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REACTIONS OF COORDINATED LIGANDS

VII *. REACTIONS OF $(CO)_5MoPPh_2Cl$ AND *cis*-$(CO)_4Mo(PPh_2Cl)_2$ WITH SELECTED DIAMINES AND AMINO ALCOHOLS. SYNTHESIS OF *cis*-$(CO)_4M(PPh_2OCH_2CH_2NMe_2)$ (M = Cr, Mo, W)

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Summary

N,N-Dimethylethanolamine reacts with $(CO)_5Mo(PPh_2Cl)$ to give $(CO)_5Mo(PPh_2OCH_2CH_2NMe_2)$ which has been characterized as a BF_3 adduct and by heating to form the mixed donor (P, N) chelate complex $(CO)_4Mo(PPh_2OCH_2CH_2NMe_2)$. The chromium and tungsten analogs of the Mo chelate complex have been made by photochemical reaction between the hexacarbonyls and the uncomplexed (P, N) ligand. A combination of chloride displacement and photochemical chelation were used to obtain *cis*-$(CO)_4Mo(PPh_2NHCH_2CH_2NMe_2)$. With *cis*-$(CO)_4Mo(PPh_2Cl)_2$, $HOCH_2CH_2NMe_2$ gives *cis*-$(CO)_4Mo(PPh_2OCH_2CH_2NMe_2)(PPh_2OH)$ and not the expected disubstituted product. Ethylenediamine, *N*-methylethylenediamine and *N,N*-dimethylethylenediamine react with *cis*-$(CO)_4Mo(PPh_2Cl)_2$ in a straight forward manner to yield *cis*-$(CO)_4Mo(PPh_2NHCH_2CH_2NR_2)_2$ where $R_2 = H_2$, HMe, and Me_2, respectively. *N,N'*-Dimethylethylenediamine yields only the chelate complex $(CO)_4Mo(PPh_2NMeCH_2CH_2NMePPh_2)$. The ^1H NMR spectra of these complexes reveal some interesting examples of PH and HH virtual coupling.

Introduction

Prior papers in this series [1] have described reactions which coordinated halophosphine ligands undergo with monofunctional amines and alcohols. In this paper, these studies are extended to include diamines and amino alcohols.

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* For part VI see ref. 1.

