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Reactivity of tri(2-furyl)phosphine (PFu₃) with [Mn₂(CO)_{10-n}(NCMe)_n] (*n* = 0–2): X-ray Structure of *mer*-[Mn(CO)₃(η¹-C₄H₃O)(PFu₃)₂]

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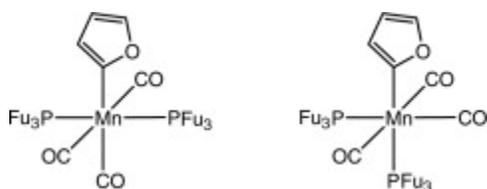
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Abstract

Reaction of tri(2-furyl)phosphine (PFu₃) with [Mn₂(CO)_{10-n}(NCMe)_n] (*n* = 1, 2) at room temperature furnished the substituted complexes [Mn₂(CO)_{10-n}(PFu₃)_n] (**1–2**). Direct reaction between Mn₂(CO)₁₀ and PFu₃ in refluxing toluene also afforded **2** together with *mer*-[Mn(CO)₃(η¹-C₄H₃O)(PFu₃)₂] (**3**) resulting from phosphorus–carbon bond cleavage of the [phosphine](#). All three new complexes have been characterized by spectroscopic data together with single crystal X-ray diffraction studies for **3**.

Graphical abstract

Reactions of tri(2-furyl)phosphine (PFu₃) with [Mn₂(CO)_{10-n}(NCMe)_n] (*n* = 0–2) have been studied.



Keywords

Manganese, Tri(2-furyl)phosphine, Carbonyl, X-ray structure

1. Introduction

Tri(2-furyl)phosphine, PFu₃, has found increasing importance in [transition metal](#) catalysis [\[1\]](#), [\[2\]](#), [\[3\]](#), [\[4\]](#), [\[5\]](#), [\[6\]](#), [\[7\]](#) since catalysts employing this ligand are often more reactive than traditional [triphenylphosphine](#) based catalysts. The different [electronic properties](#) of these two phosphines is believed to be the reason behind this difference in reactivity as their [Tolman cone angles](#) (PPh₃ – 145°; PFu₃ – 133°) are quite similar [\[8\]](#). Recently considerable effort has been devoted to explore the reactivity of PFu₃ with transition metal carbonyl complexes. For instance, the reactivity of PFu₃ with dirhenium complexes, [Re₂(CO)_{10-n}(NCMe)_n] (*n* = 0–2), has been reported by us couple of years ago where we found its behavior closely similar to that of PPh₃[\[9\]](#). More recently, we demonstrated the reactivity of PFu₃ with triosmium clusters [\[10\]](#), [Os₃(CO)_{10-n}(NCMe)_n] (*n* = 0–2) and [Os₃(CO)₁₀(μ-H)₂], and observed that here its behavior is quite different from that observed with Ru₃(CO)₁₂ which was documented by Wong et al. [\[11\]](#). Thus while the trinuclear framework is always maintained with [osmium](#), the PFu₃ substituted triruthenium clusters [Ru₃(CO)_{12-n}(PFu₃)_n] (*n* = 1, 2) and [Ru₃(CO)₉(PFu₃)₃] afford the furenyl complexes [Ru₂(CO)₆(μ-PFu₂)(μ-η¹,η²-C₄H₃O)] [\[11\]](#), [\[12\]](#) and [Ru₂(CO)₅(PFu₃)(μ-PFu₂)(μ-η¹,η²-C₄H₃O)] [\[12\]](#), respectively on heating by both ruthenium–ruthenium and phosphorus–carbon bond scission. The chemistry of these diruthenium complexes has been extensively studied in the following years [\[11\]](#), [\[12\]](#), [\[13\]](#). As a continuation of our previous work on the reactivity of PFu₃ with transition metal carbonyl complexes, we have now investigated its reactivity with [Mn₂(CO)_{10-n}(NCMe)_n] (*n* = 0–2) the results of which are the subject of this article.

2. Experimental

All manipulations were carried out under an [inert atmosphere](#) of nitrogen using standard [Schlenk techniques](#). Reagent-grade [solvents](#) were dried and distilled by standard methods prior to use. Infrared spectra were recorded on a Shimadzu FTIR 8101 spectrophotometer. [NMR spectra](#) were recorded on Bruker DPX 400 instruments. Elemental analyses were performed by BCSIR Laboratories, Dhaka. Tri(2-furyl)phosphine was purchased from Across Organic and used as received. $\text{Mn}_2(\text{CO})_{10}$ was purchased from Strem Chemicals Inc. and used without further purification and the complexes $[\text{Mn}_2(\text{CO})_9(\text{NCMe})]$ and $[\text{Mn}_2(\text{CO})_8(\text{NCMe})_2]$ were prepared according to the literature methods ^[14]. Preparative thin layer chromatography was carried out on 1 mm plates prepared from [silica gel](#) GF254 (type 60, E. Merck) at Jahangirnagar University.

