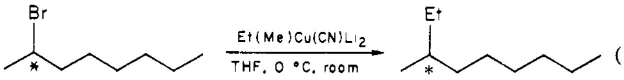
the use of an aromatic cuprate (i.e., **3**) under most atypical conditions[15] suggested that *alkyl*lithium-derived organocuprates should be investigated. Thus, reaction of (*S*)-(+)-2-bromooctane (**7**)[16] with Et(Me)Cu(CN)Li_2[17] at 0 °C to room temperature (eq 3) afforded (72%) optically active **6**[18] with a rotation clearly indicative of a net Walden inversion having taken place.[9]

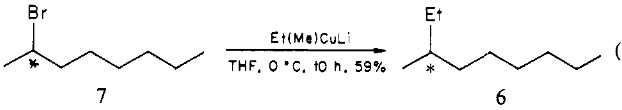


(3)

7 $[\alpha]^{19}_D$ +35.78°

6 $[\alpha]^{19}_D$ +9.60°

Similarly, treatment of **7** with the lower order counterpart Et(Me)CuLi[19] gave (*S*)-(+)-**6**[18] with essentially the same rotation (eq 4).

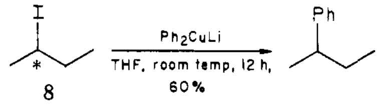


(4)

7 $[\alpha]^{19}_D$ +35.94°

6 $[\alpha]^{19}_D$ −9.53°

These experiments serve to establish that the mechanism of the coupling process at carbon is substrate and not reagent dependent. To further lend credence to this work, we prepared (*S*)-(+)-2-iodobutane (**8**)[20] and treated it with Ph_2CuLi (eq 5). Whereas



(5)

8 $[\alpha]^{19}_D$ +36.98°

$[\alpha]^{19}_D$ −0.88°

the corresponding bromide goes with inversion,[6] essentially *racemic sec*-butylbenzene was isolated by preparatory VPC,[21] in line with the data presented above.[22]

Finally, some of the implications of this study should be brought to light as they may have a considerable impact on organocopper chemistry. These include the following: (1) it has been shown that the nature of the halide undergoing displacement appears to determine the stereochemistry of the resulting carbon center. Thus, where complete stereocontrol is required, bromides are the preferred substrates.[23] (2) With proper control of experimental parameters, Gilman cuprates, in many cases, will effect substitution chemistry in synthetically useful yields.[24] (3) The substitution process at *primary* centers is also likely to be dependent upon the leaving moiety. This is strongly suggested by the observation that treatment of primary iodide **9** with **2**, R = *n*-Bu at −100 °C, for 30 min afforded 20–30% of the reduction product (eq 6).[25]

$$\underset{\mathbf{9}}{n\text{-}C_7H_{15}I} \xrightarrow[\text{THF, }-100\ ^\circ\text{C, 30 min}]{(n\text{-Bu})_2\text{Cu(CN)Li}_2} \underset{50\%}{n\text{-}C_{11}H_{24}} + \underset{30\%}{n\text{-}C_7H_{16}} + \underset{20\%}{\text{SM}}$$

Acknowledgment. Financial support from the National Institutes of Health (GM 28128), the American Cancer Society (JFRA No. 37, B.H.L.), and the Committee on Research, UCSB, Sigma Xi, is gratefully acknowledged.

Registry No. 1 (R = Et), 38297-20-0; **2** (R = Bu), 80473-69-4; **3**, 23402-69-9; (*R*)-**4**, 29117-48-4; **5**, 80473-71-8; (±)-**6**, 82373-57-7; (*S*)-**6**, 53213-47-1; (*S*)-**7**, 1191-24-8; (*S*)-**8**, 29882-56-2; 9, 4282-40-0; Et(Me)Cu(CN)Li_2, 82352-24-7; Et(Me)CuLi, 82352-23-6; (±)-*sec*-butylbenzene, 135-98-8; undecane, 1120-21-4; heptane, 142-82-5.

