η^3 -ALLYL COMPLEXES OF MOLYBDENUM—IV.* PREPARATION AND CRYSTAL STRUCTURES OF SOME SUBSTITUTED-CYCLOPENTADIENYL MOLYBDENUM ALLYL DERIVATIVES

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Abstract—A series of substituted-cyclopentadienyl molybdenum allyl derivatives has been prepared by reacting $(\eta^3-C_3H_5)_3$ MoCl with substituted-cyclopentadienyl and -indenyl alkali metal salts. In general, the isolated products are of the type Cp'Mo $(\eta^3-C_3H_5)_2$ and in three cases this has been confirmed by crystal structure determinations. Spectroscopic evidence suggests that at low temperatures compounds have the composition Cp'Mo $(\eta^3-C_3H_5)_2(\eta^1-C_3H_5)$ are also formed. The reaction with potassium fluorenyl is anomalous: the NMR spectra and a crystal structure determination show that the product $(\eta^3$ -fluorenyl)Mo $(\eta^3-C_3H_5)_3$, contains four η^3 -allyl groups bonded unsymmetrically to the metal atom.

The dormancy in the chemistry of tetra(η^3 -allyl) molybdenum was interrupted after *ca* twenty years with the isolation of (η^3 -C₃H₅)₃MoCl (1) from the reaction with gaseous HCl in pentane at -70° C [eq. (1)] and this compound has been used to prepare a series of η^3 -allyl molybdenum complexes.¹⁻³

One of the more interesting reactions studied has been that with sodium cyclopentadienide from which the paramagnetic complex $CpMo(\eta^3-C_3H_5)_2$ (2) could be isolated and its structure and electron deformation density determined by X-ray methods.⁴

$$(\eta^{3}-C_{3}H_{5})_{4}Mo \xrightarrow{HCl} (\eta^{3}-C_{3}H_{5})_{3}MoCl$$

$$1$$

$$\xrightarrow{NaCp} CpMo(\eta^{3}-C_{3}H_{5})_{2} \quad (1)$$

$$2$$

* Part III—Benn et al.¹

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Although these results have considerably extended the chemistry of the allyl molybdenum system, the compounds have proved rather unreactive, (2), in particular, (in contrast to the related chromium compound)⁵ shows no remarkable reactivity in either stoichiometric or catalytic reactions. There are two obvious approaches for improving the situation—the introduction of substituents either into the cyclopentadienyl ring or the allyl group. Here we report the synthesis of a series of substituted-cyclopentadienyl and -indenyl molybdenum allyl complexes and a subsequent publication will be devoted to their reactions.

RESULTS AND DISCUSSION

The substituted-cyclopentadienyl and -indenyl molybdenum allyl compounds shown in Table 1 have been prepared in 60-80% yield by reacting $(\eta^3-C_3H_5)_3$ MoCl (1) with the appropriate alkali metal salt in THF at $ca - 30^{\circ}$ C. With a few exceptions (see below), the products are of the type Cp'Mo $(\eta^3-C_3H_5)_2$ e.g. [eq. (2)].

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The compounds are in general orange-red solids and are thermally stable at room temperature. The anticipated and observed paramagnetism (one unpaired electron, Table 1) prevented the use of NMR-spectroscopy to obtain useful structural information and since it was not clear how the indenyl system would bond to the metal (η^3, η^5) or even η^6 - situations are conceivable), we have undertaken crystal structure determinations of the compounds containing the 2-methylindenyl, 2methoxyindenyl and 5,6-dimethoxyindenyl groups : in all cases the indenyl derivative is bonded in an η^5 - manner (see discussion below).



