

Carbonyl Insertion Reactions of Benzyl- and Allyl-tricarbonyl- π -cyclopentadienylmolybdenum¹

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Treatment of a solution of benzyltricarbonyl- π -cyclopentadienylmolybdenum in acetonitrile with phosphines and phosphites (L) affords the crystalline complexes *trans*-PhCH₂·COMo(CO)₂(L)(π -C₅H₅); allyltricarbonyl- π -cyclopentadienylmolybdenum undergoes analogous reactions forming the complexes *trans*-C₃H₅·COMo(CO)₂(L)(π -C₅H₅). *c/s*-But-2-enyltricarbonyl- π -cyclopentadienylmolybdenum reacts with triphenylphosphine in acetonitrile to form *trans*-CH₃·CH=CH·CH₂·COMo(CO)₂(Ph₃P)(π -C₅H₅). When *trans*-PhCH₂·COMo(CO)₂(Ph₃P)(π -C₅H₅) is heated (140°) decarbonylation occurs with the formation of PhCH₂Mo(CO)₂(Ph₃P)(π -C₅H₅). Kinetic measurements show that the rate-determining step in the carbonylation reactions does not involve the reacting ligand. The stereochemistry of these reactions is discussed on the basis of ¹H n.m.r. and i.r. spectroscopic measurements.

METHYL- and ethyl-tricarbonyl- π -cyclopentadienylmolybdenum both readily undergo carbonyl insertion reactions when treated with carbon monoxide,² phosphines and phosphites.³ The analogous reactions of benzyl-, allyl-, and but-2-enyl-tricarbonyl- π -cyclopentadienylmolybdenum have been studied with a view to obtaining a clearer understanding of the stereochemistry and mechanism of these reactions.

i.r. (Table 2) and ¹H n.m.r. (see Experimental section) spectroscopy.

The i.r. spectra of the complexes (I), (II), (III), (IV), (VI), and (VII) all show two terminal carbonyl bands and, in addition, bands of medium intensity in the region 1650–1600 cm.⁻¹ assignable to a metal acyl group (RCOMo). The presence of acyl groups in these complexes is confirmed by the chemical shifts of the

TABLE 1
Molybdenum complexes

Compound	Colour	Yield	M.p.	Found (%)			Reqd. (%)		
				C	H	P	C	H	P
(I) PhCH ₂ ·COMo(CO) ₂ (Ph ₃ P)(π -C ₅ H ₅)	Yellow	85	124–126°	66.2	4.6	5.4	66.2	4.6	5.2
(II) PhCH ₂ ·COMo(CO) ₂ [EtC(CH ₂ O) ₃ P](π -C ₅ H ₅)	Yellow	92	136–138	50.6	4.8	6.5	50.6	4.6	6.3
(III) PhCH ₂ ·COMo(CO) ₂ [Bu ⁿ] ₃ P](π -C ₅ H ₅)	Lemon yellow	14	75	60.5	7.2	5.9	60.3	7.3	5.8
(IV) PhCH ₂ ·COMo(CO) ₂ [(PhO) ₃ P](π -C ₅ H ₅)	Light yellow	60	42	61.7	4.7	4.7	61.3	4.2	4.8
(V) PhCH ₂ Mo(CO) ₂ (Ph ₃ P)(π -C ₅ H ₅)	Yellow-orange	39	119	67.0	5.1	5.3	67.4	4.7	5.4
(VI) C ₃ H ₅ ·COMo(CO) ₂ (Ph ₃ P)(π -C ₅ H ₅)	Yellow	38	117	62.9	4.8	5.9	63.5	4.6	5.7
(VII) C ₃ H ₅ ·COMo(CO) ₂ [EtC(CH ₂ O) ₃ P](π -C ₅ H ₅)	Yellow	20	92	45.3	5.4	6.9	45.5	4.7	6.9
(VIII) C ₄ H ₇ ·COMo(CO) ₂ (Ph ₃ P)(π -C ₅ H ₅)	Yellow	53	108	64.0	5.1	5.4	64.1	5.0	5.5

