Table I. Analytical Data for UCp_3X

compd	elemental[a] anal.	^1H NMR[b]	IR[c]	mass[d]
$UCp_3{}^{35}Cl$		3.48	3080 (w), 2910 (vw), 1440 (m), 1010 (s), 910 (w), 785 (vs)	468 (26.4), 433 (2.3), 403 (100), 368 (3.0), 338 (95.8), 303 (2.2), 273 (12.5)
$UCp_3{}^{79}Br$		3.72	3101 (w), 2925 (vw), 1440 (s), 1360 (w), 1264 (vw), 1218 (vw), 1064 (vw), 918 (m), 824–780 (vs)	512 (38.8), 447 (81.2), 382 (100), 368 (3.5), 319 (8.21), 317 (9.4), 303 (3.5)
UCp_3I	C 31.48 (32.14)	4.36	3100 (w), 2924 (vw), 1440 (s), 1360 (w), 1264 (vw), 1218 (vw), 1070 (m), 1016 (s), 910 (m), 826–780 (vs)	560 (47.6), 495 (64.3), 433 (76.2), 430 (83.3), 368 (100), 303 (40.5)

[a] Calculated values in parentheses. [b] Me_4Si as internal standard. [c] KBr pellet. In cm^{-1}. [d] Relative intensities in parentheses.

Table II. Gaseous Products from Reactions of Uranium Powder with Ethyl Bromide and Cyclopentadiene and Ethyl Bromide Uranium Powder[a]

reaction with HCp and C_2H_5Br: C_2H_4 (75), C_2H_6 (100), C_4H_8 (2), C_4H_{10} (2)

reaction of C_2H_5Br: H_2 (4), CH_4 (1), C_2H_4 (100), C_2H_6 (25), C_3H_6 (1), C_3H_8 (<1), C_4H_{10} (2)

[a] Parentheses indicate relative intensity.

evacuated system. The reacton mixture was heated and refluxed until it turned to a deep brown. After a 3-h reaction period, the mixture was filtered; the filtrate was pumped to dryness, and the solid was transferred for sublimation. The sublimate with a yield of 40% was characterized as UCp_3Cl (60% yield).

Reaction of Ethyl Bromide with Uranium Powder. An excess of ethyl bromide (5 mL) was added to a flask containing 3 g of freshly prepared uranium powder in an evacuated system. The reaction mixture was stirred. Three millimoles of gaseous products were collected after an 18-h reaction period. The results are listed in Table II. The reaction mixture was filtered; the solid was extracted with water and filtered through a glass fritt. The Br/U ratio was observed to be 2.4.

Reaction of Ethyl Bromide with Tricyclopentadienyluranium. An excess of ethyl bromide (5 mL) or ethyl iodide (5 mL) was added to a solution of tricyclopentadienyluranium (2.0 g) in 50 mL of benzene in the presence of a small amount of highly reactive uranium powder in an evacuated system. The workup was the same as the previous ones. The solid was characterized as UCp_3Br.

Results and Discussion

The direct reaction of uranium powder with cyclopentadiene and alkyl halides probably involves an oxidative-addition mechanism. A similar pathway might be followed by the reaction of UCp_3 and C_2H_5Br in the presence of a small amount of uranium powder. However, what was observed in the NMR spectra was the increase of the product, UCp_3Br, accompanied by the corresponding decrease of UCp_3, and not a trace ascribable to the intermediate, $C_2H_5UCp_3Br$. The analytical data of the gaseous products from the latter are shown in Table II. Comparable quantities of ethane and ethylene were accompanied by small quantities of butane and butene. A more complicated pattern was obtained from the reaction of uranium powder with ethyl bromide. The gaseous products could be ascribed to the decomposition of a metal alkyl or/and a precursor complex from electron-transfer dehalogenation to the formation of free radicals[9,10] and to a coupling of ethyl radicals which gave butane and butene. The evolution of methane, propane, and propene is interesting; however, this observation is not ready to be interpreted.

A similar phenomenon of coupling was also found recently in the reaction of carbon tetrachloride with uranium powder, wherein a major product, C_2Cl_6, was obviously from the coupling of the $\cdot CCl_3$ radical.[11]

The reaction of methylene chloride or carbon tetrachloride, cyclopentadiene, and uranium powder brought forth UCp_3Cl with a yield of 40% and oily products ascribable to various chloro compounds. The mass spectra of UCp_3 and UCp_3Br at 70 eV appear normal; the first two Cp ligands are easily ruptured whereas the remaining one is very difficult to remove. The U–Cl and U–Br bonds are more difficult to rupture than the more covalent U–C bonds.[12] However, U–I is more easily ruptured than U–C for UCp_3I.

