produced in the presence of ultrasound rival the reactivities of powders produced in the absence of ultrasound. Though there are no published results on the reactivity of Rieke nickel in the Ullman coupling of benzyl bromide to bibenzyl, results obtained in our laboratory (Table IV, entries 21 and 22) indicate no reactivity difference between nickel generated in the presence or absence of ultrasound. As with other reactions involving metal powders[16] the facility of the Ullman coupling of benzyl bromide to bibenzyl is not solely dependent on the powder itself but also on the presence of iodide salts. In the case of the copper-induced Ullman coupling of benzyl bromide, ultrasound significantly facilitates coupling over room-temperature stirring. This may be attributed to the ability of ultrasound to clean the surface of the copper powder.

The use of a common ultrasonic cleaner to facilitate reduction of metal halides represents an attractive modification. Reductions may be performed with substantial time savings and no increase in complexity of apparatus. We are attempting to expand the scope and synthetic applications of metal powders produced in the presence of ultrasound. We are also investigating possible means of enhancing powder reactivity.

Experimental Section

Workup of reactions were performed in air. Untreated nickel powder, when isolated, would smoke with evolution of heat on exposure to air. Unreacted metal powders were either stored under N_2 or immediately quenched with ice. Tetrahydrofuran was distilled under N_2 from sodium/benzophenone ketyl immediately before use. Benzyl bromide was distilled and protected from light. All other liquid reagents were distilled. Anhydrous $MgCl_2$ was prepared from magnesium turnings and 1,2-dichloroethane immediately before use. All other anhydrous metal halides were dried in a vacuum oven overnight (1 mm/80 °C) before use. Naphthalene was sublimed before use.

Proton NMR spectra were obtained by using an EM-390 spectrometer. Gas chromatographic analyses were performed on a Varian Series 2400 and a Varian Aerograph Model 920. Melting points were obtained on a Thomas capillary melting point apparatus.

Preparation of Ethyl 3-Phenyl-3-hydroxypropanoate from Benzaldehyde, Ethyl Bromoacetate, and Zinc. In a clean, dry, single-neck, round-bottom flask equipped with N_2 inlet and condenser were placed $ZnCl_2$ (1.65 g, 12 mmol), THF (10 mL), and lithium dispersion (0.17 g, 24 mmol, 30% in mineral oil). The mixture was sonicated. The reduction was very vigorous, so sonication was stopped intermittently to maintain control. After 1 h, sonication was stopped, and a fine, gray, dispersion of zinc was obtained. The THF was removed in vacuo and replaced by diethyl ether (10 mL) and then the flask cooled to 0 °C. Ethyl bromoacetate (1.67 g, 10 mmol) and benzaldehyde (0.85 g, 8 mmol) were weighed separately. One-tenth of the bromo ester was added to the zinc/ether dispersion. The remaining bromo ester and benzaldehyde were mixed and added dropwise to the dispersion. After addition was complete, the mixture was warmed to room temperature and stirred vigorously for 1 h, then poured into cold water, and stirred for 15 min. Extraction with ether, drying with $MgSO_4$, and vaccum evaporation left a pale yellow liquid. Analysis by GLC and NMR (by comparison to an authentic sample) showed the liquid to be the desired product, 1.39 g, 90% yield based on benzaldehyde.

Synthesis of Bibenzyl from Benzyl Bromide and Copper. In a clean, dry, three-neck round-bottom flask equipped with condenser, N_2 inlet, and rubber septum were placed $CuBr_2$ (3.80 g, 0.017 mol, 10% excess), lithium powder (0.22 g, 0.031 mol), THF (20 mL), and naphthalene (0.40 g, 3.1 mmol). The mixture was sonicated with swirling approximately every 15 s. Upon sonication, the mixture became blue-black and then began to boil for about 3 min. After completion of the boil, the mixture turned black with some lithium remaining at the surface. Swirling had to be continued (once every 15 s) to keep the lithium powder from moving up the sides of the flask. After 25 min, no visible lithium remained in the flask. Sonication was continued for an extra 30 min, and then benzyl bromide (0.57 mL, 0.0048 mol) was added through the septum via syringe to the brownish black solution without interruption of sonication. Sonication was continued for 30 min and then stopped. The reaction mixture was filtered and the filtrate evaporated at low pressure to remove THF. Carbon tetrachloride was added to the residue. After a second filtration to remove lithium salts, NMR analysis of the CCl_4 solution showed 50% unreacted benzyl bromide and 50% bibenzyl. A similar reaction run with copper iodide showed only bibenzyl after 30 min, which was confirmed by GLC analysis.