I	I	IV		
2.04	2.05	2.342(2)		
2.00	1.87	2.07		
2.01	2.01	2.07		
—	<u> </u>	2.09		
1.20(4)	1.35(4)	1.404(5)		
1.29(4)	1.60(4)	1.385(4)		
1.15(4)	1.28(4)	1.381(5)		
1.45(5)	1.39(4)	1.390(5)		
	<u></u>	1.396(4)		
—	<u> </u>	1.384(4)		
1.44(2)	1.52(2)	1.465(4)		
1.40(2)	1.42(2)	1.407(3)		
1.39(2)	1.33(2)	1.433(3)		
1.34(2)	1.51(2)	1.458(3)		
1.42(2)	1.40(2)	1.476(3)		
128.3	132.5	107.6		
130.3	127.5	107.8		
100.5	97.4	95.2		
_		103.4		
—		137.6		
_		102.2		
146(3)	127(3)	122.3(3)		
136(3)	121(2)	123.3(3)		
	_	123.3(2)		
	1.1.5(4) 1.45(5) 1.44(2) 1.40(2) 1.39(2) 1.39(2) 1.34(2) 1.42(2) 1.42(2) 1.28.3 130.3 100.5 146(3) 136(3) 	$\begin{array}{cccccccccccccccccccccccccccccccccccc$		

Table 1. Distances (Å) and angles (°)

D1: midpoint C1—C5 (in IV = C4); D2: midpoint C14—C16; D3: midpoint C17—C19; D4: midpoint C20—C21.

reaction with indenyl-lithium in ether at -78° and washing the product repeatedly with pentane at -78° C, it is possible to obtain a small amount of a relatively insoluble red-violet material whose IR spectrum (KBr, -78° C) suggests the presence of an η^1 -allyl group ($v_{C;C}$ 1640, 910 cm⁻¹) while the ¹H-NMR spectrum (D₈-THF, -78° C) although not completely interpretable is that of a diamagnetic species and shows absorptions compatible with the presence of a vinyl group (δ_{HC} ca 5.9–5.8 m, br, 4.3 d, 4.1 dd; for comparison, the vinyl protons in (t- $C_4H_9C_5H_4W(\eta^3-C_3H_5)_2C_3H_5$ are found at 5.68 m, 4.33 d and 4.23 dd ppm⁶). Similar absorptions are observed in the low temperature IR spectra of the products of the reaction with t-C4H9C5H4Li $(v_{C;C}1635, 908 \text{ cm}^{-1})$ and CpLi $(v_{C;C}1640, 912)$ phenylindenyl and 5,6-dimethoxyindenyl derivatives mentioned above, the main product of the reaction between $(\eta^3-C_3H_3)_3MoCl$ and the substitutedcyclopentadienyl or -indenyl derivatives at $ca - 30^\circ$ are the paramagnetic compounds of the type $Cp'Mp(\eta^3-C_3H_5)_2$ while at -78° these are contaminated with $Cp'Mo(\eta^3-C_3H_5)_2C_3H_5$ systems.

The reaction with fluorenyl potassium takes a different course : a purple, diamagnetic compound is formed which has the composition (fluorenyl) $Mo(\eta^3-C_3H_5)_3$. The crystal structure determination (see below) and the NMR spectral data (see Experimental) indicate that the fluorenyl group is acting as an η^3 -organic ligand and that the molecule, in addition, contains three $\eta^3-C_3H_5$ -groups [eq. (3)].



cm⁻¹) and are absent from the spectra of the related CpMo(η^3 -C₃H₅)₂ and (indenyl)Mo(η^3 -C₃H₅)₂ species.

