will be presented in a future publication.

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Registry No. BH_4^-, 16971-29-2; $B_2H_7^-$, 27380-11-6; BEt_3H^-, 75338-98-6; BH_3CN^-, 33195-00-5; BH_3, 13283-31-3; B_2H_6, 19287-45-7; BEt_3, 97-94-9; BH_2CN, 31139-16-9.

Department of Chemistry
Purdue University
West Lafayette, Indiana 47907

Derek B. Workman
Robert R. Squires*

Received November 18, 1987

First Example of a Mixed-Spin Trinuclear Manganese(II) Complex: $[MnN_6(S = {}^1/_2)][MnO_6(S = {}^5/_2)][MnN_6(S = {}^1/_2)]$

Sir:

Polynuclear manganese complexes in N,O coordination are of current interest.[1] Species of this family are believed to be active in photosystem II.[2] Herein we report the first example of a novel situation: a trinuclear N,O-coordinated complex in which manganese(II) is discretely present in both low-spin ($S = {}^1/_2$) and high-spin ($S = {}^5/_2$) configurations.[3]

The concerned complex is Mn_3L_6 in which (phenylazo)benzaldoxime, PhN=NC(=NOH)Ph (HL), acts as the ligand in the deprotonated form, L^-. Synthesis and reactions are stated in Scheme I. The complex Mn_3L_6 obtained directly from Mn^{2+} and HL occurs as a dark solid[4a] (green in solution) and is quantitatively cleaved by 2 equiv of KOH. One equivalent of the manganese is precipitated,[5] as hydroxide and the other 2 equiv remains in

Scheme I

A. Synthesis :

$$3Mn(ClO_4)_2 + 6HL \xrightarrow{(i)} Mn_3L_6 + 6HClO_4$$

B. Cleavage :

$$Mn_3L_6 + 2KOH \xrightarrow{(ii)} 2K[MnL_3] + Mn(OH)_2$$

C. Reassembly :

$$2K[MnL_3] + Mn(ClO_4)_2 \xrightarrow{(iii)} Mn_3L_6 + 2KClO_4$$

(i) 3 moles KOH, MeOH, stir. (ii) 15:1 Me_2CO-H_2O, warm, stir. (iii) MeOH, boil.

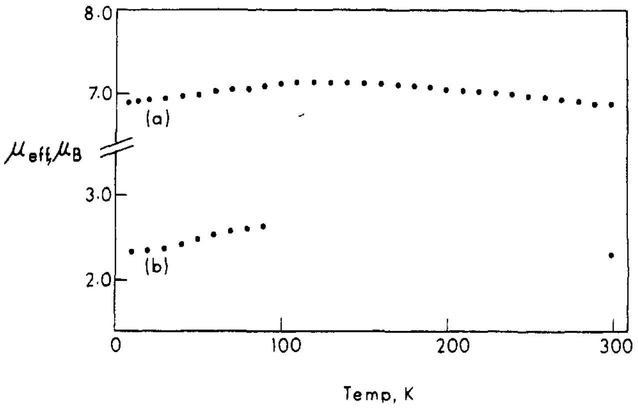

Figure 1. Variable-temperature magnetic moments of (a) Mn_3L_6 and (b) $[Ph_4As][MnL_3]$.

