

Os(OH₂)₃]³⁺, 93255-66-4; [(trpy)Os(OH₂)₃]²⁺, 93255-67-5; [(trpy)Os(O)₂(OH₂)₂]²⁺, 104033-95-6; [(trpy)Os(OH₂)₂(OH)]²⁺, 104051-36-7; [(trpy)Os(O)₂(OH)]⁺, 93255-65-3; [(trpy)Os(OH)₂(OH₂)⁺, 104033-96-7; [(trpy)Os(OH)(OH₂)₂]⁺, 93255-69-7; (trpy)Os(OH)₃, 93255-68-6; [(trpy)Os(OH)₃]⁻, 93279-99-3; (trpy)Os(O)₂(OH), 93255-70-0; [(phen)Os(O)₂(OH₂)₂]²⁺, 104033-97-8; [(phen)Os(OH₂)₄]³⁺, 104033-

98-9; [(phen)Os(OH₂)₄]²⁺, 104033-99-0; [(phen)Os(OH)(OH₂)₃]²⁺, 104034-00-6; (phen)Os(O)(OH)₂(OH), 104034-01-7; [(phen)Os(OH)₂(OH₂)₂]⁺, 104034-02-8; (phen)Os(O)₂(OH)₂, 69531-97-1; [(phen)Os(OH)₃(OH₂)⁻, 104034-03-9; [(phen)Os(O)(OH₂)₃]²⁺, 104034-04-0; [(trpy)(bpy)Os(O)]²⁺, 89463-60-5; [(trpy)(bpy)Os(OH)]²⁺, 89463-59-2.

Notes

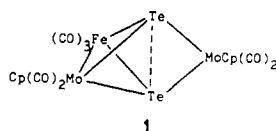
Contribution from the School of Chemical Sciences, University of Illinois, Urbana, Illinois 61801, and Department of Chemistry, University of Auckland, Auckland, New Zealand

Synthesis of Acetylene Adducts of an Iron-Molybdenum Cluster through Trapping Experiments on (C₂H₅)₂Mo₂FeTe₂(CO)_x: Conversion of arachno-(C₂H₅)₂Mo₂FeTe₂(CO)₇ to closo-(C₂H₅)₂Mo₂FeTe₂(CO)₃(RC₂H)

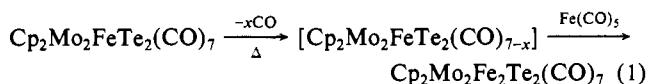
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In this note we describe an unusual substitution process for the recently prepared carbonyl cluster Cp₂Mo₂FeTe₂(CO)₇ (**1**), Cp = η⁵-C₅H₅.¹ In our previous report we showed that the ther-



molysis of **1** in toluene solutions gives a number of Fe-Mo-Te-containing clusters in low yields. Importantly, when this thermolysis was conducted in the presence of Fe(CO)₅ or CpCo(CO)₂, good yields of Mo₂FeMTe₂ clusters (M = Fe or Co) were obtained, indicating that **1** is converted to a metastable intermediate that could be trapped with the added organometallic reagents. Labeling studies showed that the Cp₂Mo₂Fe portion of **1** remains intact throughout this cluster-building process. It is therefore very likely that the thermal activation of **1** simply involves decarbonylation; intermediates of the formula Cp₂Mo₂FeTe₂(CO)₆ or Cp₂Mo₂FeTe₂(CO)₅ would be formed in this way (eq 1). It is

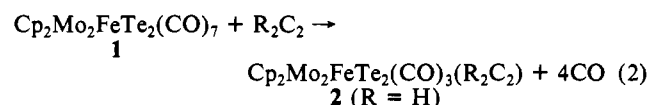


interesting that the intermediate derived from **1** is unstable since it should be able to accommodate its coordinative unsaturation through the formation of additional metal-metal bonds resulting in nido or closo clusters. In this report we describe the results of our attempts to obtain further insight into this intermediate by trapping it with other reagents. These experiments provide an example where four carbon monoxide groups are displaced by a single ligand resulting in an iron-molybdenum-acetylene complex.²

