with continuous helium flow and thereafter introduced into a high-pressure flow-tube apparatus (ca. 0.5 Torr). The target rod is aluminum, and when the laser vaporization source is impregnated with niobium and vanadium, a distribution of bare aluminum and mixed-metal clusters is observed. The clusters are well thermalized before reacting with a neutral reagent (oxygen). Reactants and products are mass filtered and detected downstream with a quadrupole mass spectrometer and pulse counting techniques. Gases such as $\mathrm{N_2}$ have been introduced without changing the original anion distribution, confirming the expected absence of collision induced dissociation processes in the flow-tube apparatus.

A portion of a typical spectrum is shown in Figure 1a. Several series of ions are seen, including the bare aluminum clusters Al_n^- (n = 7–13) and the mixed-metal clusters VAl_n^- (n = 5–12), NbAl_n^- (n = 4–10), and VNbAl_n^- (n = 2–8). The relative amounts of the mixed clusters compared with the bare aluminum clusters can be altered by changing the laser focus, and under certain conditions an additional series $\mathrm{V_2NbAl}_n^-$ (n = 0–6) is also obtained in this mass range.

In Figure 1b, a spectrum covering the same mass range shows the results of the addition of 30.6 sccm of oxygen through the reactant gas inlet. The spectrum shows that nearly one-half of the peaks from the previous spectrum have reacted away. The reactivity pattern at this intermediate reaction condition evidently results from odd–even electron alternation and its concomitant influence on reactivity. With the exception of $\mathrm{VAl_5^-}$, each of the remaining species in Figure 1b has an even number of electrons, indicating that the pairing of electrons increases the stability of the ions, or at least decreases their reactivities.

After the addition of 60.0 sccm of oxygen, three peaks dominate the spectrum. As can be seen in Figure 1c, these are $\mathrm{Al_{13}^-}$, $\mathrm{NbAl_4^-}$, and $\mathrm{VAl_6^-}$. $\mathrm{Al_{13}^-}$ has been shown[3] to be a stable product from the reaction of bare aluminum clusters with oxygen. In the current experiment the NbAl_n^- clusters react to produce $\mathrm{NbAl_4^-}$, and the VAl_n^- clusters react to product $\mathrm{VAl_6^-}$.

An important question to which the present findings provide insight concerns the counting of the d electrons of transition metals in terms of the free-electron model. In one version where the d orbitals are considered to hybridize with the other orbitals in the cluster, all the valence electrons would participate in governing the electronic structure. In this case both vanadium and niobium would contribute five electrons to the cluster. Otherwise, the d electrons would be considered as core electrons which would have V and Nb contribute two electrons and one electron, respectively.[9] The observed odd–even alternation in reactivity is a clear indication that both atoms donate an odd number of electrons. The jellium model accounts for the special stability of $\mathrm{Al_{13}^-}$ (and $\mathrm{Al_{23}^-}$), and the stability of $\mathrm{NbAl_4^-}$ can also be explained by its electronic structure if all five electrons are available; this compound has 18 valence electrons, which is a jellium shell closing, and also has special stabilities in coordination compounds (18-electron rule). If every valence electron is counted as in the previous examples, $\mathrm{VAl_6^-}$ has 24 electrons, which is not a predicted shell closing. (Note, however, if one of vanadium's electrons is promoted by hybridization, providing one free s electron, the species then becomes a 20-electron system, and its lack of reactivity would be in accord with the jellium model.)

In conclusion, the jellium model is a good guide to the reactivity patterns and related electronic structure of metal alloy clusters, but by no means provides a compendious concept. The interaction of the electronic orbitals of aluminum with those of the transition metals may be sufficiently strong to enable all of the electrons to contribute to reactivity behavior, as evidenced by the lack of differences for systems containing $4s^2$ and $5s^1$ electrons and by the general observation that odd–even electron number accounts for the reactivity of these alloy systems. Finally, it is interesting to speculate whether a structural form of $\mathrm{VAl_5^-}$ might not involve electron hybridization and hence would behave as an 18-electron system; this may account for the lack of reactivity of this species.

Acknowledgment. Financial support by the Air Force Geophysics Laboratory, Contract No. RADC-F30602-88-D-0028, is gratefully acknowledged.