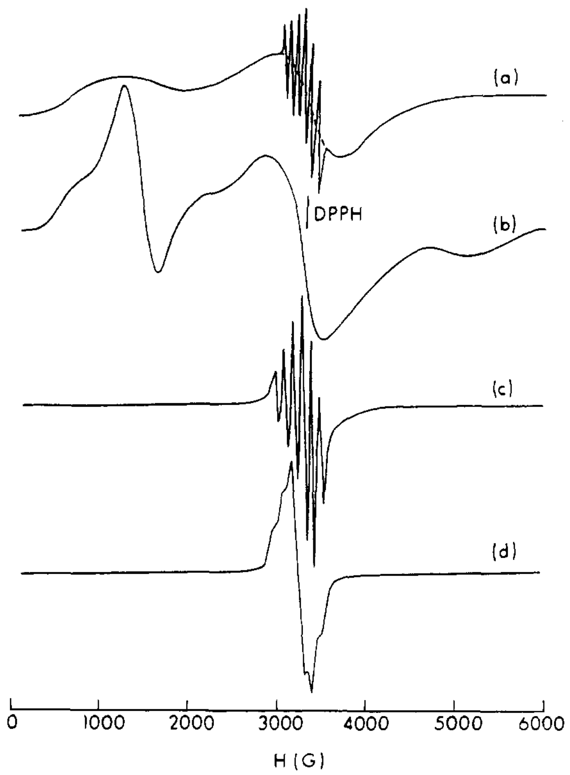

Figure 2. EPR spectra (X-band, 9.13 GHz) at 298 K: (a) Mn_3L_6 in dichloromethane; (b) Mn_3L_6 in polycrystalline form; (c) $[Ph_4As][MnL_3]$ doped (1%) into $[Ph_4As][FeL_3]$ in polycrystalline form; (d) $[Ph_4As][MnL_3]$ in polycrystalline form.

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(4) (a) Anal. Calcd for $Mn_3C_{78}H_{60}N_{18}O_6$ (Mn_3L_6): Mn, 10.92; C, 62.03; H, 3.98; N, 16.70. Found: Mn, 10.96; C, 62.10; H, 4.00; N, 16.50. (b) Anal. Calcd for $KMnC_{39}H_{30}N_9O_3$ ($KMnL_3$): Mn, 7.17; C, 61.09; H, 3.92; N, 16.45. Found: Mn, 7.09; C, 60.91; H, 4.10; N, 16.60. (c) Anal. Calcd for $MnC_{63}H_{50}N_9O_3As$ (Ph_4AsMnL_3): Mn, 4.95; C, 68.09; H, 4.50; N, 11.35. Found: Mn, 4.83; C, 68.00; H, 4.60; N, 11.10.

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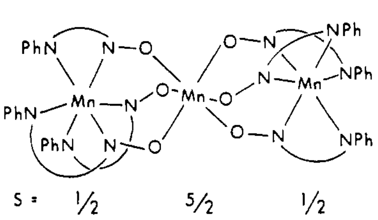
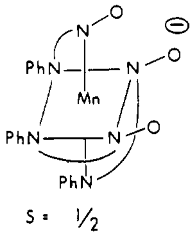
solution as MnL_3^-, which can be isolated as $K[MnL_3]$.[4b] The trinuclear complex can be quantitatively reassembled from MnL_3^- and Mn^{2+} (2:1). The salt $K[MnL_3]$ can be converted to $[Ph_4As][MnL_3]$[4c] with the help of Ph_4AsCl in aqueous acetone.

The osmometric molecular weight of Mn_3L_6 in dichloromethane is 1495 ± 20 (calculated 1509). In spite of our best attempts, we have not succeeded in growing X-ray-quality single crystals of Mn_3L_6 and salts of MnL_3^- so far. However, magnetic, spectroscopic, and electrochemical properties (some will be enumerated below) of the species have led us to suggest the provisional structures **1** and **2**. Structure **1** is an analogue of that of the iron complex $Fe_3L_6^+$, which has been X-ray-characterized along with a related complex.[6a,b] The MnL_3^- ion (**2**) is assigned facial coordination at azo and oxime nitrogen atoms as in FeL_3^-.[6a,7] In Mn_3L_6 (**1**) six oximato oxygen atoms of two *fac*-MnL_3^- units sequester the central manganese(II) atom in O_6 environment. The removal (by OH^-) and reinsertion of this atom provide the key to the understanding of cleavage and reassembly reactions of Scheme I.