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Communications

Organonickel Cyanide Chemistry. Reactions of $[(PhC{\equiv}CPh)Ni(CN)_2]^{2-}$. An Improved Synthesis of $[Ni(CN)_2(CO)_2]^{2-}$

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Summary: The reactions of $(K{\cdot}18\text{-crown-}6)_2[(\eta^2\text{-}PhC{\equiv}CPh)Ni(CN)_2]$ (**1**) with CO, $P(OMe)_3$, alkynes, and α-bromo-*p*-xylene are described. Reaction of **1** with CO provides a route to a pure, stable salt of the $Ni(CN)_2(CO)_2{}^{2-}$ anion. Compound **1** is a catalyst for the trimerization of terminal alkynes.

We recently reported that crown ethers facilitate the study of the reactivity of the long known but heretofore intractable compound $K_4Ni(CN)_4$.[1] One compound synthesized from $K_4Ni(CN)_4$ using this crown ether solid–liquid-phase transfer strategy was $(K{\cdot}18\text{-crown-}6)_2[(\eta^2\text{-}PhC{\equiv}CPh)Ni(CN)_2]$ (1), a rare example of a stable organonickel cyanide complex and the first member of the well-known (alkyne) NiL_2 class of compounds with L = CN^-. Because of the novelty of 1, its chemical reactivity is of interest. We have examined the reactivity of 1 and report the initial results of a study of the ligand substitution chemistry of 1. Of special interest are the discoveries that 1 is a precursor to a new, particularly tractable

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[1] del Rosario, R.; Stuhl, L. S. *J. Am. Chem. Soc.* **1984**, *106*, 1160–1.

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salt of the $Ni(CN)_2(CO)_2{}^{2-}$ anion (2) and that 1 is a catalyst for the trimerization of alkynes.

Reaction of 1 with excess CO is rapid; alkyne rather than cyanide is lost, as shown in eq 1. The product **2a** is a new

$$(\mathrm{K{\cdot}18\text{-}crown\text{-}6})_2[(\eta^2\text{-}\mathrm{PhC{\equiv}CPh})\mathrm{Ni(CN)_2}] \xrightarrow[\text{THF}]{\text{CO, 1 atm}} (\mathrm{K{\cdot}18\text{-}crown\text{-}6})_2[\mathrm{Ni(CN)_2(CO)_2}] \quad (1)$$

salt of the $Ni(CN)_2(CO)_2{}^{2-}$ anion[2] (2). Previously known salts of 2 are the simple potassium salt[2] **2b**, not isolated pure, and the Ph_4P^+ salt[3] **2c**. Compound **2a** can be isolated in pure form in 90% yield as yellow crystals from acetonitrile–ether.[4] In contrast to **2c**,[5] **2a** is thermally stable and readily purified by recrystallization. Thus, a pure, tractable salt of the reactive[6] complex 2 is available without resort to liquid ammonia chemistry (beyond the synthesis of $K_4Ni(CN)_4$[7]).

Expectedly,[8] the crown ether complexation in **2a** causes substantial changes in the cyanide and carbonyl IR stretching frequencies compared with **2b**. There are also interesting differences between the IR spectra of **2a** in solution and in the solid state.[4] The changes in the IR spectrum upon dissolving **2a** in acetonitrile are consistent with the transformation of a "noncation-perturbed" structure for **2a** in the solid state into a contact ion pair in solution. The shift to lower frequencies for ν_{CN} suggests that the "crowned" potassiums are interacting with 2 in solution with cyanide site preference.[9,10] Interaction of the cyanide nitrogen[10] with potassium cation should decrease the CN bond order and simultaneously increase the CO bond order.[8]

An interesting sidelight of the preparation of **2a** is that the alkyne displacement reaction is reversible. If the reaction mixture (eq 1) is allowed to stand for several hours after removal of the CO atmosphere, a trace of 1 can be detected by IR and by its deep red-purple color, suggesting that the alkyne displacement is an equilibrium process. Consistent with this observation is that diphenylacetylene reacts with preformed **2a** to give a solution which by IR is largely unreacted starting materials but with small amounts of 1 and another species ($\nu_{CN} = 2096$, $\nu_{CO} = 1973$, $\nu_{C\equiv C} = 1823$ cm^{-1}), assigned as (K·18-crown-6)[(η^2-PhC≡CPh)Ni(CO)(CN)]. We have been unable to isolate this latter complex in pure form.