Registry No. UCp_3Cl, 1284-81-7; UCp_3Br, 67507-08-8; UCp_3I, 69030-40-6; UCp_3, 54007-00-0; C_2H_5Br, 74-96-4; C_2H_5I, 75-03-6; CH_2Cl_2, 75-09-2; CCl_4, 56-23-5; HCp, 542-92-7.

---

(1) Chang, C. C.; Sung-Yu, N. K.; Hseu, C. S.; Chang, C. T. *Inorg. Chem.* **1979**, *18*, 885.
(2) Marks, T. J.; Fischer, R. D. "Organometallics of the f-Elements"; D. Reidel: Dordrecht, Boston and London, 1979; p 113.
(3) Reynolds, L. T.; Wilkinson, G. *J. Inorg. Nucl. Chem.* **1956**, *2*, 246.
(4) Fischer, E. O.; Hristidu, Y. *Z. Naturforsch, Anorg. Chem., Org. Chem. B*: **1962**, *17*, 275.
(5) Marks, T. J.; Seyam, A. M.; Wachter, W. A. *Inorg. Synth.* **1976**, *16*, 147.
(6) Wang, L. C.; Lee, H. C.; Lai, W. C.; Chang, C. T. *J. Inorg. Nucl. Chem.* **1978**, *40*, 507. Wang, L. C.; Hung, H. C.; Lee, H. C.; Chang, C. T. *J. Chem. Soc., Chem. Commun.* **1975**, 124.
(7) Fischer, R. D. *J. Organomet. Chem.* **1970**, *25*, 123.
(8) Ammon, R. V.; Kannelakopulos, B.; Fischer, R. D.; Laubereau, P. *Inorg. Nucl. Chem. Lett.* **1969**, *5*, 315.
(9) Marks, T. J. *Acc. Chem. Res.* **1976**, *9*, 223.
(10) Chock, P. B.; Halpern, J. *J. Am. Chem. Soc.* **1969**, *91*, 582.
(11) Wen, T. C.; Chang, C. C.; Chuang, Y. D.; Chang, C. T., unpublished results.
(12) Anderson, M. L.; Crisler, L. R. *J. Organomet. Chem.* **1969**, *17*, 345.

Contribution from the Department of Synthetic Chemistry,
Faculty of Engineering, Kyoto University, Kyoto 606, Japan

Preparation and Characterization of Copper(I) Amides

Tetsuo Tsuda, Katsuhiko Watanabe, Kazuyoshi Miyata,
Hirotsugu Yamamoto, and Takeo Saegusa*

Received June 11, 1980

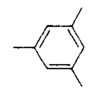
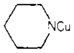
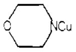
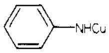
Compared to organocopper[1] and copper alkoxides,[2,3] copper amides have not been well studied. Some synthetic organic reactions involving copper amide intermediates are known.[4,5] However, isolation and characterization of copper amides derived from ordinary amines have not been reported. Recently we have prepared a unique organocopper(I) compound of mesitylcopper(I) (**1**)[6] which is thermally stable up to 100

---

(1) Jukes, A. E. *Adv. Organomet. Chem.* **1974**, *12*, 251–322.
(2) Brubaker, C. H., Jr.; Wicholas, M. *J. Inorg. Nucl. Chem.* **1965**, *27*, 59. Tsuda, T.; Hashimoto, T.; Saegusa, T. *J. Am. Chem. Soc.* **1972**, *94*, 658.
(3) Whitesides, G. M.; Sadowski, J. S.; Lilburn, J. *J. Am. Chem. Soc.* **1974**, *96*, 2829.
(4) Kauffmann, Th. *Angew. Chem.* **1974**, *86*, 321. Kitagawa, Y.; Oshima, K.; Yamamoto, H.; Nozaki, H. *Tetrahedron Lett.* **1975**, *1859*.
(5) Tsuda, T.; Miwa, M.; Saegusa, T. *J. Org. Chem.* **1979**, *44*, 3734.
(6) Tsuda, T.; Yazawa, T.; Watanabe, K.; Fujii, T.; Saegusa, T. *J. Org. Chem.* **1981**, *46*, 192.

