Table I. Reduction of Carbonyl Compounds by H_2/[1,5HDRhCl]$_2$/β-Cyclodextrin/THF

reactant	reaction time, h	product[a]	yield,[b] %
$PhCOCH_3$	8	PhC_2H_5	86[c]
		$C_6H_{11}C_2H_5$	14
$PhCOC_2H_5$	16	PhC_3H_7-*n*	75[d]
		$C_6H_{11}C_3H_7$-*n*	24
$PhCOC_3H_7$	24	$C_6H_{11}C_4H_9$-*n*	42
		$C_6H_{11}C_4H_9$-*n*	58
o-$CH_3C_6H_4COCH_3$	24	*o*-$CH_3C_6H_4C_2H_5$	92
m-$CH_3C_6H_4COCH_3$	24	*m*-$CH_3C_6H_4C_2H_5$	85
		1-ethyl-3-methylcyclohexane	15
o-$CH_3OC_6H_4COCH_3$	24	*o*-$CH_3OC_6H_4C_2H_5$	98
m-$CH_3OC_6H_4COCH_3$	24	*m*-$CH_3OC_6H_4C_2H_5$	96
p-$CH_3OC_6H_4COCH_3$	24	*p*-$CH_3OC_6H_4C_2H_5$	88
$PhCOCH_2OCH_3$	17	$PhCH_2CH_2OCH_3$	29
		$PhCH(OH)CH_2OCH_3$	71
1-tetralone	24	tetralin	93
		decalin	7
6-methoxy-1-tetralone	24	6-methoxytetralin	49
PhCHO	18	toluene	93[e]
o-$CH_3OC_6H_4CHO$	24	*o*-$CH_3OC_6H_4CH_3$	98
m-$CH_3OC_6H_4CHO$	24	*m*-$CH_3OC_6H_4CH_3$	80
		m-$CH_3OC_6H_4CH_2OH$	18
	40	*m*-$CH_3OC_6H_4CH_3$	99
o-$CH_3C_6H_4CHO$	24	*o*-xylene	92
		o-$CH_3C_6H_4CH_2OH$	8
p-$CH_3C_6H_4CHO$	48	*p*-xylene	84
		p-methylbenzyl alcohol	16
o-HOC_6H_4CHO	24	*o*-$HOC_6H_4CH_3$	98
m-HOC_6H_4CHO	24	*m*-$HOC_6H_4CH_3$	86
	36	*m*-$HOC_6H_4CH_3$	100
p-$(CH_3)_2NC_6H_4CHO$	36	*p*-$(CH_3)_2NC_6H_4CH_3$	95[d]
p-$CH_3OCOC_6H_4CHO$	48	*p*-$CH_3OCOC_6H_4CH_3$	18
		p-$CH_3OCOC_6H_4CH_2OH$	77
p-$CH_3COOC_6H_4CHO$	48	*p*-$CH_3COOC_6H_4CH_3$	54
		p-$CH_3COOC_6H_4CH_2OH$	36

[a] Products were identified by comparison of spectral data, as well as GC retention times, with authentic samples. [b] Yield determined by gas chromatography. [c] Use of 2:1 sulfolane/THF as the solvent afforded PhC_2H_5 in 48% yield. [d] Isolated yield. [e] No reaction in the absence of the metal catalyst.

is believed to be located in an exposed position and the arene ring is inside the cavity and thus protected from reaction. Consequently, the carbonyl function would be easily susceptible to attack by an in situ generated rhodium hydride intermediate. That the formation of a β-cyclodextrin–substrate inclusion complex is critical to the success of the reduction process was clearly demonstrated by comparison with the behavior of α-cyclodextrin under identical reaction conditions. α-Cyclodextrin does not form 1:1 complexes with the carbonyl substrates.[18] Reaction of *p*-methoxyacetophenone with H_2,[1,5-HDRhCl]$_2$, and α-cyclodextrin in THF afforded *p*-ethylanisole in only 14% yield (85% recovered starting material). The yield is much lower than that realized by using β-cyclodextrin (88%) and is even less than in the absence of a cyclodextrin (24%). The presence of a presumably "inert" additive such as α-cyclodextrin may have an unfavorable effect on the kinetics of the hydrogenation reaction. Irrespective of the mechanistic details,[19] the method described above for the reduction of aldehydes and ketones to hydrocarbons is simple in both execution and workup,[20] proceeds under exceptionally mild conditions and shows good functional group selectivity.