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TABLE 1

PREPARATIONS AND PROPERTIES OF NEW COMPLEXES

Compound No.	Name (Formula)	Synthetic procedure [a] (Recryst. Solvens)	Yield (%)	Color	M.p. (°C)	Elemental analysis (Found (calcd.) (%)) C	H
IV	*cis*-Tetracarbonyl(2-*N*,*N*-dimethylamino)ethyl diphenylphosphinite-(*P*,*N*))-molybdenum(0), *cis*-$Mo(CO)_4PPh_2OCH_2CH_2NMe_2$	B, C, D (CH_2Cl_2/MeOH)	41, 22, 32	yellow	170 (dec.)	49.49 (49.91)	4.17 (4.18)
V	*cis*-Tetracarbonyl(2-(*N*,*N*-dimethylamino)ethyl diphenylphosphinite-(*P*,*N*))chromium(0), *cis*-$Cr(CO)_4PPh_2OCH_2CH_2NMe_2$	D (THF/MeOH)	31	orange	176—177	53.14 (52.99)	4.67 (4.45)
VI	*cis*-Tetracarbonyl(2-(*N*,*N*-dimethylamino)ethyl diphenylphosphinite-(*P*,*N*))tungsten(0), *cis*-$W(CO)_4PPh_2OCH_2CH_2NMe_2$	D (THF/MeOH)	49	yellow	170d	42.28 (42.20)	3.70 (3.54)
VII	Pentacarbonyl(2-*N*-trifluoroboron-N,N-dimethylamino)ethyl diphenylphosphinite(*P*)molybdenum(0), $Mo(CO)_5PPh_2OCH_2CH_2NMe_2 \cdot BF_3$	B (CH_2Cl_2/hexane)	51	white	160—162	41.24 (41.62)	3.54 (3.49)
VIII	*cis*-Tetracarbonyl(*N*,*N*-dimethyl-*N'*-diphenylphosphine-1,2-diaminoethane(*P*,*N*))molybdenum(0), *cis*-$Mo(CO)_4PPh_2NHCH_2CH_2NMe_2$	B (ether/hexane)	40	yellow	135—136	49.83 (50.12)	4.61 (4.42)
IX	*cis*-Tetracarbonyl(2-(*N*,*N*-dimethylamino)ethyl diphenylphosphinite-(*P*))(diphenylphosphinous acid)molybdenum(0) · 0.5 methanol, *cis*-$Mo(CO)_4$-$PPh_2OCH_2CH_2NMe_2)(PPh_2OH) \cdot 0.5$ MeOH	A (CH_2Cl_2/MeOH)	52	white	126—128	56.01 (55.80)	4.76 (4.76)
XI	*cis*-Tetracarbonylbis(*N*,*N*-dimethyl-*N'*-diphenylphosphino-1,2 diaminoethane(*P*))molybdenum(0), *cis*-$Mo(CO)_4(PPh_2NHCH_2CH_2NMe_2)_2$	A, B (CH_2Cl_2/ether)	55, 60	white	155—159	57.40 (57.45)	5.56 (5.62)
XII	*cis*-Tetracarbonylbis(*N*-diphenylphosphino-1,2-diaminoethane-(*P*))molybdenum(0), *cis*-$Mo(CO)_4(PPh_2NHCH_2CH_2NH_2)_2$	A (see below)	92	white	156—156.5	54.88 (55.18)	4.98 (4.92)
XIII	*cis*-Tetracarbonylbis(*N'*-methyl-*N*-diphenylphosphino-1,2-diaminoethane(*P*))molybdenum(0), *cis*-$Mo(CO)_4(PPh_2NHCH_2CH_2NHMe)_2$	A (CH_2Cl_2/hexane)	65	white	139—140.5	56.02 (56.36)	5.12 (5.29)
XIV	*cis*-Tetracarbonyl(*N*,*N'*-dimethyl-*N*,*N'*-bis(diphenylphosphino-1,2-diaminoethane(*P*,*P'*))molybdenum(0), *cis*-$Mo(CO)_4PPh_2NMeCH_2CH_2NMePPh_2$	A (CH_2Cl_2/MeOH)	37	white	199—204	57.63 (57.81)	4.49 (4.55)

[a] Procedure A: A solution of 3 mmol of II in 20—30 ml of THF was added dropwise with stirring at room temperature to 40—80 mmol of a diamine or 2-(*N*,*N*-dimethylamino)-ethanol over 0.5—2.5 h. Evaporation of the THF under vacuum left a white residue from which the products were extracted by various procedures. Compound XII was obtained directly by washing the residue with absolute ethanol and ether. Compounds IX, XI, XIII and XIV were obtained by washing the respective residues with water and recrystallizing the water-insoluble fractions from the solvent mixtures indicated in the Table.

Procedure B: A solution of 3—6 mmol of an amino alcohol or diamine in 10 ml of THF was added dropwise with stirring at room temperature to a solution of a stoichiometric amount of I or II and 2—3 ml of dry triethylamine in 10 ml of THF. After stirring for a period of several hours, the solution was filtered to remove triethylamine hydrochloride. Complex XI was obtained by evaporation of the filtrate and recrystallization of the residue. Compounds IV and VIII were prepared by a 3—7 h irradiation of the THF filtrate. These products were isolated by evaporating the solvent and recrystallization of the residues. Complex VII was prepared by adding a 25% excess of $BF_3 \cdot$ etherate to a filtered ether solution of $(CO)_5MoPPh_2OCH_2CH_2NMe_2$.

Procedure C: The thermal reaction of stoichiometric amounts (5.00 mmol) of III and $Mo(CO)_6$ in diglyme (25 ml) at 60°C for 30 min yielded IV.

Procedure D: Irradiation of a THF (30 ml) solution of stoichiometric amounts (5 mmol) of the Group VI metal hexacarbonyls and III was carried out for 2 days. The residues obtained after evaporation of the reaction mixtures were recrystallized as indicated in the Table.