2.1. Reaction of $[\text{Mn}_2(\text{CO})_9(\text{NCMe})]$ with PFu_3

PFu_3 (84 mg, 0.370 mmol) was added to a CH_2Cl_2 solution (20 mL) of $[\text{Mn}_2(\text{CO})_9(\text{NCMe})]$ (150 mg, 0.372 mmol) and the reaction mixture was then stirred at room temperature for 24 h. The solvent was removed under reduced pressure and the residue chromatographed by TLC on silica gel. Elution with hexane/ CH_2Cl_2 (7:3, v/v) developed two bands. The first band was unreacted $[\text{Mn}_2(\text{CO})_9(\text{NCMe})]$ (trace) while the second band afforded $[\text{Mn}_2(\text{CO})_9(\text{PFu}_3)]$ (**1**) (115 mg, 52%) as yellow crystals from hexane/ CH_2Cl_2 at $-20\text{ }^\circ\text{C}$. Spectral data for **1**: *Anal.* Calc. for $\text{C}_{21}\text{H}_9\text{Mn}_2\text{O}_{12}\text{P}$: C, 42.45; H, 1.53. Found: C, 42.88; H, 1.61%. IR (ν_{CO} , CH_2Cl_2): 2093 m, 2017 m, 1993 vs, 1965 sh, 1945 w cm^{-1} . ^1H NMR (CDCl_3): δ 7.81 (br s, 3H), 7.22 (br s, 3H), 6.63 (br s, 3H). $^{31}\text{P}\{^1\text{H}\}$ NMR (CDCl_3): δ -10.2 (s). MS (m/z): 594 (M^+).

2.2. Reaction of $[\text{Mn}_2(\text{CO})_8(\text{NCMe})_2]$ with PFu_3

To a CH_2Cl_2 solution (20 mL) of $[\text{Mn}_2(\text{CO})_8(\text{NCMe})_2]$ (150 mg, 0.361 mmol) was added PFu_3 (168 mg, 0.724 mmol) and the reaction mixture was then stirred at room temperature for 24 h. A similar [work up](#) and [chromatographic](#) separation described as above developed three bands on TLC plate. The first band was unreacted $[\text{Mn}_2(\text{CO})_8(\text{NCMe})_2]$ (trace) while the second and third bands gave $[\text{Mn}_2(\text{CO})_9(\text{PFu}_3)]$ (**1**) (13 mg, 6%) and $[\text{Mn}_2(\text{CO})_8(\text{PFu}_3)_2]$ (**2**) (43 mg, 15%) as yellow crystals, respectively, from hexane/ CH_2Cl_2 at $-20\text{ }^\circ\text{C}$. Spectral data for **2**: *Anal.* Calc. for $\text{C}_{32}\text{H}_{18}\text{Mn}_2\text{O}_{14}\text{P}_2$: C, 48.14; H, 2.28. Found: C, 48.63; H, 2.41%. IR (ν_{CO} , CH_2Cl_2): 2003 m, 1960 vs cm^{-1} . ^1H NMR (CDCl_3): δ 7.73 (br s, 6H), 6.75 (br s, 6H), 6.50 (br s, 6H). $^{31}\text{P}\{^1\text{H}\}$ NMR (CDCl_3): δ -35.0 (s). MS (m/z): 798 (M^+).

2.3. Reaction of $\text{Mn}_2(\text{CO})_{10}$ with PFu_3

A [toluene](#) solution (20 mL) of $\text{Mn}_2(\text{CO})_{10}$ (150 mg, 0.256 mmol) and PFu_3 (120 mg, 0.519 mmol) was heated to reflux for 4 h. The volatiles were removed by [rotary evaporation](#) and the residue was separated by TLC on silica gel. Elution with hexane/ CH_2Cl_2 (7:3, v/v) developed four bands. The third and fourth bands was unreacted $\text{Mn}_2(\text{CO})_{10}$ (trace) and PFu_3 (trace) while the second and third bands gave *mer*- $[\text{Mn}(\text{CO})_3(\eta^1\text{-C}_4\text{H}_3\text{O})(\text{PFu}_3)_2]$ (**3**) (31 mg, 18%) and $[\text{Mn}_2(\text{CO})_8(\text{PFu}_3)_2]$ (**2**) (60 mg, 29%) as yellow crystals, respectively, from

