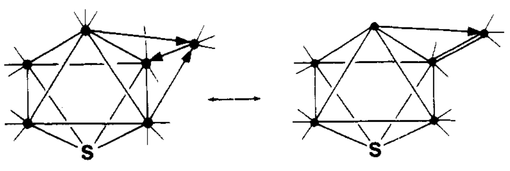
shape of the cluster is that of an $Os(CO)_3$ "capped" octahedron if the sulfido ligand is considered as a part of the cluster. Mingos has shown recently that capping groups should not change the number of bonding cluster valence orbitals so the observed structure of V is also an acceptable one.[27]

Of further interest is the highly unsymmetrical bonding of the capping group to the cluster. For example, both the shortest (Os(1)–Os(6) = 2.625 (1) Å) and longest (Os(5)–Os(6) = 2.930 (1) Å) metal–metal bonds in the molecule involve the capping metal atom Os(6). While the metal–metal bonds to capping groups are usually shorter than those within the polyhedron, the distortion observed in V is unprecedented.[15,28–30]

The bonding in V can also be described with two-center two-electron metal–metal bonds if one employs a series of heteropolar bonds such as those indicated in structure Va by the double-headed arrows.[28] In addition, a component of the resonance form Vb which implies the absence of a bond between Os(5) and Os(6) and a double bond between Os(1) and Os(6) would explain the observed distortions.



(Va) (Vb)

Discussion

The condensation of I and II to yield III is clearly a multistep process involving ligand loss and metal–metal bond formation. It is believed that this process is initiated via the formation of a coordinate bond between the sulfido ligand in I and a metal atom in II, but in the absence of isolated intermediates further steps cannot be delineated at this time. Equation 1 shows schematically where bonds in I and II must be made and broken in order to produce III.

The formation of V from III is less complex; see eq 2. One carbonyl ligand is probably lost from each of the edge bridging $Os(CO)_4$ groups. One $Os(CO)_3$ group then becomes the capping group while the other inserts into one of the metal–metal bonds of the cluster and leads to its enlargement.

Acknowledgment. We wish to thank the National Science Foundation for support of this research through Grant No. CHE 80-19041 and Engelhard Industries for a loan of osmium tetraoxide.

Registry No. I, 88746-45-6; II, 61817-93-4; III, 88730-74-9; IV, 86437-19-6; V, 88730-75-0.

Supplementary Material Available: A table of structure factor amplitudes and a listing of interatomic angles for each structural analysis (30 pages). Ordering information is given on any current masthead page.

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Communications

Monoalkylation of Dichloroarenes with Grignard Reagents Catalyzed by a Nickel Complex[†]

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Summary: Dichloroarenes react with Grignard reagents in the presence of a catalytic amount of $[Ni(triphos)Cl]PF_6$ (triphos = bis(2-(diphenylphosphino)ethyl)phenylphosphine) to give mainly monoalkylated products.

Although the cross coupling of aromatic halides with Grignard reagents catalyzed by transition metals has been known for several years,[1] the desired monoalkylation of dichloroarenes has not been successfully accomplished. Cross-coupling reactions of dichlorobenzene using conventional nickel catalysts give predominant formation of dialkylated products.[2] While dibromobenzene can be selectively monoalkylated with Grignard reagents in the presence of palladium catalysts,[3] the use of chlorinated arenes is preferred because they are more readily available than their brominated analogues. Specifically, monoalkylation of dichloroarenes would be a useful route to pharmaceutical and agricultural intermediates.[4]

We have discovered that $[Ni(triphos)Cl]PF_6$[5] (triphos = bis(2-(diphenylphosphino)ethyl)phenylphosphine) is an effective catalyst for the monoalkylation of dichloroarenes. Some examples are shown in Table I.[6] In a typical reaction, 4.7 mL (13.6 mmol) of 2.9 M EtMgBr in ether was added to 2.00 g (13.60 mmol) of *o*-dichlorobenzene and 0.050 g (0.065 mmol) of $[Ni(triphos)Cl]PF_6$ in 35 mL of

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[†] Contribution No. 3409.

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(6) $[Ni(triphos)Cl]PF_6$ catalyzes the reaction of chlorobenzene and EtMgBr to yield 88% ethylbenzene.