TABLE 2
I.r. spectra (cm.⁻¹) *

Compound	Terminal carbonyl bands	Acyl band
(I) PhCH ₂ ·COMo(CO) ₂ (Ph ₃ P)(π -C ₅ H ₅)	1943s	1866vs
(II) PhCH ₂ ·COMo(CO) ₂ [EtC(CH ₂ O) ₃ P](π -C ₅ H ₅)	1963s	1892vs
(III) PhCH ₂ ·COMo(CO) ₂ [Bu ⁿ] ₃ P](π -C ₅ H ₅)	1933s	1849vs
(IV) PhCH ₂ ·COMo(CO) ₂ [(PhO) ₃ P](π -C ₅ H ₅)	1956s	1882vs
(V) PhCH ₂ Mo(CO) ₂ (Ph ₃ P)(π -C ₅ H ₅)	1973s	1896vs
(VI) C ₃ H ₅ ·COMo(CO) ₂ (Ph ₃ P)(π -C ₅ H ₅)	1943s	1866vs
(VII) C ₃ H ₅ ·COMo(CO) ₂ [EtC(CH ₂ O) ₃ P](π -C ₅ H ₅)	1965s	1898vs
(VIII) C ₄ H ₇ ·COMo(CO) ₂ (Ph ₃ P)(π -C ₅ H ₅)	1941s	1865vs

* Recorded with a Perkin Elmer 257 spectrophotometer in cyclohexane solution. s = Strong, vs = very strong, m = medium

The reaction of RMo(CO)₃(π -C₅H₅) (R = alkyl group) with carbon monoxide and phosphorus ligands is strongly solvent-dependent,³ and in acetonitrile rapid reaction occurs at room temperature thus avoiding complications arising from decarbonylation. Benzyl- and allyl-tricarbonyl- π -cyclopentadienylmolybdenum both react with phosphorus ligands in acetonitrile to afford the coloured crystalline acyl complexes listed in Table 1, characterised by elemental analysis (Table 1), and by

PhCH₂ and CH₂·CH·CH₂ protons, which occur at lower field than in the corresponding complexes where the benzyl and allyl groups are directly bonded to molybdenum. Thus the reaction of RMo(CO)₃(π -C₅H₅) (R = benzyl or allyl) with phosphorous ligands results in the displaced carbon monoxide formally inserting into the carbon-molybdenum bond, as has been established^{3a} with the related ethyl- and methyl-molybdenum complexes.

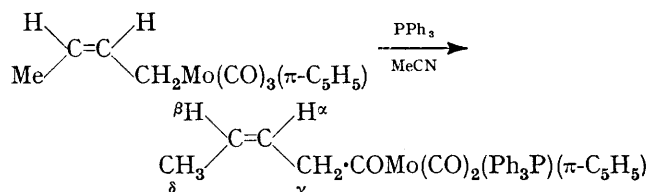
¹ For a preliminary report see P. J. Craig and M. Green, *Chem. Comm.*, 1967, 1246.

² J. A. McCleverty and G. Wilkinson, *J. Chem. Soc.*, 1963, 4096.

³ (a) K. W. Barnett and P. M. Treichel, *Inorg. Chem.*, 1967, 6, 294; (b) I. S. Butler, F. Basolo, and R. G. Pearson, *Inorg. Chem.*, 1967, 6, 2074; (c) P. J. Craig and M. Green, *J. Chem. Soc. (A)*, 1968, 1978.

