


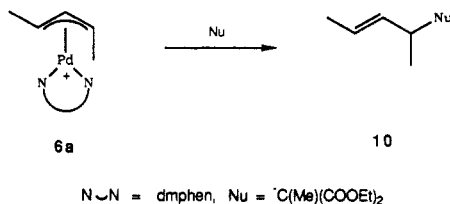


Table I. Stoichiometric and Catalytic Allylic Substitutions Using 2,9-Dimethyl-1,10-phenanthroline Palladium Complexes

starting material (% isom purity)	nucleophile	products, %		
				
1s ^a (95)	⁻ C(Me)(COOEt) ₂	94	1	5
1a ^a (95)		7	37	56
1s ^b (92)	NHMe ₂	70	10	20
1a ^c (95)		6	12	82
(E)-MeCH=CH- CH ₂ OAc ^d (98)	⁻ C(Me)(COOEt) ₂	95	1	4
(Z)-MeCH=CH- CH ₂ OAc ^d (53)		45	21	34

^a Stoichiometric reaction in DMF, counterion fluoroborate, argon atmosphere, 0 °C, 2 equiv of sodium diethyl methylmalonate. ^b Stoichiometric reaction in H₂O, counterion trifluoroacetate, 0 °C, 0.1 mmol/mL, 5 equiv of Me₂NH, 5 equiv of NaCN added after 30 s, 45% reacted. ^c As above; 35% reacted. ^d Catalytic reaction in DMF, argon atmosphere, 20 °C, 1 h, 0.2 mmol/mL substrate, 1% Pd catalyst (1), 4% dmphen, 2 equiv of sodium diethyl methylmalonate.

diethyl ether and rapid crystallization immediately after preparation while the anti complex could be crystallized from the equilibrium mixture. The same anti/syn equilibrium ratio was observed for all terminally monosubstituted allylic groups, indicating that the destabilization of the syn configuration by dmphen is general.⁵ In order to determine the chemical consequences of the stereochemistry of the η³-allyl group, essentially pure (~95%) syn and anti complexes, 1s and 1a, were reacted separately with the anion of diethyl methylmalonate. The syn complex gave essentially pure E product 7, and the anti complex gave the Z product 8 together with the regioisomer 9 in the ratio 37/56 (Table I). Essentially the same product pattern was observed when the hexenyl complex 3 was used, suggesting that this is a general behavior of complexes of monosubstituted allyl groups. We also prepared the complex [Pd(dmphen)(MeCH=CH=CHMe)]⁺BF₄⁻ (6), which gave an 83/17 equilibrium mixture of syn,anti (6a) and syn,syn (6s) isomers, respectively. When this was reacted with the anion of methylmalonate, only the E product 10 could be detected, confirming a higher relative reactivity at the anti substituted carbon. The effect of a different nucleophile on the



product pattern was studied by reacting the crotyl complexes 1a and 1s with dimethylamine. In order to minimize isomerization of the primary products,^{6c} the reaction was performed in water solution at 0 °C by using the trifluoroacetate salts of 1a and 1s and quenching the reaction after 30 s with an excess of sodium cyanide. Despite appreciable isomerization also under these conditions, the trends from addition of malonate were confirmed, i.e., retention of E and Z stereochemistry and preference for reaction at the secondary position in the case of the anti isomer 1a. The latter result could be a problem in catalytic reactions, but it obviously represents a novel concept for regiocontrol.

Although pyridine type ligands are generally not useful in catalytic reactions,⁶ a few alkylations of (Z)- and (E)-2-butenyl acetates were performed, with a palladium dmphen as catalyst.

(5) Unsubstituted 1,10-phenanthroline gave an equilibrium amount of ~10% anti isomer.

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Unexpectedly, both complete retention of configuration (Table I) and faster reaction than with Pd(PPh₃)₄ were observed, perhaps due to more rapid oxidative addition.

These results show that the initial concept of stereocontrol is correct. However, truly catalytic conversion of E substrates into Z products requires further studies of ways to influence the relative rates of isomerization and nucleophilic attack. Such studies are under way in our laboratories.

Acknowledgment. We thank the Swedish Board for Technical Development, the Swedish National Science Research Council, and the National Research Council of Italy (CNR) for financial support.

Supplementary Material Available: Elemental analyses and NMR spectral data of compounds 1a and 1s (1 page). Ordering information is given on any current masthead page.