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Table I. Monoalkylation of Dichlorobenzenes[a]

compound	Grignard	% mono	% di
o-dichlorobenzene	EtMgBr	71	11
	MeMgI[b]	65	17
	sec-BuMgCl[c]	45	5
	$PhCH_2MgCl$[b,d]	49	19
	PhMgBr[b]	53	17
m-dichlorobenzene	*n*-BuMgCl	68	23
m-dichlorobenzene	*sec*-BuMgCl[e]	21	11
p-dichlorobenzene	EtMgBr	75	9
p-dichlorobenzene	*sec*-BuMgCl[f]	31	13
2,6-dichloroanisole	EtMgBr	60	14
2,6-dichlorotoluene	*n*-BuMgCl[g]	49	30

[a] Yields were determined by GC and were not optimized. Columns used were either (1) 10% SP1000 on 100/120 Chromosorb WAW, (2) 10% Carbowax 20M on 80/100 Supelcoport, or (3) 10% SP2100 on 100/120 Supelcoport; mono = monoalkylated product; di = dialkylated product. [b] Active catalyst was initially generated with EtMgBr. Three days of reflux was needed for completion of reaction. [c] Secondary Grignard reactions were done in refluxing THF. Chlorobenzene (9%), 1-chloro-2-*n*-butylbenzene (6%), and 1-*n*-butyl-2-*sec*-butylbenzene (3%) along with 26% *o*-dichlorobenzene were found. [d] Bibenzyl (10%) was also found. [e] Chlorobenzene (30%), 1-chloro-3-*n*-butylbenzene (5%), 1-*sec*-butyl-3-*n*-butylbenzene (4%), and *m*-dichlorobenzene (28%) were found. [f] Chlorobenzene (20%), 1-chloro-4-*n*-butylbenzene (8%), 1-*sec*-butyl-4-*n*-butylbenzene (4%), and *p*-dichlorobenzene (23%) were found. [g] Reaction was done in diethyl ether at room temperature.

diethyl ether at 0 °C. After refluxing overnight, the mixture was analyzed by GC to contain 71% 1-chloro-2-ethylbenzene along with 11% 1,2-diethylbenzene. When the disappearance of *o*-dichlorobenzene under the above conditions was monitored by GC, it was found to obey first-order kinetics with $k = 6.8 \pm 0.4 \times 10^{-3}$ min^{-1}. For comparison, when this reaction was catalyzed by $NiCl_2$(dppp) (dppp = $Ph_2PCH_2CH_2CH_2PPh_2$), 39% dialkylated product, 12% monoalkylated product, and 47% starting dichloride were found.

Linear alkyl Grignard reagents react more cleanly than secondary ones because they generate fewer reduced and rearranged products (see Table I). These side products are more prevalent for *m*- and *p*-dichlorobenzene than for *o*-dichlorobenzene. The reaction is slower with secondary Grignards, and boiling THF conditions are needed for a reasonable reaction time. With methyl, benzyl, and phenyl Grignard reagents, reactions with *o*-dichlorobenzene proceed more smoothly when the active catalyst is initially generated with EtMgBr.[7] When EtMgBr was omitted, the reaction with MeMgI yielded only 10% of *o*-chlorotoluene after refluxing for 4 days. Presumably, the Ni(II) complex must be reduced to either Ni(I) or Ni(0) to be an active catalyst. Vinyl, allyl, and *tert*-butyl Grignard reagents do not react with *o*-dichlorobenzene.

Table II. Substituted Dichloroarenes and EtMgBr[a]

compound	products
3,4-dichlorotoluene	3-ethyl-4-chlorotoluene (62), **1** 3,4-diethyltoluene (17)
2,5-dichlorobenzotrifluoride	2-chloro-5-ethylbenzotrifluoride (41), **2** 2-ethyl-5-chlorobenzotrifluoride (18) 2,5-diethylbenzotrifluoride (17)
2,5-dichlorotoluene	2-ethyl-5-chlorotoluene (40), **3** 2-chloro-5-ethyltoluene (26), **4** 2,5-diethyltoluene (19)
2,4-dichlorotoluene	2-ethyl-4-chlorotoluene (57), **5** 2-chloro-4-ethyltoluene (10) 2,4-diethyltoluene (16)
2,3-dichloroanisole	2-chloro-3-ethylanisole (70), **6** 2-ethyl-3-chloroanisole (4)

[a] Yields are in parentheses and all yields except the last example have been determined by GC on 10% SP1000 on 100/120 Chromosorb WAW. With the last example, yields are isolated yields.