Addition of three or more equivalents of phenylacetylene to an acetonitrile solution of 1 results in the complete disappearance of the added alkyne, but there is no evidence for the formation of $[(PhC{\equiv}CH)Ni(CN)_2]^{2-}$. Instead, 1,2,4-triphenylbenzene is produced as well as some polymer and a trace of a tetraphenylbenzene.[11] A new organonickel species is produced, but it has not been possible to obtain it as a pure compound. This trimerization reaction is catalytic. Using a large excess of phenylacetylene (300 equiv) gives a 40% yield (based on phenylacetylene) of 1,2,4-triphenylbenzene plus traces of phenylacetylene dimers (*m/e* 204) and tetramers (*m/e* 382). Most of the remaining organic material is present as nonvolatile polymers which have not been further examined.

Compound 1 reacts extremely slowly with excess (40 equiv) diphenylacetylene, producing only traces of hexaphenylbenzene after 6 days of reaction at ambient temperature (The thermal stability of 1 is limited much above room temperature: in refluxing acetonitrile decomposition is detectable in 30 min). Similarly, a small amount of 1,2-diphenyl-3,4,5,6-tetraethylbenzene was isolated from a 5-day reaction of 1 with 20 equiv of 3-hexyne. Other *terminal* alkynes are oligomerized catalytically, however. For example, 1-butyne gives a mixture of trimers and tetramers (probably cyclic).[12] 3-Hexyne does not appear to oligomerize under these conditions.

Compound 1 does not react with PPh_3 (25 °C, 24 h), in contrast to $K_4Ni(CN)_4$ which undergoes complete ligand displacement to give $Ni(PPh_3)_4$.[1] This observation is consistent with phosphine substitution in $Ni(NO)(CN)_3{}^{2-}$[13] and $(C_5H_5)Co(CN)(CO)^-$,[14] in which equilibria are set up involving phosphine displacement of cyanide but lying on the side of free phosphine and complexed cyanide. Less bulky trimethyl phosphite reacts rapidly with 1. With 4 equiv of phosphite an equilibrium mixture of $Ni[P(OCH_3)_3]_4$ and (K·crown)$[Ni[P(OCH_3)_3]_3(CN)]$ (3) is formed. The same equilibrium may be established from $Ni[P(OCH)_3)_3]_4$ and (K·crown)CN. Monitoring this reaction by IR and ^{31}P NMR shows the mechanism of this substitution to be complex, with several intermediates being formed. Attempts to isolate products from reactions with lower ratios of phosphite to nickel complex indicate that at least some of the intermediate steps are equilibria as well.

Reactions of 1 with electrophiles are complex. We have examined reactions with CH_3I, $C_5H_{11}Br$, $(CH_3)_3CC(O)Cl$, and p-$CH_3C_6H_4CH_2Br$; only the last gives a simple organic product, 1,2-di-*p*-tolylethane, in 33% yield (from a 1:2 reaction, based on Ni). The organonickel products are thermally sensitive and have defied isolation. This reactivity pattern is suggestive of the involvement of electron-transfer processes. Another possible indication of the reluctance of 1 to undergo oxidative addition is its failure to react with H_2 (1 atm, 25 °C, 48 h).

Several conclusions can be drawn from this study at this point. First, 1 is a reactive compound, in apparent contrast to other (alkyne)NiL_2 complexes for which little reaction chemistry is known; this conclusion emphasizes the special attributes of the cyanide ligand, a sterically nondemanding, strong σ-donor and reluctant π-acceptor. This study also provides another example[14] of the nongeneral nature of electrophilic N-alkylation of metal cyanides[15] when low-valent complexes are taken into consideration. The observation of catalytic activity of 1 shows that the cyanide

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(4) The compound crystallizes as an acetonitrile solvate. Anal. Calcd for $C_{30}H_{51}N_3O_{14}K_2Ni$: c, 44.23; H, 6.31; N, 5.16; Ni, 7.21. Found: C, 44.01; H, 6.31; N, 5.16; Ni, 7.26. IR (CH_3CN): 2076 (sh), 2067 (s) cm^{-1} ($\nu_{C\equiv N}$), 1940 (s), 1865 (s) cm^{-1} (ν_{CO}) IR (mineral oil mull): 2089 (sh), 2074 (s) cm^{-1} ($\nu_{C\equiv N}$), 1927 (s), 1844 (s) cm^{-1} (ν_{CO}).