TABLE 2

^{1}H NMR (60 MHz) DATA [a]

Complex	M	E	$\delta(CH_2E)$	$\delta(CH_2N)$	$\delta(CH_3N)$	$\delta(HN)$	$\lvert ^2J(HP)\rvert$	$\lvert ^3J(HP)\rvert$	$\lvert ^3J(H_EH_N) + {}^3J(H_EH_{N'})\rvert$
A. *cis*-$M(CO)_4(PPh_2ECH_2CH_2NMe_2)$									
IV	Mo	O	4.13 d, vct	2.75 vct	2.03 s	—	—	18.8	7.0
V	Cr	O	4.10 d, vct	2.62 vct	2.53 s	—	—	17.8	7.6
VI	W	O	4.21 d, vct	2.94 vct	2.91 s	—	—	19.3	7.3
VIII	Mo	NH	3.30 m	2.70 vct	2.63 s	2.04 d	7.8	—	6.0

Complex	*n*	L	E	R	R′	$\delta(CH_2E)$	$\delta(CH_2N)$	$\delta(CH_3N)$	$\delta(HEP)$	$\delta(HNC)$	$\lvert ^2J(H_CP)\rvert$	$\lvert ^3J(HH)\rvert$
B. *cis*-$Mo(CO)_4L_n(PPh_2ECH_2CH_2NRR')_{2-n}$												
VII [b]	1	CO	O	Me	Me	4.15 dt	3.74 t	3.14 s	—	—	4.8	4.8
X [c]	1	PPh_2OH	O	Me	Me	3.42 dt	2.25 t	2.02 s	9.88 bs [c]	—	5.0	5.0
XI	0	—	NH	Me	Me	2.53 bt	2.1 [d]	2.06 s	3.05 m	—	—	5.0
XII	0	—	NH	H	H	2.41 bs	2.41 bs	—	2.41 bs	0.85 s	—	
XIII	0	—	NH	H	Me	2.39 bs	2.38 bs	2.25 s	2.5 [e]	0.84 s	—	

Complex	R	CH_2N		CH_3N	
		$\delta(CH_2N)$	$\lvert ^3J(HP) + {}^5J(HP)\rvert$	$\delta(CH_3N)$	$\lvert ^3J(HP) + {}^5J(HP)\rvert$
C. *cis*-$Mo(CO)_4PR_2NMeCH_2CH_2NMePR_2$					
XIV	Ph	3.50 vct	15.2	2.44 vct	6.5
Ref. 4 (CH_2Cl_2)	Me	3.15 vct	13.2	2.55 vct	8.2

[a] Chemical shifts are in ppm from internal TMS at 0 ppm. The coupling constants are in Hz. The solvent is $CDCl_3$ except for VII in which case CD_3COCD_3 was used. Symbols used: s, singlet; d, doublet; t, triplet (normal); vct, virtual coupled triplet; m, multiplet; br, broad. [b] BF_3 adduct. [c] In the NMR spectrum of IX, the δ 9.88 band of the Ph_2POH ligand is absent owing perhaps to exchange broadening with the alcohol proton of the methanol of crystallization. Also, a sharp signal of the intensity expected for 0.5 CH_3OH of crystallization appears at δ 3.22 ppm and overlaps the NCH_2 signal of X. [d] Overlaps with the CH_3N signal. [e] Overlaps with the CH_2N resonances.

TABLE 3

INFRARED SPECTRA

Complex		M	E	ν(C≡O)	ν((P)—E—C)	ν(P—E—(C))	δ(P—E—C)
A. *cis*-$M(CO)_4(PPh_2ECH_2CH_2NMe_2)$ [a]	V	Cr	O	2014m, 1914m, 1902s, 1867m	947s, 895m	798s	1042s
	IV	Mo	O	2023m, 1921sh, 1908s, 1874m	945s, 896m	798s	1043s
	VI	W	O	2018m, 1913m, 1897s, 1871m	943s, 894m	796s	1042s
	VIII	Mo	NH	2021m, 1906s, 1861m,	956s, 900m	818s	1158s
Complex		**R**	**R′**	**ν(C≡O)**	**ν(NH)**	**ν((P)—N—C)**	**ν[(P—N—(C)]**
B. *cis*-$Mo(CO)_4(PPh_2NHCH_2CH_2NRR')_2$ [b]	XI	Me	Me	2024m, 1927m, 1902s	3305sh, 3266m	1104w, 1083w	810wb, 774m
	XII	H	H	2018m, 1931m, 1901s	3408m, 3356m	1106w, 1089w	800wb, 771m
	XIII	H	Me	2023m, 1925sh, 1905s	3340sh, 3291m	1100w, 1081s	809wb,
C. $(CO)_4Mo(PPh_2PCH_2CH_2NMe_2)(PPh_2OH)$ [b]	X			2020m, 1937m, 1906s			
C. $(CO)_4\overline{MoPPh_2NMeCH_2CH_2NMeP}Ph_2$ [b]	XIV			2024m, 1936m, 1898s			