The regioselectivity in certain cases is high (Table II)[8]; with 3,4-dichlorotoluene and EtMgBr, 3-ethyl-4-chlorotoluene is the only monoalkylated product detected.[9,10] The reaction of 2,3-dichloroanisole with EtMgBr gives 70% 2-chloro-3-ethylanisole and only 4% 2-ethyl-3-chloroanisole.[11] Competition reactions between chlorotoluenes indicate that the chloride ortho or meta to the methyl group displaces more readily than the chloride in the para position. Reaction of a 1:1:1 mixture of *m*- and *p*-chlorotoluene and EtMgBr gives 62% *m*-ethyltoluene and 34% *p*-ethyltoluene. With a 1:1 mixture of *o*- and *p*-chlorotoluene, 66% of *o*-ethyltoluene and 33% of *p*-ethyltoluene are obtained. A 0.8:1 mixture of *m*- to *o*-ethyltoluene is obtained from a 1:1 mixture of *m*- and *o*-chlorotoluene.[12]

The regioselectivity is dependent on the Grignard reagent used; for example, the reaction of 2,5-dichlorotoluene with MeMgI gives 47% 2-chloro-5-methyltoluene and 30% 4-chloro-*o*-xylene. The major isomer is derived from alkylation at the C_5 position. With MeMgBr, 39% of 4-chloro-*o*-xylene and 38% of 2-chloro-5-methyltoluene are obtained. With EtMgBr, the major isomer is derived from alkylation at the C_2 position (see Table II).

The site of alkylation in the benzene ring is unequivocally determined through the use of nuclear Overhauser enhancements (NOE) between the methylene protons of the ethyl group and the various aromatic protons. As an example, the assignment of the aromatic protons and the location of the Et group for the major isomer from the reaction of 2,3-dichloroanisole and EtMgBr are discussed. The NMR spectrum of the product in the aromatic proton region showed a triplet at 7.13 ppm, a doublet of doublets at 6.84 ppm, and another doublet of doublets at 6.76 ppm. The triplet at 7.13 ppm can be assigned unambiguously to H_5. The NOE measurements were done by presaturating the desired NMR resonances and measuring the NOE by difference techniques. When the $-OCH_3$ protons

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[7] EtMgBr (1.5 mmol) was added to 0.065 mmol of catalyst in ether at 0 °C. $KBEt_3H$ can also be used in place of EtMgBr.

[8] 360-MHz ^1H NMR spectra: **2** (δ, CD_2Cl_2) 7.55 (d, $J = 2$ Hz, 1 H), 7.44 (d, $J = 8$ Hz, 1 H) 7.35 (dd, $J = 2$ and 8 Hz, 1 H), 2.70 (q, $J = 8$ Hz, 2 H), 1.25 (t, J = 8 Hz, 3 H); **3** (δ, CD_2Cl_2) 7.13 (m, 3 H), 2.60 (q, $J = 8$ Hz, 2 H), 2.29 (s, 3 H), 1.19 (t, $J = 8$ Hz, 3 H); **5** (δ, CD_2Cl_2) 7.14 (m, 1 H), 7.08 (m, 2 H), 2.60 (q, $J = 8$ Hz, 2 H), 2.27 (s, 3 H), 1.20 (t, $J = 8$ Hz, 3 H). Assignments of **3** and **5** were done by comparison with the ^1H NMR spectrum (aromatic region) of authentic 4-chloro-1,2-dimethylbenzene, and the minor isomer from the reaction of 2,5-dichlorotoluene with EtMgBr was determined by NOE measurements to be **4** (δ, CD_2Cl_2) 7.24 (d, $J = 8$ Hz, 1 H), 7.09 (d, $J = 2$ Hz, 1 H), 6.97 (dd, $J = 8$, 2 Hz, 1 H), 2.60 (q, $J = 8$ Hz, 2 H), 2.35 (s, 3 H), 1.22 (t, $J = 8$ Hz, 3 H).