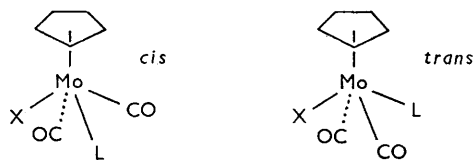
cis-But-2-enyltricarbonyl- π -cyclopentadienylmolybdenum, prepared from *cis*-1-chlorobut-2-ene and $[(\pi\text{-C}_5\text{H}_5)\text{Mo}(\text{CO})_3]^- \text{Na}^+$ in tetrahydrofuran, reacts with triphenylphosphine in acetonitrile to form the yellow crystalline complex (VIII). Elemental analysis (Table 1), the presence in the i.r. spectrum (Table 2) of two terminal carbonyl bands and an acyl band established the gross structure of the complex. The ^1H n.m.r. spectrum showed bands at τ 2.60 (15H, $\text{C}_5\text{H}_5\text{P}$), 4.61 (1H, H_α , $J_{\alpha\beta}$ 6.0, $J_{\alpha\gamma}$ 5.0 c./sec.), 4.81 (1H, H_β , $J_{\beta\alpha}$ 6.0, $J_{\beta\gamma}$ 6.0 c./sec.), 5.05 [5H, $\pi\text{-C}_5\text{H}_5$, $J(\text{HP})$ 1.0 c./sec.], 6.32 (2H, H_γ , $J_{\gamma\alpha}$ 5.0 c./sec.), and 8.39 (3H, H_δ , $J_{\delta\beta}$ 6.0 c./sec.).

This reaction therefore involves carbonyl insertion with retention of the *cis*-stereochemistry of the double bond:



It is interesting that the reaction proceeds without an allylic rearrangement, *i.e.* $\text{CH}_3\cdot\text{CH}:\text{CH}\cdot\text{CH}_2\text{Mo} \rightarrow \text{CH}_2=\text{CH}\cdot\text{CHMe}\cdot\text{COMo}$. The absence of products corresponding to the 'turn-round' of the allylic system has also been observed⁴ with the related reactions of $\text{CH}_3\cdot\text{CH}:\text{CH}\cdot\text{CH}_2\text{Mn}(\text{CO})_5$. These observations are of particular interest in view of the reported⁵ occurrence of allylic isomerisation during the course of the sulphur dioxide insertion reaction.

The relative intensities of the two terminal carbonyl bonds of the complexes $\text{XMo}(\text{CO})_2(\text{L})(\pi\text{-C}_5\text{H}_5)$ ($\text{X} = \text{H}$,⁶ I ,⁷ SnPh_3 ,⁸ MeCO ,^{3c} and EtCO ;^{3c} $\text{L} =$ phosphorus ligands) can be tentatively related to the CO-Mo-CO bond angles, which are different for *cis*- and *trans*-complexes, *i.e.* *cis*- CO-Mo-CO *ca.* 78° , *trans*- CO-Mo-CO



ca. 106° .^{9,10} With *trans*-complexes the carbonyl band at highest wavenumbers would be expected^{7,10} to have the lowest intensity, whereas, the reverse is predicted for the corresponding *cis*-complexes. It has also been noted,^{3c,6,7} that so-called *trans*-complexes show the cyclopentadienyl proton n.m.r. signal as a doublet arising from ^1H - ^{31}P coupling, whereas, no coupling is observed with *cis*-complexes. Recently,¹¹ an X-ray diffraction study with $\text{MeCOMo}(\text{CO})_2(\text{Ph}_3\text{P})(\pi\text{-C}_5\text{H}_5)$ established a *trans*-stereochemistry, which had previously^{3c} been suggested for this complex on the basis of relative

terminal carbonyl intensities and $(\pi\text{-C}_5\text{H}_5)\text{-}^{31}\text{P}$ coupling. Thus the relative intensities of the carbonyl bonds of the complexes (I)–(VIII) (Table 2) and the appearance of the $(\pi\text{-C}_5\text{H}_5)$ -proton signal of all of the complexes as a doublet, strongly suggests that these complexes have a *trans*-stereochemistry.