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(15) This displacement was performed at 51 °C for 72 h.

(16) Formed from (−)-2-octanol with PBr_3 (neat); cf ref 8.

(17) It was necessary to switch to the still more highly mixed cuprate since use of **5** led to a considerable amount of competing reduction under a variety of conditions. The selectivity of transfer (ethyl/methyl) was ca. 12:1.

(18) Isolated by preparatory VPC with purity of >99% (by VPC).

(19) As in ref 17, Et_2CuLi gave low yields (~18%) of **6**.

(20) Goodwin, D. G.; Hudson, H. R. *J. Chem. Soc. B* **1968**, 1333. In this work, 2-iodobutane is prepared from PI_3/CS_2 and shows α^{20}_D 60.4° or $[\alpha]^{20}_D$ 37.99° with a density of 1.59 as listed in the CRC Handbook of Chemistry and Physics.[7]

(21) With a 3-m column of 4% SE-30 on Chromosorb P (60/80 mesh). The purity of *sec*-butylbenzene isolated was determined to be >99% (by VPC).

(22) In view of the coupling between an aryl ligand in **3** and a secondary iodide (**8**), it is apparent that there is *some* dependence of the reaction on the cuprate involved since $Ph_2Cu(CN)Li_2$ reacts with 2-iodooctane affording only trace amounts of the desired product.[3b]

(23) Tosylates are also known to react with inversion: Johnson, C. R.; Dutra, G. A. *J. Am. Chem. Soc.* **1973**, *95*, 7783.

(24) In addition to the examples shown in the text, we have also found that *n*-Bu_2CuLi reacts with iodocyclohexane at −50 °C in ~1 h to afford *n*-butylcyclohexane in quantitative yield; cf.: Corey, E. J.; Posner, G. H. *J. Am. Chem. Soc.* **1968**, *90*, 5615.

(25) We have from this observation gone on to show that primary bromides are functionally equivalent (in terms of leaving-group ability) to iodides by switching from reagents **1** to **2**. Hence, e.g., 5-bromovaleronitrile reacts with *n*-$Bu_2Cu(CN)Li_2$ (1.3 equiv) at −50 °C (2.5 h) to afford pelargononitrile in 92% isolated yield (Kozlowski, J.; Parker, D., unpublished work). A general study will be reported in due course. Typically, considerably higher temperatures (ca. 0 °C) are required with Gilman reagents.[5]

6-Methyl-1,6-diazabicyclo[6.2.2]dodecane: An Inside, Outside Six-Atom Bridged Boat Piperidine

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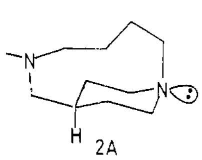
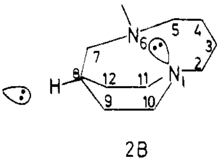
Received February 1, 1982

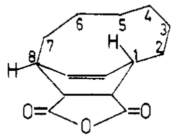
2-Methyl-1,2-diazabicyclo[2.2.2]octane **1**[1] was bis-alkylated with 1,4-diiodobutane in two steps, following the methodology of Alder and co-workers[2] to give hexaalkylhydrazine dication $\mathbf{2}^{2+}$,[3] which was reduced to the neutral diamine **2**[4] (see Scheme I). Two classes of conformations are available for **2** depending on the configuration at the piperidine nitrogen N_1, the chair piperidine class 2A (in which the piperidine ring could reverse) and the boat piperidine class **2B**, which is the inside, outside isomer of 6-methyl-1,6-diazabicyclo[6.2.2]dodecane. Gassman and Hoye[5] recently described the X-ray structure of the substituted (i,o)-bicyclo[6.2.2]dodecane **3**, which is a very strained compound, as evidenced by the large bond-angle deformations found for its 6-bridge. The nitrogen at position 1 of **2B** replaces the inner C_1H bond of **3** with a sterically less demanding nitrogen lone pair, which should reduce the strain in **2B** relative to **3**, and N_1 of **2B** can also flatten more easily to further relieve strain in **2B** relative to **3**.