0020-1669/81/1320-2728$01.25/0

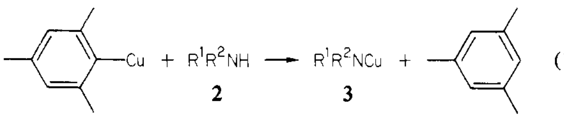
Table I. Analytical Data and Properties for R^1R^2NCu (**3**)[a]

	% 	% Cu content		% R^1R^2-	dec temp,
R^1R^2NCu	formation	found	calcd	NCO_2Me	°C
H_2NCu (**3a**)	100	80.0	79.9	17	ca. 90
n-BuNHCu (**3b**)	98	46.1	46.8	44	ca. 70
t-BuNHCu (**3c**)	87	46.2	46.8	68	>140
Et_2NCu (**3d**)	92	47.0	46.8	71	ca. 90
n-Bu_2NCu (**3e**)	95	33.5	33.1	76	ca. 110
 (**3f**)	90	42.1	43.0	56	ca. 105
 (**3g**)	100	46.0	46.8	60	ca. 125
 (**3h**)	88	38.5	40.8	55	ca. 90

[a] Due to high oxygen and moisture sensitivity, C, H, and N analyses of the copper(I) amides required special handling not available to us. Iodometry is an alternative to the usual analytical data; the sampling can be done on a mmol scale in a drybox under nitrogen. Cf.: Tsuda, T.; Hashimoto, T.; Saegusa, T. *J. Am. Chem. Soc.* 1972, *94,* 658. Tsuda, T.; Habu, H.; Horiguchi, S.; Saegusa, T. *Ibid.* 1974, *96,* 5930. Tsuda, T.; Chujo, Y.; Saegusa, T. *Ibid.* 1980, *102,* 431. Tsuda, T.; Sanada, S.-I.; Ueda, K.; Saegusa, T. *Inorg. Chem.* 1976, *15,* 2329.

°C and is soluble in common organic solvents. Here we report preparation of copper(I) amides derived from ordinary amines as well as ammonia with use of **1** as an efficient metalation reagent and their characterization.

1 abstracted the active hydrogen of aliphatic primary and secondary amines to produce corresponding copper(I) amides with simultaneous quantitative formation of mesitylene (eq 1). Ammonia and aniline similarly reacted with **1**. Metalation was carried out in tetrahydrofuran (THF) at ambient temperature with an excess of **2** to **1**. Copper(I) amides were easily isolated by evaporating the reaction mixtures or by filtering the precipitates produced. This method is useful for the preparation of copper(I) amides free of coordinating ligands or salts. The preparation of copper(I) amides by the metathesis of copper(I) halides with lithium amides is accompanied inevitably by the formation of lithium halides.



(1)

1

Analytical data and properties of various copper(I) amides prepared (**3a**–**3h**) are summarized in Table I. Copper(I) amides thus obtained are sensitive to air. The compounds **3a**, **3c**, **3g**, and **3h** are insoluble in THF, but **3b**, **3d**, and **3f** are partly soluble. Interestingly, **3e** is soluble in common organic solvents such as benzene, THF, and hexamethylphosphoric triamide (HMPA). The molecular weight determination by cryoscopy showed that **3e** exists as a tetramer in benzene. ^1H NMR absorptions of **3e** in C_6D_6 appeared at δ 2.95 (2 H, t), 2.00 (2 H, m), 1.35 (2 H, m) and 0.95 (3 H, t) which showed a downfield shift compared to *n*-Bu_2NH. Previously we have reported that a copper(I) carbamato complex, $R^1R^2NCO_2Cu\cdot(t\text{-BuNC})_n$, is prepared by the reaction of *t*-BuOCu, R^1R^2NH, and CO_2 in the presence of *t*-BuNC, which reacts with MeI to give methyl carbamate $R^1R^2NCO_2Me$.[7] In the present study, we observed that isolated copper(I) amides **3a**–**3h** absorbed CO_2 in benzene at ambient temperature in the presence of 3 equiv of *t*-BuNC. Treatment of the resulting solution with MeI gave $R^1R^2NCO_2Me$ probably via an intermediacy of $R^1R^2NCO_2Cu\cdot(t\text{-BuNC})_n$ (eq 2 and Table I).

$$R^1R^2NCu + CO_2 \xrightarrow{t\text{-BuNC}} R^1R^2NCO_2Cu\cdot(t\text{-BuNC})_n \xrightarrow{\text{MeI}} R^1R^2NCO_2Me \quad (2)$$

