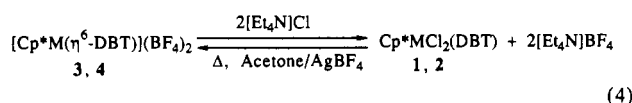


is easily displaced by acetone, but **4** is stable in nitromethane. DBT in **3** is not displaced by acetone because of the insolubility of the complex in this solvent; **3** is stable in nitromethane.

Assignments of the ^1H and ^{13}C NMR data for **3** and **4** indicate that coordination occurs through the benzene ring ($\text{C}_6\text{-C}_{10}$, C_{12} ; Chart I) of DBT. In the ^1H NMR spectra, the resonances of the coordinated DBT are slightly downfield with respect to the free ligand, as was also observed in $\text{Cp}^*\text{Ir}(\eta^6\text{-BT})^{2+}$.²² This contrasts with $\text{Cr}(\text{CO})_3(\eta^6\text{-DBT})$,³ $[\text{Cp}^*\text{M}(\eta^6\text{-DBT})]\text{PF}_6$,^{2,4} and $[(\text{CpM})_2(\mu\text{-}\eta^6,\eta^6\text{-DBT})]^{2+}$,^{2,4} where upfield shifts are observed presumably because of the lower oxidation state of the metal. However, the ^{13}C signals of the arene carbons directly bonded to the metal in **3** and **4** are upfield with respect to the free ligand. In complex **4**, all of the coordinated arene ^{13}C signals split into doublets due to coupling with Rh.

Complex **3** reacts immediately with 2 equiv of $[\text{Et}_4\text{N}]\text{Cl}$ in CH_3NO_2 to give the $\eta^1(\text{S})$ -DBT complex **1** (eq 4) at 25 °C.



Likewise **4** reacts immediately with 2 equiv of $[\text{Et}_4\text{N}]\text{Cl}$ in CHCl_3 at room temperature to give complex **2**. These reactions illustrate an interesting linkage isomerization of DBT from η^6 - to $\eta^1(\text{S})$ -coordination. These reactions are reversed by adding 2 equiv of AgBF_4 to **1** and **2** in acetone solvent and refluxing for 5 min. Treatment of **3** and **4** in CH_3NO_2 with dppe at room temperature overnight gave no reaction. Also, **3** did not react with bpy under the same conditions.

In summary, these studies demonstrate that the sulfur of DBT is sufficiently strongly coordinating that it is capable of cleaving the chloro bridging ligands in $[\text{Cp}^*\text{MCl}_2]_2$ to give Cp^*MCl_2 -DBT, **1** and **2**; in contrast, benzo[*b*]thiophene, 2,5-dimethylthiophene, and thiophene do not react similarly. This suggests that DBT would be a stronger S-coordinating ligand at a metal site on HDS catalysts.

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Registry No. **1**, 137516-63-3; **2**, 137540-28-4; **3**, 137516-65-5; **4**, 137516-67-7; **5**, 115565-17-8; $\text{Cp}^*\text{IrCl}_2(\text{PPh}_3)$, 66517-28-0; $\text{Cp}^*\text{IrCl}_2(\text{PMe}_3)$, 80298-81-3; $[\text{Cp}^*\text{IrCl}(\text{dppe})]\text{Cl}$, 130353-02-5; $[\text{Cp}^*\text{IrCl}_2]_2$, 12354-84-6; $[\text{Cp}^*\text{RhCl}_2]_2$, 12354-85-7.

Supplementary Material Available: Tables of crystal data, calculated hydrogen atom positions, bond angles, least-squares planes, and displacement parameters (8 pages); a table of calculated and observed structure factors (21 pages). Ordering information is given on any current masthead page.

