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Synthesis and solid-state structures of (triphos)iridacyclopentadiene complexes as models for vinylidene intermediates in the [2 + 2 + 1] cyclotrimerization of alkynes

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His coauthors dedicate this manuscript to Prof. Arnold L. Rheingold: friend, colleague and scholar

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1. Introduction

We previously reported that the reactions of iridacyclopentadiene complexes with terminal alkynes depend in a dramatic way on the nature of the ancillary ligands (Scheme 1). For example, in the bis(triphenylphosphine)iridacyclopentadiene complex [{ $\kappa^2(C^1, C^4)$ - $CR=CRCR=CR{(PPh_3)_2Ir(NCMe)(CO)]BF_4$ (1), the acetonitrile and κ^2 -butadiendiyl ligands occupy meridional coordination sites [1,2]; whereas, in triphos complex { $\kappa^2(C^1,C^4)$ -CR=CRCR=CR}(triphos)Ir(NCMe)]BF₄ (**2**-*NCMe*) the acetonitrile and κ^2 -butadiendiyl ligands occupy facial coordination sites (Scheme 1) [3]. Metallacycle **1** undergoes reaction with phenylacetylene to give the bicyclic metallalactone complex 3 [1]; whereas, 2-NCMe undergoes reaction with phenylacetylene to give fulvene complex **4** [3,4]. In both cases, the reaction mechanism appears to involve the formation of a vinylidene intermediate (I and II). In the case of I, the vinylidene ligand is trapped by the metallacycle ester substituent. For II, the vinylidene ligand undergoes a reductive coupling with the butadiendiyl ligand.

ABSTRACT

The iridium 1,1,1-tris(diphenylphosphinomethyl)ethane (triphos) complexes [{ $\kappa^2(C^1,C^4)$ -CR=CRCR=CR}-{CH_3C(CH_2PPh_2)_3}]r(NCMe)]BF_4 (**2**-*NCMe*, R = CO_2Me) and [{ $\kappa^2(C^1,C^4)$ -CR=CRCR=CR}{CH_3C(CH_2PPh_2)_3}]r(CO)]BF_4 (**2**-*CO*, R = CO_2Me) serve as models for proposed iridium–vinylidene intermediates of relevance to the [2 + 2 + 1] cyclotrimerization of alkynes. The solid-state structures of **2**-*NCMe*, **2**-*CO*, and [$\kappa^2(C^1,C^4)$ -CR=CRCR=CR]{CH_3C(CH_2PPh_2)_3}]r(Cl) (**2**-*Cl*), were determined by X-ray crystallography.

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In order to better understand the structural parameters in intermediates of type **II**, we have now structurally characterized both **2**-*NCMe* and the carbon monoxide analogue, [{ $\kappa^2(C^1,C^4)$ -CR=CRCR=CR}{CH_3C(CH_2PPh_2)_3}Ir(CO)]BF_4 (**2**-*CO*, R = CO₂Me), both of which may be viewed as structural models for the presumed vinylidene intermediate **II**. In addition, we report the solid-state structure of [{ $\kappa^2(C^1,C^4)$ -CR=CRCR=CR}{CH_3C(CH_2PPh_2)_3}Ir(CI)]BF_4 (**2**-*Cl*, R = CO₂Me).