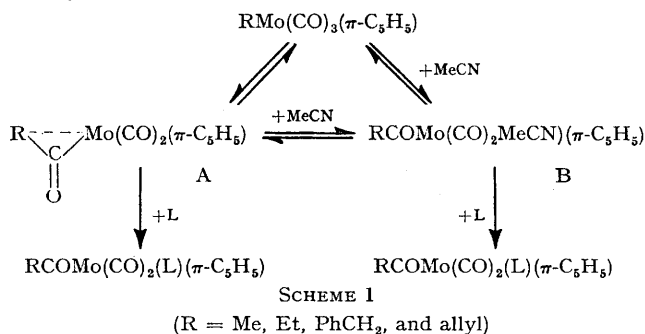
When the formation of these complexes was followed in deuterioacetonitrile by ^1H n.m.r. spectroscopy there was no evidence for the initial formation of a *cis*-complex, and therefore, it is likely that the *trans*-complexes (I)–(VIII) are formed directly and not in a subsequent thermodynamically controlled process, as has been observed¹² with the reaction of $\text{MeMn}(\text{CO})_5$ with triphenylphosphine.

TABLE 3

Kinetic parameters for the reaction of $\text{PhCH}_2\text{Mo}(\text{CO})_3(\pi\text{-C}_5\text{H}_5)$ ($1.69 \times 10^{-4}\text{M}$) and $\text{C}_3\text{H}_5\text{Mo}(\text{CO})_3(\pi\text{-C}_5\text{H}_5)$ ($1.69 \times 10^{-4}\text{M}$) with phosphorus ligands in acetonitrile

Compound	Ligand	Ligand concn. (L) (10^{-2}M)	Temp.	$10^4 K_{\text{obs}}$ (sec. $^{-1}$)
$\text{PhCH}_2\text{Mo}(\text{CO})_3(\pi\text{-C}_5\text{H}_5)$	PPh_3	6.60	30.5°	2.39
		13.40	30.5	2.39
		19.60	30.5	2.39
	$\text{Ph}(\text{OPh})_3$	6.62	45	8.25
		13.30	45	8.10
		8.87	30	1.85
$\text{C}_3\text{H}_5\text{Mo}(\text{CO})_3(\pi\text{-C}_5\text{H}_5)$	PPh_3	17.0	30	1.95
		24.0	30	2.00
		7.65	45	1.00
	$\text{P}(\text{OPh})_3$	13.52	45	1.05
		36.88	45	1.10
		65.64	45	1.11
		9.39	45	1.00
		67.80	45	1.70

Kinetic measurements (Table 3) on the reaction of phosphorus ligands with benzyl- and allyl-tricarbonyl- π -cyclopentadienylmolybdenum in acetonitrile clearly demonstrates that the rate of reaction is virtually independent of ligand and ligand concentration, which implies the rate-determining formation of a reactive intermediate. Two alternative formulations for the intermediate in these reactions have been previously briefly considered:^{3c}



⁴ M. Green and D. J. Westlake, unpublished observations.
⁵ F. A. Hartman, P. J. Pollick, R. L. Downs, and A. Wojcicki, *J. Amer. Chem. Soc.*, 1967, **89**, 2495.
⁶ Alison Bainbridge, P. J. Craig, and M. Green, *J. Chem. Soc. (A)*, 1968, 2715.
⁷ A. R. Manning, *J. Chem. Soc. (A)*, 1967, 1984.
⁸ A. R. Manning, *J. Chem. Soc. (A)*, 1968, 65.

⁹ M. J. Bennett and R. Mason, *Proc. Chem. Soc.*, 1963, 273; M. R. Churchill and J. P. Fennessey, *Chem. Comm.*, 1966, 695.

¹⁰ W. Beck, A. Melnikoff, and R. Stahl, *Chem. Ber.*, 1966, **99**, 3721.

¹¹ M. R. Churchill and J. P. Fennessey, *Inorg. Chem.*, 1968, **7**, 953.

