—Mn(1)—C(18)	130.6(2)	C(4a)—C(5a)—C(5)	131.4(4)	C(10)—C(15)—C(14)	120.9(4)
C(4a)—Mn(1)—C(18)	96.0(2)	Mn(1)—C(5a)—C(8a)	70.4(2)	Mn(1)—C(16)—O(1)	177.8(4)
C(5a)—Mn(1)—C(18)	91.5(2)	C(4a)—C(5a)—C(8a)	107.2(4)	Mn(1)—C(17)—O(2)	178.2(4)
C(8a)—Mn(1)—C(18)	122.0(2)	C(5)—C(5a)—C(8a)	121.4(4)	Mn(1)—C(18)—O(3)	178.4(5)
C(9)—Mn(1)—C(18)	157.0(2)	C(5)—C(6)—C(7)	121.1(4)		

−42.2°, respectively. Apparently, different repulsions between the *ortho*-C atom of the phenyl substituent and the carbonyl carbon atoms [C(15)···C(16) and C(15)···C(17) are 3.886 and 3.756 Å, respectively] may account for a slight distortion from the ideal staggered conformation of the $Mn(CO)_3$ fragment. Actually, the C(18)—Mn(1)—X(1)—C(5a) torsion angle, where X(1) is the center of the C(1a)C(4a)C(5a)C(8a)C(9) cycle, is 28.4°, which slightly differs from the value of 36° corresponding to an ideal staggered conformation.

The splitting of the asymmetric stretching vibration band of carbonyl groups (band *E*) is an interesting feature of the IR spectra of complexes **1** and **2** in hexane: ν(CO) 1948 and 1942 cm^{-1} (**1**) or 1942 and 1937 cm^{-1} (**2**). We believe that because of the increased barrier to rotation of the tricarbonylmanganese group with respect to the π-ligand, which is caused by steric hindrance, complexes **1** and **2** occur in solutions in the form of *anti*-C(9) conformers; the presence of this conformer in the crystal of **1** was confirmed by the data of X-ray structural analysis (see Fig. 1). Therefore, effective local C_{3v} symmetry for complexes of this type is disrupted, and band *E* becomes nondegenerated. In the ^{13}C NMR spectra of compounds **1** and **2**, the signals of the carbonyl C atoms are observed in the form of significantly broadened singlets (12.0 and 11.75 Hz, respectively). Usually, the widths of these signals of carbonylmetal groups are no more than 3 Hz.

A comparison of these data with the observed degeneration of bands *E* in the νCO region of the IR spectra allows the conclusion that in compounds **1** and **2** the rotation of the carbonylmetal group is substantially impeded because of steric hindrance by phenyl and *tert*-butyl groups. This leads to broadening of the corresponding signals in the NMR spectra. The results of high- and low-temperature NMR studies will be published elsewhere.

Experimental

All reactions were carried out under a dry purified argon atmosphere with the use of anhydrous solvents. Silpearl silica gel (40—100 μm) (Sklo Union, Czechoslovakia) was used for column chromatography. IR spectra were recorded on a Specord 75IR instrument; NMR spectra were obtained on a Varian VRX-400 instrument.

Initial 9-phenyl- and 9-*tert*-butylfluorenes were prepared by the known procedure.[14]

Synthesis of the complex (η^5-9-$PhC_{13}H_8$)$Mn(CO)_3$ (1). A solution of Bu^nLi (2.42 mL, 1.86 *M*) in hexane was added dropwise to a solution of 9-phenylfluorene (0.988 g, 4.081 mmol) in THF (15 mL) with stirring at room temperature; the colorless reaction mixture developed a crimson color during this step. A saturated solution of $[Mn(CO)_4Br]_2$ (0.998 g, 2.02 mmol) in THF (70 mL) was slowly added to the obtained solution at room temperature with active stirring for 60 min, and then the mixture was stirred for 2 h under the same conditions; during this step, the solution changed in color to dark-cherry. The reaction mixture was transferred to a hexane solution by gradual distillation of THF using a water bath with simultaneous addition of hexane. The obtained solution was refluxed for 1—1.5 h until the color changed from dark-red to light-yellow. The reaction mass was cooled to room temperature, evaporated, and chromatographed on a column with SiO_2 (20 : 80 benzene—hexane as eluent). The isolated second orange fraction was evaporated to dryness, and the mixture of white and orange crystals was dissolved in 30 mL of THF. Lithium bis(trimethylsilyl)amide (0.686 g, 4.1 mmol) was added to the obtained solution with stirring at room temperature, and after 30 min, solid CO_2 (20 g) was added to the mixture. An aqueous HCl solution (20 mL, 0.1 *M*) was added dropwise to the mixture at 0 °C. THF was removed on a rotary evaporator,

Table 3. Atomic coordinates (×10⁴) and equivalent isotropic temperature factors (U_{iso}^{eq}×10³) in the structure of **1**