Acknowledgment. We are indebted to British Petroleum, and to the Natural Sciences and Engineering Research Council, for support of this research.

Registry No. $PhCOCH_3$, 98-86-2; $PhCOC_2H_5$, 93-55-0; $PhCOC_3H_7$, 495-40-9; *o*-$MeC_6H_4COCH_3$, 577-16-2; *m*-$MeC_6H_4COCH_3$, 585-74-0; *o*-$MeOC_6H_4Ac$, 579-74-8; *m*-$MeOC_6H_4Ac$, 586-37-8; *p*-$MeOC_6H_4Ac$, 100-06-1; $PhCOCH_2OCH_3$, 4079-52-1; PhCHO, 100-52-7; *o*-$MeOC_6H_4CHO$, 135-02-4; *m*-$MeOC_6H_4CHO$, 591-31-1; *o*-MeC_6H_4CHO, 529-20-4; *p*-MeC_6H_4CHO, 104-87-0; *o*-HOC_6H_4CHO, 90-02-8; *m*-HOC_6H_4CHO, 100-83-4; *p*-$Me_2NC_6H_4CHO$, 100-10-7; *p*-$MeOCOC_6H_4CHO$, 1571-08-0; *p*-$AcOC_6H_4CHO$, 878-00-2; PhEt, 100-41-4; $C_6H_{11}Et$, 1678-91-7; PhC_3H_7-*n*, 103-65-1; $C_6H_{11}C_3H_7$-*n*, 1678-92-8; $C_6H_{11}C_4H_9$-*n*, 1678-93-9; *o*-MeC_6H_4Et, 611-14-3; *m*-MeC_6H_4Et, 620-14-4; *o*-$MeOC_6H_4Et$, 14804-32-1; *m*-$MeOC_6H_4Et$, 10568-38-4; *p*-$MeOC_6H_4Et$, 1515-95-3; $Ph(CH_2)_2OMe$, 3558-60-9; $PhCH(OH)CH_2OMe$, 3587-84-6; MePh, 108-88-3; *o*-$MeOC_6h_4Me$, 578-58-5; *m*-$MeOC_6H_4Me$, 100-84-5; *m*-$MeOC_6H_4CH_2OH$, 6971-51-3; *o*-MeC_6H_4Me, 95-47-6; *o*-$MeC_6H_4CH_2OH$, 89-95-2; *p*-MeC_6H_4Me, 106-42-3; *p*-$MeC_6H_4CH_2OH$, 589-18-4; *o*-HOC_6H_4Me, 95-48-7; *m*-HOC_6H_4Me, 108-39-4; *p*-$Me_2NC_6H_4Me$, 99-97-8; *p*-$MeOCOC_6H_{Me}$, 99-75-2; *p*-$MeOCOC_6H_4CH_2OH$, 6908-41-4; *p*-$AcOC_6H_4Me$, 140-39-6; *p*-$AcOC_6H_4CH_1OH$, 6309-46-2; [1,5HDRhCl]$_2$, 32965-49-4; 1-tetralone, 529-34-0; 6-methoxy-1-tetralone, 1078-19-9; 1-ethyl-3-methylcyclohexane, 3728-55-0; tetralin, 119-64-2; decalin, 91-17-8; 6-methoxytetralin, 1730-48-9; β-cyclodextrin, 7585-39-9.

(17) Fornasier, R.; Reniero, F.; Scrimin, P.; Tonellato, U. *J. Org. Chem.* **1985**, *50*, 3209.
(18) Reference 17, footnote 18.
(19) It is conceivable, as an alternate pathway, that the rhodium(I) catalyst (either itself or a hydride formed by reaction with hydrogen) first binds to β-cyclodextrin and then reacts with the organic substrate. Several cyclodextrin–rhodium complexes have recently been isolated: Alston, D. R.; Slawin, A. M. Z.; Stoddart, J. F.; Williams, D. J. *Angew. Chem., Int. Ed. Engl.* **1985**, *24*, 786.
(20) The following general procedure was used. Hydrogen is bubbled through a stirred THF solution (9 mL) of the substrate (3.2 mmol), [1,5-HDRhCl]$_2$ (0.065 mmol), and β-cyclodextrin (0.32 mmol) at room temperature and 1 atm. After 8–48 h (see Table I), the solvent was removed by rotary evaporation, and the product was purified either by distillation or by column chromatography.