¹² K. Noack, M. Ruch, and F. Calderazzo, *Inorg. Chem.*, 1968, **7**, 345.

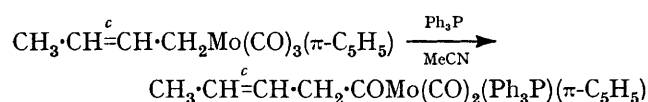
Inorg. Phys. Theor.

Examination of the ^1H n.m.r. spectrum of benzyl-, allyl-, and but-2-enyl-tricarbonyl- π -tricarbonyl- π -cyclopentadienylmolybdenum in deuterioacetonitrile which is a good donor solvent, provided no evidence for the intermediate B. If the solvated intermediate B is the precursor of the *trans*-acyl complexes then the displacement of acetonitrile by the phosphorus ligand must involve retention of stereochemistry. This follows from the earlier observation^{3c} that the bimolecular reaction of $\text{EtMo}(\text{CO})_3(\pi\text{-C}_5\text{H}_5)$ with phosphorus ligands in chloroform also gives *trans*-acyl complexes; and the formation of the intermediate B involves the bimolecular reaction of acetonitrile with $\text{RMo}(\text{CO})_3(\pi\text{-C}_5\text{H}_5)$.

Although no stereochemical studies are available it seems unlikely that the bimolecular displacement of acetonitrile from intermediate B by phosphorus ligands would proceed stereospecifically with retention of configuration, and therefore, the present evidence is accommodated more satisfactorily by the rate-determining formation of the non-solvated intermediate A. The function of acetonitrile in facilitating the rate-determining formation of A must then be to stabilise the transition state for the migration of the R group from the molybdenum to co-ordinated carbon monoxide.

The reactivity sequence $\text{Et} > \text{Me} > \text{PhCH}_2 > \text{CH}_2\cdot\text{CH}\cdot\text{CH}_2$ established in this and a previous study^{3c} suggests that little or no charge separation is involved in

the step $\text{Mo}-\text{CO} \xrightarrow{\text{R}} \text{MoCOR}$. The absence of *cis-trans* isomerisation about the C=C bond during the reaction



provides a more sensitive measure of the degree of charge separation involved in the reactions, and emphasises the intramolecular nature of these 1,2-migration reactions.

The decarbonylation of the complex (I) was briefly studied. At 140° in the absence of a solvent rapid loss of carbon monoxide occurred to give the complex (V) (Table 1), characterised by i.r. (absence of an acyl bond) (Table 2) and ^1H n.m.r. (the presence of the $\text{C}_6\text{H}_5\text{CH}_2$ signal at high field corresponding to the absence of an acyl group) spectroscopy. The relative intensities of the terminal carbonyl bands suggests a *trans*-stereochemistry for (V), however, the cyclopentadienyl proton signal appears as a singlet, and therefore, the stereochemistry of the complex remains undecided.

EXPERIMENTAL

Proton n.m.r. spectra were recorded in deuteriochloroform on a Varian Associates HA 100 spectrometer. All reactions were carried out in a nitrogen atmosphere. Acetonitrile was distilled from phosphorus pentoxide.

Reactions with Benzyltricarbonyl- π -cyclopentadienylmolybdenum.—(a) *Triphenylphosphine*. A solution of triphenylphosphine (0.90 g., 3.44 mmoles) in acetonitrile (15 ml.) was added dropwise with stirring to a solution of benzyltri-

carbonyl- π -cyclopentadienylmolybdenum (1.0 g., 2.98 mmoles) in acetonitrile (30 ml.). After $4\frac{1}{2}$ hr. at room temperature the solvent was removed *in vacuo*, and the residue recrystallised (-78° , methylene chloride-hexane) to give (I) (1.50 g., 85%). The ^1H n.m.r. spectrum showed bands at τ 2.58 [20H, $\text{C}_6\text{H}_5\text{P}$ and $\text{C}_6\text{H}_5\text{CH}_2$], 5.02 [5H,d, $\pi\text{-C}_5\text{H}_5$, $J(\text{HP})$ 1.2 c./sec.], and 5.70 (2H,s, $\text{COCH}_2\text{C}_6\text{H}_5$).