hexane/CH₂Cl₂ at -20 °C. Spectral data for **3**: *Anal.* Calc. for C₃₂H₂₁MnO₁₀P₂: C, 55.54; H, 3.16. Found: C, 56.03; H, 3.28%. IR (ν_{CO} , CH₂Cl₂): 2040 w, 1959 vs, 1934 s cm⁻¹. ¹H NMR (CDCl₃): major [isomer](#): δ 7.66 (br s, 1H), 7.62 (m, 7H), 6.49 (br s, 1H), 6.44 (br s, 6H), 6.37 (br s, 6H); minor isomer: δ 7.37 (br s, 3H), 6.55 (br s, 3H), 6.10 (m, 9H), 5.86 (m, 6H). ³¹P{¹H} NMR (CDCl₃): major isomer: δ -29.6 (s); minor isomer: δ -26.5 (d, J = 36 Hz, 1P), -29.9 (d, J = 36 Hz, 1P). MS (m/z): 670 (M⁺).

2.4. X-ray crystallography

[Single crystals](#) **3** suitable for [X-ray diffraction](#) were obtained by recrystallization from hexane/CH₂Cl₂ at -20 °C and mounted on Nylon fibers with a [mineral oil](#), and diffraction data were collected at 100(2) K on a Bruker AXS SMART diffractometer equipped with an Bruker APEX2 CCD detector using graphite-monochromated Cu K α radiation (λ = 1.54178 Å). Integration of intensities and data reduction was performed using the saint program [\[15\]](#). Numerical (based on the real shape of the crystals) absorption correction was applied in all cases followed by the multi-scan sadabs procedure [\[16\]](#). The structures were solved by direct methods [\[17\]](#) and refined by full-matrix least squares on F^2 [\[18\]](#). All non-hydrogen atoms were refined anisotropically. The details of the data collection and structure refinement are given in [Table 1](#).

Table 1. Crystallographic data and structure refinement for **3**.

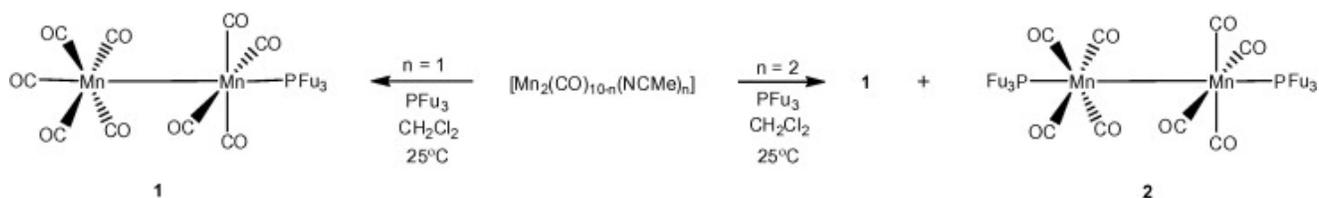
Compound	3
Empirical formula	C ₃₁ H ₂₁ MnO ₁₀ P ₂
Formula weight	670.36
Temperature (K)	100(2)
Wavelength (Å)	1.54178
Crystal system	monoclinic
Space group	$P 2_1/n$
Unit cell dimensions	
a (Å)	8.5311(4)
b (Å)	23.417(1)
c (Å)	14.4346(7)
α (°)	90
β (°)	90.390(2)
γ (°)	90
V (Å ³)	2883.6(2)
Z	4
D_{calc} (mg/m ³)	1.544
Absorption coefficient (mm ⁻¹)	5.299

Compound	3
$F(0\ 0\ 0)$	1368
Crystal size (mm ³)	0.24 × 0.13 × 0.11
θ range for data collection (°)	3.60–61.32
Index ranges	$-9 \leq h \leq 9, 0 \leq k \leq 26, 0 \leq l \leq 16$
Reflections collected	23 622
Independent reflections (R_{int})	4377 (0.0318)
Refinement method	full-matrix least-squares on F^2
Maximum and minimum transmission	0.5933 and 0.3628
Data/restraints/parameters	4377/0/481
Goodness-of-fit on F^2	1.038
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0316, wR_2 = 0.0777$
R indices (all data)	$R_1 = 0.0371, wR_2 = 0.0799$
Largest difference in peak and hole (e Å ⁻³)	0.304 and -0.247