2. Results and discussion

The reaction of $[\kappa^2(C^1,C^4)-CR=CRCR=CR](PPh_3)_2Ir(CI)$ (5, $R = CO_2Me)$ [5] with 1,1,1-tris(diphenylphosphinomethyl)ethane (triphos) has previously been demonstrated to give the neutral iridacycle–chlorido complex $[\kappa^2(C^1,C^4)-CR=CRCR=CR](triphos)$ Ir(CI) (2-*Cl*, $R = CO_2Me)$ [3] and upon subsequent reaction with AgBF₄ in acetonitrile, the cationic acetonitrile complex 2-*NCMe* (Scheme 2) [6]. Alternatively, treatment of **5** with bis(2-diphenylphosphinoethyl)phenylphosphine generated the facial tris(phosphine) complex $[\kappa^2(C^1,C^4)-CR=CRCR=CR]{PhP(CH_2CH_2PPh_2)_2}$ Ir(CI) (**6**-*Cl*, $R = CO_2Me$) [7]. Subsequent halide abstraction and exposure to CO gave the cationic meridional complex $[{\kappa^2(C^1,C^4)-CR=CRCR=CR}]{PhP(CH_2CH_2PPh_2)_2}Ir(CO)]BF_4$ (**7**-*CO*, $R = CO_2Me$).



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Scheme 1. The effect of ancillary ligands on the reactions of iridacyclopentadiene complexes.

When CO is bubbled through a dichloromethane solution of **2**-*NCMe*, followed by stirring under an atmosphere of CO for 24 h, the cationic carbonyl complex **2**-*CO* is formed in 86% isolated yield. In the IR spectrum (KBr) of **2**-*CO* the CO stretching vibration (2094 cm⁻¹) is at significantly higher wavenumber than for **7** (2069 cm⁻¹, KBr), which indicates that the trans phosphine ligand is a better donor ligand than the trans κ^2 -butadiendiyl ligand. In [{ $\kappa^2(C^1,C^4)$ -CR=CRCR=CR}(PPh_3)_2Ir(CO)_2]BF₄ (**8**, R = CO₂Me), the CO stretching vibrations are also observed at higher wavenumber (2080, 2115 cm⁻¹; CH₂Cl₂) than for **2**-*CO* [2c]. Thus, the vinylidene

ligand in intermediate **II** should be stabilized by π -backbonding to a greater extent than the vinylidene ligand in **I** (Scheme 1).

In the ¹H NMR spectrum (CDCl₃) of **2**-*CO*, the hydrogen resonances for the methoxycarbonyl groups are observed as singlets at δ 3.86 (6H) and 2.64 (6H). These values are very similar to those observed for the triphos complexes **2**-*NCMe* (δ 3.76 and 2.67) and **2**-*Cl* (δ 3.70 and 2.52). In contrast, the bis(phosphine) complexes, **1** (δ 3.50 and 3.49), **5** (δ 3.46 and 3.26), and **8** (δ 3.52 and 3.09) exhibit no methyl hydrogen resonances at higher field than δ 3.0. The tris(phosphine) complex **6** also exhibits relatively downfield



Scheme 2. Synthesis of iridacyclopentadiene complexes with tridentate phosphine ligands.



Fig. 1. Structural drawing of 2-NCMe (most hydrogen atoms and BF₄⁻ counter ion omitted for clarity).

Table 1		
Selected bond distances () for 2 -NCMe, 2 -CO, and 2	2 -Cl.

A-B	2 -NCMe	2 -CO	2 -Cl
Ir-C(1)	2.117(11)	2.076(6)	2.117(4)
Ir-C(4)	2.092(12)	2.109(7)	2.116(5)
Ir-N(1)	2.053(12)		
Ir-C(18)		1.891(7)	
Ir-Cl			2.411(1)
Ir-P(1)	2.410(3)	2.414(2)	2.403(1)
Ir-P(2)	2.326(3)	2.416(2)	2.309(1)
Ir-P(3)	2.426(3)	2.413(2)	2.386(1)
N(1)-C(18)	1.155(18)		
C(18)-C(19)	1.432(20)		
C(1)-C(2)	1.357(17)	1.348(9)	1.340(6)
C(2) - C(3)	1.473(12)	1.446(7)	1.451(7)
C(3) - C(4)	1.376(18)	1.340(10)	1.361(6)
C(1) - C(8)	1.477(13)	1.514(6)	1.469(7)
C(2) - C(7)	1.459(16)	1.516(10)	1.496(6)
C(3) - C(6)	1.532(19)	1.512(9)	1.482(7)
C(4) - C(5)	1.512(14)	1.509(8)	1.481(7)
C(8) - O(1)	1.200(14)	1.203(9)	1.210(5)
C(8) - O(2)	1.348(15)	1.329(11)	1.348(5)
C(7) - O(3)	1.229(14)	1.205(9)	1.206(5)
C(7) - O(4)	1.339(16)	1.316(10)	1.331(6)
C(6) - O(5)	1.202(20)	1.201(10)	1.211(6)
C(6) - O(6)	1.343(17)	1.326(10)	1.343(6)
C(5) - O(7)	1.221(14)	1.188(11)	1.210(5)
C(5)-O(8)	1.327(16)	1.329(7)	1.327(6)
C(18)-O(9)		1.143(9)	