[a] ν(C≡O) measured on CCl_4 solutions; other values obtained with KBr discs. [b] ν(C≡O) measured on CS_2 solutions; other values obtained with KBr discs.

Of particular interest are those products in which only one terminus of the difunctional amine or amino alcohol has reacted with the halophosphine ligand, thus leaving the other end of the ligand free to undergo subsequent reactions.

Experimental

Reagents and solvents. *cis*-Tetracarbonylbis(chlorodiphenylphosphine)-molybdenum(0) (I) [2] and pentacarbonyl(chlorodiphenylphosphine)molybdenum(0) (II) [3] were prepared by literature methods. Chlorodiphenylphosphine (Aldrich) was vacuum distilled prior to use. Diethyl ether, tetrahydrofuran, diamines and aminoalcohols were distilled from calcium hydride under dry nitrogen. Other solvents were used as received from various suppliers. Triethylamine was dried by distillation from potassium hydride.

Analyses. Elemental analyses were provided by Baron Consulting Company, Orange, Conn. Infrared spectra were obtained with a Perkin—Elmer Model 283 instrument. ^1H NMR spectra were obtained with a Hitachi Perkin—Elmer R20A spectrometer. TMS was the internal standard and all spectral parameters were obtained with a Takeda—Riken 3824X frequency counter. Melting points were determined on samples sealed under nitrogen in capillary tubes. Physical properties and analyses are given in Tables 1—3.

Syntheses and isolation procedures

All reactions were performed under a positive pressure nitrogen atmosphere. Photochemical reactions were carried out in Pyrex Schlenk tubes. Water circulating through an internal probe kept the solutions cool during external irradiation with an unfiltered mercury lamp.

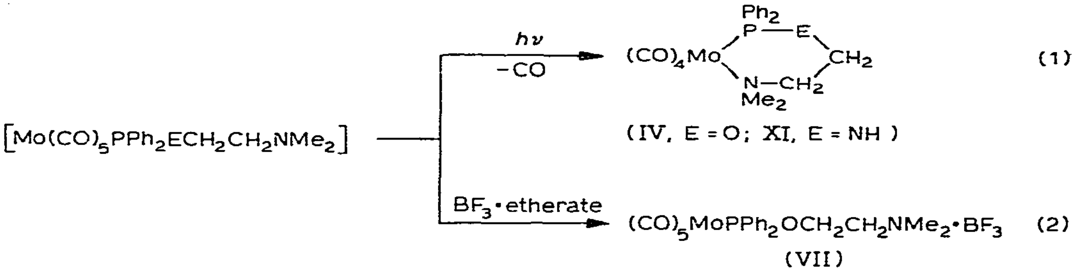
Recrystallizations were carried out with mixed solvents according to the following general procedure. A saturated solution (r.t.) of the impure product in the more polar solvent was heated to boiling whereupon the less polar solvent was added to the point of incipient precipitation. The hot solution was filtered under nitrogen pressure through a 1 cm layer of alumina on the frit of a glass funnel. The filtrate was heated to boiling and an additional amount of the less polar solvent was added to the cloud point. Cooling in a refrigerator (—10° C) and subsequent work-up yielded one or more crops of crystalline product. A summary of the synthetic procedures is given in Table 1.