In the absence of *t*-BuNC, **3d** slowly absorbed CO_2 in benzene at ambient temperature to produce Et_2NCO_2Cu.[7]

The thermal stability of copper(I) amides prepared was qualitatively examined by observing the deposition of metallic copper by heating them in tetralin. For example, a suspension of **3d** in tetralin was dissolved at 60 °C to produce a homogeneous solution, from which metallic copper began to deposit at 90 °C. Heating **3e** in tetralin at 110 °C for 1 h produced *N*,*N*-di-*n*-butylamine in 30% yield and *N*-*n*-butylidene-*n*-butylamine in 20% yield, respectively. The results concerning thermal stability are included in Table I. It is noteworthy that copper(I) amides having a β-hydrogen atom are thermally stable compared to the corresponding organocoppers(I) and copper(I) alkoxides which are known to be thermally unstable. For example, as for *n*-butyl compounds, an *n*-butylcopper(I) phosphine complex rapidly decomposes at room temperature to butane and 1-butene,[8] and copper(I) *n*-butoxide slowly decomposes at room temperature.[3]

3e did not absorb carbon monoxide of ordinary pressure in THF–HMPA (4:1) at room temperature. **3e** also did not react with *n*-butyl iodide under a similar condition. On the contrary, lithium bis(*N*,*N*-di-*n*-butylamino)cuprate, $(n\text{-}Bu_2N)_2CuLi$, which was prepared by the equimolar reaction of **3e** and *n*-Bu_2NLi reacted with CO and *n*-BuI to give lithium bis(*N*,*N*-di-*n*-butylcarbamoyl)cuprate, $(n\text{-}Bu_2NCO)_2CuLi$,[5] and *n*-Bu_3N, respectively. *n*-Bu_3N was also produced in a good yield by the reaction of **3e** and *n*-BuI in the presence of LiI. These findings suggest the low nucleophilic reactivity of copper(I) amides.[9]

Experimental Section

All operations described below were carried out under nitrogen.

Preparation of Mesitylcopper(I) (1). The reaction mixture resulted from the addition of a 1.0 M THF solution of mesitylmagnesium bromide (100 mL, 100 mmol) to a suspension of CuCl (10.9 g, 110 mmol) in 100 mL of THF at −20 °C was allowed to react overnight at ambient temperature under stirring. The filtrate obtained after the addition of 50 mL of dioxane to complete the precipitation of MgBrCl was evaporated under reduced pressure to produce a solid residue. Dissolution of this solid in 50 mL of benzene, filtration of a small amount of insoluble matters, and evaporation of the filtrate in vacuo at ambient temperature gave a pale yellow solid of **1** (13.7 g, 75 mmol): Cu content by iodometry 34.0% (calcd 34.8%); mesitylene formation by acetolysis 98%; NMR (C_6D_6) δ 6.54 (phenyl protons, m, 2 H), 2.80 (*o*-methyl protons, s, 6 H), 1.90 (*p*-methyl protons, s, 3 H); IR (Nujol) 1592 (ν(C═C)), 845 cm^{-1} (δ(C═C—H)).

Preparation of Copper(I) *N*,*N*-Di-*n*-butylamide (3e). Di-*n*-butylamine (2.09 mL, 12.4 mmol) was added at ambient temperature to 5 mL of THF containing **1** (0.451 g, 2.47 mmol). The mixture was stirred overnight at ambient temperature. GLPC analysis of the resulting homogeneous solution on 20% Silicone DC 550 on a Celite 545 column using tetralin as an internal standard showed the formation of mesitylene in a yield of 95%. The reaction mixture was evaporated in vacuo to produce a pale yellow solid of **3e**, which was washed with 10 mL of hexane and was dried in vacuo. **3e** was crystallized in hexane at −15 °C.

Reaction of Copper(I) *N*,*N*-Di-*n*-butylamide (3e) with Carbon Dioxide. In a flask equipped with a three-way stopcock were placed **3e** (0.109 g, 0.568 mmol), *t*-BuNC (0.19 mL, 1.70 mmol), and 5 mL

---

(7) Tsuda, T.; Washita, H.; Watanabe, K.; Miwa, M.; Saegusa, T. *J. Chem. Soc., Chem. Commun.* **1978**, *815*.

(8) Whitesides, G. M.; Stedronsky, E. R. *J. Am. Chem. Soc.* **1970**, *92*, 1426.