methyl hydrogen chemical shifts at δ 3.86, 3.54, 3.48, and 3.32. These chemical shift comparisons suggest that the triphos ligand adopts a favored conformation in which one or more of the phenyl groups shields the α -methoxycarbonyl hydrogens.

In order to understand the steric parameters in intermediate **II**, X-ray crystallographic analyses were performed on the model complexes **2**-*NCMe* and **2**-*CO* (Figs. 1 and 2, Tables 1 and 2). Both structures exhibit a distorted octahedral geometry about iridium with C(1)–Ir–C(4) angles constrained by the ring structure to 77.0(2)–78.4(5)°. The P(1)–Ir–P(3) angles are significantly larger at 85.6(1)–86.2(1)°. The axial ligands are bent away from Ph^A and Ph^E with Ir–N(1)–C(18) and Ir–C(18)–O(1) angles in **2**-*NCMe* and **2**-*CO* of 175.9(10)° and 172.9(6)°, respectively. The largest deviation of the metallacycle ring atoms from the Ir–C(1)–C(2)–C(3)–C(4) mean plane is 0.04(1) Å at C(4) in **2**-*NCMe* and 0.04(1) Å at C(1) in **2**-*CO*. Within the metallacycle ring, the C(1)–

Table 2							
Selected bond	angles ((°) for	2 -NCMe	and	2 -CO,	and 2	2 -Cl.

А-В-С	2 -NCMe	2 -CO	2 -Cl
C(1)– Ir – $C(4)$	78.5(4)	77.0(2)	77.8(2)
C(1)-Ir-N(1)	84.8(4)		
C(1)-Ir- $C(18)$		81.2(3)	
C(1)-Ir-Cl			83.3(1)
C(1) - Ir - P(1)	98.0(3)	97.7(2)	97.1(1)
C(1)-Ir- $P(2)$	93.9(3)	95.6(2)	97.7(1)
C(1)-Ir- $P(3)$	176.0(3)	173.5(2)	172.5(1)
C(4)-Ir-N(1)	82.4(4)		
C(4)-Ir- $C(18)$		81.6(3)	
C(4)–Ir–Cl			81.4(1)
C(4)-Ir- $P(1)$	172.2(3)	174.0(1)	174.9(1)
C(4)-Ir-P(2)	96.8(3)	95.7(2)	91.9(1)
C(4)-Ir- $P(3)$	97.7(3)	99.0(2)	99.5(1)
N(1)-Ir-P(1)	90.4(3)		
N(1)-Ir-P(2)	178.5(3)		
N(1)-Ir-P(3)	93.5(3)		
C(18)-Ir- $P(1)$		95.0(2)	
C(18)– Ir – $P(2)$		176.2(2)	
C(18)–Ir–P(3)		93.3(2)	
Cl-Ir-P(1)			97.1(1)
Cl-Ir-P(2)			97.7(1)
Cl-Ir-P(3)			172.5(1)
P(1)– Ir – $P(2)$	90.3(1)	87.4(1)	88.9(1)
P(1)– Ir – $P(3)$	85.6(1)	86.2(1)	85.6(1)
P(2)-Ir-P(3)	89.8(1)	89.8(1)	89.4(1)
Ir-C(1)-C(2)	114.7(6)	116.7(4)	114.8(4)
Ir - C(1) - C(8)	128.8(10)	124.5(5)	129.4(3)
Ir-C(4)-C(3)	114.8(7)	115.1(4)	114.4(4)
Ir - C(4) - C(5)	130.8(9)	126.8(6)	129.1(3)
C(1)-C(2)-C(3)	115.8(10)	114.8(6)	116.8(4)
C(2)-C(3)-C(4)	115.8(10)	116.2(6)	116.1(4)