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Reversible Opening and Closing of Hetero Trimetallic Units in $\mathrm{(C_5H_5)_2Y(THF)Re_2H_7(PMe_2Ph)_4}$ and $\mathrm{(C_5H_5)_2LuRe_2H_7(PMe_2Ph)_4}$[1]

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Mixed-metal complexes[2] containing two substantially different types of metal have the potential to effect substrate transformations not achievable with either of the individual metallic components. Complexes that can accomplish such bimetallic reactions must contain some ligands that can hold the metals together during the reaction and other ligands that react with substrates. Although suitable bridging ligands have been identified for mixed-metal complexes containing f elements and transition metals,[3] these compounds generally do not contain reactive ligands that can subsequently transfer to substrate. Transition-metal polyhydride phosphine complexes[4] appeared to be an attractive set of starting materials for the synthesis of mixed-transition-metal/f-element species, since the several hydride ligands could provide the desired reactive ligands and also could comprise the necessary linkages to bridge to the lanthanide. Hydride-bridged heterometallic transition-metal complexes are known[5] as well as hydride-bridged polymetallic lanthanide and yttrium complexes including mixed-metal species such as $\{[\mathrm{Cp'_2Y}(\mu\text{-H})]_2[\mathrm{Cp'_2Zr}(\mu\text{-H})](\mu_3\text{-H})\}$ ($\mathrm{Cp'} = \mathrm{C_5H_4Me}$).[6] In addition, given parallels in lanthanide and alkali-metal chemistry[7] and the existence of complexes such as $\mathrm{KOsH_3(PMe_2Ph)_3}$[8] and $\mathrm{(THF)_2KReH_6(PPh_3)_2}$,[9] it seemed likely

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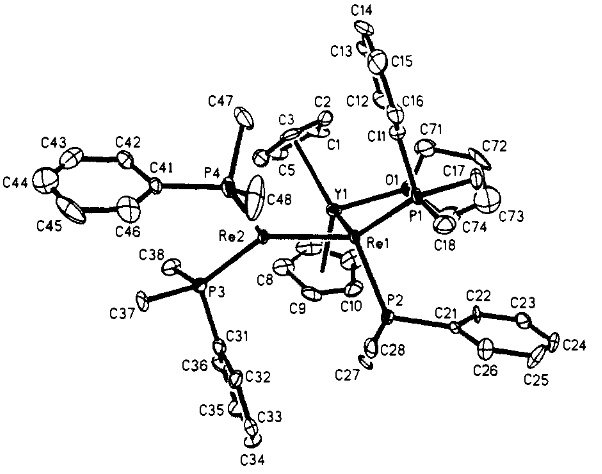


Figure 1. ORTEP drawing (30% level) of non-hydrogen atoms of $Cp_2Y(THF)Re_2H_7(PMe_2Ph)_4$. Hydride hydrogens were not located. Selected structural parameters: Re–Re, 2.576 (1) Å; Re–Y, 3.090 (2) Å; Re–P, 2.295 (4)–2.356 (4) Å; Y–O, 2.405 (11) Å. Re2–Re1–Y1, 94.8 (1)°; O1–Y1–Re1, 98.1 (3)°. (Cp midpoint)–Y–(Cp midpoint), 122.1°.

that related species containing lanthanide ions could exist. Finally, we also anticipate a synergic effect in which the electropositive lanthanide metal will enhance the electron density of the hydride ligands. We report here the successful execution of these objectives via a new synthetic method for linking lanthanides to transition metals. As a consequence, we have observed the opening and closing of a trimetal triangle as THF adds and dissociates.

$(C_5H_5)_2YMe(THF)$[10] reacts with $Re_2H_8(PMe_2Ph)_4$[11] in THF at 25 °C to generate methane (identified by ^1H NMR) and a quantitative yield of a compound characterized by NMR spectroscopy[12] and X-ray crystallography[13] as $Cp_2Y(THF)Re_2H_7(PMe_2Ph)_4$. An NMR sample of the molecule in C_6D_6 at 25 °C shows one C_5H_5 resonance, one broad hydride resonance, one phosphorus resonance, and signals for THF that are distinct from those of free THF in benzene. The solid-state structure (Figure 1) shows that the NMR results are oversimplified by some dynamic averaging phenomenon. The molecule has an open-triangular or L-shaped form with inequivalent rhenium (and thus phosphorus) centers. One rhenium atom is clearly not bonded to yttrium (Re–Y = 4.186 Å). The other Re–Y distance, 3.090 (2) Å, is reasonable for a hydride-bridged linkage (cf. the 3.664 (1) Å Y···Y distance in $[Cp'_2Y(THF)(\mu\text{-}H)]_2$).[14,15] The 2.576 (1) Å Re–Re distance is similar to the 2.538 (4) Å Re–Re distance in $Re_2H_8(PEt_2Ph)_4$,[16] but the positions of the phosphine ligands are substantially different. Although the hydrides were not located from the X-ray diffraction data, a structure in which two hydrides connect yttrium and rhenium is proposed. The presence of bridging hydride ligands is indicated by the phosphorus-decoupled ^1H NMR spectrum at 65 °C, where the hydride resonance becomes a doublet (J_{Y-H} = 7.2 Hz).