2-(N,N-Dimethylamino)ethyl diphenylphosphinite, $Me_2NCH_2CH_2OPPh_2$ *(III).* A solution containing 9.67 ml (50.0 mmol) of chlorodiphenylphosphine and 25 ml of triethylamine in 150 ml of diethyl ether was stirred at room temperature as 4.45 g (50.0 mmol) of 2-*N*,*N*-dimethylaminoethanol in 150 ml of diethyl ether was added dropwise over a 4 h period. The filtrate, obtained after collection of solid triethylamine hydrochloride, was evaporated under vacuum to yield the crude product as an oily residue. Distillation at 138° C (0.05 mmHg) gave 8 ml of pure product (^1H NMR) as a clear colorless oil. The product decomposes on standing and was used as prepared in subsequent synthesis.

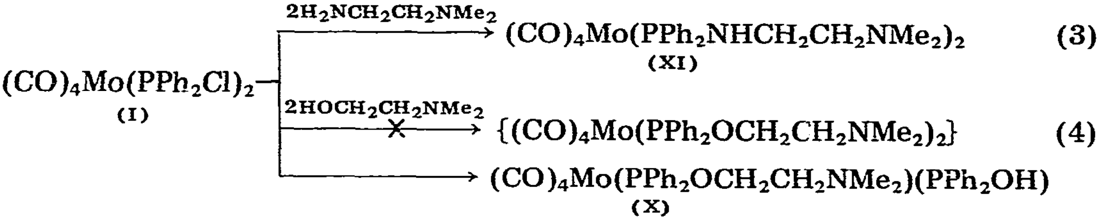
Results and discussion

When $Mo(CO)_5PPh_2Cl$ (II) reacts with $NH_2CH_2CH_2NMe_2$ or $HOCH_2CH_2$-NMe_2 in the presence of triethylamine or excess diamine, the expected products $Mo(CO)_5PPh_2ECH_2CH_2NMe_2$ (E = O, NH) although formed are difficult to purify in as much as they do not crystallize.

As shown in reactions 1 and 2, characterization of these substances has been possible by effecting photochemically induced chelation and in one case by adduct formation with boron trifluoride. Direct thermal reaction of $PPh_2OCH_2CH_2NMe_2$ with $Mo(CO)_6$ also yields IV. Photochemical reactions of this mixed donor ligand with each of the Group VI hexacarbonyls to yield IV and the Cr and W analogs were also successful. Attempts to obtain $Mo(CO)_5PPh_2NHCH_2CH_2NMe_2 \cdot BF_3$ were unsuccessful. An incompletely characterized product containing $Mo(CO)_5$ (IR), and Ph, CH_2 and N-Me (NMR) moieties was obtained. It is possible that this product arose from reaction between BF_3 and the NH part of the ligand.

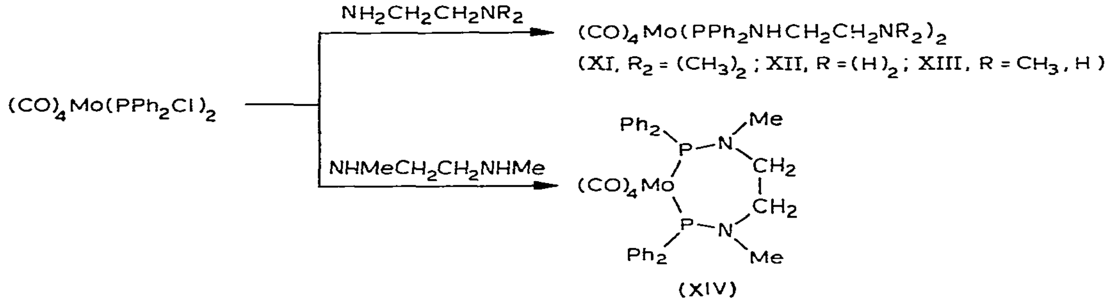


The disubstituted complex, *cis*-$Mo(CO)_4(PPh_2Cl)_2$ (I) when treated with either a stoichiometric amount of $NH_2CH_2CH_2NMe_2$ in the presence of triethylamine or with an excess of the diamine yields the readily crystallized and expected product XI shown in reaction 3. In contrast, $HOCH_2CH_2NMe_2$ did not yield the oxygen analog of XI shown in reaction 4. The product isolated has been characterized as *cis*-$Mo(CO)_4(PPh_2OCH_2CH_2NMe_2)(PPh_2OH)$ (X) based on infrared and ^1H NMR observations. In particular, the metal carbonyl stretching bands agree in number and frequency with those expected for a *cis*-$Mo(CO)_4$-(P donor)$_2$ complex (See Table 1).