1



2

In the temperature interval 10–90 K the magnetic moment of $[Ph_4As][MnL_3]$ slowly increases from 2.3 to 2.6 μ_B; at 300 K the moment is 2.30 μ_B (Figure 1). The presence of a significant temperature-dependent orbital contribution to the magnetic moment is normal for octahedral low-spin $3d^5$ complexes.[8] Due to high pairing energy, low-spin grossly octahedral complexes of manganese(II) are rare.[9] The salts of MnL_3^- are noteworthy in this respect.

By cocrystallization $[Ph_4As][MnL_3]$ can be grown freely in the lattice of $[Ph_4As][FeL_3]$, in which the geometry of the anion is definitely facial.[6a,7] That genuine lattice incorporation of manganese(II) takes place is revealed by the well-resolved ^{55}Mn hyperfine structure ($g = 2.047$; $A = 100$ G) in EPR spectra (Figure 2). In pure $[Ph_4As][MnL_3]$, the resonance is broad ($g = 2.022$) and hyperfine splitting is ill-defined. In dichloromethane solution the spectrum ($g = 2.041$; $A = 100$ G) is entirely analogous to that of $[Ph_4As][MnL_3]$ doped into $[Ph_4As][FeL_3]$. The IR spectrum of $[Ph_4As][MnL_3]$ has a 1:1 correspondence with that of $[Ph_4As][FeL_3]$.[10] Clearly, MnL_3^- is a structural analogue of FeL_3^-.

There is a similar 1:1 correspondence in the IR spectra[10] of Mn_3L_6 and $MnFe_2L_6$, which is an authentic trinuclear complex of structural type **1** with terminal manganese(II) atoms replaced by low-spin iron(II) atoms.[6b] The complexes display a structured feature (absent in MnL_3^-) at 390 cm^{-1} assignable to the Mn–O stretch[11] of the central MnO_6 sphere. The two complexes also have cognate electrochemistry.[12] Both display a quasi-reversible one-electron cyclic voltammetric oxidation of the central manganese atom to the trivalent state. A pair of quasi-reversible one-electron reductions are also seen in each case on the negative side of the SCE. The $E_{1/2}$ values in volts vs SCE and peak-to-peak separations in millivolts (given in parentheses) for the three responses are as follows. (i) Mn_3L_6: 0.51 (120); −0.20 (120), −0.39 (100). (ii) $MnFe_2L_6$: 0.56 (110); −0.79 (100), −0.90 (80). A pair of reduction waves corresponding to successive electron addition to the ligands (azo function) attached to the two terminal metal atoms is diagnostic of trinucleation.[6] In the mononuclear species a single reduction is observed: MnL_3^-, −0.90 V; FeL_3^-, −1.20 V.

Table I. Magnetic Moment (μ_B) Data

temp, K	$[Ph_4As]$-$[MnL_3]$[a]	Mn_3L_6[a,b]	temp, K	$[Ph_4As]$-$[MnL_3]$[a]	Mn_3L_6[a,b]
10	2.31	6.90 (6.83)	69	2.56	7.04 (7.01)
29	2.33	6.93 (6.84)	90	2.63	7.09 (7.06)
51	2.47	6.99 (6.94)	300	2.30	6.95 (6.83)

[a] Moment = $2.84(\chi_M T)^{1/2}$, where χ_M is the experimental molar susceptibility after diamagnetic correction. [b] Values in parentheses are calculated with the help of the following relation: moment = $(2x^2 + 6^2)^{1/2}$, where x is the experimental moment (column 2) of $[Ph_4As]$-$[MnL_3]$.

In the temperature interval 10–300 K the effective magnetic moment of Mn_3L_6 varies in the range 6.9–7.2 μ_B (Figure 1). Assuming 6.0 μ_B (temperature independent) to be the moment of the central metal atom ($S = {}^5/_2$), the moments of Mn_3L_6 at different temperatures were calculated according to the procedure stated in Table I. Selected values are set out in the same table. The agreement between calculated and experimental values is generally satisfactory, showing that Mn_3L_6 contains one high-spin and two low-spin metal atoms. The slight temperature dependence of the moment is primarily due to the corresponding dependence of the MnL_3^- units. There is no evidence of any large magnetic exchange interaction within Mn_3L_6 down to 10 K.