[(MeC₅H₄)₃U]₂[μ-1,4-N₂C₆H₄]: A Bimetallic Molecule with Antiferromagnetic Coupling between the Uranium Centers

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Antiferromagnetic coupling of electron spins on two or more metal centers that are connected by bridging ligands, referred to as indirect coupling or superexchange coupling, is a topic of considerable interest in d-transition-metal chemistry.¹ In the f-block metals, a few examples of antiferromagnetic exchange coupling have been observed in solid-state compounds with extended lattices such as UCl₃^{2a,b} and EuCl₂,^{2c} where the ordering temperatures are ca. 22 K and 1.6 K, respectively. Antiferromagnetic coupling has been observed in a molecular lanthanide system, Cp₄Dy₂(μ-Br)₂, with an ordering temperature of 6 K, though no coupling was observed in Cp₄Er₂(μ-Br)₂, Cp₄Yb₂(μ-Br)₂,^{3a-d} or [(Me₅C₅)₂Yb]₂(μ-E) where E is O, S, Se, or Te.^{3e} Recently, inelastic neutron scattering experiments have been interpreted in terms of electron exchange coupling in the facial bioctahedral compounds Cs₃M₂Br₉ where M is Yb, Tb, or Ho.^{3f,g} In the actinide series, no examples of coupling have been documented in molecular systems; the U(IV) compounds [(MeC₅H₄)₃U]₂(μ-E)^{4a} and [(Me₃Si)₂N]₃U₂(μ-E)^{4b} where E is

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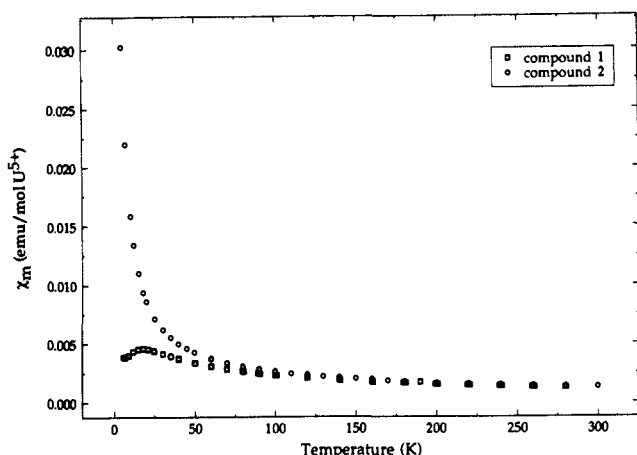


Figure 1. Experimental magnetic susceptibility data of **1** and **2** as a function of temperature.

Table I. Magnetic Susceptibility Values for Uranium(V) Organoimides

compd	5–40 K		140–280 K	
	μ_{eff}^a	θ^b	μ_{eff}^a	θ^b
$\text{Cp}_3\text{UNSiMe}_3$	1.19	-0.7	1.83	-82
$(\text{MeC}_5\text{H}_4)_3\text{UNPh}$	1.25	1.03	1.96	-110
2	1.30	-3.95	2.12	-134
1	coupled		2.08	-147

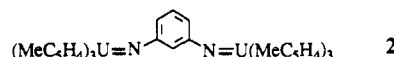
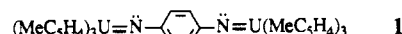
^a $\chi_M(\text{corr}) = C/(T - \theta)$. The effective magnetic moment, μ_{eff} , is calculated as $\mu_{\text{eff}} = 2.828C^{1/2}$, where C and θ , the Curie and Weiss constants, respectively, are obtained by fitting the magnetic susceptibility data to the equation $\chi_M(\text{corr}) = C(T - \theta)^{-1}$. Moments are expressed in Bohr magnetons per U(V). The values reported were determined at 5 kG; the values at 40 kG were identical to within 2%. The $\chi_M(\text{corr})$ values are corrected for container and sample diamagnetism. ^b In Kelvins.

S, Se, or Te and where the U–E–U angles are nearly linear show no coupling to 5 K.