Additional IR bands at 1058, 1020 and 938 cm^{-1} can be assigned to P—O—C and P—O—H groups. ^1H NMR signals are readily assigned to the various types of protons in X: δ (ppm) 9.88, broad singlet, POH; 7.58, multiplet, phenyl groups; 3.42, apparent doublet of triplets, P—O—CH_2; 2.25, apparent triplet, CH_2—N; 2.02, singlet, N—CH_3. The respective intensity ratios are 1/20/2/2/6 and support the chemical shift assignments. Exactly how this particular reaction product could arise is not clear. One possible route involves nucleophilic displacement of $CH_2CH_2NMe_2$ from oxygen of a coordinated ligand by either $HOCH_2CH_2NMe_2$ or the NMe_2 base site of a second coordination ligand. The resulting coordinated Ph_2PO^- ligand could then be protonated by $HOCH_2CH_2NMe_2$. This unexpected reaction is not thought to be the result of water present in the reaction mixture. The major consequence of this unusual observation in so far as our immediate studies are concerned is to place a limitation on the type of $(CO)_4Mo(Ph_2PE{-}R)_2$ which may prepared by this general method. Thus, the remaining work reported here was focused on reactions of $(CO)_4Mo(PPh_2Cl)_2$ with ethylenediamine and some *N*-substituted ethylene diamines

The reactions of I with ethylenediamine, *N*-methylethylenediamine and *N,N*-dimethylethylenediamine were found to proceed to give the *cis* disubstituted complexes, XI, XII, and XIII, with no evidence of formation of a chelate complex. In contrast, the only pure product we have been able to obtain with *N,N'*-dimethylethylenediamine is the chelate complex XIV even when a large excess of the diamine was present.



The assigned structures of XII—XIV are based on ^1H NMR and IR observations as well as elemental analyses. That the NH_2 end of $NH_2CH_2CH_2NHMe$ reacts preferentially over the more nucleophilic end suggests the reaction is sterically controlled. This observation agrees with our earlier results which showed secondary amines to be considerably much less reactive to coordinated Ph_2PCl than either ammonia or primary amines. The reaction of I with *sym*-dimethylethylenediamine yields the chelate complex XIV and is possibly another manifestation of a kinetic steric effect. Once one of the Ph_2PCl ligands has reacted with the disecondary amine, the second phosphorus donor ligand is susceptible to attack by either a second molecule of the diamine or can undergo ring formation to yield XIV. Apparently, the reaction with a second molecule of diamine is too slow to compete with the cyclization reaction. For the other two amines, the reaction with a second molecule of the diamine is faster than internal cyclization. The methods of isolation employed here gave no indication of the two types of reaction occurring with the same diamine. Johannsen and coworkers [4] also observed cyclization exclusively with *cis*-$Mo(CO)_4(PMe_2Cl)_2$ and *N,N'*-dimethylethylenediamine.

^1H NMR spectra

The ^1H NMR spectra of complexes were obtained routinely for characterization purpose. Data has been tabulated in Table 2. Although most of the assignments are routine, it is important to note that N—H resonance signals overlap with CH_2 resonances in several cases and assignments had to be based upon peak integration values. An interesting general observation of the complexes reported here is the common occurrence of virtual coupling [5,6]. The spectrum of the chelate complex XIV contains two virtual coupling triplets, one each for the N—CH_3 and N—CH_2 moieties, and is very much like that reported earlier for the $P(CH_3)_2$ analog [4] of XIV. Values of $|{}^3J(HP) + {}^5J(HP)|$ have been measured from the outer lines of the apparent triplets and for XIV are 15.2 Hz (N—CH_2) and 6.5 Hz (N—CH_3). Assuming the $^3J(HP)$ values are 12 Hz based upon observations of PPh_2 NHMe and PPh_2NMe_2 when coordinated to Mo^0 [7], approximate $^5J(HP)$ values would have to be +3 Hz (N—CH_2) and —5 Hz (N—CH_3). Should the two $^3J(HP)$ values not be equal, although we expect them to have the same sign, then the values and signs of $^5J(HP)$ are quite unpredictable. Further synthetic and spectral studies to determine separately the three-band and five-band coupling constants in ring systems of this type are needed.