Results and Discussion

Synthesis. We began this study by searching for ligands that would form adducts of the metastable intermediates thermally generated from **1**. When solutions of **1** were heated at 110 °C in the presence of H₂, N₂, azobenzene, or ethylene, the product distribution appeared to be independent of the substrate and only

a small quantity of the products were soluble in organic solvents. However, when solutions of **1** were treated with certain acetylenes, we were able to isolate compounds of the formula Cp₂Mo₂FeTe₂(CO)₃(C₂R₂) (**2** for R = H). Such compounds could be prepared with acetylene or phenylacetylene but not with diphenylacetylene (eq 2). In this regard the intermediate resembles



Cp₂Mo₂(CO)₄, which reacts more readily with electron-rich acetylenes than with Ph₂C₂.³

The new compounds were obtained as black-green materials that are soluble in organic solvents, giving air-sensitive green solutions. They were formulated on the basis of microanalysis, mass spectrometry, and conventional spectroscopic data. The proton-coupled ¹³C NMR spectrum of Cp₂Mo₂FeTe₂(CO)₃(C₂H₂) showed that the acetylene moiety was intact since we observed both |¹J(¹³C, ¹H)| (212 Hz) and |²J(¹³C, ¹H)| (8 Hz). The observed value for ¹J(C,H) is intermediate between that for C₂H₂ (249 Hz) and C₂H₄ (156 Hz) and is consistent with a dimetallatetrahedrane structure.⁴ The ¹H NMR spectrum of (MeCp)₂Mo₂FeTe₂(CO)₃(PhC₂H) provided further information on the symmetry of the molecule: the methyl groups are equivalent but the MeCp signals appeared as several multiplets, indicating that the MeCp centroids do not lie on a symmetry plane in this derivative.

We have previously shown that ¹²⁵Te NMR was a sensitive probe for geometry of the M₃Te₂ core.⁵ These previous measurements were however obtained only for nido or arachno clusters, as there existed no examples of closo M₃Te₂ clusters. The ¹²⁵Te chemical shift for **2** was found to be 931 ppm downfield with respect to Me₂Te. This shift is in the range found for the nido clusters. In contrast, δ_{Te} for **1** occurs at 1100 ppm *upfield* of the same standard.¹ This indicates that δ_{Te} for M₃Te₂ clusters is not directly influenced by the metal-metal bonding within the M₃ subunit. In fact the unusually high-field position of δ_{Te} for **1** may be a consequence of the magnetic anisotropy effects arising from the close Te...Te contact of 3.14 Å.⁶

The compound Cp₂Mo₂FeTe₂(CO)₃C₂H₂ was studied by cyclic voltammetry in THF solution and found to have two reduction waves at -1.27 (i_a/i_c = 1.02, ΔE_p = 92 mV) and at -1.89 V (i_a/i_c = 2.77, ΔE_p = 95 mV) vs. Ag/AgCl.

Structure of (C₂H₅)₂Mo₂FeTe₂(CO)₃C₂H₂ (2**).** In the lattice, the Mo₂FeTe₂ core is of approximate C_{2v} symmetry (Figures 1

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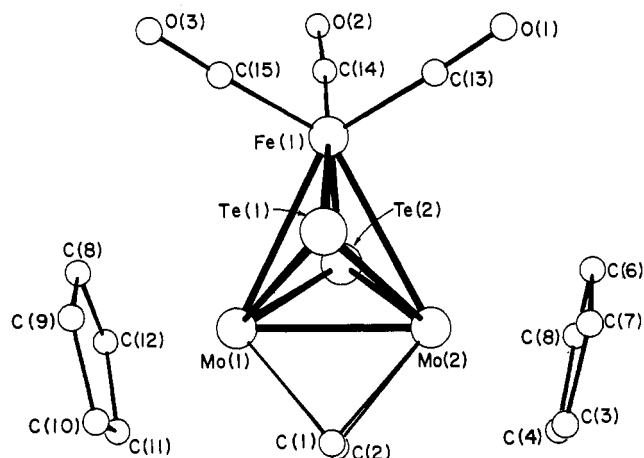


Figure 1. Structure of the non-hydrogen atoms in $\text{Cp}_2\text{Mo}_2\text{FeTe}_2(\text{CO})_3(\text{C}_2\text{H}_2)$.