A good synthetic route to higher valent uranium compounds, particularly for synthesis of U(V) organoimides, has been discovered recently, as shown in eq 1.⁵ Extending this reaction to $(\text{RC}_5\text{H}_4)_3\text{U}(\text{thf}) + \text{R}'\text{N}_3 \rightarrow (\text{RC}_5\text{H}_4)_3\text{UNR}' + \text{N}_2 + \text{thf}$ (1)

diazidobenzene derivatives gives **1**⁶ or **2**.⁶ The bimetallic, pen-

taivalent uranium derivatives of the 5f¹ electron configuration show antiferromagnetic coupling in **1** and the lack of coupling in **2**. The



plot of χ_M vs T is shown in Figure 1 for both derivatives, and the values of the magnetic moments are listed in Table I for these and related mononuclear organoimides of U(V). The similarity of the curves for **1** and **2** is obvious from 50 to 300 K, as is the difference from 5 to 50 K, the difference being that the spins on the two U(V) centers are antiferromagnetically coupled in **1** with an ordering temperature of ~ 20 K and the two U(V) centers in **2** behave as independent paramagnets to 5 K.⁷

Magnetic susceptibility and electron paramagnetic resonance (EPR) measurements have been carried out on a number of mononuclear, pentavalent uranium compounds of the type $(\text{MeC}_5\text{H}_4)_3\text{UNR}$. The magnetic susceptibility curves as a function of temperature are all very similar, Table I, Figure 1, but no EPR spectra at ~ 4 K have been observed.

In the magnetic measurements where no coupling is observed, there are two distinct temperature regions with different slopes in the $(1/\chi_M)$ vs T plots: a low-temperature region from 5 to ~ 40 K and a higher temperature region from 140 to 300 K. For a 5f¹ electron system, a plausible model that explains this behavior is an isolated crystal field ground state and an excited crystal field state that becomes populated at the higher temperatures which then contributes to the total magnetization. At low temperatures, the magnetism can be attributed primarily to the population of the ground crystal field state.^{2a,b} Since no major differences are observed in the magnetic susceptibility per U(V) between monomeric compounds of the type $(\text{MeC}_5\text{H}_4)_3\text{UNR}$ and the bimetallic molecule **2**, the bimetallic compound **2** can be considered as the sum of two $(\text{MeC}_5\text{H}_4)_3\text{U}$ -imide units.

The local symmetry about the U(V) center in these compounds is approximately C_{3v} . The ground term for a U(V) 5f¹ ion is a $^2F_{5/2}$. Under C_{3v} symmetry, the $J = 5/2$ state splits into three magnetic doublets, two $\mu = \pm 1/2$ states and one $\mu = \pm 3/2$ state where μ is the crystal quantum number.⁸ The $\mu = \pm 1/2$ states should show a normal g_{\parallel}, g_{\perp} EPR spectrum as the selection rule $\Delta J_z = \pm 1$ is valid when these doublets are split by a magnetic field. For the $\mu = \pm 3/2$ states, $\Delta J_z > 1$, and g_{\perp} is necessarily 0. Thus a normal EPR spectrum is not expected if the crystal quantum number of the ground crystal field state is $\mu = \pm 3/2$, even when the doublet is split by a magnetic field. The absence of an EPR spectrum is indicative though not proof of a $\mu = \pm 3/2$ ground state. This argument is valid even when the crystal field is large enough to substantially mix the higher lying $J = 7/2$ crystal field levels into the ground state.⁹