When the same synthetic and product isolation procedures are applied to the reaction of $Cp_2LuMe(THF)$ with $Re_2H_8(PMe_2Ph)_4$ in THF, the product isolated is the THF-free molecule $Cp_2LuRe_2H_7(PMe_2Ph)_4$.[17,18] The NMR patterns of this complex are analogous to those of $Cp_2Y(THF)Re_2H_7(PMe_2Ph)_4$, but the solid-state structure[18] (Figure 2) is markedly different. In this case, THF is absent and the three metals adopt a closed-triangular shape with an idealized C_{2v} structure which contains symmetry-equivalent phosphorus nuclei. While hydride ligands were not located from the X-ray data, μ_2-hydride bridges and possibly a μ_3-hydride ligand are proposed to connect lutetium to the rhenium atoms.

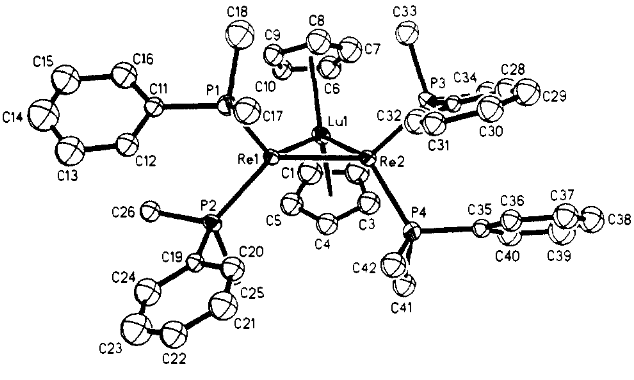
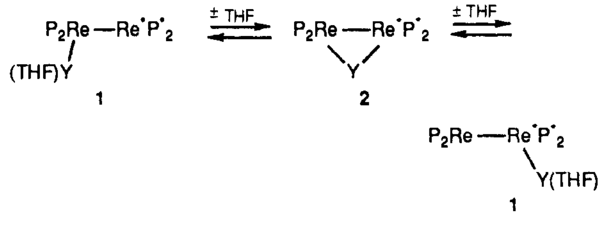


Figure 2. ORTEP drawing (40% level) of the non-hydrogen atoms of $Cp_2LuRe_2H_7(PMe_2Ph)_4$. Hydride hydrogens were not located. Selected structural parameters: Re–Re, 2.561 (1) Å; Lu1–Re1, 3.068 (1) Å; Ln1–Re2, 3.025 (1) Å; Re–P, 2.319 (4)–2.337 (5) Å. Lu1–Re1–Re2, 64.3 (1)°, (Cp, midpoint)–Lu–(Cp midpoint), 124.2 °C.

The lutetium structure provides an attractive intermediate for the fluxional process which averages the NMR signals of $Cp_2Y(THF)Re_2H_7(PMe_2Ph)_4$. One possible dynamic process, involving dissociation of THF, is shown in eq 1.[19] Consistent with this



(1)

hypothesis, the dynamic process can be slowed by lowering the temperature: the $^{31}P\{^1H\}$ NMR spectrum of $Cp_2Y(THF)Re_2H_7(PMe_2Ph)_4$ at −88 °C in 1:3 toluene–THF shows two signals for **1** and an additional singlet due to compound **2** with a 1:2 ratio of 1.[20] Equation 1 also suggests that there should be rapid exchange between free and coordinated THF. In fact, the ^1H NMR spectrum of $Cp_2Y(THF)Re_2H_7(PMe_2Ph)_4$ in THF-d_8 shows only the resonances of free OC_4H_8, i.e., coordinated OC_4H_8 has been completely displaced in the time required to record the spectrum. Finally, dissolving $Cp_2Y(THF)Re_2H_7(PMe_2Ph)_4$ in toluene, followed by removal of solvent in vacuo, yields the THF-free yttrium species $Cp_2YRe_2H_7(PMe_2Ph)_4$.[21] This complex can be readily resolvated and, hence, contains latent unsaturation.