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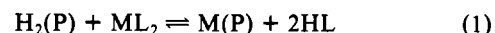
A Superior Method for the Metalation of Hydroporphyrins

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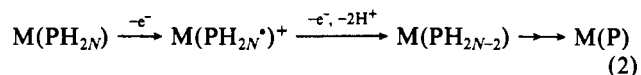
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The metalation of hydroporphyrin compounds is a more formidable challenge than the metalation of porphyrins. Both reactions are metatheses in which bases, the tetrapyrrole dianion and the ligand(s) in the metal carrier complex, compete with each

other for acids, the metal ion and protons (eq 1). The hydro-



porphyrin reaction is complicated by the tendency of free-base- and metallohydroporphyrins to undergo oxidative dehydrogenation in the presence of trace amounts of oxygen or other oxidants.¹ This reaction transforms the initial hydroporphyrin compound, whose dianion is designated PH_{2N} in eq 2, into the next most



unsaturated tetrapyrrole compound and eventually into a porphyrin compound. Thus, metalation of a hydroporphyrin can afford mixtures of complexes, which may not be easy to separate. In extreme cases, a metalloporphyrin complex may be the sole product of the reaction.

Typical reaction systems for the metalation of porphyrins^{2,3} represent a compromise between several conflicting requirements. Ideally, the solvent should dissolve both the porphyrin and the metal carrier but should not coordinate the metal ion so strongly that it competes with the porphyrin. The ligand(s) in the metal carrier complex should provide good solubility for the metal, be labile to permit ready displacement by the porphyrin, and have a weak conjugate acid. Commercial availability, stability, and ease of handling of the metal carrier complex are also desirable. In some cases, an additional base is required to shift eq 1 in favor of products, either by deprotonation of the free-base porphyrin or by complexation of the metalloporphyrin product.

Two commonly used methods for metalation of porphyrins, the acetate method^{2,3} and the dimethylformamide method,^{2,4} are less than ideal when applied to hydroporphyrins. For porphyrins, the high boiling point of DMF (153 °C) is advantageous because the solubilities of the porphyrin and metal chlorides are relatively high, reactions are rapid, and the expulsion of the HCl byproduct from an open reaction system drives metalation to completion.⁴ Hydroporphyrins typically do not survive metalation in DMF completely intact, even if the reaction is run anaerobically at temperatures much lower than the boiling point of the solvent. This behavior is mirrored by the irreversibility of the electrochemical oxidations of many hydroporphyrins in DMF, which contrasts with their reversibility in other solvents.^{5,6} Although less severe, oxidative dehydrogenation is not negligible in the acetate method. In both methods significant losses can also occur during the multistep workups and the inevitable chromatographic purification of the product.

A third method for metalation of porphyrins employs metal acetylacetonate complexes as the metal carrier.⁷ It has not achieved as wide popularity as the other methods, perhaps because most reports imply that molten phenol at 180–240 °C or refluxing 1,2,4-trichlorobenzene (214 °C) is the solvent of choice for the reaction.^{2,3,7,8} The high reaction temperature and the difficulty of removing the solvent would appear to obviate the use of this method for hydroporphyrins. Nonetheless, the *acac*⁹ method has

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some attractive features. Acac complexes are readily available, are highly soluble in weakly coordinating organic solvents, and have a weak and volatile conjugate acid. Furthermore, many anhydrous $M(\text{acac})_2$ complexes are oligomers of coordinatively unsaturated monomers and undergo facile ligand addition reactions with Lewis bases.¹⁰

Our investigation of the reactions of hydroporphyrin compounds with metal acac complexes was prompted by reports that the reaction of porphyrins with metal acac complexes in low-boiling organic solvents can result in partial to complete metalation.^{7a,11} In our experience, hydroporphyrins often metalate more rapidly than porphyrins. Thus, one might expect that hydroporphyrins would react more completely and more rapidly under the same conditions. Our results establish that the reaction is a very gentle method for metalation of hydroporphyrins. With proper care, chromatographic purification of the product is not necessary. Direct metalation to the readily oxidized Fe(II) and Mn(II) porphyrin and hydroporphyrin complexes is also possible.

Results and Discussion

The reactions of $M(\text{acac})_2$ complexes with porphyrin or hydroporphyrin free bases were investigated in several organic solvents, including benzene, toluene, and chloroform. Each combination of metal and ligand was examined at two different sets of concentrations. One was appropriate for direct monitoring by UV-vis spectroscopy. The second was typical of an actual synthetic preparation (3–10 mM or greater). The progress of each reaction was spectroscopically monitored. Reactions were continued for 30–60 min after judged complete. With the exception of $\text{Pd}(\text{acac})_2$ reactions (see below), reactions at both scales behave identically and afford the expected metalloporphyrin complex.