C(2) and C(3)–C(4) bond distances are consistent with localized carbon–carbon double bonds. There is a relatively large displacement of the two ester substituents on the α -carbons of the metal-lacycle from the mean plane of the metallacycle ring, in a direction that is away from the P(2) phenyls which extend under the metal-lacycle ring. Thus, in **2**-*NCMe* the carbonyl carbons exhibit the following displacements from the mean plane of the metallacycle ring: C(5) – 0.49(1), C(6) – 0.04(2), C(7) – 0.04(1), and C(8) – 0.24(1) Å. For **2**-CO the carbonyl carbon displacements from the mean plane of the metallacycle ring are C(5) – 0.276(8), C(6) – 0.097(8), C(7) – 0.094(8), and C(8) – 0.547(8) Å. The α -methoxycar-



Fig. 2. Sructural drawings of 2-CO (most hydrogen atoms and BF₄⁻ counter ion omitted for clarity).

bonyl metallacycle-substituents are positioned in the shielding cone of Ph^B and Ph^F, which is consistent with the relatively upfield chemical shift observed for the ester methyl hydrogens in the ¹H NMR spectra.

The Ph^A and Ph^E phenyl rings in **2**-CO and **2**-NCMe extend above the metallacycle plane and adopt conformations in which the dihedral angle between the two six-member rings is 32.1(2)° and 35.5(2)°, respectively (see the second structure in Figs. 1 and 2). These angles generate a slight wedge shape with the open end directed toward the axial CO/NCMe ligand and result in relatively short non-bonded distances between the axial CO/NCMe ligands and an ortho-carbon on each of the phenvls. For **2**-NCMe, the $C(6A) \cdots N$ and $C(6E) \cdots N$ distances are 3.053(11) and 3.148(12) Å. respectively. With hydrogen atoms placed in idealized positions. the $H(6A) \cdots N(1)$ and $H(6E) \cdots N(1)$ non-covalent bond distances in 2-NCMe are 2.82 and 2.64 Å. Similar interactions are observed for the ortho-carbons of Ph^{A} and Ph^{E} in **2**-CO. The C(6A) \cdots C(18) and C(6E)...C(18) distances are 3.083(9) and 3.140(10) Å, and the $H(6A) \cdots C(18)$ and $H(6E) \cdots C(18)$ non-covalent bond distances are 2.61 and 2.81 Å, respectively. Thus, the hydrogens are directed toward the π -system of the C–N and C–O triple bonds. There are also relatively short distances between the same ortho-carbons (hydrogens) of Ph^A/Ph^E and an oxygen atom in each of the two α -methoxycarbonyl ring-substituents. In **2**-NCMe, the two α -methoxycarbonyls adopt a conformation that places O(1) at a 2.30 Å distance from H(6A) and O(8) at a 2.48 Å distance from H(6E). The C(6A)-H(6A)-O(1)/C(6E)-H(6E)-O(8) angles are $159.1^{\circ}/146.6^{\circ}$ for 2-NCMe and the C(6A)-H(6A)-O(1)/C(6E)-H(6E)-O(7) angles are $147.4^{\circ}/160.9^{\circ}$ for **2**-CO. In **2**-CO, the O(1)···H(6A) and O(7)···H(6E) distances are very short at 2.51 and 2.20 Å, respectively. These O...H distances are well within the sum of the van der Waals radii for hydrogen and oxygen (2.62 Å) and taken together with the C-H-O angles indicate that these are moderate to weak hydrogen-bonding interactions [8].