In the case of the reaction with 5,6-dimethoxyindenyl lithium, the IR spectrum and elemental analysis indicate that the major component containing an η^1 -allyl group ($v_{\rm C}$ 1622, 905 cm⁻¹) is stable at room temperature and here repeated washing has also enabled a sample to be isolated whose ¹H-NMR spectrum suggests the presence of a vinyl group (δ_{HC} 5.9–5.8 m,br, 4.26 d, 4.13 d) although the broadness of the signals suggests contamination by paramagnetic material (the structure of what is presumably the minor component, viz. (η^{5} -5,6dimethoxyindenyl)Mo(η^3 -C₃H₅)₂, is presented below). The elemental analysis of the product of the reaction with 2-phenylindenyl lithium suggests that this compound should also be formulated as (2phenylindenyl)Mo(C_3H_5), in agreement with the observed diamagnetism. Unfortunately, we were unable to prepare sufficient of this compound for an NMR-spectoscopic study or to obtain suitable crystals for an X-ray diffraction study so that we cannot decide whether this compound should be formulated as $(\eta^3-2-\text{phenylindenyl})Mo(\eta^3-C_3H_5)_3$ (see the fluorenyl compound discussed below) or $(\eta^{5}-2-\text{phenylindenyl})Mo(\eta^{3}-C_{3}H_{5})_{2}C_{3}H_{5}$. The analytical data for the product of the reaction with 1-CyCH₂indenyl lithium lies between that expected for tris-allyl and bis-allyl systems, so that presumably a mixture of the two species is formed.

To summarize, with the exception of the 2-

All fifteen H-atoms of these three groups are magnetically inequivalent indicating that they are asymmetrically bonded to the metal atom. Particularly remarkable in the ¹H-NMR spectrum is the difference in the chemical shifts of the *meso*-H-atoms: two have "normal" values of *ca* 4 ppm whereas one experiences a high field shift to 0.22 ppm. We assign this signal to the *meso*-H-atom which lies above the fluorenyl plane (H-21, see crystal structure). The allylic H-atoms of the fluorenyl group give rise to a singlet at 6.14 ppm (9') and a doublet at 4.35 ppm (1') while the remaining fluorenyl H-atoms are assigned to four doublets and three triplets lying between 8.35 and 7.05 ppm.

CRYSTAL STRUCTURE DETERMINATIONS

The molecular structure of three substitutedindenyl molybdenum derivatives viz. $(\eta^{5}-2$ -methylindenyl)Mo $(\eta^{3}-C_{3}H_{5})_{2}$ (I), $(\eta^{5}-2$ -methoxyindenyl) Mo $(\eta^{3}-C_{3}H_{5})_{2}$ (II) and $(\eta^{5}-5,6$ -dimethoxyindenyl)Mo $(\eta^{3}-C_{3}H_{5})_{2}$ (III), as well as of (fluorenyl)Mo $(\eta^{3}-C_{3}H_{5})_{3}$ (IV) have been determined by single crystal X-ray diffraction methods and are shown in Figs 1 and 2. Selected bond distances and bond angles and interplanar angles are summarized in Tables 1 and 2.

The molybdenum atom in the three substitutedindenyl derivatives is bonded to the five membered ring in an η^{5} -manner. In the case of the 5,6-dimethoxyindenyl derivative, the unit cell contains two



Fig. 1. The molecular structures of $[(\eta^{5}-2\text{-methylindenyl})Mo(\eta^{3}-C_{3}H_{5})_{2}]$ (I), $[(\eta^{5}-2\text{-methoxy-indenyl})Mo(\eta^{3}-C_{3}H_{5})_{2}]$ (II) and $[(\eta^{5}-5,6\text{-dimethoxyindenyl})Mo(\eta^{3}-C_{3}H_{5})_{2}]$ (III).

independent molecules but the poor quality of the data (Table 2) precludes any further discussion. The structure of the 2-methylindenyl and 2-methoxyindenyl derivatives are similar to that observed for $(\eta^5-C_5H_5)Mo(\eta^3-C_3H_5)_2^2$ with the metal lying in a trigonal planar environment. The allyl groups are arranged in a non-parallel, *cis*-configuration with the *meso*-C-atoms directed away from the metal.



Fig. 2. The molecular structure of [(fluorenyl)Mo(η^3 -C₃H₅)₃] (IV).