Many of the anhydrous $M(\text{acac})_2$ complexes are trimeric in noncoordinating solvents.¹⁰ Thus, we arbitrarily chose to employ a 3:1 metal to tetrapyrrole ratio. Although convenient, the 3:1 excess is not necessary. In most cases, complete metalation can be achieved with a slight excess of metal.

The solubilities of anhydrous Cu-, Mn-, and $\text{Zn}(\text{acac})_2$ in benzene or toluene are somewhat limited. The more concentrated reactions for these metals were initially heterogeneous but progressed satisfactorily and became homogeneous. We also investigated the reactions of these metal complexes in such mixed-solvent systems as chloroform/methanol, toluene/methanol, and toluene/chloroform. Although homogeneous from the start, reactions in these solvent mixtures do not offer any special advantage in time or product purity over the heterogeneous reactions.

There is not a consistent correlation between the saturation level of the tetrapyrrole and the time required to achieve complete metalation. For Co, Ni, and Zn, the reaction times in benzene are ordered porphyrin \geq chlorin $>$ isobacteriochlorin. However, the chlorin reacts substantially slower than the others with Cu, Mn, and Pd. In general, reactions are of convenient duration when toluene is used as the solvent for porphyrins and when benzene is used as the solvent for hydroporphyrins. With the exception of the reactions involving Fe and Mn, which are relatively slow, metalation of the porphyrin and isobacteriochlorin in these solvents is complete in 1 h or less.

The workups of the reactions are particularly mild. For $M(\text{acac})_2$ complexes with good aqueous solubility (Co, Fe, and Ni), the reaction mixture is washed with water to remove excess $M(\text{acac})_2$. For the others, workup consists of solvent evaporation followed by extraction of the desired complex from the residue

with diethyl ether (Cu, Mn, and Zn) or hexane (Pd). In most cases, the complex can be recrystallized without chromatographic purification.

The Cu,⁶ Ni,^{12,13} and Zn¹ complexes of OEP, OEC, and OEiBC⁹ have been reported previously. The UV-vis spectra of the complexes prepared by reaction of $M(\text{acac})_2$ with the free-base ligands were identical with the spectra of the respective authentic complexes. In some cases, metalation reactions employing $M(\text{acac})_2$ complexes are appreciably slower than the corresponding reactions of either metal chloride in DMF or metal acetate in acetic acid. No detectable oxidative dehydrogenation of the hydroporphyrin ligands takes place during the $M(\text{acac})_2$ reactions, however, in contrast to the reactions in DMF or acetic acid. The improved yield and elimination of a chromatographic purification step more than compensate for the longer reaction time.

The generality of the $M(\text{acac})_2$ method for hydroporphyrins with different substitution patterns is demonstrated by the success of the reactions with $\text{H}_2(\text{TPC})$ and $\text{H}_2(\text{TPiBC})$.⁹ Aside from the lower solubility of the *meso*-tetraphenyl compounds, the reactions behaved essentially the same as for the octaethyl compounds. The UV-vis spectra of $M(\text{TPC})$ and $M(\text{TPiBC})$ ($M = \text{Cu}$,^{14–16} Ni,¹³ and Zn¹⁷) agreed well with those reported previously.