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(13) Crystallographic data for $C_{46}H_{69}OP_4YRe_2$: a = 11.9546 (16) Å, b = 31.613 (4) Å, c = 12.571 (2) Å, β = 92.376 (11)° with Z = 4 in space group $P2_1/n$. $R(F)$ = 6.1% and $R_w(F)$ = 9.0% for 6616 absorption-corrected reflections ($F > 4\sigma(F)$).

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(17) Selected spectroscopic data for $Cp_2LuRe_2H_7(PMe_2Ph)_4$: ^1H NMR (25 °C, C_6D_6) δ 1.72 (pseudo d, J = 5.4 Hz, PMe), −8.62 (br s, ReH), 6.48 (s, LuCp); ^{31}P NMR (25 °C, C_6D_6) δ −8.77 (s, ReP); IR (Nujol mull) 1966, 1936 cm^{-1} (weak) (terminal H).

(18) Crystallographic data for $C_{42}H_{61}P_4LuRe_2$: a = 17.677 (4) Å, b = 12.960 (2) Å, c = 19.199 (3) Å, β = 90.990 (16)° with Z = 4 in space group $P2_1/c$. $R(F)$ = 4.9% and $R_w(F)$ = 5.8% for 5253 absorption-corrected reflections ($F > 4\sigma(F)$).

(19) Exchange involving intramolecular migration of PMe_2Ph is less likely since the necessary bridging phosphine is unknown. Dissociation into the solvent-separated ion pair $[Cp_2Y(THF)]^+[Re_2H_7P_4]^-$ is excluded by the retention of $^{89}Y-^1H$ coupling at 65 °C.

(20) No decoalescence is observed at −60 °C in the absence of added THF since species **1** is significantly populated only in the presence of added THF. This is consistent with the fact that the ^1H chemical shifts of **1** in toluene at 25 °C are very close to those of free THF.

(21) Selected spectroscopic data for $Cp_2YRe_2H_7(PMe_2Ph)_4$: ^1H NMR (25 °C, C_6D_6) δ 1.73 (pseudo d, J = 4.7 Hz, PMe), −8.88 (br s, ReH), 6.49 (s, YCp); ^{31}P NMR (25 °C, C_6D_6) δ −9.04 (Re–P).

In contrast to the yttrium system, in which products with and without coordinated THF are accessible, treatment of $Cp_2LuRe_2H_7(PMe_2Ph)_4$ with 1 equiv of THF in C_6D_6 gives no spectral shifts; no evidence for THF binding is observed. The marginally smaller radius of lutetium (0.848 Å vs 0.88 Å for yttrium)[22] may cause this difference in chemical behavior.[23]

These results suggest that the $Cp_2LnRe_2H_7(PMe_2Ph)_4$ complexes may be particularly useful as a class of mixed-metal species: size-selective substrate binding may be possible by proper choice of Ln. For example, substrates more slender than THF may add to the lutetium complex. More importantly, these heterometallic polyhydrides appear to be able to accommodate addition and elimination of substrates without reverting back to their homometallic constituents. For example, when THF is lost from the yttrium complex, the vacant coordination position is filled by a hydride ligand, with concomitant conversion of the open L-shaped form to a closed metal triangle. Hence, selective and truly polymetallic chemistry may be achievable with complexes of this class.

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Supplementary Material Available: ORTEP diagrams and tables of crystal data, positional parameters, bond distances and angles, and thermal parameters for $(C_5H_5)_2Y(THF)Re_2H_7(PMe_2Ph)_4$ and $(C_5H_5)_2LuRe_2H_7(PMe_2Ph)_4$ (24 pages). Ordering information is given on any current masthead page.