[9] The products were isolated by flash column chromatoraphy on silica gel eluted with hexane and also by preparative GC. 360-MHz ^1H NMR spectrum of major isomer **1** (δ, CD_2Cl_2): 7.20 (d, $J = 8$ Hz, 1 H), 7.07 (d, $J = 8$ Hz, 1 H), 6.95 (dd, $J = 8$, 2 Hz, 1 H), 2.69 (q, $J = 8$ Hz, 2 H), 2.27 (s, 3 H), 1.20 (t, $J = 8$ Hz, 3 H).

[10] The reaction of 3,4-dichlorotoluene with *n*-BuMgCl gives 60% 3-*n*-butyl-4-chlorotoluene and 13% 3-chloro-4-*n*-butyltoluene.

[11] The products were isolated by the use of the chromatotron from Harrison Research. 360-MHz ^1H NMR spectrum of the major isomer **6** (δ, $CDCl_3$): 7.13 (t, $J = 8$ Hz, 1 H), 6.84 (dd, 8, 1 Hz, 1 H), 6.76 (dd, 8, 1 Hz, 1 H), 3.80 (s, 3 H), 2.76 (q, $J = 8$ Hz, 2 H), 1.22 (t, $J = 8$ Hz, 3 H).

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are presaturated, only the resonances at 6.76 ppm showed NOE, allowing assignment of this set of resonances to H_6. Presaturation of the methylene protons of the ethyl group showed only H_4 to give appreciable NOE, locating the ethyl group on C_3. Similar NOE measurements were carried out on other products and their regioisomers determined.

We are studying the mechanism of this reaction and the influence of other tridentate ligands and nucleophiles.

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Registry No. EtBr, 74-96-4; MeI, 74-88-4; $PhCH_2Cl$, 100-44-7; PhBr, 108-86-1; *n*-BuCl, 109-69-3; $[Ni(triphos)Cl]PF_6$, 33847-47-1; *sec*-BuCl, 78-86-4; $NiCl_2(dppp)$, 15629-92-2; *o*-dichlorobenzene, 95-50-1; *m*-dichlorobenzene, 541-73-1; *p*-dichlorobenzene, 106-46-7; 2,6-dichloroanisole, 1984-65-2; 2,6-dichlorotoluene, 118-69-4; 3,4-dichlorotoluene, 95-75-0; 2,4-dichlorobenzotrifluoride, 320-50-3; 2,5-dichlorotoluene, 19398-61-9; 2,4-dichlorotoluene, 95-73-8; 2,3-dichloroanisole, 1984-59-4; *o*-chloroethylbenzene, 89-96-3; *o*-diethylbenzene, 135-01-3; *o*-chlorotoluene, 95-49-8; *o*-xylene, 95-47-6; *o*-chloro-*sec*-butylbenzene, 36383-12-7; *o*-di-*sec*-butylbenzene, 1013-03-2; *o*-chlorobenzylbenzene, 29921-41-3; *o*-dibenzylbenzene, 792-68-7; *o*-chlorobiphenyl, 2051-60-7; *o*-diphenylbenzene, 84-15-1; *m*-chlorobutylbenzene, 15499-28-2; *m*-dibutylbenzene, 17171-74-3; *m*-chloro-*sec*-butylbenzene, 36383-13-8; *m*-di-*sec*-butylbenzene, 1079-96-5; *p*-chloroethylbenzene, 622-98-0; *p*-diethylbenzene, 105-05-5; *p*-chloro-*sec*-butylbenzene, 36383-14-9; *p*-di-*sec*-butylbenzene, 1014-41-1; 6-chloro-2-ethylanisole, 89031-98-1; 2,6-diethylanisole, 2944-51-6; 2-butyl-6-chlorotoluene, 89031-99-2; 2,6-dibutyltoluene, 89032-00-8; 1-butyl-2-*sec*-butylbenzene, 89032-01-9; bibenzyl, 103-29-7; 1-*sec*-butyl-3-butylbenzene, 89032-02-0; 1-*sec*-butyl-4-butylbenzene, 37909-91-4; 3-ethyl-4-chlorotoluene, 89032-03-1; 3,4-diethyltoluene, 13732-80-4; 2-chloro-5-ethylbenzotrifluoride, 89032-04-2; 2-ethyl-5-chlorobenzotrifluoride, 89032-05-3; 2,5-diethylbenzotrifluoride, 89032-06-4; 2-ethyl-5-chlorotoluene, 89032-07-5; 2-chloro-5-ethyltoluene, 89032-08-6; 2,5-diethyltoluene, 13632-94-5; 2-ethyl-4-chlorotoluene, 89032-09-7; 2-chloro-4-ethyltoluene, 89032-10-0; 2,4-diethyltoluene, 1758-85-6; 2-chloro-3-ethylanisole, 89032-11-1; 2-ethyl-3-chloroanisole, 89032-12-2.