3. Results and discussion

3.1. Reactions of $[\text{Mn}_2(\text{CO})_{10-n}(\text{NCMe})_n]$ ($n = 1, 2$) with PFu_3

Reaction between $[\text{Mn}_2(\text{CO})_9(\text{NCMe})]$ and PFu_3 at room temperature yields the mono-phosphine substituted product $[\text{Mn}_2(\text{CO})_9(\text{PFu}_3)]$ (**1**) in 52% yield ([Scheme 1](#)). The carbonyl region of the [IR spectrum](#) is very similar to those of other mono-phosphine substituted compounds $[\text{Mn}_2(\text{CO})_9(\text{phosphine})]$ [[19](#), [20](#), [21](#), [22](#)] and the [mass spectrum](#) shows a parent [molecular ion](#) peak at m/z 594 and ions due to successive loss of nine carbonyls. The $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum shows a singlet at δ -10.2, while the ^1H NMR spectrum has three equal intensity broad singlets at δ 7.81, 7.22, and 6.63 attributed to the furyl-ring protons.



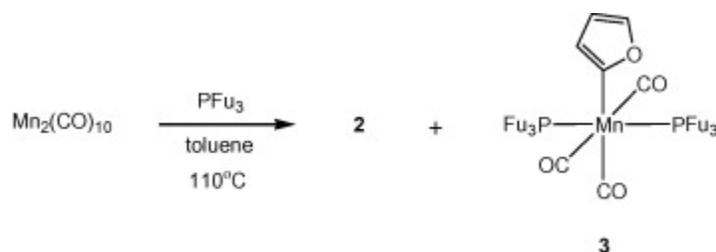
Scheme 1. Reactions of $[\text{Mn}_2(\text{CO})_{10-n}(\text{NCMe})_n]$ ($n = 1, 2$) with PFu_3 .

A similar reaction between $[\text{Mn}_2(\text{CO})_8(\text{NCMe})_2]$ and PFu_3 gives both **1** and the bis-phosphine product $[\text{Mn}_2(\text{CO})_8(\text{PFu}_3)_2]$ (**2**) in 6% and 15% yields, respectively, ([Scheme 1](#)). The pattern of the carbonyl stretching region of the infrared spectrum are similar to other $[\text{Mn}_2(\text{CO})_8(\text{phosphine})_2]$ [[23](#)] complexes containing two phosphines at axial positions while the mass spectrum shows a parent molecular ion peak at m/z 798 together with other ions due to stepwise loss of all eight carbonyls. The ^1H NMR spectrum shows only aromatic resonances attributable to the furyl ring protons while the $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum exhibits a singlet at δ

-35.0 due to two magnetically equivalent [phosphorus atoms](#) present in the molecule. A preliminary [X-ray structure](#) confirmed this assignment, **2** being isostructural with the analogous [rhenium](#) complex $[\text{Re}_2(\text{CO})_8(\text{PFu}_3)_2]$ [\[19\]](#). Unfortunately the major disorder of oxygen and furyl sites in the ligands and also the rotational positions of the furyl rings lead to a poor overall structural model. Further information is given in the electronic supplementary information.

3.2. Direct reaction between $\text{Mn}_2(\text{CO})_{10}$ and PFu_3 : carbon–phosphorus bond cleavage

Thermal treatment of $\text{Mn}_2(\text{CO})_{10}$ and PFu_3 in boiling [toluene](#) afforded **2** together with [mononuclear](#) *mer*- $[\text{Mn}(\text{CO})_3(\eta^1\text{-C}_4\text{H}_3\text{O})(\text{PFu}_3)_2]$ (**3**) in 29% and 18% yields, respectively, ([Scheme 2](#)). Complex **3** was characterized by a combination of [spectroscopic data](#) and single crystal X-ray diffraction analysis.



Scheme 2. Direct reaction between $\text{Mn}_2(\text{CO})_{10}$ and PFu_3 .