Trapping of Silylenes by 9,10-Dimethylanthracene: 2,3:5,6-Dibenzo-7-silabicyclo[2.2.1]hepta-2,5-dienes

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Received March 4, 1986

Summary: Silylenes generated thermally from 7-silanorbornadienes **1a–c** react with 9,10-dimethylanthracene (DMA) to give 7-silabicyclo[2.2.1]hepta-2,5-dienes **3a–c**. For dimethylsilylene, a small amount of 2,3:5,6-dibenzo-1,4,7,7,8,8-hexamethyl-7,8-disilabicyclo[2.2.2]octa-2,5-diene (**4a**) is also obtained, arising via Diels–Alder reaction of tetramethyldisilene with DMA.

Although silylenes are known to add to alkenes, alkynes, dienes, and carbonyl compounds,[1] addition of silylenes to

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(1) Gaspar, P. P. In *Reactive Intermediates*; Jones, M., Jr., Moss, R. A., Eds.; Wiley: 1978; Vol. 1, p 229. *Ibid.*, Vol. 2, p 335, 1981. *Ibid.*, Vol. 3, p 333, 1985.

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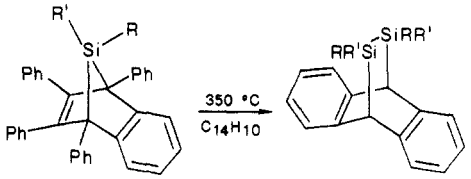
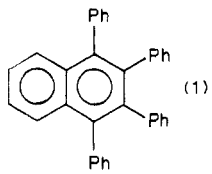
anthracene has not previously been observed. In 1979 Sakurai et al. reported that cothermolysis of 2,3-benzo-1,4,5,6-tetraphenyl-7-silanorbornadienes **1a–c** with anthracene at 350 °C gave 2,3:5,6-dibenzo-7,8-disilabicyclo-[2.2.2]octa-2,5-dienes **2a–c** as the sole products.[2] These products were believed to result from initial dimerization of silylenes to form disilenes, followed by cycloaddition to the 9,10-positions of anthracene (eq 1), but stepwise reaction of silylenes with anthracene could not be excluded.



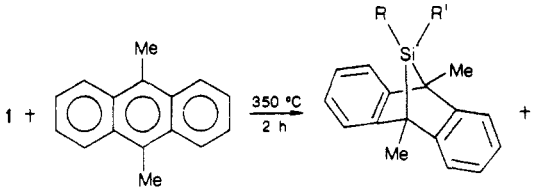
1a–c **2a–c**



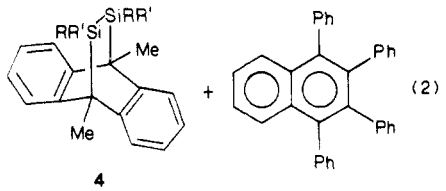
(1)

a, R = R' = Me; **b**, R = Me; R' = Ph; **c**, R = R' = Ph

We now find that the reaction of silylenes generated thermally from **1a–c** with 9,10-dimethylanthracene (DMA) takes a different course, leading to the formation of 7-silabicyclo[2.2.1]hepta-2,5-dienes **3a–c** as the major or exclusive silicon-containing products. When a mixture of **1a** (4.02 mmol), 9,10-dimethylanthracene (4.06 mmol), and benzene (ca. 1 g) was heated in a sealed tube at 350 °C for 2 h, **3a** was obtained in 33% yield along with the disilicon adduct **4a** (13%) and 1,2,3,4-tetraphenylnaphthalene (97%) (eq 2).[3] Since compound **3a** decomposes on silica



3

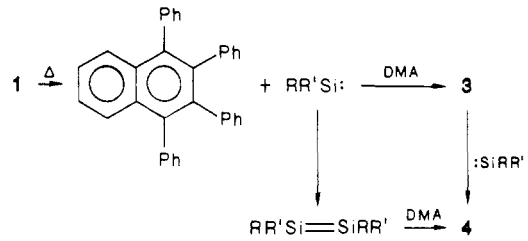


(2)

4

gel but is rather stable thermally, it was isolated by preparative gas chromatography: mp 96–97 °C; ^{1}H NMR (200 MHz, $CDCl_3$) δ −0.29 (s, 6 H, SiMe), 1.78 (s, 6 H, CMe), 7.00–7.20 (m, 8 H, ArH); ^{13}C NMR ($CDCl_3$) δ −6.3, 10.1, 43.6, 119.8, 124.3, 147.1; ^{29}Si NMR ($CDCl_3$) 67.7 ppm; high-resolution mass (EI, 30 eV) 264.1339; calcd for $C_{18}H_{20}Si$ 264.1334. The ^{29}Si resonance at very low field is characteristic for 7-silanorbornadiene derivatives.[4]