2A



2B



3

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(1) Prepared from 1,2-diazabicyclo[2.2.2]octane (Nelsen, S. F.; Gannett, P. M.; Steffek, D. J. *J. Org. Chem.* **1980**, *45*, 3857) by formaldehyde/cyanoborohydride reductive methylation (Nelsen, S. F.; Weisman, G. R. *Tetrahedron Lett.* **1973**, 2321).

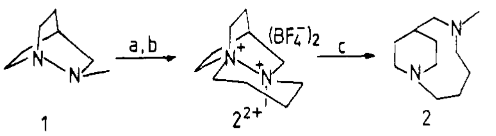
(2) (a) Alder, R. W.; Goode, N. C.; King, T. J.; Mellor, J. M.; Miller, B. W. *J. Chem. Soc., Chem. Commun.* **1976**, 173. (b) Alder, R. W.; Gill, R.; Goode, N. C. *Ibid.* **1976**, 973. (c) Alder, R. W.; Sessions, R. B.; Mellor, J. M.; Rawlins, M. G. *Ibid.* **1977**, 747. (d) Alder, R. W.; Sessions, R. B. *J. Am. Chem. Soc.* **1979**, *101*, 3651. (e) Alder, R. W.; Sessions, R. B.; Bennet, A. J.; Moss, R. E. *J. Chem. Soc., Perkin Trans. 1* **1982**, 603. (f) For a review of the special properties of diamines, see: Alder, R. W.; Sessions, R. B. "The Chemistry of Functional Groups; Supplement F: The Chemistry of Amines, Nitroso, and Nitro Compounds"; Patai, S., Ed.; Wiley: New York, 1982; Chapter 18, p 763.

(3) $\mathbf{2}^{2+}(BF_4^-)_2$ (dec 244–245 °C) gave a satisfactory combustion analysis and reasonable spectral data.

(4) **2** (mp 54–56 °C (sealed tube)) gave a high-resolution MS mass of 187.1783, an acceptable deviation of 0.1 ppm from $C_{11}H_{22}N_2$.

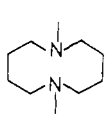
(5) Gassman, P. G.; Hoye, R. C. *J. Am. Chem. Soc.* **1981**, *103*, 215.

Scheme I



A major difference between conformational classes **2A** and **2B** is the distance between the nitrogens, which are far apart in **2A** but are forced to be rather close in **2B**, imposing through-space interaction between the nitrogen lone pairs. We will argue that the unusual spectral and electron-loss properties of **2** require that it is in the **2B** class of conformations, which resemble the geometry of $\mathbf{2}^{2+}$.

Intense UV absorption (λ_{max} (pentane) 212 nm (ϵ 4800), sh 245 (ϵ 2500)) is shown by **2**, and its photoelectron (PE) spectrum has two lone-pair ionizations, IP_v = 7.21 and 7.84 eV. The 0.6-eV vertical ionization potential difference is only reasonable if there is significant lone pair–lone pair interaction or flattening at one of the nitrogens. The monocyclic analogue of **2**, 1,6-dimethyl-1,6-diazabicyclodecane **4**, shows only end absorption in its UV spectrum (ϵ_{210} 2990, ϵ_{240} 310) and a single lone-pair PE peak, IP_v = 7.81 eV. Both types of data for **2** are precedented only for medium ring bridgehead amines that are flattened at N such as HC[3.3.3]N (λ_m 240 nm (ϵ 2935), IP_v = 7.13 eV)[6] and HC[4.4.4]N (λ_m 220 nm (ϵ 4830), IP_v = 7.84 eV)[7] and for bis-bridgehead diamines that combine flattening with through-space NN interaction such as N[3.3.3]N (λ_m 217 nm (ϵ 5700), 278 sh (ϵ 700), IP_v = 6.85, 7.94 eV)[2c,7] and N[4.4.4]N (λ_m 233 nm (ϵ 8100), 261 sh (ϵ 3800), IP_v = 6.75, 7.87 eV).[2c,7] The broad-band shape for both lone-pair ionization peaks of **2** shows that neither of its nitrogens can be nearly as flat as that of HC[3.3.3]N, which exhibits a π-type band, showing vibrational fine structure.[6b] Strong Bohlman bands are shown at 2730 and 2770 cm^{-1} in the IR spectrum of **2**, indicating alignment of $C_\alpha H$ bonds with a lone-pair orbital axis.