The solid-state molecular structure of **3** is depicted in [Fig. 1](#) with selected inter-atomic distances and angles listed in the figure caption. The molecule consists of a single [manganese](#) atom ligated by three carbonyls, two PFu_3 and one 2-furyl ligands. The coordination geometry around the manganese atom is a distorted [octahedron](#) with three carbonyls arranged in a *meridional* fashion. The distortion from octahedral coordination geometry is evident from reduction of the *trans*-OC–Mn–CO angle from 180° in the perfect octahedron to $169.76(10)^\circ$ in **3**. The two PFu_3 ligands are mutually *trans* to each other while the 2-furyl ligand is coordinated to the manganese atom in η^1 fashion. The Mn–P distances [Mn(1)–P(1) 2.2594(7) and Mn(1)–P(2) 2.2597(7) Å] observed in **3** are the same to those found in **2** [2.2594(7) Å]. The Mn(1)–C(4) covalent distance [2.050(2) Å] is also very similar to those reported in literature for related complexes [\[19\]](#), [\[21\]](#), [\[22\]](#).

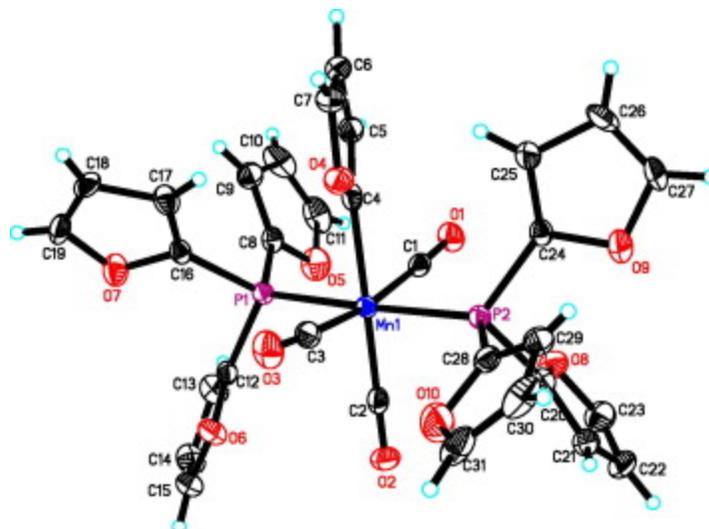


Fig. 1. Molecular structure of *mer*-[Mn(CO)₃(η¹-C₄H₃O)(PFu₃)₂] (**3**). Selected bond distances (Å) and angles (°): Mn(1)–P(1) 2.2594(7), Mn(1)–P(2) 2.2597(7), Mn(1)–C(1) 1.837(3), Mn(1)–C(2) 1.815(3), Mn(1)–C(3) 1.855(3), Mn(1)–C(4) 2.050(2), P(1)–Mn(1)–P(2) 177.17(3), P(1)–Mn(1)–C(4) 87.24(6), P(2)–Mn(1)–C(4) 91.52(6), P(1)–Mn(1)–C(1) 90.94(7), P(2)–Mn(1)–C(1) 86.37(7), C(1)–Mn(1)–C(3) 169.76(10), C(1)–Mn(1)–C(2) 95.15(10), C(1)–Mn(1)–C(4) 83.92(10), C(2)–Mn(1)–C(4) 178.35(10).

The [NMR spectra](#) of compound **3** show that it exists in two isomeric forms in solution. Thus the ¹H NMR spectrum displays two distinct set of resonances in the aromatic region. The resonances at δ 7.66, 7.62, 6.49, 6.44 and 6.37 are due to the major [isomer](#) while those at δ 7.37, 6.55, 6.10 and 5.86 are assigned to the minor isomer. Consistent with this the ³¹P{¹H} NMR spectrum displays a singlet at δ –29.6 for the major isomer and two doublets at δ –26.5 (*J* = 36 Hz) and –29.9 (*J* = 36 Hz) for the minor isomer in 4:1:1 intensity ratio. A total of three isomers (**3a**, **3b** and **3c** shown in [Chart 1](#)) are possible for compound **3**. We assume that the isomer observed in the solid-state is the major isomer (**3a**) in solution as we should observed only a singlet on the ³¹P{¹H} NMR spectrum for it. The two phosphorus atoms in **3b** are magnetically non-equivalent while those of **3c** are magnetically equivalent for which we should also get a singlet on the ³¹P{¹H} NMR spectrum. Thus the presence of two doublets ruled out isomer **3c**, so **3b** is likely to be the minor isomer of compound **3** in solution.

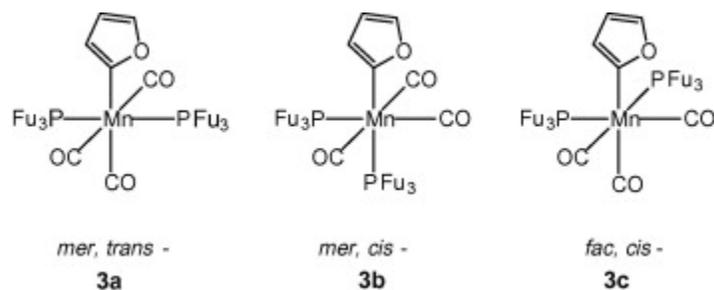


Chart 1. Possible [isomers](#) of **3**.