Scheme I



Similarly, thermolysis of **1b** and **1c** with DMA led to the silylene adducts **3b** and **3c** in 29% and 11% yields, respectively, along with 1,2,3,4-tetraphenylnaphthalene.[5] No **4b** or **4c** was observed in these reactions; if they were formed, it was at most in trace amounts.

In the thermolysis of **1a** with DMA, compound **4a** could conceivably be formed either (1) by dimerization of dimethylsilylene to tetramethyldisilene and Diels–Alder addition to DMA or (2) by insertion of dimethylsilylene into a silicon–carbon bond of **3a**. When **3a** (0.095 mmol) was heated with **1a** (0.096 mmol) in benzene at 350 °C for 1 h, the **1a** was completely consumed with quantitative formation of 1,2,3,4-tetraphenylnaphthalene along with an insoluble pale yellow polymer, $(Me_2Si)_n$.[6] Compound **3a** was recovered essentially unchanged, and only traces of **4a** and DMA were found. The second pathway is therefore excluded, and product **4a** must arise from tetramethyldisilene addition DMA (Scheme I).

Thus in our reaction, addition of silylene to DMA competes favorably with dimerization and trapping of the disilene, although in the reaction investigated earlier by Sakurai, only dimerization and disilene addition were observed.[2] Both steric and electronic factors might influence the nature of the products. The methyl groups on DMA may make it more reactive than anthracene toward silylenes; DMA is known to be much more reactive than anthracene toward electrophilic reagents.[7] In addition the methyl groups may sterically inhibit the addition of disilenes to DMA to form **4**.[8]

Other methods for generating silylenes in the presence of DMA do not necessarily lead to products like **3a**. Thus when *sym*-dimethoxytetramethyldisilane was thermolyzed with DMA, no **3a** or **4a** were formed; instead silylene insertion took place into the Si–O bonds of the precursor. Insertion of silylenes into MeO–Si bonds is known to prevail over other reactions involving only moderately active silylene trapping agents.[2,9] Similarly when $(Me_2Si)_6$ was photolyzed at 254 nm in the presence of DMA, it was converted to $(Me_2Si)_5$, indicating that dimethylsilylene was formed, but no **3a** was obtained.[10]

(2) Nakadaira, Y.; Kobayashi, T.; Otsuka, T.; Sakurai, H. *J. Am. Chem. Soc.* **1979**, *101*, 486. Repetition of the reaction between **1a** and anthracene described in this publication gave results identical with those reported by Nakadaira et al.

(3) **4a**: ^{1}H NMR ($CDCl_3$, δ) −0.20 (s, 12 H, SiMe), 2.09 (s, 6 H, CMe), and 7.08–7.28 (m, 8 H, ArH). Since it is difficult to separate **4a** from DMA, the mixture of **4a** and DMA after chromatography was treated with MCPBA in $CHCl_3$. Compound **4a** was smoothly oxidized to give 2,3:5,6-dibenzo-1,4,7,7,9,9-hexamethyl-8-oxabicyclo[2.2.3]nona-2,5-diene: mp 231–232 °C; NMR ($CDCl_3$, δ) −0.08 (s, 12 H, SiMe), 1.73 (s, 6 H, CMe), 7.15–7.36 (m, 8 H, ArH); high-resolution mass 338.1517, calcd for $C_{20}H_{26}OSi_2$: 338.1522.

(4) (a) Sakurai, H.; Sakaba, H.; Nakadaira, Y. *J. Am. Chem. Soc.* **1982**, *104*, 6156. (b) Sekiguchi, A.; Zigler, S. S.; West, R.; Michl, J. *J. Am. Chem. Soc.* **1986**, *108*, 4241. (c) Sakurai, H.; Nakadaira, Y.; Koyama, T.; Sakaba, H. *Chem. Lett.* **1983**, 213.