(9) Slow CO_2 absorption of **3d** to form Et_2NCO_2Cu may probably proceed via a carbamic acid mechanism. See: Chisholm, M. H.; Extine, M. W. *J. Am. Chem. Soc.* **1977**, *99*, 792.

of benzene. CO_2 (42 mL, 1.70 mmol) was introduced at ambient temperature through the serum-stoppered three-way stopcock with a hypodermic syringe, and the resulting benzene solution was stirred for 3 h at ambient temperature. MeI (0.11 mL, 1.70 mmol) was added, and the reaction mixture was stirred overnight at room temperature. GLPC analysis of the reaction mixture on 20% Silicone DC 550 on a Celite 545 column with tetralin as an internal standard indicated the formation of $(n\text{-Bu})_2NCO_2Me$ in a yield of 76%, which was identified by the agreement of its IR spectra and retention time of GLPC with those of the authentic sample.

Thermal Decomposition of Copper(I) *N*,*N*-Di-*n*-butylamide (3e). A solution of 5 mL of tetralin containing **3e** (0.407 g, 2.12 mmol) was gradually heated to about 110 °C where a black precipitate began to deposit. After being heated at 110 °C for 1 h, the reaction mixture was analyzed by GLPC on 20% Silicone DC 550 on a Celite 545 column with ethylbenzene as an internal standard. Di-*n*-butylamine and *N*-*n*-butylidene-*n*-butylamine were produced in 30% and 20% yields, respectively, which were identified by the agreement of their IR spectrum and retention times of GLPC with those of authentic samples.

Registry No. **1**, 75732-01-3; **3a**, 77590-45-5; **3b**, 77590-46-6; **3c**, 77590-47-7; **3d**, 71426-07-8; **3e**, 77590-48-8; **3f**, 73680-02-1; **3g**, 77590-49-9; **3h**, 77590-50-2; H_2NCO_2Me, 598-55-0; $n\text{-BuHNCO}_2Me$, 2594-21-0; $t\text{-BuHNCO}_2Me$, 27701-01-5; Et_2NCO_2Me, 4652-44-2; $(n\text{-Bu})_2NCO_2Me$, 56475-79-7; $(CH_2)_5NCO_2Me$, 1796-27-6; $O(CH_2)_4NCO_2Me$, 6906-13-4; $C_6H_5NHCO_2Me$, 2603-10-3; CO_2, 124-38-9.

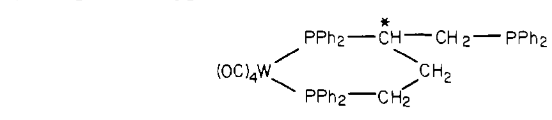
Contribution from the Departments of Chemistry, State University of New York at Buffalo, Buffalo, New York 14214, and Eastern Illinois University, Charleston, Illinois 61920

Crystal Structure and Molecular Geometry of $(OC)_4WPPh_2CH(CH_2CH_2PPh_2)CH_2PPh_2{\cdot}CH_3CO_2C_2H_5$. Free Radical Addition of PPh_2H to a Coordinated Phosphinoalkene

Melvyn Rowen Churchill,*[1] Arnold L. Rheingold,[1,2]
and Richard L. Keiter*[3]

Received October 20, 1980

Base and free radical catalyzed additions of secondary phosphines to vinylphosphines are convenient syntheses for a variety of poly(tertiary phosphines).[4] The availability of starting materials limits the variety of products, however, with most containing phosphorus atoms connected by $(CH_2)_n$ units. Some variation in the backbone substituency can be introduced by carrying out addition reactions on coordinated phosphinoalkenes[5] or phosphinoalkynes.[6] The recently reported reaction of *trans*-$(OC)_4W(PPh_2CH{=}CH_2)_2$ with PPh_2H is such an example[5] (eq 1). This reaction generates a tris(tertiary



I

phosphine) (I) which is capable of forming five- or six-membered chelating rings and which contains a chiral carbon center in the backbone. Wilkinson-type catalytic systems incorporating chiral bidentate phosphines are very useful for asymmetric synthesis of optically active amino acids.[7] Furthermore, rhodium complexes of tridentate phosphorus ligands may serve as hydrogenation catalysts if the nonphosphorus ligand of the four-coordinate complex is reactive.[8]

Table I. Experimental Data for the X-ray Diffraction Study on Crystalline $(OC)_4WPPh_2CH(CH_2CH_2PPh_2)CH_2PPh_2$