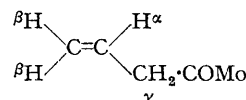
(b) *4-Ethyl-2,6,7-trioxa-1-phosphabicyclo[2,2,2]octane*. 4-Ethyl-2,6,7-trioxa-1-phosphabicyclo[2,2,2]octane (0.35 g., 2.16 mmoles) in acetonitrile (15 ml.) was treated similarly with benzyltricarbonyl- π -cyclopentadienylmolybdenum (0.60 g., 1.79 mmoles) in acetonitrile (15 ml.). Removal of the solvent *in vacuo* followed by recrystallisation of the solid residue (-78° , methylene chloride-hexane) gave (II) (0.53 g., 92%). The ^1H n.m.r. spectrum showed bands at τ 2.71 (5H, $\text{C}_6\text{H}_5\text{CH}_2$), 4.75 [5H,d, $\pi\text{-C}_5\text{H}_5$, $J(\text{HP})$ 1.3 c./sec.], 5.67 [6H,d, $\text{P}-\text{OCH}_2$, $J(\text{HP})$ 5.0 c./sec.], 5.85 [2H,s, $\text{COCH}_2\text{C}_6\text{H}_5$], 8.16 (2H,m, CH_2CH_3), and 9.19 (3H,m, CH_2CH_3).

(c) *Tri-n-butylphosphine*. Reaction of tri-n-butylphosphine (0.40 g., 1.98 mmoles) with benzyltricarbonyl- π -cyclopentadienylmolybdenum (0.46 g., 1.37 mmoles) in acetonitrile (20 ml.) proceeded similarly. Removal of the solvent *in vacuo*, and recrystallisation (-78° , methylene chloride-hexane) gave (III) (0.10 g., 14%). The ^1H n.m.r. spectrum showed bands at τ 2.95 (5H, $\text{C}_6\text{H}_5\text{CH}_2$), 4.95 [5H,d, $\pi\text{-C}_5\text{H}_5$, $J(\text{HP})$ 1.1 c./sec.], 5.81 (2H, $\text{COCH}_2\text{C}_6\text{H}_5$), and 7.81–9.32 [18H, complex m, $\text{P}-\text{CH}_2\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{CH}_3$].

(d) *Triphenylphosphite*. A similar reaction of triphenylphosphite (0.33 g., 1.06 mmoles) with benzyltricarbonyl- π -cyclopentadienylmolybdenum (0.50 g., 1.49 mmoles) in acetonitrile (30 ml.) gave a solid residue, which was recrystallised 5 times (78° , methylene chloride-hexane) to give (IV) (0.6 g., 60%). The ^1H n.m.r. spectrum showed bands at τ 2.64 (20H, C_6H_5 and $\text{C}_6\text{H}_5\text{CH}_2$), 5.08 [5H,d, $\pi\text{-C}_5\text{H}_5$, $J(\text{HP})$ 1.2 c./sec.], and 6.21 (2H, s, $\text{COCH}_2\text{C}_6\text{H}_5$).

Decarbonylation of Dicarboxyphenylacetyl(triphenylphosphine)- π -cyclopentadienylmolybdenum (I).—Compound (I) (0.75 g., 1.2 mmoles) was heated (140°) in a sealed evacuated tube for $\frac{1}{2}$ hr., when the solid melted with evolution of gas. The residue was recrystallised twice (methylene chloride-hexane) to give (V) (0.28 g., 39%). The ^1H n.m.r. spectrum showed bands at τ 2.4–2.9, (20H, $\text{C}_6\text{H}_5\text{P}$ and $\text{C}_6\text{H}_5\text{CH}_2$), 4.73 (5H,s, $\pi\text{-C}_5\text{H}_5$), and 8.75 (2H,s, $\text{MoCH}_2\text{C}_6\text{H}_5$).