(5) Compound **3b**: mp 132–133 °C; NMR ($CDCl_3$, δ) −0.05 (s, 3 H, SiMe), 1.85 (s, 6 H, CMe), 6.74–6.89 (m, 2 H, ArH), 7.03–7.31 (m, 11 H, ArH); ^{29}Si NMR ($CDCl_3$) 51.9 ppm; high-resolution mass 326.1502, calcd for $C_{23}H_{22}Si$ 326.1491. Compound **3c**: mp 213–214 °C; NMR ($CDCl_3$, δ) 2.01 (s, 6 H, CMe), 6.80–7.33 (m, 18 H, ArH); ^{29}Si NMR ($CDCl_3$) 37.7 ppm; high-resolution mass 388.1644, calcd for $C_{28}H_{24}Si$ 388.1647.

(6) Gilman, H.; Cottis, S. G.; Atwell, W. H. *J. Am. Chem. Soc.* **1964**, *86*, 1596.

(7) Kloetzel, M. C. In *Organic Reactions*; Adams, R., Ed.; Wiley: New York, 1967; Vol. 4, p 29.

(8) A competition experiment involving thermolysis of **1a** in the presence of equimolar amounts of both anthracene and DMA produced only **2a**, and no **3a** or **4a**. However, **2a** may be the thermodynamic rather than kinetic product.

(9) Atwell, W. H.; Weyenberg, D. R. *J. Am. Chem. Soc.* **1968**, *90*, 3438; *Angew Chem., Int. Ed. Engl.* **1969**, *5*, 1021.

Acknowledgment. Research was sponsored by the Air Force Office of Scientific Research, Air Force Systems Command, USAF, under Contract No. F49620-86-C-0011. The United States Government is authorized to reproduce and distribute reprints for governmental purposes notwithstanding any copyright notation thereon. We thank Xia Zhaorong for technical assistance.

Registry No. **1a**, 18816-24-5; **1b**, 103530-75-2; **1c**, 1762-11-4; **3a**, 103457-11-0; **3b**, 103477-29-8; **3c**, 103457-12-1; **4a**, 103457-13-2; DMA, 781-43-1.

(10) A control experiment showed that **3a** is photolabile under the conditions employed.

Intramolecular Conversion of a Five-Membered Iridacycle to a Three-Membered Counterpart by CO_2 Extrusion

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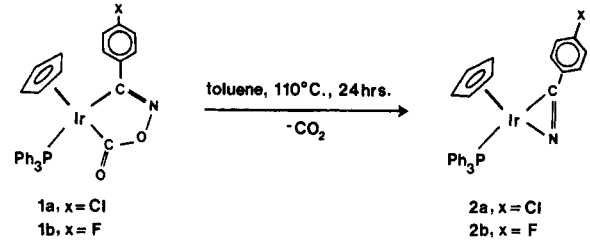
Summary: Thermolysis of the metallacycles **1a** and **1b** in refluxing toluene for 24 h results in loss of CO_2 and the formation of a product characterized by the formal oxidative addition of the 16-electron Ir(I) metal fragment "$CpIrPPh_3$" into the nitrile triple bond, generating the kinetically very stable side-bonded nitrile complexes **2a** and **2b**, in high yield. An X-ray diffraction study was undertaken of **2a** confirming its structure as that containing a Ir^{III}—C=N metallacycle.

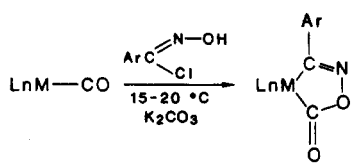
We have been investigating the reactivity of metallacycles generated by the cycloaddition of aryl nitrile oxides to low-valent metal carbonyl complexes.[1] We wish to report the formation of side-bonded nitrile complexes whose chemical characteristics appear to be more readily attributed to the result of oxidative addition across the nitrile triple bond by a metal fragment than by π-complexation of a nitrile to a low-valent metal.