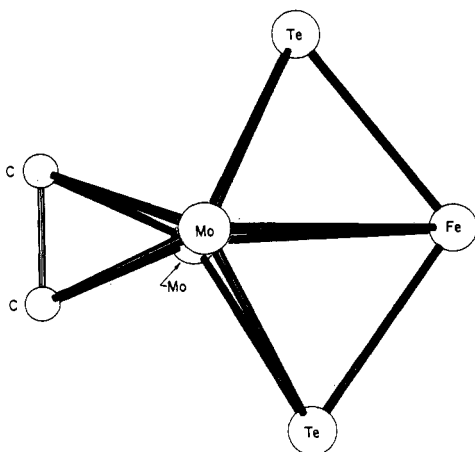
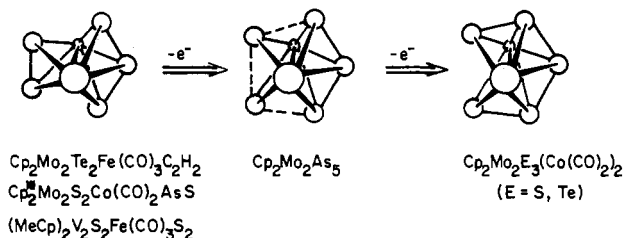


Figure 2. Side-on view of the $\text{Mo}_2\text{FeTe}_2\text{C}_2$ core in **2**.

and **2**). The most important aspects of the structure are that it is indeed closo and that the acetylene is symmetrically bridging the two molybdenum centers. Selected bond distance and angles are summarized in Table I; the following discussion focuses on the structural characteristics of the cluster core.

Geometrically, **2** closely resembles the isoelectronic $(\text{MeCp})_2\text{V}_2\text{S}_2\text{Fe}(\text{CO})_3(\text{S}_2)^7$ and Wachter's $\text{Cp}^*\text{Mo}_2\text{S}_2\text{Co}(\text{CO})_2(\text{AsS})^8$ ($\text{Cp}^* = \text{C}_5\text{Me}_5$). These are members of a growing family of compounds of the type $\text{Cp}_2\text{M}_2(\mu\text{-X})_5$; other recent examples are $\text{Cp}_2\text{Mo}_2\text{As}_5^9$ and $\text{Cp}_2\text{Mo}_2\text{E}_3(\text{Co}(\text{CO})_2)_2$ ($\text{E} = \text{S},^{10} \text{Te}^{11}$). This relationship is shown below, where the central, mutually bonded atoms are molybdenum or vanadium.



The Fe-Mo distances of 2.917 (2) and 2.954 (2) Å in **2** are long in comparison with those of **1**, where the bonding Mo-Fe

Table I. Selected Structural Parameters for $(\text{C}_5\text{H}_5)_2\text{Mo}_2\text{FeTe}_2(\text{CO})_3\text{C}_2\text{H}_2$

bond distances, Å		bond angles, deg	
Mo(1)-Mo(2)	2.651 (1)	Mo(1)-Mo(2)-Fe	62.5 (0)
Mo(1)-Fe	2.917 (2)	Mo(2)-Fe-Mo(1)	53.7 (0)
Mo(2)-Fe	2.954 (2)	Fe-Mo(1)-Mo(2)	63.9 (0)
Te(1)···Te(2)	3.794	Te(1)-Mo(1)-Te(2)	100.3 (0)
		Te(1)-Mo(2)-Te(2)	100.4 (0)
		Te(1)-Fe-Te(2)	105.8 (1)
Mo(1)-Te(1)	2.677 (1)		
Mo(1)-Te(2)	2.654 (1)	Mo(1)-Te(1)-Mo(2)	59.4 (0)
Mo(2)-Te(1)	2.674 (1)	Mo(1)-Te(2)-Mo(2)	60.0 (0)
Mo(2)-Te(2)	2.649 (1)	Mo(1)-Te(1)-Fe	67.6 (0)
Fe(1)-Te(1)	2.567 (2)	Mo(1)-Te(2)-Fe	68.0 (0)
Fe(1)-Te(2)	2.562 (2)		
C(1)-C(2)	1.356 (15)	Mo(1)-C(1)-Mo(2)	75.6 (4)
C(1)-Mo(1)	2.172 (11)	Mo(1)-C(2)-Mo(2)	75.5 (4)
C(1)-Mo(2)	2.155 (11)		
		C(13)-Fe-C(15)	109.1 (5)
		C(14)-Fe-C(15)	90.7 (6)

