

(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 Ablett, 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