Thermolysis of **1a** and **1b**[2] in boiling toluene for 24 h leads to the formation of the remarkably stable **2a** and **2b**, respectively, with extrusion of CO_2 (Scheme I). All ^1H, ^{19}F, and ^{31}P NMR data, as well as elemental analyses, are consistent with the structures shown for **2a** and **2b**.[3] The structure of **2a** was also confirmed by an X-ray diffraction study described below. The IR spectra of **2a** and **2b** exhibit a CN stretching frequency at 1758 and 1756 cm^{-1}, respectively, a decrease of 472 and 468 cm^{-1} from the corresponding free nitriles. Similar large decreases in the CN stretching frequencies have been observed in other complexes which are believed to contain side-bonded nitriles,[4-8] as opposed to the more common mode of nitrile coordination which occurs by σ-bonding through the nitrile nitrogen lone electron pair.[9] In order to establish whether the formation of free nitrile occurred by decomposition of **1**, to generate the 16-electron metal fragment "$CpIrPPh_3$" which then coordinates free nitrile, or if an intramolecular mechanism was involved, **1b** was decomposed in the presence of a 20-fold excess of p-ClC_6H_4CN. If nitrile formation occurred by the former mechanism, **2a** would be the predominant product, whereas if an intramolecular process was involved, then compound **2b** should be obtained. Both ^{31}P and ^{19}F NMR identified **2b** as the predominant product (80% yield by NMR); no resonance in the ^{31}P NMR was observed for **2a**. This result indicated that no nitrile exchange had occurred and that the formation of **2** involved an intramolecular process. The ^{19}F NMR of the products of decomposition of **1b** gave two resonances, one of which corresponded to **2b** and the other to free p-FC_6H_4CN. The yield of p-FC_6H_4CN was 9% by NMR in the absence of p-ClC_6H_4CN and 20% in the presence of p-ClC_6H_4CN; the ^{31}P NMR contained a minor resonance at 17.09 ppm together with the major resonance due to **1b** in both cases. The ^1H NMR spectrum of the reaction products gave no evidence of hydrides which could be formed as a result of C–H oxidative addition of the solvent or intramolecular hydride abstraction. The nature of the minor product resulting from loss of p-FC_6H_4CN from **1b** and having a ^{31}P NMR resonance at 17.09 ppm was not determined.

Scheme I



(1) We have synthesized a number of metallacycles by cycloaddition of aryl nitriles oxides with low-valent metal carbonyl complexes. A preliminary communication has been published (Walker, J. A.; Knobler, C. B.; Hawthorne, M. F. *J. Am. Chem. Soc.* **1983**, *105*, 3370) and a complete report of this synthetic route to these metallacycles and their reactivity will be submitted shortly; the general reaction is outlined.



Ar = p-ClC_6H_4–, 2,4,6-$(CH_3)_3C_6H_2$–, and p-FC_6H_4–. Metallacycle yields vary between 60 and 80%.

(2) Selected data for **1a** and **1b** (full details will be reported elsewhere[1]). **1a**: ^1H NMR (CD_2Cl_2) δ 7.37–7.15 (complex multiplets, 19 H), 5.39 (d, 5 H, J = 1.0 Hz); $^{31}P\{^1H\}$ NMR (C_6D_6) δ –2.22. Anal. Calcd for $C_{31}H_{24}ClIrNO_2P$: C, 53.17; H, 3.46; Ir, 27.45; N, 2.00; P, 4.42. Found: C, 52.92; H, 3.57; Ir, 27.12; N, 1.91; P, 4.33. **1b**: ^1H NMR (CD_2Cl_2) δ 7.46–6.74 (complex multiplets, 19 H), 5.39 (d, 5 H, J = 0.88 Hz). $^{31}P\{^1H\}$ NMR ($C_7D_5CD_3$ δ –2.09. Anal. Calcd for $C_{31}H_{24}FIrO_2P$: C, 54.37; H, 3.54; Ir, 28.07; N, 2.05; P, 4.52. Found: C, 54.12; H, 3.66; Ir, 27.92; N, 2.01; P, 4.44.

(3) Selected data for **2a** and **2b** (full details will be reported elsewhere[1]). **2a**: ^1H NMR (C_6D_6) δ 7.15–6.24 (complex multiplets, 19 H), 5.90 (d, 5 H, J = 1.46 Hz); $^{31}P\{^1H\}$ NMR (C_6D_6) δ 16.56. Anal. Calcd for $C_{30}H_{24}ClIrNP$: C, 54.83; H, 3.69; N, 2.13; P, 4.71. Found: C, 54.66; H, 3.60; N, 1.97; P, 4.08. **2b**: ^1H NMR (CD_2Cl_2) δ 7.67–6.82 (complex multiplets, 19 H), 5.27 (d, 5 H, J = 1.2 Hz); $^{31}P\{^1H\}$ NMR ($C_6D_5CD_3$) δ 16.29. Anal. Calcd for $C_{30}H_{24}FIrNP$: C, 56.23; H, 3.78; Ir, 29.99; N, 2.19; P, 4.83. Found: C, 55.70; H, 3.94; Ir, 29.42; N, 2.15; P, 4.71.

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