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 (6) (a) $[(\text{MeC}_5\text{H}_4)_3\text{U}]_2[\mu\text{-}1,4\text{-N}_2\text{C}_6\text{H}_4]$, (**1**). To 0.68 g of $(\text{MeC}_5\text{H}_4)_3\text{U}$ (thf) (1.2 mmol) dissolved in 30 mL of diethyl ether was added 0.10 g of 1,4-diazidobenzene^{6c} (0.62 mmol) in 10 mL of diethyl ether. Gas was evolved immediately, and the color of the solution changed from red to deep purple with formation of a dark precipitate. After stirring for 15 min, the volatile material was removed under reduced pressure, leaving a dark solid. This solid was extracted into toluene (60 mL), the purple solution was filtered, and the filtrate was concentrated to 40–45 mL and cooled to -20 °C. Purple-black flakes were isolated by filtration and dried under reduced pressure, yielding 0.19 g of product. Concentrating the remaining solution to ca. 15 mL and cooling to -20 °C allowed isolation of an additional 0.12 g of product. Total yield was 48% (0.31 g), mp 261–263 °C. ¹H NMR (C_6D_6 , 30 °C): δ 4.69 (9 H, $\nu_{1/2} = 6$ Hz), -2.46 (6 H, $\nu_{1/2} = 23$ Hz), -9.01 (6 H, $\nu_{1/2} = 17$ Hz), 15.11 (4 H, $\nu_{1/2} = 14$ Hz). Anal. Calcd for $\text{C}_{42}\text{H}_{46}\text{N}_2\text{U}_2$: C, 47.8; H, 4.40; N, 2.66. Found: C, 48.1; H, 4.54; N, 2.64. Mass spectrum: 1054, 1055 (calcd relative intensity, observed relative intensity; 100, 100; 47, 36). IR: 1578 w, 1493 m, 1283 m, 1260 m, 1090 w, 1047 w, 1032 m, 928 w, 903 w, 849 m, 833 m, 764 s, 610 m, 592 m, 538 w cm^{-1} . (b) $[(\text{MeC}_5\text{H}_4)_3\text{U}]_2[\mu\text{-}1,3\text{-N}_2\text{C}_6\text{H}_4]$, (**2**). This compound was prepared similarly from $(\text{MeC}_5\text{H}_4)_3\text{U}(\text{thf})$ and 1,3-diazidobenzene^{6d} and isolated as brown-red needles from toluene in 37% yield, mp 213–215 °C. ¹H NMR (C_6D_6 , 59 °C): 4.38 (18 H, $\nu_{1/2} = 17$ Hz), -2.27 (12 H, $\nu_{1/2} = 62$ Hz), -9.22 (12 H, $\nu_{1/2} = 55$ Hz), 28.23 (1 H, $\nu_{1/2} = 25$ Hz), -0.46 (1 H, $\nu_{1/2} = 32$ Hz), -6.89 (2 H, $\nu_{1/2} = 32$ Hz). The NMR spectrum was recorded at 59 °C because the peak at -0.46 ppm was too broad to be observed at room temperature. Anal. Calcd for $\text{C}_{42}\text{H}_{46}\text{N}_2\text{U}_2$: C, 47.8; H, 4.40; N, 2.66. Found: C, 47.5; H, 4.41; N, 2.63. Mass spectrum: 1054 (observed by FAB MS using 18-crown-6 and tetraglyme). IR: 1545 m, 1490 w, 1292 w, 1250 m, 1200 m, 1148 m, 1048 w, 1029 m, 990 m, 861 w, 854 w, 842 m, 797 m, 765 s, 682 m, 604 w, 330 w cm^{-1} . (c) Herring, D. L. *J. Org. Chem.* **1961**, *26*, 3998. (d) Foster, M. O.; Fierz, H. E. *J. Chem. Soc.* **1907**, *91*, 1942.

(9) This argument can be given in another way. From group theory, an ion with a $J = 5/2$ free ion state placed in a crystal at a site of C_{3v} symmetry decomposes into two Γ_4 states (which are each doubly degenerate) and one degenerate doublet ($\Gamma_5 + \Gamma_6$).¹⁰ The Zeeman operator ($L_x + 2S_x$) transforms as Γ_3 under C_{3v} symmetry. The direct product $\Gamma_4 \times \Gamma_3 \times \Gamma_4$ contains Γ_1 , so an EPR spectrum is expected for a Γ_4 ground state. The direct product (Γ_5 or Γ_6) \times Γ_3 (Γ_5 or Γ_6) does not contain Γ_1 , so $g_{\perp} = 0$ if the degenerate doublet (Γ_5 or Γ_6) is the ground state. Since the transition probability for the usual EPR transition is proportional to the square of the matrix element of the $(L_x + 2S_x)$, (g_{\perp}^2), no EPR spectrum is observed.¹¹ The crystal field Hamiltonian can only mix crystal field states of the same symmetry (but differing in J value), so the above proof is valid even if there is appreciable mixing of the ground $J = 5/2$ and the excited $J = 7/2$ states by a strong crystal field.

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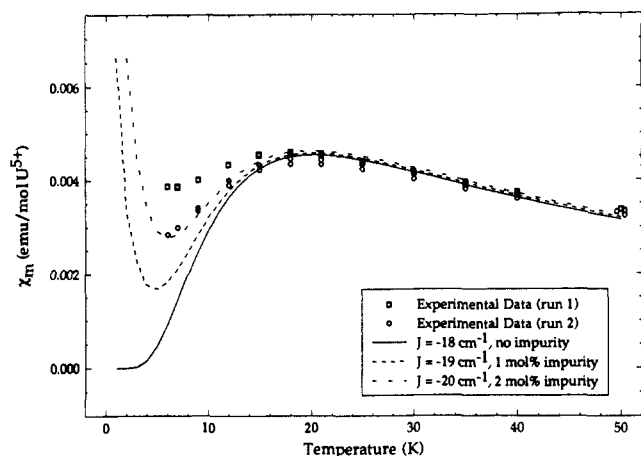


Figure 2. Comparison of experimental magnetic susceptibility data with calculated values for **1**. The calculations are with $g_{\perp} = 2.6$. The impurity is assumed to be $(\text{MeC}_5\text{H}_4)_3\text{U}(\text{thf})$, and three calculated curves are shown for $J = -18 \text{ cm}^{-1}$ (no impurity), $J = -19 \text{ cm}^{-1}$ (1 mol % impurity), and $J = -20 \text{ cm}^{-1}$ (2 mol % impurity).