Table 2. Angles between planes

	I	II	Ι	II	IV
allyl(a)/Cp	72.2	75.5	79.6	94.9	58.8
allyl(b)/Cp	76.1	76.4	91.5	79.3	52.2
allyl(c)/Cp		_	_	_	32.0
allyl(a)/allyl(b)	25.7	28.2	14.1	18.9	36.0
allyl(a)/allyl(c)					44.9
allyl(b)/allyl(c)		—	_	—	62.0

allyl(a): C14,C15,C16; allyl(b): C17,C18,C19; allyl(c): C20,C21,C22; Cp: C1--C5.

The angles between the allyl planes (25.7° for I, 28.2° for II) are similar to that observed for $(\eta^5 - C_5H_5)Mo(\eta^3 - C_3H_5)_2$ (25.6°).

The four organic ligands in (fluorenyl) $Mo(\eta^3-C_3H_5)_3$ are arranged in an essentially tetrahedral manner around the central metal atom. The fluorenyl group is bonded asymmetrically to molybdenum [Mo-C(4) 2.342(2), Mo-C(3) 2.611(2) and Mo-C(10) 2.856(3)] indicating considerable rehybridization towards an η^1 -allyl situation.

 Γ_a ble 3. Analytical data for the Cp'Mo(η^3 -C₃H₅)₂ compounds and related systems

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EXPERIMENTAL

All experiments were carried out under argon using anhydrous and oxygen-free solvents. $(\eta^3 C_{3}H_{5}$ MoCl was prepared according to Benn et $al.^3$ The following compounds were prepared by published procedures: MeC₅H₄Na⁷ i- $C_3H_7C_5H_4Na$,⁷ $t-C_4H_9C_5H_4Na$,⁸ Me_5C_5Tl ,⁹ indenylNa,¹⁰ 2-Me-indenylLi,¹¹ 2-MeO-indenylLi,¹² 2-Ph-indenylLi,¹³ 5,6-dimethoxyindenylLi¹⁴ and fluorenylK.¹⁵¹H NMR spectra were recorded on a Brucker WH 400 instrument and mass spectra using a Varian CH 5 with ³⁸Mo as the standard. Microanalyses were carried out by Dornis and Kolbe, Microanalytical Laboratory, Mülheim a. d. Ruhr.

$[(\eta^{5}-t-C_{4}H_{9}C_{5}H_{4})Mo(\eta^{3}-C_{3}H_{5})_{2}]$

A solution of t-butylcyclopentadienyl lithium (1.23 g, 9.6 mmol) in monoglyme (80 cm³) was adubed to a sinred suspension of $(p^3 + 5.55) \ge 500$ (2.15 g, 8.5 mmol) in THF (50 cm³) at -35° over a period of 30 min and stirred at this temperature for a further 18 h. The solvent was evaporated at room temperature and the orange-brown residue extracted with pentane. The pentane-extract was filtered through Avicel, evaporated and the solid residue recrystallized from hexane at -35° C to give the compound as red-orange crystals. Yield 1.89 g (75% theory). The analytical data is summarized in Table 3.

$[(\eta^{5}-MeC_{5}H_{4})Mo(\eta^{3}-C_{3}H_{5})_{2}]$

Prepared as above from methylcyclopentadienyl lithium in THF at -30° C and purified by vacuum sublimation (70° C/ 10^{-4} mmHg).

 $[(\eta^{5}-i-C_{3}H_{7}C_{5}H_{4})Mo(\eta^{3}-C_{7}H_{5})_{2}]$

 $[(\eta^{5}-Me_{5}C_{5})Mo(\eta^{3}-C_{3}H_{5})_{2}]$

Prepared as above from pentamethylcyclopentadienyl thallium in THF at -78 to -10° C.

 $[(\eta^{5}-indenyl)Mo(\eta^{3}-C_{3}H_{5})_{2}]$

Prepared as above from indenyl lithium or sodium in THF or either at -35° C

$[(\eta^{5}-2-methylindenyl)Mo(\eta^{3}-C_{3}H_{5})_{2}]$

Prepared as above from 2-methylindenyl lithium in THF at -78 to -30° C.