Atom	*x*	*y*	*z*	U_{iso}^{eq}/Å²
Mn(1)	1243(1)	764(1)	3993(1)	25(1)
O(1)	1270(3)	4139(4)	3505(1)	46(1)
O(2)	2894(2)	1788(5)	4512(1)	46(1)
O(3)	−7(3)	2245(6)	4599(1)	67(2)
C(1)	2575(3)	−2759(6)	4142(2)	35(1)
C(1a)	1791(3)	−1984(5)	3920(1)	26(1)
C(2)	2438(4)	−3669(6)	4505(2)	43(2)
C(3)	1560(4)	−3809(7)	4666(2)	49(2)
C(4)	806(4)	−3038(7)	4464(2)	41(2)
C(4a)	883(3)	−2118(5)	4085(1)	29(1)
C(5)	−741(3)	−1115(6)	3792(2)	34(1)
C(5a)	220(3)	−1313(6)	3789(1)	27(1)
C(6)	−1204(3)	−380(6)	3450(2)	37(1)
C(7)	−732(3)	202(6)	3103(2)	34(1)
C(8)	195(3)	69(6)	3093(2)	29(1)
C(8a)	713(3)	−727(5)	3439(1)	27(1)
C(9)	1689(3)	−1057(5)	3523(1)	26(1)
C(10)	2406(3)	−840(5)	3218(1)	26(1)
C(11)	2220(3)	−1459(6)	2813(1)	31(1)
C(12)	2864(3)	−1314(7)	2518(2)	36(1)
C(13)	3719(3)	−535(7)	2623(2)	38(1)
C(14)	3917(3)	52(7)	3025(2)	39(2)
C(15)	3265(3)	−90(6)	3320(2)	33(1)
C(16)	1268(3)	2847(6)	3701(1)	31(1)
C(17)	2241(3)	1410(6)	4304(2)	32(1)
C(18)	486(3)	1668(7)	4369(2)	43(2)
H(1)	3202(25)	−2715(53)	4036(11)	12(9)
H(2)	3026(33)	−4364(67)	4658(16)	43(14)
H(3)	1691(33)	−4485(71)	4939(17)	50(15)
H(4)	223(33)	−3012(69)	4591(16)	39(13)
H(5)	−988(32)	−1414(69)	4018(15)	36(13)
H(6)	−1868(46)	−235(96)	3462(21)	81(20)
H(7)	−1105(38)	587(77)	2846(19)	61(17)
H(8)	396(24)	389(51)	2891(12)	7(10)
H(11)	1759(41)	−2147(93)	2779(19)	70(20)
H(12)	2729(24)	−1707(53)	2265(13)	11(9)
H(13)	4171(35)	−479(73)	2442(17)	48(15)
H(14)	4531(36)	521(69)	3079(16)	45(14)
H(15)	3356(31)	323(67)	3584(16)	35(13)

and the residue was extracted three times with CH_2Cl_2. The combined organic layers were dried over $CaCl_2$, filtered, and evaporated *in vacuo*; the obtained dry residue was extracted with hexane. After filtration and evaporation of the hexane extract under an Ar flow, large orange crystals precipitated. Complex **1** (0.725 g), m.p. 128 °C (from hexane), was obtained in 47 % yield. Found (%): C, 69.31; H, 3.48. $C_{22}H_{13}O_3Mn$. Calculated (%): C, 69.49; H, 3.45. IR (hexane), ν/cm^{-1}: 2023, 1948, 1942 (CO). ^1H NMR (CD_2Cl_2), δ: 8.20 (d, 2 H, J = 7.5 Hz); 7.90 (d, 2 H, J = 7.8 Hz); 7.69 (d, 2 H, J = 7.8 Hz); 7.59 (t, 2 H, J = 7.8 Hz); 7.49 (t, 1 H, J = 7.5 Hz); 7.30 (m, 4 H) (uncoordinated rings of the fl ligand and 9-Ph). ^{13}C NMR (CD_2Cl_2), δ: 226.114 (3 CO); 128.90, 125.95, 125.71, 124.33, 107.15, 94.59 (uncoordinated rings of the fl ligand); 83.48 (C(9)); 133.98, 131.82, 129.72, 128.63 (9-Ph).

X-ray structural analysis of complex 1. Crystals of **1** are monoclinic, at −95 °C a = 14.569(5) Å, b = 7.364(3) Å, c = 32.22(1) Å, β = 93.67(2)°, V = 3449(2) Å^3, d_{calc} = 1.465 g cm^{-3}, Z = 8, space group C 2/c. The unit-cell parameters and intensities of 3401 independent reflections were measured on an automated four-circle Syntex $P2_1$ diffractometer (−95 °C, Mo-Kα radiation, graphite monochromator, θ/2θ scanning technique, θ ≤ 27°).

The structure was solved by the direct method and was refined by the least-squares method first with isotropic temperature factors and then with anisotropic temperature factors. All H atoms were located from the difference Fourier synthesis and refined isotropically. The final R values were R = 0.0611, R_w = 0.0755 for 2575 reflections with $I \geq 3\sigma(I)$. All calculations were performed using the SHELXTL PLUS program (the PC version)[15] on an IBM PC computer. Atomic coordinates are given in Table 3.

Synthesis of (η^5-Bu^t$C_{13}H_8$)Mn$(CO)_3$ (2). Bu^{t}OK (0.23 g, 2.1 mmol) was added to a solution of [(η^6-9-Bu^t$C_{13}H_9$)Mn$(CO)_3$]PF_6 [8] (0.375 g, 0.84 mmol) in 30 mL of THF at −20 °C, the mixture was stirred for 30 min, and then the temperature of the reaction mixture was brought to room temperature. The mixture was filtered, THF was distilled off from the mixture by boiling with a simultaneous addition of heptane. Boiling of the obtained heptane solution was continued for no more than 30 min. After the solution was chromatographed on a column with SiO_2 (20 : 80 benzene—hexane as an eluent), complex **2** (0.192 g) was obtained in 64 % yield, m.p. 196 °C (from hexane) (see Ref. 11).

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