(5) Although we found that $(Ph_4P)_2Ni(CN)_2(CO)_2$, prepared according to ref 3b, gave satisfactory elemental analyses, the IR spectrum revealed contamination with $Ni(CN)_4{}^{2-}$ and an unidentified extra carbonyl absorption. We found this material to be unstable at ambient temperature under N_2 and were unable to increase its purity by recrystallization.

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(10) Darensbourg, M. Y.; Barros, H. L. C. *Inorg. Chem.* **1979**, *18*, 3286–3288. Contact ion pairs of $NaFe(CO)_5CN$ and $NaMo(CO)_5$ in ether show cyanide site preference.

(11) 1,2,4-Triphenylbenzene was identified by GC–MS and ^1H NMR.

(12) GC–MS analysis shows both 1,2,4- and 1,3,5-triethylbenzene and several tetramers, probably cyclic (*m/e* 216, strong).

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Organometallics 1986, 5, 1262–1264

ligand is as compatible as carbonyl or phosphine with catalytic activity. The effect of counterion variation on cyanometallate stability, i.e., **2a** vs. **2c**, is consistent with other observations from this laboratory;[16] the causes of these counterion effects are not well understood, however, and are worthy of further study. Perhaps most importantly, this work emphasizes that organometallic cyanide chemistry in aprotic solvents is possible. This is not a trivial point. There are few previous studies of the reactivity of organocyanometalates in aprotic solvents and, to our knowledge, none in which one low-valent organocyanometalate was converted to another (i.e., **1** → **2**).

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Registry No. **1**, 88657-44-7; **2a**, 88728-24-9; **3**, 101834-94-0; $Ni[P(OCH_3)_3]_4$, 14881-35-7; p-$CH_3C_6H_4CH_2Br$, 104-81-4; phenylacetylene, 536-74-3; 1,2,4-triphenylbenzene, 1165-53-3; diphenylacetylene, 501-65-5; 3-hexyne, 928-49-4; 1-butyne, 107-00-6; trimethyl phosphite, 121-45-9; 1,2-di-p-tolylethane, 538-39-6.

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Novel Cationic Dinuclear Ruthenium(I) Complexes Containing Carbonyl and Isocyanide Ligands. The X-ray Crystal Structures of $[Ru_2(CO)_2(CN\text{-}t\text{-}Bu)_8](PF_6)_2$ and $[Ru_2(CNCH_2C_6H_5)_{10}](PF_6)_2$

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Summary: Nucleophilic substitution reactions on *catena*-$[Ru(CO)_2(O_2CCH_3)]$ with isocyanides provide synthetic routes to the dinuclear ruthenium(I) complexes $[Ru_2(CO)_2(CN\text{-}t\text{-}Bu)_8](PF_6)_2$, $[Ru_2(CO)(CNR)_9](PF_6)_2$, and $[Ru_2(CNR)_{10}](PF_6)_2$ ($R = C_6H_5CH_2$, 2,6-$Me_2C_6H_3$) which are derivatives of the unknown d^7–d^7 dimer $[Ru_2(CO)_{10}]^{2+}$. The X-ray crystal structures of $[Ru_2(CO)_2(CN\text{-}t\text{-}Bu)_8](PF_6)_2$ and $[Ru_2(CNCH_2C_6H_5)_{10}](PF_6)_2$ have been determined.

Homoleptic metal isocyanide complexes and complexes which contain predominantly isocyanide ligands are surprisingly rare when compared to the diversity of metal carbonyl complexes known.[1-3] Why this is so is still not clear, but it can probably be related to the different steric and electronic properties of the isocyanide ligand when compared to the carbonyl ligand. We have recently begun investigating the chemistry of the polymeric ruthenium(I) complexes *catena*-$[Ru(CO)_2(O_2CR)]$[4] and of the dinuclear osmium(I) complexes $[\{Os(CO)_3(O_2CR)\}_2]$[4] as precursors to ruthenium(I) and osmium(I) chemistry. Herein we report on the reactions of *catena*-$[Ru(CO)_2(O_2CR)]$ with isocyanides in labilizing alcohol solvents which produce rare examples of dinuclear homoleptic isocyanide complexes and provide the first rational[5] synthetic route to derivatives of the unknown $[M_2(CO)_{10}]^{2+}$ (M = Fe, Ru, Os) dicationic dimers. Confirmatory X-ray structure determinations on two of these salts have been carried out.