Table II. Te···Te Distances and ^{125}Te Chemical Shifts for Metal Carbonyl Telluride Clusters

cluster formula	description	Te···Te, Å	$\delta(\text{Te})^a$
$[\text{CpMoFeTe}_2(\text{CO})_5]\text{SbF}_6^b$	tetrahdrene	unknown ^b	-839
$\text{Cp}_2\text{Mo}_2\text{FeTe}_2(\text{CO})_7$	arachno	3.134 (1)- 3.150 (1)	-1092
$\text{Fe}_3\text{Te}_2(\text{CO})_9\text{PPh}_3$	arachno	3.138 (1)	-887, -938
$\text{Fe}_3\text{Te}_2(\text{CO})_7(\text{PPh}_3)_2^c$	nido	3.305 (1)	1123
$\text{Cp}_2\text{Mo}_2\text{FeTe}_2(\text{CO})_3(\text{C}_2\text{H}_2)$	closo	3.794 (1)	931

^a Upfield shifts (vs. neat Me_2Te) are negative. ^b The Te-Te distances for the bromide and dithiocarbamate derivatives are 2.81 and 2.95 Å, respectively: Bogan, L. E., Jr.; Rauchfuss, T. B.; Rheingold, A. L. *Inorg. Chem.* **1985**, *24*, 3722. ^c Day, V. W.; Lesch, D. A.; Rauchfuss, T. B., unpublished results (see also: Lesch, D. A. Ph.D. Thesis, University of Illinois at Urbana-Champaign, 1983).

distances are in the range of 2.828 (1)-2.850 (1) Å for the four crystallographically independent molecules.¹ The Mo-Mo distance of 2.651 (1) Å in **2** is intermediate between that of 2.448 (1) Å in $\text{Cp}_2\text{Mo}_2(\text{CO})_4^{12}$ and 2.980 (1) Å in $\text{Cp}_2\text{Mo}_2(\text{CO})_4\text{C}_2\text{H}_2$. Because of the large size of tellurium, the M-Te-M angles are acute, particularly so (59-60°) across the Mo(1)-Mo(2) vector. The Te(1)···Te(2) distance of 3.794 Å is much longer than other *noncloso* $\text{M}_3(\mu_3\text{-Te})_2$ structures (Table II). The long Te(1)-Te(2) distance is a direct structural consequence of the bonded Mo_2Fe triangle.

The acetylenic C-C distances of 1.356 (15) Å is elongated relative to that of C_2H_2 (1.21 Å) but is comparable to the C-C distances in free C_2H_4 (1.34 Å)¹³ and $\text{Cp}_2\text{Mo}_2(\text{CO})_4\text{C}_2\text{H}_2$ (1.337 (5) Å).³

Conclusions. This project was undertaken in order to shed some light on the unusually reactive species formed when **1** is decarbonylated thermally. The activation of the Mo_2FeTe_2 core by decarbonylation contrasts with the behavior of $\text{Fe}_3\text{Te}_2(\text{CO})_{10}$. The arachno-nido interconversion of $\text{Fe}_3\text{Te}_2(\text{CO})_{10,9}$ is facile, clean, and reversible.¹⁰

The conversion of **1** to **2** does conform with the mechanism proposed for the assembly of $\text{Cp}_2\text{Mo}_2\text{Fe}_2\text{S}_2(\text{CO})_8$ and $\text{Cp}_2\text{Mo}_2\text{Fe}_2\text{Te}_2(\text{CO})_7$.¹ An early stage in this mechanism is proposed to involve the formation of a cluster of the type $\text{Cp}_2\text{Mo}_2\text{Fe}_2(\text{CO})_5$. Such a species has a valence electron count appropriate for a closo cluster¹¹ but is susceptible to attack by an acetylene or a coordinatively unsaturated metal carbonyl.¹ This line of reasoning leads one to inquire as to why the compounds