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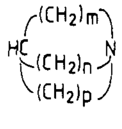
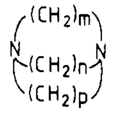


HC[mnp]N

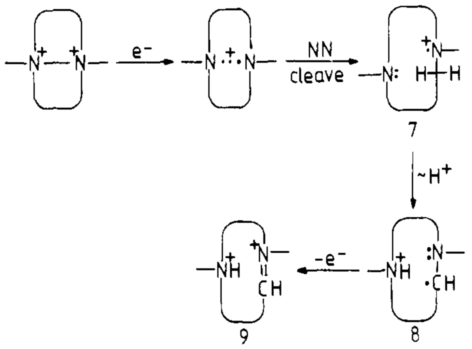


N[mnp]N

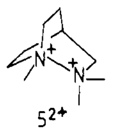
Neutral **2** is rather air sensitive and shows easy oxidation by cyclic voltammetry (CV). Irreversible oxidation peaks are observed at −0.13 and +0.39 V vs. SCE (0.2 V/s scan rate, acetonitrile/0.1 M n-Bu_4NClO_4, 23 ± 1 °C, gold electrode, the CV conditions used in this work unless otherwise noted). Oxidation of **2** occurs much more easily than expected from models that lack NN interaction: **4** shows an irreversible CV wave peaking at +0.73 V, and 9-*tert*-butyl-9-azabicyclo[3.3.1]nonane, which has IP_v = 7.30 V (only 0.1 eV higher than that of **2**), shows a reversible oxidation wave, $E^{0\prime}$ = 0.74 V. Alder's bis-bridgehead diamines N[3.3.3]N and N[4.4.4]N show oxidation comparably easy to **2** but differ in having chemically reversible, though electrochemically rather slow, electron transfer: for N[3.3.3]N, $E_1^{0\prime} = -0.17$ V, $E_2^{0\prime} = +0.1$ V, and for N[4.4.4]N $E_1^{0\prime} = -0.1$ V, $E_2^{0\prime} = +0.2$ V. The odd electron in Alder's cation radicals is in an orbital dominated by the NN antibonding orbital; they have "three-electron σ bonds".[2] Their easy oxidation reflects the NN interaction being destabilizing to the neutral form but stabilizing in the radical cation.

Reduction of $\mathbf{2}^{2+}$ is also facile, and broad overlapping CV reduction peaks occur at −0.21, −0.50, and −0.98 V. The rather positive first reduction peak for $\mathbf{2}^{2+}$ is comparable to those for