Cp'	Yield (%)	Colour (m.p. °C)	$\mu_{\mathrm{eff}}\left(\mathrm{BM}\right)$	MS (70 eV)	%Mo (calc.)	%C (calc.)	%H (calc.)
C ₅ H ₅ ^d	}	nrange(120–122dec.)					
MeC ₅ H ₄	20.4	orange(120-122dec.)	ł	259(M ⁺)	37.1(37.3)	56.2(56.0)	6.6(6.7)
i-C ₃ H ₇ C ₅ H ₄	2	range(47–49)	ł	$288(M^+ + 1)$	33.4(33.4)	59.0(58.9)	7.4(7.4)
<i>t</i> -C ₄ H ₉ C ₅ H ₄	75	red-orange(89-90)	1.6/1.5	301(M+)	31.9(32.1)	60,2(60.2)	7.9(7.7)
Me _s C _s	60	α range($86-88$)	- {	、 ~ }	30.7(30.7)	-	
Indenyl	49	wine-red(103-104)	1.7	295(M ⁺)	32.7(32.7)	61.2(61.4)	5.9(5.8)
2-Meindenyl	58	range-red(121-122)	1.6/1.6	305(M ⁺ – 4H)	31.0(31.2)	62.6(62.5)	6.2(6.2)
1-CyCH ₂ indeny ₁	59	crange-red	• {	•	$23.5(24.7)^{b}$	69.1(67.9)	7.4(7.5)
2-MeOindenyl	54	red(136-138)	1.7/1.6	$362(M^+ + C_3 H_3)$	29.2(29.7)	60.6(59.4)	6.1(5.9)
2-Phindenyl	7	red(153-163)	diamagn.	$371(M^+ - C_3 H_3)$	23.3(23.4) ^c	70.2(70.2)	6.4(6.4)
5,6-(MeO) ₂ inde ₀ yl	83(crude)	wine-red(145–148)	1.7	$351(M^+ - C_3H_s)$	24.3(24.3) ^c	61.7(60.9)	6.7(6.6)
Fluorenyl	40	purple(175–180)	diamagn.	$345(M^+ - C_3 H_{15})$	25.2(25.0) ^c	68.8(68.7)	6.4(6.3)
" See Jolly et c_{ij} .							

Calc. for (C_{1,A}H₆CH₂Cy)Mo(C₃H₅)₃ %Mo ₂2.33, %₆C 69.77, %H 7.90.

 $(\eta^{5}-\text{organyl}) (\eta^{3}-\text{C}_{3}\text{H}_{5})_{3} \text{ compound}$

 $[(\eta^{5}-2-methoxyindenyl)Mo(\eta^{3}-C_{3}H_{5})_{2}]$

Prepared as above from 2-methoxy indenyl lithium in THF at -78 to -15° C.

$[(\eta^{5}-2-phenylindenyl)Mo(C_{3}H_{5})_{3}]$

Prepared as above from 2-phenylindenyl lithium in THF at -78° C.

$[(\eta^{5}-1-\text{CyCH}_{2}indenyl)\text{Mo}(\text{C}_{3}\text{H}_{5})_{n}]$ (*n* = 2, 3)

Prepared as above from 1-CyCH₂indenyl lithium in THF at -30° C.

$[(\eta^{5}-5,6-dimethoxyindenyl)Mo(C_{3}H_{5})_{3}]$

Prepared as above from 5,6-dimethoxy indenyl lithium in THF at -78 to -25° C.