The triphos Ph^C and Ph^D rings extend under the metallacycle with the following closest non-bonded distances: For **2**-*NCMe*; $H(2C)\cdots C(3) 2.72$, $H(2C)\cdots C(4) 2.61$, $H(2D)\cdots C(1) 2.71$, $H(2D)\cdots C(2) 2.45$. For **2**-*CO*; $H(2C)\cdots C(3) 2.56$, $H(2C)\cdots C(4) 2.51$, $H(2D)\cdots C(1) 2.75$, $H(2D)\cdots C(2) 2.85$ Å. As shown in Fig. 3, these non-bonded interactions between the triphos ligand and the butadiendiyl and axial CO ligands results in relatively small 83° and 80° angles between the axial ligand (NCMe/CO) and the centroid



Fig. 3. Angles between the axial ligands and the centroids of the C(1)-Ir-C(4) and P(1)-Ir-P(3) mean-planes.

of the metallacycle ring in **2**-*NCMe* and **2**-*CO* (Fig. 3). For **2**-*CO*, these small angles result in a $C(1) \cdots C(18)$ and $C(4) \cdots C(18)$ non-bonded distances of 2.586(10) and 2.619(8) Å.

Whereas **2**-*NCMe* and **2**-*CO* are similar in terms of bond and torsion angles, the structure of **2**-*Cl* exhibits much different conformational preferences with respect to the iridium–phosphorus and phosphorus–carbon bonds. The C(1A)–P(1)–Ir–N dihedral angles are $9.6(3)^{\circ}$ (**2**-*NCMe*), $30.8(3)^{\circ}$ (**2**-*CO*) and $86.8(2)^{\circ}$ (**2**-*Cl*), and the C(1E)–P(3)–Ir–L (L=N, C(18), Cl) dihedral angles are $-28.4(4)^{\circ}$ (**2**-*NCMe*), $-12.0(3)^{\circ}$ (**2**-*CO*), and $-56.7(10)^{\circ}$ (**2**-*Cl*). Most apparent from viewing the right hand structure in Fig. 4 is the dihedral angle between the mean planes of the Ph^A and Ph^E rings, which at $47.5(1)^{\circ}$, is much larger than in the case of **2**-*NCMe* ($32.1(2)^{\circ}$) and **2**-*CO* ($35.5(2)^{\circ}$). Along with these changes in torsion angles, the non-covalent bond distances between Cl and the ortho-carbons



Fig. 4. Structural drawings of 2-Cl (most hydrogen atoms omitted for clarity).

of Ph^A and Ph^E are significantly longer than the corresponding distances to the axial ligand in **2**-*NCMe* and **2**-*C*0: C(6A)···Cl 3.391(5), C(6E)···Cl 3.296(4) Å. The corresponding ortho-hydrogen – chlorido distances however are similar to the hydrogen-axial ligand (N or C(18)) interactions in **2**-*NCMe* and **2**-*C*0: H(6A)···Cl 2.783 and H(6E)···Cl 2.57 Å, which are within the 2.85 Å sum of the van der Waals radii for hydrogen and chlorine. The chlorido ligand of M–Cl bonds is highly polar and X–H···Cl–M hydrogen bonds in crystals typically have an angular range of 80–140° [8]. In the case of **2**-*Cl*, the Ir–Cl–H(6A) and Ir–Cl–H(6E) angles are 89° and 87°, respectively. There is also a short hydrogen-bonding interaction between H(6A) and O(2) of 2.28 Å in **2**-*Cl*.