Ideally, the MnO_6 octahedron in Mn_3L_6 (**1**) should have 3-fold axial symmetry. Two allowed transitions near 1100 and 3200 G in X-band EPR spectra are characteristic of $S = {}^5/_2$ ions in strongly axial geometry.[13] Since these resonances originate in different Kramers doublets, they should in principle be observable in both solid and fluid media. A solution of Mn_3L_6 in dichloromethane indeed displays two broad EPR features at 298 K (Figure 2) in the axial regions. A six-line ^{55}Mn hyperfine structure ($g = 2.035$; $A = 80$ G) due to the terminal low-spin ions lies over the feature at 3200 G.[14,15] In the microcrystalline phase the hyperfine structure is no longer resolved but the low-field resonance becomes sharper and is centred at 1440 G. Several weak resonances (800, 2120, and 5080 G) are also present (Figure 2). It is estimated that D, the axial coefficient of the spin Hamiltonian,

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(5) The precipitate was quantitatively estimated as MnO_4^- after dissolving it in 2 N sulfuric acid followed by oxidation with $NaBiO_3$. Mn: calcd, 3.64% (one-third of Mn_3L_6); found, 3.84%.

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(10) Spectra were run in the region 350–4000 cm^{-1} in a KBr disk. The region up to 1600 cm^{-1}, where most of the bands occur, is displayed in supplementary material (Figures 3 and 4).

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(12) Cyclic voltammetry and coulometry were performed in dichloromethane (0.1 M in TEAP), with a platinum working electrode and an SCE reference electrode (298 K).

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(14) We have also measured the solution (dichloromethane) EPR spectrum of $MnFe_2L_6$[6b] under the same conditions as Mn_3L_6. Two broad resonances are again seen at ~1200 and ~3200 G. However, no hyperfine structure is observable on the latter resonance in this case.

(15) A reviewer asked whether the hyperfine-split spectrum could be due to a monomeric impurity rather than Mn_3L_6. The lines are too intense to arise from an impurity. The spectra are invariant from preparation to preparation. Molecular weight data are in disagreement with the presence of large concentrations of supposed "MnL_2". On the other hand, all evidence strongly supports the trinuclear formulation. If "free" MnL_3^- is considered to be the monomeric impurity, it will need a cation for charge balance. Molecular weight as well as analytical data disprove this possibility.

lies somewhat below 0.2 cm^{-1} and a small rhombic component is present.[13] The resonance positions of Figure 2b–d remain unaffected upon cooling the sample to 77 K.

Currently, we are engaged in a search for other examples of low-spin mononuclear and mixed-spin polynuclear manganese(II) complexes. The possible transformation of the latter into reactive mixed-valence species via chemical and/or electrochemical electron transfer is also under scrutiny. The $Mn_3L_6^+$ species observed cyclic voltammetrically is however too unstable to be isolated as salts.

Acknowledgment. We are very grateful to Professor W. E. Hatfield for some of the magnetic measurements, to Dr. L. R. Falvello for making crystallographic examinations of several preparations, and to Dr. R. N. Mukherjee for some preliminary experiments. Financial assistance received from the Department of Science and Technology and Council of Scientific and Industrial Research, New Delhi, India, is gratefully acknowledged.

Registry No. Mn_3L_6, 114201-32-0; $K[MnL_3]$, 114220-78-9; $[Ph_4As][MnL_3]$, 114201-34-2; $[Ph_4As][FeL_3]$, 114201-36-4.

Supplementary Material Available: Figures comparing the IR spectra (KBr disk) of $[Ph_4As][FeL_3]$ and $[Ph_4As][MnL_3]$ (Figure 3) and of $MnFe_2L_6$ and Mn_3L_6 (Figure 4) in the region 350–1600 cm^{-1} (3 pages). Ordering information is given on any current masthead page.