The reaction of $\text{Fe}(\text{acac})_2$ with $\text{H}_2(\text{OEP})$ is reported to afford the μ -oxo complex $[\text{Fe}(\text{OEP})_2]\text{O}$.² We find that, when conducted anaerobically in toluene, the reaction affords directly the four-coordinate heme complex $\text{Fe}(\text{OEP})$. Similarly, reaction of $\text{Fe}(\text{acac})_2$ with the hydroporphyrin free-bases affords the known complexes $\text{Fe}(\text{OEC})$ and $\text{Fe}(\text{OEiBC})$.¹⁸ The complexes are pure with respect to oxidation both of the metal and of the hydroporphyrin ligand. This method for producing ferrous complexes is superior in our hands to direct metalation with FeBr_2 in the presence of a hindered base.¹⁹ The latter method involves filtration through a bed of degassed alumina, which usually results in partial oxidation of the iron and of the macrocycle. Although iron can be rereduced easily by treatment with excess ethanethiol, mixtures of $\text{Fe}(\text{OEC})$ and $\text{Fe}(\text{OEiBC})$ complexes are not readily separated.¹⁸

The Co(II) complexes of hydroporphyrins have been prepared by reaction of the free-base ligand with cobalt acetate.^{20–22} Chlorins can be reacted either in acetic acid or in pyridine. Isobacteriochlorins survive the reaction only if pyridine is the solvent. Even then, the cobalt isobacteriochlorin complexes prepared in this manner are of rather limited stability.²³ In contrast to these results, stable complexes are readily isolated from the reaction of the hydroporphyrin free bases with $\text{Co}(\text{acac})_2$ in benzene. The features of the UV-vis spectra of $\text{Co}(\text{OEC})$ and $\text{Co}(\text{OEiBC})$ correspond to those observed in a spectrum of a mixture of these complexes that was prepared by reaction of cobalt chloride and octaethylporphyrinogen.²⁴ The spectra also resemble

(9) Abbreviations: acac, 2,4-pentanedione anion; OEP, 2,3,7,8,12,13,17,18-octaethylporphyrin dianion; OEC, *trans*-2,3-dihydro-2,3,7,8,12,13,17,18-octaethylporphyrin dianion (chlorin); OEiBC, mixture of *trans*- and *cis*-2,3,7,8-tetrahydro-2,3,7,8,12,13,17,18-octaethylporphyrin dianion (isobacteriochlorin); TPC, 2,3-dihydro-5,10,15,20-tetraphenylporphyrin dianion; TPiBC, 2,3,7,8-tetrahydro-5,10,15,20-tetraphenylporphyrin dianion.
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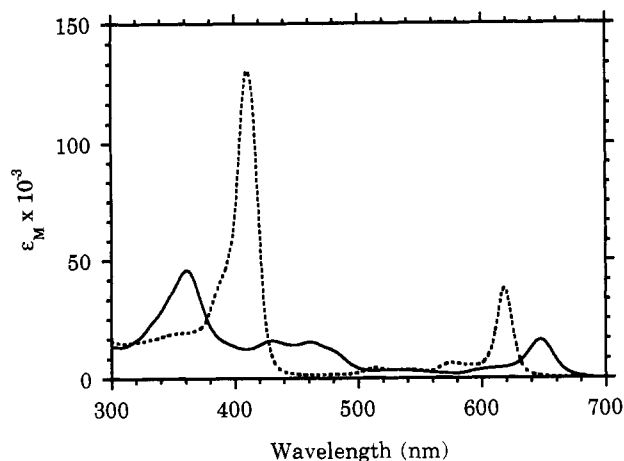


Figure 1. UV-vis spectra of Mn(OEC)Cl (—) and Mn(OEC) (---) in THF.

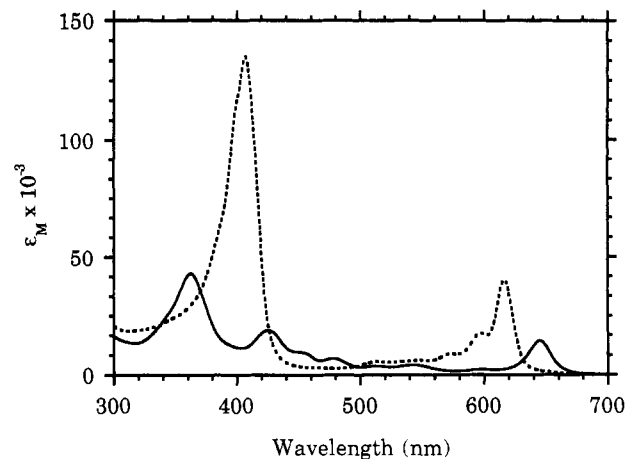


Figure 2. UV-vis spectra of Mn(OEiBC)Cl (—) and Mn(OEiBC) (---) in THF.