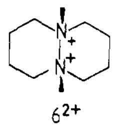
Scheme II



Alder's dications, but in strong contrast to the CV behavior of hexaalkylhydrazine dications in which the nitrogens are not forced together in the neutral form. Although $\mathbf{5}^{2+}$ is an excellent structural analogue for $\mathbf{2}^{2+}$, its CV shows a single distended wave peaking at −1.39 V, closely resembling the behavior of $\mathbf{6}^{2+}$ (the dicationic analogue of **4**), which gives $E_p^{red} = -1.37$ V in butyronitrile at −84 °C. We suggest that the CV peak potentials for $\mathbf{5}^{2+}$ and $\mathbf{6}^{2+}$ reduction reflect neither the thermodynamic nor the kinetic ease of adding an electron to these species because of following chemical reactions. Pulse radiolysis experiments on $\mathbf{6}^{2+}$, N[3.3.3]N^{2+}, and N[4.4.4]N^{2+} showed that all three compounds react at a diffusion-controlled rate with solvated electrons in water,[8] which seems inconsistent with a far slower or thermodynamically more difficult reduction of $\mathbf{6}^{2+}$ than the other two dications, which might have been inferred from the over 1 V more negative reduction peak potential observed for $\mathbf{6}^{2+}$ by CV. $\mathbf{6}^{2+}$ was found to give a three-electron σ-bonded radical cation upon reduction, but its lifetime was only 5 ms. The weak $(N{\cdots}N)^{+\cdot}$ bond (estimated at 14.5 kcal/mol from the lifetime of $\mathbf{6}^{+\cdot 8}$) breaks rapidly, and when the nitrogens move apart, spin and charge become localized on one nitrogen as indicated for a generalized bicyclic hexaalkylhydrazine in Scheme II (structure **7**). Because aminium cations are strong acids, a rapid intramolecular proton transfer generating α-amino radical **8** ought to occur. Because α-amino radicals are very easily oxidized to immonium cations, an electron should be transferred back to the electrode by the α-amino radical (known electrochemically reversible α-amino radicals lose an electron with $E^{0\prime}$ of about −1.5 V[9]). If the reactions of Scheme II are fast enough, then one should not see significant reduction current until the potential where the immonium cation **9** is reduced. We suggest that this is the reason that $\mathbf{5}^{2+}$ and $\mathbf{6}^{2+}$ show such negative reduction waves compared to the those of other hexalkyl hydrazines dications discussed. For $\mathbf{2}^{2+}$ reduction, a combination of slower cleavage, proton transfer, and/or oxidation reactions is suggested to result in observation of reduction current near where $\mathbf{2}^{2+}$ takes up an electron.



5^{2+}



6^{2+}

Despite the easy oxidation of **2** and reduction of $\mathbf{2}^{2+}$, neither electron-transfer reaction is either chemically or electrochemically reversible, in contrast to Alder's compounds, in which the ionic forms have propellane structures. We have failed to observe $\mathbf{2}^{+\cdot}$ by ESR under any conditions.[10] The $NOPF_6$ oxidation of **2** gives a complex product mixture in which we have failed to detect $\mathbf{2}^{2+}$. Sodium/ammonia reduction of $\mathbf{2}^{2+}$ does give **2**, but under these conditions all of the intermediates of Scheme II would reduce to

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(6) (a) Alder, R. W.; Arrowsmith, R. J.; Casson, A.; Sessions, R. B.; Heilbronner, E.; Kovac, B.; Huber, H.; Taagepera, M. *J. Am. Chem. Soc.* **1981**, *103*, 6137. (b) Aue, D. H.; Webb, H. M.; Bowers, M. T. *Ibid.* **1974**, *97*, 4136.

(7) Alder, R. W.; Arrowsmith, R. J. *J. Chem. Res., Synop.* **1980**, 163; *Miniprint* **1980**, 2301.

(8) Nelsen, S. F.; Alder, R. W.; Sessions, R. B.; Asmus, K.-D.; Hiller, K.-O.; Göbl, M. *J. Am. Chem. Soc.* **1980**, *102*, 1429.

(9) Nelsen, S. F.; Kessel, C. R.; Brien, D. J. *J. Am. Chem. Soc.* **1980**, *102*, 702.

(10) Low-temperature oxidation at mercury does generate a complex ESR spectrum, but mercury is required for this result, and the species observed seems unlikely to be $\mathbf{2}^{+\cdot}$.

2. It is apparent that the structural freedom to move the nitrogens of $\mathbf{2}^{+\cdot}$ relative to each other causes it to decompose rapidly.