3. Conclusions

The most important structural feature of these triphos complexes, with respect to the [2 + 2 + 1] cyclotrimerization of alkynes, is summarized in Fig. 3 where it is demonstrated that the α -carbons of the butadiendiyl ligand and the axial ligand (CO, NCMe, Cl), which occupies the site of the vinylidene ligand in **II** (Scheme 1), are brought into close proximity as a result of the bulky triphos ancillary ligand. Such close proximity may facilitate the reductive coupling of the butadiendiyl and vinylidene ligands to give the observed fulvene products. This suggests that a less bulky triphos analogue would lead to slower reductive coupling and perhaps allow for the observation of the vinylidene intermediate by IR or NMR spectroscopy. Efforts to obtain X-ray crystallographic analyses of related metallacyclopentadiene complexes with alternative, sterically less-demanding tridentate phosphine ligands, as well as the trispyrazolylborate (Tp) ligand, have yet to be successful.

4. Experimental section

All reactions and reaction workups were performed in the air unless otherwise noted. Organic solvents were obtained commercially and dried over either calcium hydride or sodium metal. IR spectra were recorded on a Nicolet Magna-IR 550 FTIR spectrometer. ¹H and ¹³C NMR spectra were taken on Varian Hg 300 (76 MHz), Hg 400 (101 MHz), or UN 500 (126 MHz) spectrometers. ¹H and ¹³C chemical shifts were referenced to residual protio-solvent signals. FAB mass spectra were determined at the University of California, Riverside Mass Spectrometry Facility. Elemental analyses were performed by NuMega Resonance Labs Inc. of San Diego, California.

Preparation of $[{\kappa^2(C,C)-CR=CRCR=CR}{CH_3C(CH_2PPh_2)_3}$ Ir-(CO)]BF₄ (**2**-CO, R = CO₂Me).

A 100-mL flask equipped with a magnetic stir bar was charged with $[{\kappa^2(C,C)-CR=CRCR=CR}{CH_3C(CH_2PPh_2)_3}Ir(NCMe)]BF_4$ (2-NCMe, $R = CO_2CH_3$; 199.7 mg, 0.16 mmol) and wet CH_2Cl_2 (30 mL). Carbon monoxide gas was bubbled through the solution for 30 min, and the solution was allowed to stir at rt under an atmosphere of CO for 24 h. The volatiles were removed under vacuum and the residue was recrystallized from CH₂Cl₂/Et₂O to give 2-CO as an off-white powder in 86% yield (171.3 mg). IR (KBr): 3066 (w), 2952 (w), 2094 (s), 1724 (s), 1703 (s), 1437 (s), 1333 (w), 1236 (s) cm⁻¹; ¹H NMR (CDCl₃, 400 MHz): δ 8.06 (t, 4H, J = 4.8 Hz), 7.68 (s, 4H), 7.56 (s, 7H), 7.32 (s, 5H), 7.17 (t, 2H, J = 7.2 Hz), 6.84 (t, 4H, J = 7.2 Hz), 6.15 (dd, 4H, J = 11.1, 8.1 Hz), 3.8 (s, 6H), 3.13 (m, 4H), 2.64 (s, 6H), 2.12 (dd, 2H, J = 13.2, 3.6 Hz), 1.83 (d, 3H, J = 1.5 Hz); ¹³C NMR (CDCl₃, 100 MHz): δ 170.68 (m), 165.61, 164.40, 152.44, 141.03 (t, *J* = 8.2 Hz), 140.29 (t, *J* = 7.7 Hz), 132.36 (t, *J* = 2.2 Hz), 132.13, 132.02 (m), 131.81, 131.57, 131.51 (m), 130.58 (d, J = 54.6 Hz), 130.44, 129.81 (d, J = 10.9 Hz), 129.36 (t, J = 5.4 Hz), 128.67 (t, J = 5.5 Hz), 127.80 (m), 127.23 (m), 52.43, 51.51, 38.59, 38.43 (m), 33.61 (m), 26.54 (d, J = 32.3 Hz). Anal. Calc. for C₅₄H₅₁O₉IrP₃BF₄: C, 53.34; H, 4.22. Found: C, 52.97; H, 3.92%.

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Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.ica.2010.07.052.

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