Reactions with Allyl tricarbonyl- π -cyclopentadienylmolybdenum.—(a) *Triphenylphosphine*. A solution of triphenylphosphine (1.1 g., 4.2 mmoles) in acetonitrile (5 ml.) was added dropwise with stirring to a solution of allyl tricarbonyl- π -cyclopentadienylmolybdenum (1.1 g., 3.85 mmoles) in acetonitrile (30 ml.) at room temperature. When the yellow reaction mixture gradually became green. After 22 hr. the solvent was removed *in vacuo*, and the residue recrystallised (methylene chloride-hexane) to give (VI) (0.80 g., 38%). The ^1H n.m.r. spectrum showed bands at τ 2.64 (15H, $\text{C}_6\text{H}_5\text{P}$), 4.20 (1H, H_α , $J_{\alpha\beta}$ 17.0, $J_{\alpha\gamma}$ 10.8, $J_{\alpha\delta}$ 6.9 c./sec.), 5.08 [5H,d, $\pi\text{-C}_5\text{H}_5$, $J(\text{HP})$ 1.0 c./sec.], 5.14 (1H, H_β , $J_{\beta\alpha}$ 17.0, $J_{\beta\gamma}$ 2.40 c./sec.), 5.25 (1H, H_β , $J_{\beta\delta}$ 2.40, $J_{\beta\alpha}$ 10.8 c./sec.), and 6.31 (2H,d, H_γ , $J_{\gamma\alpha}$ 6.90 c./sec.); the assignments were confirmed by double-irradiation experiments.



(b) *4-Ethyl-2,6,7-trioxa-1-phosphabicyclo[2,2,2]octane*. A

solution of allyltricarbonyl- π -cyclopentadienylmolybdenum (0.70 g., 2.44 mmoles) and 4-ethyl-2,6,7-trioxa-1-phosphabicyclo[2,2,2]octane (0.40 g., 2.97 mmoles) in acetonitrile (25 ml.) was heated (70°) for 4 hr. The solvent was removed *in vacuo* to give an oily residue which was recrystallised three times (−78°, methylene chloride–hexane) to give (VII) (0.20 g., 20%). The ^1H n.m.r. spectrum showed bands at τ 4.14 (1H, H_α , $J_{\alpha\beta}$ 17.0, $J_{\alpha\gamma}$ 10.0, $J_{\alpha\delta}$ 7.5 c./sec.), 4.48 [5H, d, $\pi\text{-C}_5\text{H}_5$, $J(\text{HP})$ 1.20 c./sec.], 5.10 (1H, H_β , $J_{\beta\alpha}$ 17.0, $J_{\beta\gamma}$ 2.1 c./sec.), 5.29 (1H, H_β , $J_{\beta\alpha}$ 2.1, $J_{\beta\gamma}$ 10.0 c./sec.), 5.49 [6H, d, POCH_2 , $J(\text{HP})$ 5.2 c./sec.], 6.16 (2H, d, H_γ , $J_{\gamma\alpha}$ 7.5 c./sec.), and 8.93 [5H, m, CH_2CH_3].

Reaction Between Triphenylphosphine and But-2-enyltricarbonyl- π -cyclopentadienylmolybdenum.—A solution of triphenylphosphine (0.55 g., 2.1 mmoles) and but-2-enyltricarbonyl- π -cyclopentadienylmolybdenum (0.50 g., 1.67 moles) in acetonitrile (30 ml.) was stirred at room temperature for 21 hr. The solvent was removed *in vacuo*, and the residue recrystallised (−78°, pentane) to give (VIII) (0.50 g., 53%).

Kinetic Measurements.—The rates of reactions were followed by the methods previously^{3c} described.

[8/1141 Received, August 7th, 1968]