We suggest that the spectral data that demonstrate flattening at nitrogen and/or through-space NN interaction in **2**, along with its facile oxidation, require that **2** exists in the inside, outside conformation **2B**. The 1-azabicyclo[6.2.2]dodecane **2** prefers i,o over o,o conformations, which models suggest have a great deal of nonbonded steric interaction between the piperidine and six-bridge methylene groups.

Acknowledgment. We thank Roger Alder (Bristol) for voluminous and fruitful correspondence, and the National Science Foundation (CHE 802611) and National Institute of Health (GM25428) for financial support of this work.

Registry No. 1, 6523-29-1; **2**, 82415-30-3; $\mathbf{2}^{2+}\cdot 2BF_4^-$, 82415-32-5; **4**, 82415-33-6; $\mathbf{5}^{2+}$, 82415-34-7; $\mathbf{6}^{2+}$, 82415-35-8; N[4.4.4]N, 71058-67-8; N[3.3.3]N, 283-58-9; 1,4-diiodobutane, 628-21-7; 9-*tert*-butyl-9-azabicyclo[3.3.1]nonane, 64776-36-9.

Metal-Templated Synthesis of a Macrocyclic Triphosphine–Molybdenum Complex, *fac*-$(CO)_3Mo(PHC_3H_6)_3$

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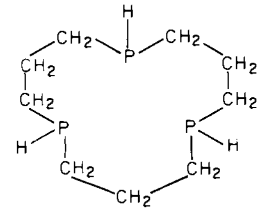
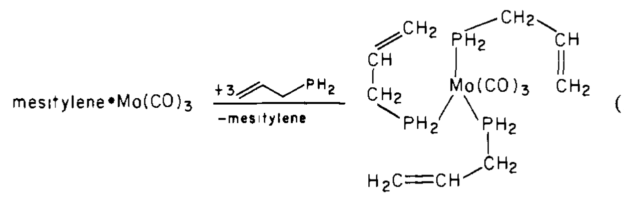
Received March 29, 1982

Numerous examples of acyclic polyphosphine ligands have been reported in recent years.[1] In contrast, many fewer examples of cyclic polyphosphines, phosphine macrocycles, have appeared.[2] The paucity of phosphine macrocycles is unfortunate in view of their potential for formation of a wide variety of novel and important transition-metal complexes. In this communication we report, from a reaction taking advantage of a kinetic template effect, the first synthesis of a metal complex containing the [12]ane-P_3 triphosphine macrocycle $(HPCH_2CH_2CH_2)_3$, **1**.[3]



1

Reaction of mesitylene·$Mo(CO)_3$ with allylphosphine[4] in benzene at 30 °C for 48 h, removal of solvent and unreacted allylphosphine in vacuo, redissolution of product in benzene, and its filtration through a 2-cm alumina column, followed by removal of solvent in vacuo, yield *fac*-$(CO)_3Mo(H_2PCH_2CH{=}CH_2)_3$ **2** (98% yield): characteristic[5,6] IR absorptions (neat, thin film or



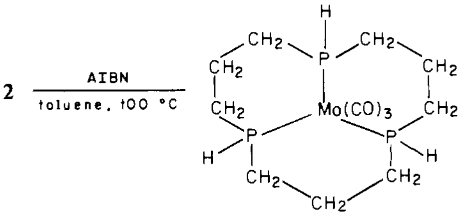
(1)

2

C_6H_{12} solution) 3084 (vinylic CH), 3007, 2980, 2940, 2908 (aliph CH str), 2302, 2294 (PH str), 1954, 1864 (CO str), 1633 (C=C) cm^{-1}; ^{31}P NMR (C_6D_6, rel to 85% H_3PO_4) δ 59.1 (tr, $^1J_{PH} = 295$ Hz, $^1J_{^{95}Mo-^{31}P} = 118$ Hz); ^{1} H NMR ($CDCl_3$) δ 3.92 (2, PH_2), 2.36 (2, CH_2), 5.9 (1, =CH), 5.0 (1, =CH′H, trans), 5.1 (1, =CCH′, cis); mass spectral parent ion at *m*/*e* 402.