Bis-phosphine substituted mononuclear carbonyl complexes of manganese and rhenium are not uncommon in literature, but complexes of the type $[M(\text{CO})_3(\eta^1\text{-R})(\text{PR}'_3)_2]$ ($M = \text{Mn}, \text{Re}$) are rare [24], [25], [26], [27], [28]. Examples of complexes having similar structures include the rhenium complexes *fac*- $[\text{Re}(\text{CO})_3(\eta^1\text{-C}_5\text{H}_7)(\text{PR}_3)_2]$ ($R = \text{Me}, \text{Et}$) [24] and *fac*- $[\text{Re}(\text{CO})_3(\eta^1\text{-C}_5\text{H}_5)(\text{PMe}_3)_2]$ [25] which have structures akin to **3c**, and the manganese complexes *mer*- $[\text{Mn}(\text{CO})_3(\eta^1\text{-fluorenyl})(\text{PR}_3)_2]$ ($R = \text{Et}, \text{Bu}$) [26], *mer*- $[\text{Mn}(\text{CO})_3(\eta^1\text{-cpp})(\text{PEt}_3)_2]$ (cppH = cyclopenta[def]phenanthrene) (**D**) [27], and *mer*- $[\text{Mn}(\text{CO})_3(\eta^1\text{-CCPh})\{\text{P}(\text{OPh}_3)\}_2]$ (**E**) [28] whose structures are similar to **3a**. The later two manganese complexes (**D** and **E**) were crystallographically characterized and line drawings of their molecular structures are presented in Chart 2 together with Mn–P and Mn–C (involving hydrocarbonyl ligand) bond distances. Among the three structurally characterized $[\text{Mn}(\text{CO})_3(\eta^1\text{-R})(\text{PR}'_3)_2]$ complexes, the Mn–P and Mn–C bond distances of complex **D** that contains a bulky cpp group are significantly longer than those found in **E** and **3**. The lengthening of Mn–P bonds can be easily explained by the presence of more sterically demanding bulky cpp ligand in **D**, but it does not alone explain the unusual lengthening of Mn–C bond (ca. 0.25 Å) in this complex compare to **E** and **3**. In complex **D**, the carbon atom of the cpp ligand through which it is bonded to Mn is a sp^3 hybrid carbon whereas the organic groups in **E** and **3** used either sp or sp^2 hybrid carbon to form bond with manganese. As a result of both these effects, the length of the Mn–C bond in **D** is unusually long compare to **E** and **3**.

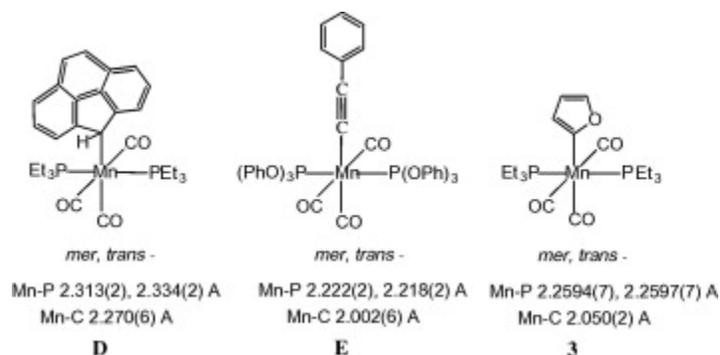


Chart 2. Examples of $[\text{Mn}(\text{CO})_3(\eta^1\text{-R})(\text{PR}'_3)_2]$ complexes.

4. Conclusions

In summary, three new manganese complexes have been synthesized and characterized in the present study. The reactions between the labile acetonitrile complexes $[\text{Mn}_2(\text{CO})_{10-n}(\text{NCMe})_n]$ ($n = 1, 2$) and PFu_3 give only simple phosphine-substituted complexes **1** and **2** at ambient temperature whereas compound **3** which contains a $\eta^1\text{-C}_4\text{H}_3\text{O}$ ligand is formed at high temperature by carbon–phosphorus bond cleavage of the PFu_3 ligand. The fate of the difurylphosphido (PFu_2) moiety is remained unknown from the present study.

Acknowledgements

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