The ^1H NMR spectra (Fig. 1) of the three chelate complexes *cis*-$(CO)_4M$-$(Ph_2POCH_2CH_2NMe_2)$ (M = Cr, Mo, W) are nearly identical. The $POCH_2CH_2$ section of the chelate ligand constitutes an AA'MM'X system. Interactions of the nuclear moments give rise to three apparent triplets. An apparent triplet assigned to N—CH_2 falls close to the N—CH_3 singlet with the extent of overlap varying among the metals. The O—CH_2 resonance appears as a doublet of

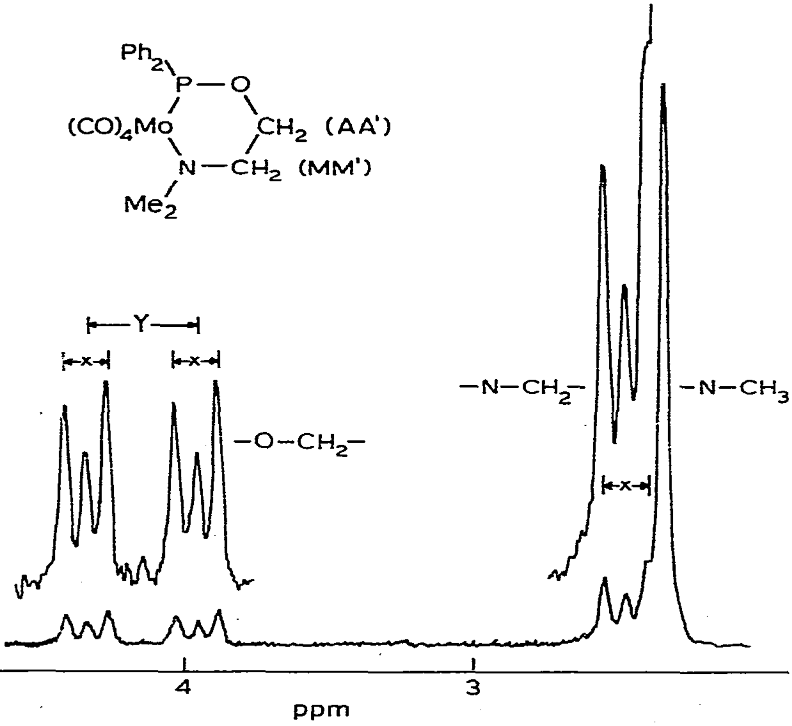


Fig. 1. ^1H NMR (60 MHz) partial spectrum of *cis*-$(CO)_4Mo(PPh_2OCH_2CH_2NMe_2)$ in $CDCl_3$. TMS is the internal standard at 0 PPM. The indicated line separations are: $x = |{}^3J(H_AH_M) + {}^3J(H_AH_{M'})|$; $y = |{}^3J(H_AP)| = |{}^3J(H_{A'}P)|$.

apparent triplets with ^3J(HP) of 18—19 Hz for all three metals. We feel that these apparent triplets also represents virtual coupling and are of a type less commonly seen in metal complexes. As such then, the separations of the outer lines of each OCH_2 apparent triplet yield consistent values of 7.0 to 7.3 Hz for $|^3J(AM) + {}^3J(AM')|$. As is apparent from the Fig. 1, this value is not measurable directly from the N—CH_2 resonance in any case. However, twice the separations between the one visible sharp line and the center of the broad middle peak gives $|^3J(AM) + {}^3J(AM')|$ values in agreement with those obtained from the OCH_2 resonances for Cr and Mo. A value for W cannot be obtained at 60 Mhz owing to overlap with the N—CH_3 singlet.

Infrared spectra

Table 3 contains infrared data obtained for these complexes. The metallocarbonyl bands for the various tetracarbonyl species indicate all of the species studied to have a *cis* geometry [8]. Assignments of bands in the 1100—700 cm^{-1} region are not without some uncertainty, but are consistent with prior assignments [1,9].

Acknowledgements

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