The bimetallic molecule **1** can be considered to be two monomeric $(\text{MeC}_5\text{H}_4)\text{U}$ units that are connected by the diimide through which an intramolecular antiferromagnetic exchange takes place. Because of the steric bulk of the $(\text{MeC}_5\text{H}_4)_3\text{U}$ unit, intermolecular exchange is negligible, as shown by the data for **2**. The magnetism at low temperature in **1** and **2** is attributed to the population of the ground crystal field state only. Thus the Curie constant obtained from the plot of $1/\chi_M$ vs T at low temperature for **2** is directly related to the average g value. Since $g_{\perp} = 0$ with the assignment of the ground state as $\mu = \pm^3/2$, $g_{\parallel} = 2.6$ for the $\mu = \pm^3/2$ ground crystal field state of the $\text{U}(\text{V})$ ion. It is assumed that the ground state for **1** is the same as that for **2** in the absence of electron exchange, and that there is a one-dimensional exchange interaction along the 3-fold or z axis for **1** between the two U centers. With the second assumption, the exchange interaction in **1** may be treated with the model used for an isolated dimer.^{1f,12} The Hamiltonian for such a dimer is written as

$$\mathcal{H} = -2JS_{z1}S_{z2} + g_{\perp}\mu_B H_z(S_{z1} + S_{z2}) \quad (2)$$

where S_{z1} and S_{z2} are effective spin $1/2$ operators, J is the exchange constant, and μ_B is the Bohr magneton. The magnetic susceptibility for a randomly oriented powder (per uranium ion) is (with $g_{\perp} = 0$)^{1f}

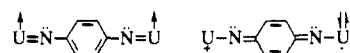
$$\chi_{\text{av}} = \frac{N g_{\parallel}^2 \mu_B^2}{2kT} (1 + e^{-J/kT})^{-1} \quad (3)$$

where N is Avogadro's number, k is the Boltzmann constant, and T is the absolute temperature. This model is applicable to **1** only for $T \lesssim 50 \text{ K}$.

The calculated susceptibilities for **1** (with $g_{\perp} = 2.6$) as a function of temperature for various values of J are shown in Figure 2. The experimental deviation from the theoretical model at low temperatures is assumed to be due to a small amount of a paramagnetic impurity as different preparations of **1** show differing susceptibilities in this temperature range. Assuming that the impurity is the starting material, $(\text{MeC}_5\text{H}_4)_3\text{U}(\text{thf})$, then ca. 1–2 mol % is sufficient to cause the observed deviations at low temperature. It is concluded that $J \sim -19 \text{ cm}^{-1}$ and the antiferromagnetic state is lowest in energy.

The observation that the spins on each uranium center of **1** antiferromagnetically couple while those on **2** do not couple to 5 K may be rationalized by a superexchange pathway. The imido nitrogens in **1** are in the 1,4-positions of the benzene ring, and they can form a conjugated ring while those on **2** cannot. It is reasonable to postulate that the spin on each uranium center can

communicate across the conjugated ligand in **1** though not in **2**. This may be illustrated by the two resonance structures shown below.



These two resonance structures imply that the two spins communicate by way of the ligand π -system and the electron on each uranium is in a π -symmetry orbital. A spin polarization model can be postulated, and this model also rationalizes the observation of antiferromagnetic coupling.^{1c,8} We know of no simple way to distinguish between these two physical processes.

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Helical and Nonhelical Palladium(II) Complexes of Oligopyridine Ligands: The Ligand-Directed Assembly of Polynuclear Complexes

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There has been considerable recent interest in the design of ligands that facilitate the spontaneous self-assembly of complexes¹ and host-guest compounds² with novel chemical, biological, or topological properties. The assembly of double-helical polynuclear complexes has been studied by several groups.^{3,4} We have investigated the ability of the oligopyridines to assemble flexible but nonlabile double-helical ligand environments with a view to

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