Organometallic Chemistry in Aqueous Solution: Water-Soluble Bis(dimethylphosphino)methane-Bridged Dipalladium Complexes[†]

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Summary: A new series of binuclear palladium complexes bridged by the diphosphine bis(dimethylphosphino)methane (dmpm) is reported. The complex, $Pd_2Cl_2(dmpm)_2$, has been prepared and found to have remarkably high solubility and stability in aqueous solutions. In aqueous solutions, the complex exists as $Pd_2(OH)_2(dmpm)_2$ at pH > 4. In either aqueous or nonaqueous solvents, $Pd_2X_2(dmpm)_2$ (X = Cl, OH, OR) inserts one equivalent of CO into its Pd–Pd bond to give $Pd_2X_2(\mu\text{-CO})(dmpm)_2$. The structure of $Pd_2Cl_2(\mu\text{-CO})(dmpm)_2$ has been determined by X-ray diffraction.

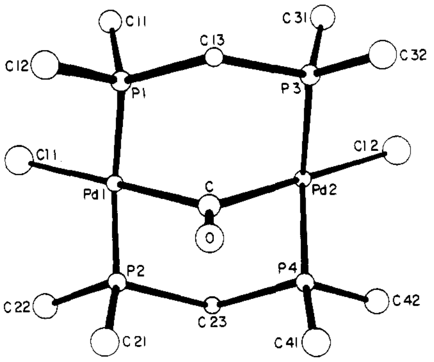

Figure 1. Drawing of $Pd_2Cl_2(\mu\text{-CO})(dmpm)$ with all non-hydrogen atoms.

We report the synthesis and reactivity of bis(dimethylphosphino)methane-bridged binuclear palladium complexes which have an unusual property: high solubility in aqueous solutions. Numerous reports of diphosphine-bridged binuclear transition-metal complexes have appeared in recent years.[1-26,32] The impetus for many of

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[†] This paper is dedicated to the memory of Professor Earl L. Muetterties.

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(27) $Pd_2Cl_2(dmpm)_2$: Anal. Calcd for $C_{10}H_{28}P_4Cl_2Pd_2$: C, 21.60; H, 5.08. Found: C, 21.92; H, 5.25. ^1H NMR (CD_2Cl_2) δ 2.65 (m), 1.60 (m); ^1H NMR (D_2O) δ 2.80 (m), 1.57 (m); $^{13}C\{^1H\}$ NMR (CH_2Cl_2) δ 42.0 (CH_2, t), 16.9 (CH_3, q); $^{31}P\{^1H\}$ NMR (H_3PO_4 external, CD_2Cl_2/CH_2Cl) δ −31 (s); $^{31}P\{^1H\}$ NMR (D_2O) δ −33 (s); IR (CD_3CN) 1420 (w), 1300 (w), 1290 (w), 1290 (w), 1180 (m) cm^{-1}; UV–vis (λ_{max}, nm (ϵ)) (H_2O) 261 (17 000), 290 (9890), 339 (4670), 393 (2760), (CH_3CN) 242 (8899), 274 (17 909), 318 (9948), 382 (4531), (CH_2Cl_2) 243 (10 863), 278 (22 383), 320 (14 167), 385 (5729); $\Lambda_{0.001M}$ = 51.5 Ω^{-1} cm^2 mol^{-1} in H_2O.

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