those reported for other Co(II) chlorins and isobacteriochlorins.^{20,25}

Manganese hydroporphyrin complexes have received minimal attention prior to this work. The presence of both manganese and chlorin compounds in chloroplasts led to early speculation that manganese chlorin complexes were involved in photosynthesis. Hence, the preparation and UV-vis spectra of a few manganese chlorin and manganese chlorophyll complexes were investigated.²⁶ Like porphyrins, Mn(II) chlorin complexes are readily oxidized to Mn(III), Mn(III) chlorins have spectra of the hyper type,²⁷ and Mn(II) chlorins have spectra of the normal type.

Reaction of Mn(acac)₂ with H₂(OEC) or H₂(OEiBC) affords a product which we concluded was a mixture of Mn(II) and Mn(III) hydroporphyrin complexes, on the basis of comparisons to reported manganese chlorin spectra. This finds support in the results of the reaction of Mn(acac)₂ with H₂(OEP). The spectrum of the crude product is a superposition of the known spectra of Mn(OEP) and Mn(OEP)X. The conclusion was confirmed by independent synthesis of the hydroporphyrin complexes. Mn(OEC)Cl and Mn(OEiBC)Cl were prepared by metalation with manganese acetate in DMF and reduced to their respective Mn(II) complexes by reaction with zinc amalgam. The spectra of Mn(OEC)Cl and Mn(OEiBC) prepared in this manner, Figure 1, resemble closely the spectra reported for the analogous TPC complexes.^{26b} The spectra of Mn(OEiBC)Cl and Mn(OEiBC), the first reported Mn isobacteriochlorin complexes, are presented in Figure 2. Pure Mn(II) complexes can be isolated directly from the Mn(acac)₂ reactions if zinc amalgam is present during the metalation. Thus, production of Mn(III) complexes appears to take place during the metal insertion phase of the reaction rather than during workup. Mn(acac)₂ might not be as resistant to oxidation as would be implied by its aerobic preparation.

Reactions with Pd(acac)₂ were examined to test whether the method is useful for a relatively inert, second-row transition metal. Reaction with H₂(OEP) in toluene results in rapid formation of Pd(OEP). Reactions with hydroporphyrins are considerably more complicated. When the initial concentration of H₂(OEC) or H₂(OEiBC) is 7–10 mM or greater, the reaction is rapid and affords the known,⁶ expected product Pd(OEC) or Pd(OEiBC), respectively. As the initial concentration of macrocycle is decreased, the reaction slows and a second species appears and eventually becomes the dominant product. At low concentrations, both H₂(OEC) and H₂(OEiBC) afford blue-violet species with identical UV-vis spectra. The spectra have a prominent band at 617 nm, a wavelength considerably longer than that of the visible bands of either Pd(OEC) or Pd(OEiBC),⁶ and bear a striking

resemblance to that of the protonated free-base chlorin H₃-(OEC)⁺.²⁸ Owing to the limited scale of reactions which afford these products, we have not been able to obtain sufficient material to effect a good purification. However, preliminary data suggest that both products are six-coordinate Pd complexes in which Pd lies above the N₄ plane of an OEC macrocycle and is also coordinated to acac or other ligands. Complexes with metals substantially out of a macrocycle plane and simultaneously coordinated to other ligands (including acac) have been reported for transition-metal and lanthanide porphyrins,^{3,8} lanthanide phthalocyanines,²⁹ and Pd macrocyclic complexes.³⁰ A product with a long-wavelength visible band was also reported to be formed in the reaction of H₂(OEiBC) with bis(benzonitrile)palladium chloride.³¹

Conclusion

The reaction of metal acetylacetonate complexes with free-base porphyrin and hydroporphyrin compounds in noncoordinating organic solvents provides a very gentle method for metalation of hydroporphyrins. The method offers no particular advantage in preparing relatively robust porphyrin complexes with most of the metals that we examined. Two significant exceptions are the reactions of iron and manganese, where direct metalation to the readily oxidizable four-coordinate Fe(II) and Mn(II) porphyrin complexes can be achieved. The acac method is far superior to other methods for the metalation of hydroporphyrins. Although metal insertion can proceed more slowly than with other methods, it is accompanied by negligible loss of the desired hydroporphyrin product through oxidative dehydrogenation of the ligand. Chromatographic purification of the product is usually not required.