(A) Crystal Parameters at 24 °C[a]

cryst system: monoclinic	$V = 4448.5\ (2)$ Å^3
space group: $P2_1/c$ [C^5_{2h}, No. 14]	$Z = 4$
$a = 10.869\ (2)$ Å	ρ(calcd) = 1.49 g cm^{-3}
$b = 17.030\ (3)$ Å	mol wt: 994.60
$c = 24.063\ (6)$ Å	
$\beta = 92.80\ (2)^\circ$	

(B) Measurement of Intensity Data

diffractometer: Syntex $P2_1$
radiation: Mo Kα ($\lambda = 0.710\,73$ Å)
monochromator: highly oriented graphite
reflcs measd: $\pm h,+k,+l$
2θ range: 3.0–45.0°
scan type: coupled θ(crystal)–2θ(counter)
scan speed: 3.0°/min
scan width: $[2\theta(\text{Mo K}\alpha_1) - 0.7]^\circ$–$[2\theta(\text{Mo K}\alpha_2) + 0.7]^\circ$
std reflcs: 1,0,12; $\bar{6}11$; $\bar{2}$,10,0 (these were measured after each 97 reflcs; examination of the data indicated that no correction for decay was warranted)
reflcs collected: 6009 total, yielding 5844 symmetry-independent data
abs coeff: $\mu = 29.1$ cm^{-1}
ignorance factor: $p = 0.015$

[a] Unit-cell parameters were derived from a least-squares fit to the setting angles of the unresolved Mo K$\bar{\alpha}$ components of 24 reflections of the forms {711}, {617}, {3,10,4}, {1,11,3}, {3,1,13}, and {1,4,15}.

NMR and IR spectra led to the postulation of structure I, but these data alone could not totally rule out other isomeric possibilities. A feature of the assignment that was particularly puzzling was that it required proposing phosphorus–phosphorus coupling through five bonds (3.6 Hz). Observation of long-range coupling is not uncommon when nuclear sites are separated by olefinic or aromatic groups but would not be expected through five aliphatic linkages.[9]

Experimental Section

A. Preparation of Sample. Crystals suitable for an X-ray diffraction study were obtained by a procedure similar to that previously reported[5] except for details of the final workup. During evaporation of the petroleum ether/ethyl acetate column chromatography eluting solvent, pale yellow crystals separated which were used in this study without the subsequent recrystallization from $MeOH/CH_2Cl_2$ as employed previously. Both NMR spectroscopy and elemental analysis results

---

*To whom correspondence should be addressed: M.R.C., State University of New York at Buffalo; R.L.K., Eastern Illinois University.

(1) State University of New York at Buffalo.
(2) On sabbatical leave from the State University of New York at Plattsburgh.
(3) Eastern Illinois University.
(4) King, R. B.; Cloyd, J. C., Jr. *J. Am. Chem. Soc.* **1975**, *97*, 53. Uriate, R.; Mazanec, T. J.; Tau, K. D.; Meek, D. W. *Inorg. Chem.* **1980**, *19*, 79. Grim, S. O.; Del Gaudio, J.; Molenda, R. P.; Tolman, C. A.; Jesson, J. P. *J. Am. Chem. Soc.* **1974**, *96*, 3416.
(5) Keiter, R. L.; Sun, Y. Y.; Brodack, J. W.; Cary, L. W. *J. Am. Chem. Soc.* **1979**, *101*, 2638.
(6) Carty, A. J.; Johnson, D. K.; Jacobson, S. E. *J. Am. Chem. Soc.* **1979**, *101*, 5612. Carty, A. J.; Taylor, N. J.; Johnson, D. K. *Ibid.* **1979**, *101*, 5422. Carty, A. J.; Jacobson, S. E.; Taylor, N. J.; Chieh, P. C. *J. Chem. Soc., Dalton Trans.* **1976**, 1375. Carty, A. J.; Jacobson, S. E.; Simpson, R. T.; Taylor, N. J. *J. Am. Chem. Soc.* **1975**, *97*, 7254.
(7) Brunner, H. *Acc. Chem. Res.* **1979**, *12*, 250. James, B. R. *Adv. Organomet. Chem.* **1979**, *17*, 319.
(8) Meek, D. W.; Niewahner, J.; Kreter, P., paper presented at the Biennial Inorganic Chemistry Symposium, Guelph, Canada, 1980. Dubois, D. L.; Meek, D. W. *Inorg. Chim. Acta* **1979**, *19*, L29.
(9) Finer, E. G.; Harris, R. K. *Prog. Nucl. Magn. Reson. Spectrosc.* **1971**, *6*, 61.

0020-1669/81/1320-2730$01.25/0