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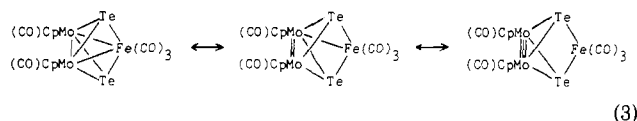
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Table III. Crystal Data and Refinement Parameters

formula	C ₁₅ H ₁₂ FeMo ₂ O ₃ Te ₂
cryst syst	monoclinic
space group	P2 ₁ /n
a, Å	7.137 (1)
b, Å	24.012 (3)
c, Å	10.457 (3)
α, deg	90
β, deg	92.43 (2)
γ, deg	90
V, Å ³	1790.44
Z	4
ρ _{calcd} , ρ _{obsd} , g cm ⁻³	2.76, 2.86
cryst dims, mm	0.04 × 0.1 × 0.12
radiation	Mo Kα
diffractometer	CAD-4
abs coeff, cm ⁻¹	53.4
max, min transmission coeff	0.7087, 0.5305
2θ scan range, deg	2.25
no. of unique data	1988 (I > 3σ(I))
no. of data processed	2682
R _F	0.0315
R _{wF}	0.0341

Cp₂Mo₂FeE₂(CO)_x (x = 6 or 5; E = S, Te) should be so reactive. An intriguing possibility is that such species contain a reactive multiple bond between the two molybdenum centers, i.e. eq 3.



Experimental Section

Materials and Methods. The general procedures followed those described in the previous paper.¹ Reagent grade chemicals were obtained commercially. Electrochemical measurements were made by using a BAS-100 instrument and a three-electrode cell with a Pt working electrode. For these measurements 10⁻³ M THF solutions of the compound were prepared in a 0.1 M Bu₄NClO₄ solution in THF. The data reported here were obtained at a scan rate of 50 mV/s.

Reactions of (RCp)₂Mo₂FeTe₂(CO)₇ with Acetylenes. In a typical experiment Cp₂Mo₂FeTe₂(CO)₇ (0.301 g, 0.363 mmol) was heated at reflux in toluene (125 mL) under a vigorous C₂H₂ purge for 1 h. The filtered reaction mixture was reduced to one-sixth volume and diluted with an equal volume of hexane. Flash chromatography (silica gel with 7:3 CH₂Cl₂-hexane) of this solution yielded a yellow-green band of the acetylene complex followed by a green band (1). The product, 65 mg, was obtained as microcrystals by precipitation with hexane. At ~50% conversion, the yields were ~25%; longer reaction times lead to lower yields. Compound 1 did not react with acetylene even when treated with anhydrous Me₃NO in refluxing THF. Anal. Calcd for C₁₅H₁₂FeMo₂O₃Te₂: C, 24.24; H, 1.63; Fe, 7.51. Found: C, 24.01; H, 1.69; Fe, 7.73. FABMS: m/e 745 (M⁺), 719, 691, 661, 634, 613, 602, 585, 557, 520. IR (c-C₆H₁₂ solution): 2000 s, 1952 sh, 1946 m cm⁻¹. ¹³C NMR (CDCl₃ solution): δ 218.4, 91.6 (¹J(¹H,¹³C) = 212 Hz, ²J(¹H,¹³C) = 8 Hz), 47.5 (¹J(¹H,¹³C) ≈ 212 Hz, ²J(¹H,¹³C) = 8 Hz). ¹H NMR (CDCl₃ solution): δ 5.16 s (10 H), 4.37 s (2 H).

The compound Cp₂Mo₂FeTe₂(CO)₃(PhCCH) was prepared similarly from 1. Anal. Calcd for C₂₁H₁₆FeMo₂O₃Te₂: C, 30.79; H, 1.97; Fe, 6.78; Te, 31.15. Found: C, 30.57; H, 2.08; Fe 6.97; Te, 29.3. FABMS: m/e 819 (M⁺), 791, 673, 735, 665, 633. IR (c-C₆H₁₂ solution): 2002 s, 1954 m, 1948 m cm⁻¹. ¹H NMR (CDCl₃ solution): δ 7.31 and 6.84 m (5 H, 5.14 s (10 H), 4.62 s (1 H)).