We are currently investigating the extension of this method to other, more substitutionally inert metal acetylacetonate complexes.

Experimental Section

M(acac)₂ complexes (M = Co,³² Cu,³³ Fe,³⁴ Mn,³⁵ Ni,³² and Zn³⁶) were prepared by literature methods. Pd(acac)₂ was purchased from Aldrich. All were dried overnight in a vacuum oven at an appropriate temperature (between 65 and 100 °C). H₂(OEP),³⁷ H₂(OEC),³⁸ H₂-

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(OEiBC),¹ H₂(TPC),³⁸ and H₂(TPiBC)³⁸ were prepared by literature methods. Zn amalgam was 3% by weight. Other reagents and solvents were reagent or HPLC grade, were purified as previously reported,³⁹ and were thoroughly degassed prior to use. All reactions and manipulations were carried out under a nitrogen or argon atmosphere by use of Schlenk techniques or in a Vacuum/Atmospheres Co. drybox.

Generalized M(acac)₂ Metalation Procedure. In a typical reaction, 50 mg of the tetrapyrrole free base, 3 equiv of the M(acac)₂ complex, and 25 mL of degassed solvent were placed in a 100-mL Schlenk flask which contained a magnetic stirbar and was equipped with a reflux condenser topped by a gas Schlenk inlet. Larger amounts of reactants and more concentrated solutions can be used, if desired. When M = Mn, a spatula tip of 3% Zn amalgam was also added to the reaction mixture. The solution (or in some cases mixture) in the flask was degassed, stirring was initiated, and the contents were heated to reflux under a nitrogen atmosphere. The progress of the reaction was monitored periodically by UV-vis spectroscopy. Heating was continued for 30 min longer than the time judged necessary to complete the reaction. The solution was then cooled to room temperature and worked up by the method indicated below.

For reactions with Co, Fe, or Ni, the reflux condenser was replaced with a rubber septum. A 50-mL portion of degassed water was added, and the resulting mixture was stirred to extract the excess M(acac)₂ complex. The aqueous layer was removed with a cannula. Washing was repeated until the aqueous layer was free of the color of the M(acac)₂ complex. Typically, 3 × 50 mL was required. The organic layer was dried over MgSO₄ and filtered, and the solvent was removed under vacuum. The yield of crude product was nearly quantitative. The crude product was recrystallized from an appropriate solvent.

For reactions with Cu, Mn, Pd, and Zn, the reaction solvent was removed under vacuum. The residue was extracted with diethyl ether (hexane for Pd), and the resulting mixture was filtered to remove the excess M(acac)₂. The extraction was repeated until the ether was no longer colored by the metallotetrapyrrole product. The ether extracts were combined, and the ether was removed under vacuum. The crude product was recrystallized from an appropriate solvent.

Co(OEC). UV-vis (C₆H₆) λ_{max}, nm (10⁻³ε_M, M cm⁻¹): 391 (126.7), 558 (8.1), 613 (33.2).

Co(OEiBC). UV-vis (C₆H₆) λ_{max}, nm (10⁻³ε_M, M cm⁻¹): 380 (50.0), 517 (7.0), 547 (10.0), 590 (38.5).

Mn(OEC). UV-vis (THF) λ_{max}, nm (10⁻³ε_M, M cm⁻¹): 411.3 (130.6), 514.8 (4.3), 542.4 (3.6), 576.4 (6.3), 619 (38.0).

Mn(OEiBC). UV-vis (THF) λ_{max}, nm (10⁻³ε_M, M cm⁻¹): 406 (135), 511.2 (5.6), 571.6 (8.8), 597.4 (17.6), 616.4 (40.3).