The new dinuclear ruthenium(I) complexes are prepared by treating a suspension of the polymer *catena*-$[Ru(CO)_2(O_2CCH_3)]$ (**1**) in refluxing ethanol with an excess of isocyanide (typically a 6:1 isocyanide:ruthenium molar ratio), followed by precipitation with NH_4PF_6. Short reaction times (<10 min) produce the salts $[Ru_2(CO)_2(CN\text{-}t\text{-}Bu)_8](PF_6)_2$ (**2**) and $[Ru_2(CO)(CNR)_9](PF_6)_2$ (**3**, $R = C_6H_5CH_2$; **4**, R = 2,6-$Me_2C_6H_3$), whereas more prolonged reaction times (72 h) have been found to yield the homoleptic dimers $[Ru_2(CNR)_{10}](PF_6)_2$ (**5**, $R = C_6H_5CH_2$; **6**, R = 2,6-$Me_2C_6H_3$[5]). Complexes **3** and **4** convert on heating with isocyanide to **5** and **6**, but we have found no evidence, under either thermal or photochemical conditions, for the further substitution of **2** by isocyanide.

Lower substituted dinuclear cations have not been observed in the reactions of **1** with isocyanides in alcohol solvents even with stoichiometric additions of isocyanide. What is observed in these reactions though is the complete consumption of isocyanide with a corresponding partial conversion of **1**. The process by which **1** reacts with isocyanide in alcohol possibly occurs via a combination of routes involving depolymerization of dimeric Ru(I) units by either isocyanide[6] or solvent molecules[7,9] giving soluble $[\{Ru(CO)_2L(O_2CCH_3)\}_2]$ complexes, and the labilization[10] of the acetato ligands in such species to give cationic intermediates of the type $[Ru_2(CO)_nL_m(O_2CCH_3)]^+$ or $[Ru_2(CO)_nL_m]^{2+}$. The effects of the positive charge residing on such intermediates would be expected to favor substitution of carbonyl ligands by the more nucleophilic isocyanide ligands, thus leading to high levels of substitution.

Complexes **2**–**6** exhibit in solution an unusual spectrum of stereochemical flexibility. For **4** and **6** single methyl

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[†] On study leave from the Department of Chemistry, University of South Africa, Pretoria.

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(5) We have recently also obtained the cation $[Ru_2(CNC_6H_3Me_2\text{-}2,6)_{10}]^{2+}$ as a byproduct of melting $[(\eta^1,\eta^2\text{-}C_8H_{13})Ru(CNC_6H_3Me_2\text{-}2,6)_4]PF_6$. The X-ray crystal structure of $[Ru_2(CNC_6H_3Me_2\text{-}2,6)_{10}](BPh_4)_2$ has been determined showing this molecule to possess an eclipsed configuration of isocyanide ligands and a ruthenium–ruthenium bond of 3.003 Å: Chalmers, A. A.; Liles, D. C.; Meintjies, E.; Oosthuizen, H. E.; Pretorius, J.; Singleton, E. *J. Chem. Soc., Chem. Commun.* **1985**, 1340.

(6) Neutral complexes of the type $[\{Ru(CO)_2L(O_2CCH_3)\}_2]$ (L = donor ligand) can be obtained directly from **1** by reaction in solvents such as benzene or diethyl ether. Lewis and co-workers[4] have obtained an extensive series of phosphine-substituted products in this way.

(7) The polymer **1** is soluble in donor solvents such as acetonitrile and dimethyl sulfoxide from which the labile complexes $[\{Ru(CO)_2(solvent)(O_2CCH_3)\}_2]$[4,8] can be isolated. In ethanol this solvolysis reaction does not occur as readily, but warming of **1** in ethanol does produce a slight yellow coloration, suggesting the formation of similar species.

(8) Albers, M. O.; Singleton, E., unpublished observations.

(9) Complexes of the type $[\{Ru(CO)_2(solvent)(O_2CCH_3)\}_2]$ have been found[8] to react readily with ligands L to give as products $[\{Ru(CO)_2L(O_2CCH_3)\}_2]$. Indeed, addition of t-BuNC to the pale yellow solution obtained by heating **1** in ethanol[7] gives $[\{Ru(CO)_2(CN\text{-}t\text{-}Bu)(O_2CCH_3)\}_2]$, providing evidence for the formation of solvolyzed dinuclear Ru(I) species in ethanol.

(10) We have found that alcohol solvents are particularly effective at promoting cation formation. When reactions are carried out in solvents such as toluene, heptane, or tetrahydrofuran, a range of *neutral* substitution products are obtained. These will, however, be discussed in more detail elsewhere.

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