$[(\eta^{3}-1,9,10-fluorenyl)Mo(\eta^{3}-C_{3}H_{5})_{3}]$

Fluorenyl potassium (3.0 g, 14.6 mmol) was added to a stirred suspension of $(\eta^3-C_3H_5)_3MoCl$ (3.6 g, 14.3 mmol) in THF (300 cm³) at $-78^{\circ}C$ over a period of 2 h and stirred for a further 18 h. The solvent was evaporated and the dark purple residue extracted with pentane at 5°C, the pentane-extract was filtered through Avicel at 5°C, the solvent evaporated at $-35^{\circ}C$ and the residue recrystallized twice from hexane to give the compound as small purple crystals. Yield 2.2 g (40% theory). ¹H NMR (D₈-THF, 243 K, 400 MHz): δ 4.50 (m, 2), 3.45 (dd, 1a/3s), 2.74 (dd, 1s), 0.65 (dd, 3a), J (1a, 1s) ~ 0, J (2, 3a) 11.0, J (2, 1s) 8.3, J (2, 1a) 12.8, J (2, 3s) 8.3; 2.82 (dd, 6s), 2.53 (dd, 4a), 0.33 (dd, 4s), 0.22 (m, 5), -0.30 (dd, 6a), J (4s, 6s) 2.1, J (5, 6a) 12.7, J (5, 4s) 8.8, J (5, 4a) 13.0, J (5, 6s) 8.8; 4.04 (m, 8), 3.80 (dd, 7s), 2.50 (dd, 7a), 1.58 (dd, 9s), -0.52 (dd, 9a), J (7s, 9s) 2.0, J (8, 9a) 10.3, J (8, 9s) 8.1, J (8, 7a) 13.0, J (8, 7s) 8.3; 8.35 (d, 4'), 8.03 (d, 5'), 7.71 (d, 8'), 7.50 (t, 3'), 7.37 (t, 7'), 7.24 (t, 6'), 7.05 (t, 2'), 6.14 (s, 9'), 4.35 (d, 1'), J (1', 2') 7.0, J (2', 3') 7.8, J (3', 4') 7.4, J (5', 6') 7.5, J (6', 7') 7.2, J (7', 8') 7.6—numbering scheme shown below.

¹³C NMR (D₈-THF): 104.4/104.1/99.4 (2/5/8, J 160/160/158), 51.1/53.3/60.7/65.8/65.9/80.3 (1/3/4/ 6/7/9. J 158,160/158,160/160,164/154/154/156). 55.1 (9', J 164), 104.7 (1', J 160), 125.0 (10'), 122.2 (4', J 158), 123.5 (3', J 159), 124.0 (2', J 160), 136.2 (11'), 141.9 (12'), 120.0 (5', J 158), 127.8 (6', J 158), 128.6 (7', J 160), 126.0 (8', J 160), 152.7 (13') numbering scheme shown below.



	Ι	II	ш	IV
	C ₁₆ H ₁₉ Mo	C ₁₆ H ₁₉ OMo	$C_{17}H_{21}O_2Mo$	$C_{22}H_{24}Mo$
a (Å)	6.114(1)	17.771(6)	11.568(1)	15.500(2)
b	7.532(1)	9.719(2)	7.945(1)	
с	15.304(1)	8.178(3)	17.868(2)	
α (°)	99.74(1)	90	90	111.46(1)
β	93.22(1)	90	104.02(1)	
γ	105.07(1)	90	90	
V (Å ³)	667	1412	1593	2634
$d (\mathrm{g} \mathrm{cm}^{-3})$	1.53	1.52	1.47	1.45
μ (Mo) (cm ⁻¹)	9.36	8.92	8.02	7.26
Ζ	2	4	4	6
Space group	ΡĪ	$Pna2_1$	P21	R3
Independent reflections	2999	4066	4904	4824
Observed reflections	2770	3280	3331	3590
Number of variables	230	163	300	304
R	0.026	0.028	0.063	0.023
R _w	0.035	0.032	0.074	0.023

Table 4. Crystal data

Crystal structure determination

A detailed description of the experimental procedure has been reported previously.¹⁶ Crystal data for I-IV are summarized in Table 4.

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