The MeCp-PhC₂H compound was prepared analogously but was an oil and was not obtained in analytically pure form. ¹H NMR (CDCl₃ solution): δ 7.19-6.97 m (5 H); 4.97 m, 4.91 m, 4.83 m, and 4.73 m (8 H); 4.45 s (1 H); 1.90 s (6 H).

X-ray Crystallography. Preliminary X-ray photography of the black rhombic crystals showed monoclinic diffraction symmetry. The space group P2₁/n was assigned on the basis of systematic absences for the 0k0 (k = 2n + 1) and h0l (h + l = 2n + 1) reflections. A suitable crystal was epoxied to a thin glass fiber and positioned on a Nonius CAD-4 diffractometer. Unit cell dimensions were derived from a least-squares fit to the setting angles of 25 reflections, obtained by using Zr-filtered Mo Kα radiation (λ = 0.7107 Å) (Table III). Intensity data were collected by using a variable-width, variable-speed 2θ/ω scan. There was no significant decay during the data collection, as revealed by monitoring

Table IV. Atomic Positions

	x	y	z
Te(1)	-0.0927 (1)	0.10762 (3)	0.6075 (1)
Te(2)	0.4315 (1)	0.17689 (3)	0.6271 (1)
Mo(1)	0.2249 (1)	0.10841 (4)	0.4803 (1)
Mo(2)	0.2396 (1)	0.09566 (4)	0.7324 (1)
Fe	0.0818 (2)	0.2009 (1)	0.6286 (1)
O(1)	-0.1168 (14)	0.2178 (4)	0.8634 (8)
O(2)	0.2414 (12)	0.3131 (4)	0.6313 (8)
O(3)	-0.164 (1)	0.2359 (5)	0.4154 (9)
C(1)	0.2309 (14)	0.0312 (4)	0.5888 (10)
C(2)	0.406 (2)	0.0533 (5)	0.5932 (10)
C(3)	0.239 (4)	0.0262 (6)	0.8849 (1)
C(4)	0.421 (3)	0.0484 (7)	0.8883 (13)
C(5)	0.404 (3)	0.1043 (7)	0.9305 (12)
C(6)	0.220 (3)	0.1162 (8)	0.9512 (12)
C(7)	0.112 (3)	0.0657 (10)	0.9258 (14)
C(8)	0.184 (2)	0.1505 (5)	0.2778 (10)
C(9)	0.071 (2)	0.1030 (6)	0.2773 (11)
C(10)	0.193 (2)	0.0564 (5)	0.2923 (10)
C(11)	0.384 (2)	0.0770 (6)	0.3036 (11)
C(12)	0.373 (2)	0.1347 (6)	0.2943 (12)
C(13)	-0.038 (2)	0.2100 (5)	0.7728 (11)
C(14)	0.186 (2)	0.2679 (5)	0.6338 (11)
C(15)	-0.068 (2)	0.2211 (5)	0.4971 (11)

three standard reflections for every one hundred data. Absorption corrections were applied after carefully measuring and indexing each of the ten crystal faces. Fourteen reflections were struck from the data set because of extremely uneven background counts.

The structure was readily solved by locating a trigonal-bipyramidal array of five heavy atoms on the E map with the use of SHELX. Subsequent difference Fourier maps revealed the positions of the remaining eighteen nonhydrogen atoms (Table IV). No attempts were made to refine the hydrogen atom positions, although the acetylenic hydrogens were apparent in the final difference Fourier map.

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Registry No. 1, 94820-11-8; 2, 104198-45-0; Cp₂Mo₂FeTe₂(CO)₃-(PhC₂H), 104198-46-1; C₂H₂, 74-86-2; PhC₂H, 536-74-3; (MeCp)₂Mo₂FeTe₂(CO)₇, 94820-12-9; (MeCp)₂Mo₂FeTe₂(CO)₃-(PhC₂H), 104198-47-2.

Supplementary Material Available: A table of thermal parameters (1 page); a table of structure factors (9 pages). Ordering information is given on any current masthead page.

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Chelation of *cis*-Halo(oxalato)tetraamminechromium(III) Complexes: Evidence for Direct Displacement of a Coordinated Anion by Another

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The factors controlling substitution reactions of octahedral complexes have been extensively investigated and reviewed.^{1,2}

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