Characterization of **2** as a facially substituted tris(allylphosphino) complex is supported by the single ^{31}P NMR spectral resonance and the presence of two characteristic carbonyl IR absorptions.[6] That **2** is only phosphorus coordinated and not olefin coordinated is substantiated by the ^{31}P NMR coordination chemical shift, the magnitude of the $^1J_{PH}$ coupling constant, the presence of ^{95}Mo satellites in the ^{31}P NMR spectrum, and the lack of significant perturbation of the vinylic ^1H NMR or IR spectral features.

Heating of **2** in benzene 1.5 h at 75 °C with ca. 1 mol% AIBN [2,2′-azobis(isobutyronitrile)] added as free-radical initiator filtration of the solution through alumina, and removal of solvent in vacuo, followed by recrystallization from toluene, yield pure macrocyclic triphosphine complex *fac*-$(CO)_3Mo(HPC_3H_6)_3$, **3** (~85% yield).[7] ^{31}P NMR spectral analyses of reaction solutions prior to product isolation show traces of products characterized tentatively as intermediate ring cyclization products. Spectral data for **3** are as follows: characteristic[5,6] IR absorptions (cyclohexane)2330 (P–H), 1945, 1844 (CO) cm^{-1}; ^{31}P NMR (C_6D_6, rel to 85% H_3PO_4) δ 32.0 ($^1J_{PH} = 318$ Hz, $^1J_{^{95}Mo-^{31}P} = 118.4$ Hz); ^1H NMR ($CDCl_3$) two complex multiplets collapse on ^{31}P decoupling to δ 4.9 (area 1) and a broad complex multiplet (area 6) at δ 1.8. Homonuclear irradiation of the area-1 multiplet yield sharpened lines for the P–H resonance allowing its unambiguous assignment. The mass spectrum exhibits a parent ion at *m*/*e* 402 (assigned to $^{96}Mo^{12}C_{12}P_3{}^{16}O_3H_{21}{}^+$). Crystalline **3** undergoes only slight decomposition after several hours exposure to air or ambient moisture; however, **3** decomposes rapidly in O_2-containing solutions to as yet uncharacterized materials.



3

The macrocycle complex crystallizes from toluene in two distinct crystal habits, very thin plates[8] and needles.[9] Samples of each

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(1) (a) Uriarte, R.; Mazanec, T. J.; Tau, K. D.; Meek, D. W. *Inorg. Chem.* **1980**, *19*, 79–85. (b) Mason, R.; Meek, D. W. *Angew. Chem., Int. Ed. Engl.* **1978**, *17*, 183–94. (c) Ghilardi, C. A.; Midolini, S.; Orlandini, A.; Sacconi, L. *Inorg. Chem.* **1980**, *19*, 301. (d) Christol, H.; Cristau, H. J.; Fallouk, F.; Hullot, P.; *Tetrahedron Lett.* **1979**, *28*, 2591–2594. (e) King, R. B. *Acc. Chem. Res.* **1972**, *5*, 177–188. (f) Hewertson, W.; Watson, H. R. *J. Chem. Soc.* **1962**, 1490–1493.

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(7) All new compounds have acceptable analytical data.

(8) Crystal data for $(CO)_3Mo(PHC_3H_6)_3$ (thin plates): $a = 13.14$ (2) A°, $b = 9.05$ (1) A°, $c = 13.79$ (3) A°, $\beta = 92.4$ (1)°, $V = 1638$ (5) Å^3. The thin plates were twinned; both fragments were indexed and gave the cell dimensions given above; the crystal was of insufficient quality for single-crystal X-ray diffraction analysis. The space group was not determined.

0002-7863/82/1504-4700$01.25/0