Mn(OEC)Cl was prepared by a modification of the literature method for the synthesis of Mn(OEP)Cl.⁴⁰ A 50-mg (0.094-mmol) sample of H₂(OEC) was dissolved in 20 mL of warm dimethylformamide contained in a Schlenk flask, and 50 mg (0.20 mmol) of Mn(CH₃CO₂)₂·4H₂O was added. The mixture was degassed and brought to reflux under a nitrogen atmosphere. The reaction was complete after 4 h. The green solution was cooled to room temperature and was added to an ice-cold solution of 1.6 g of NaCl in 20 mL of water. A yellow-green precipitate formed. It was collected by filtration, washed with water, and briefly air-dried. It was then redissolved in 10 mL of methanol, the resulting solution was poured into 10 mL of 6 M HCl, and the mixture was shaken well. A green precipitate formed. It was collected by filtration, washed with water until the washings were neutral, and then dried in vacuo. The crude product was purified by chromatography on an alumina column (activity 3, neutral). Elution was performed with 100:1 CH₂Cl₂/methanol. The product eluted in a yellow-green band. A red band of Mn(OEP)Cl remained on the column. Evaporation of the eluate afforded the product, Mn(OEC)Cl. UV-vis (THF) λ_{max}, nm (10⁻³ε_M, M cm⁻¹): 360.6 (46.0), 431.7 (17.0), 464.4 (17.2), 479.8 sh (12.9), 531.7 (4.3), 601.9 (4.4), 648.1 (16.8).

Mn(OEiBC)Cl was prepared by the same procedure as Mn(OEC)Cl. One modification was that the NaCl and HCl washes were performed anaerobically. UV-vis (THF) λ_{max}, nm (10⁻³ε_M, M cm⁻¹): 362.3 (43.1), 427.9 (18.8), 453.5 (9.6), 478.2 (7.2), 512.3 (4.0), 541.3 (4.3), 598.2 (2.4), 645.2 (14.5).

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(39) Stolzenberg, A. M.; Glazer, P. A.; Foxman, B. M. *Inorg. Chem.* **1986**, *25*, 983.

(40) Boucher, L. J.; Garber, H. K. *Inorg. Chem.* **1970**, *9*, 2644.

Additions and Corrections

1991, Volume 30

Ghazar Aharonian, Lilliane G. Hubert-Pfalzgraf,* Abdenasser Zaki, and Guy Le Borgne: Preparation and Structure of a Mixed Niobium(I) Isocyanide Carbonyl Complex with a Bent C-N-C Linkage.

Page 3105. In the Introduction, the fourth sentence should be deleted and replaced by "Some niobium and tantalum carbonyl isocyanide derivatives have been mentioned" (Warnock, G. F. P.; Sprague, J.; Fjare, K. L.; Ellis, J. E. *J. Am. Chem. Soc.* **1983**, *105*, 672). At the end of the paragraph, add "A related complex, Nb(CO)Cl(CNMe)(dmpe)₂, has been obtained quite recently by the group of Lippard and used for CO/CNR reductive coupling" (Carnahan, E. M.; Lippard, S. J. *J. Am. Chem. Soc.* **1990**, *112*, 3230).

Page 3107. At the end of the third complete paragraph, add "Strongly bent (129–139°) C-N-R angles have been observed on other M-(CNR)(CO)(dmpe)₂Cl complexes (M = Nb, R = Cy; M = Ta, R = Et, *t*-Bu)" (Carnahan, E. M.; Bott, S. G.; Lippard, S. J. To be submitted for publication).—Lilliane G. Hubert-Pfalzgraf

Allen Applett, Tristram Chivers,* A. Wallace Cordes,* and Rainer Vollmerhaus: Synthesis and Structure of the Norbornene Adduct of 1,3,5,2,4,6-Trithiatriazinium Tetrachloroaluminate [C₇H₁₀S₃N₃][AlCl₄].

Page 1393. In Table I, the assignments for two of the ¹³C NMR resonances were inadvertently transposed. The correct assignment column is given below:

C(6) and C(7)
C(4)
C(3) and C(5)
C(1) and C(2)

—Tristram Chivers