Department of Inorganic Chemistry
Indian Association for the Cultivation
of Science
Calcutta 700032, India

Partha Basu
Samudranil Pal
Animesh Chakravorty*

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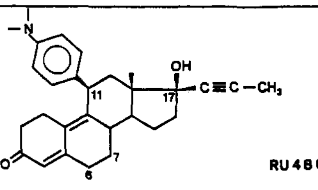
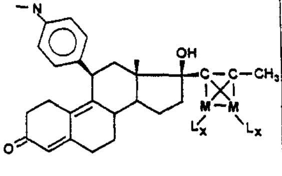
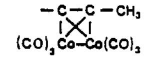
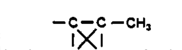
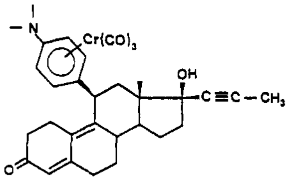
Transition-Metal Carbonyl Complexes in Progesterone Receptor Assay

Sir:

Current biochemical steroid hormone receptor assays, particularly assays for estradiol and progesterone receptors, are important in therapy[1,2] but remain expensive, are difficult to perform, and require radiolabeled ligands.[3] An alternative receptor assay would ideally involve: a sensitivity comparable to that of radioisotopic detection methods, together with a lower cost; a marker with an indefinite shelf life exempt from any legal restrictions or licensing requirements.

A receptor assay based on metal carbonyl complexes $[M_x(CO)_y]$ as markers and FT-IR spectroscopy as the detection method may fulfill very satisfactorily the above criteria.[4] Metal carbonyl complexes exhibit strong infrared absorption bands in the ν(CO) region between 2200 and 1800 cm^{-1}, a region normally devoid of strong absorption by other species, and thus can be detected by FT-IR spectroscopy in very low concentrations. In this paper, we will demonstrate for the first time the utilization of metal carbonyl moieties as markers in progesterone receptor assay.

Table I. Relative Binding Affinities (RBA) of RU 486 and RU 486 Complexes for the Cytosol Progestin (PR) and Glucocorticoid (GR) Receptors[a]

		RBA	
		PR	GR
 1 RU486		389	251
	 $\begin{array}{c} -C-C-CH_3 \\ (CO)_3Co-Co(CO)_3 \end{array}$ 2	13	17
	 $\begin{array}{c} -C-C-CH_3 \\ (CO)_2Cp\text{-}Mo\text{-}Mo\text{-}Cp(CO)_2 \end{array}$ 3	10	8
 4		20	29

[a] The RBA values for progestin (PR) and glucocorticoid (GR) receptors were measured in a routine screening assay as previously described.[22,23] Rabbit uterus and $[^3H]$R 5020 were used for PR; rat thymus and $[^3H]$dexamethasone were used for GR. Cytosol was incubated for 24 h at 0 °C. The RBA value of progesterone for PR and that of dexamethasone for GR were taken to be equal to 100.

While therapeutically useful synthetic antiestrogens, antiandrogens, antiglucocorticoids, and antimineralocorticoids have been available for some time,[5-8] only quite recently has a promising new synthetic antiprogestin called mifepristone (**1**, RU 486 [17β-hydroxy-11β-(4-(dimethylamino)phenyl)-17α-(1-propynyl)estra-4,9-dien-3-one]; see Table I) been developed.[9,10] This compound has high affinity for both the progesterone (PR) and the glucocorticoid (GR) receptor and has potent antiprogestin and antiglucocorticoid activity in vitro and in vivo.[11-13] Furthermore, mifepristone (**1**) appears to be a good candidate for transition-metal carbonyl labeling owing to the existence of several potential sites for complexation. The mapping of PR binding sites suggests close and rigid contact between the receptor and both the steroid A-ring and its "C_4, C_7, C_{14}" surface and a more labile

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