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Asymmetric Synthesis through the Topochemical Reaction in a Chiral Crystal of a Prochiral Diolefin Molecule Having a "Cisoid" Molecular Structure and Amplification of Asymmetry by a Seeding Procedure[1]

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Topochemically controlled reactions of organic crystals have become of interest in recent years as one means of asymmetric synthesis because these reactions proceed stereoselectively under the control of the crystal lattice. There have been a few successful examples regarding the production of optically active materials from achiral molecules by using the chiral environment of a crystal as the sole source of chirality in a photoreacting process.[2] Our earlier communication demonstrated that an alternating arrangement of both enantiomers was generated by a topochemical induction during the reaction of some prochiral molecules in achiral crystals.[3]

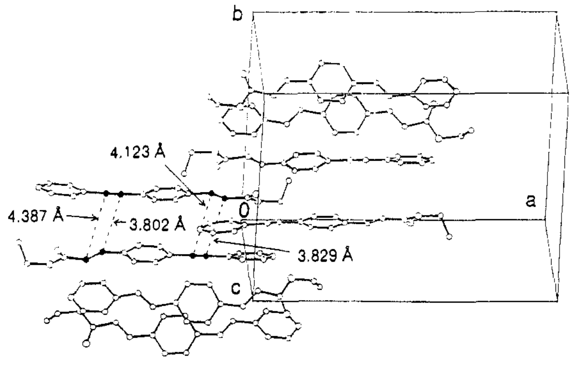
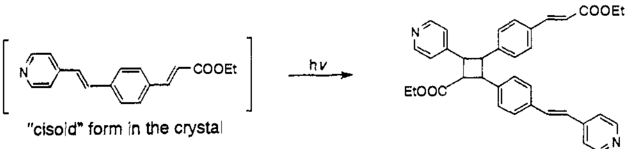


Figure 1. Crystal structure of **1**. The double bonds that may form cyclobutane are linked by the dotted lines.

Scheme I



Ethyl 4-[2-(4-pyridyl)ethenyl]cinnamate[4a] (**1**) is highly photoreactive in the crystalline state and was converted by [2 + 2] photocyclodimerization into dimer **2** in high yield (Scheme I). The cyclobutane structure of **2** was confirmed by ^1H NMR and MS spectroscopy[4b] and by topochemical considerations based on X-ray crystal structure analysis. It was proved that optically active **2** was obtained by the photoirradiation of a single crystal of **1** and, furthermore, that the amplification of asymmetry was successfully performed by seeding during the recrystallization of **1**.

Light-yellow prisms (mp 115-116 °C, 10-17 mg) were obtained from an ethanolic solution of **1** by slow evaporation. The single crystal was cut into two pieces, and one piece was then finely powdered and photoirradiated by a 500-W super-high-pressure mercury lamp for 5 h at room temperature. A cut-off filter ($\geq$365 nm) was employed for exciting only the monomer molecule in order to maximize the chemical yield of dimer **2**. After photoirradiation, **2** was purified by TLC and the sign of optical rotation confirmed. The single crystal that gave (+)-**2** or (−)-**2** was called (+)-**1** or (−)-**1**, respectively. Crystals of (+)-**1** or (−)-**1** (ca. 200 mg) were obtained from an ethanolic solution of **1** respectively by seeding with finely powdered crystals of another piece of (+)-**1** or (−)-**1** (3-6 mg). Finely powdered crystals of **1**, obtained by seeding, were dispersed in 30% aqueous $CaCl_2$ containing a few drops of surfactant (NIKKOL TL-10FF) to avoid the flotation of crystals and were irradiated, with vigorous stirring, by a mercury lamp located outside of the flask, through the same cut-off filter under a nitrogen atmosphere at −40 °C for 10 h. The enantiomeric excess of the resulting **2**, after purification by TLC, was determined by HPLC on an optically active solid phase.[5] The enantiomeric excess for (+)-**2** was 92%, $[\alpha]_D$ +72° (*c* 1.0 in CH_2Cl_2), and for (−)-**2**, 95%, $[\alpha]_D$ −75°. Although the crystals gradually became amorphous as the photoreaction proceeded, the chemical yield of **2** was more than 60%.

The photochemical behavior and asymmetric induction could be readily interpreted by an X-ray structure analysis of a crystal of **1**.[6] The crystal structure of **1** is shown in Figure 1.

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(1) The term "amplification of asymmetry by seeding" means that a large quantity of crystals that gave a dimer of the same optical rotation as that of the dimer obtained from seed crystals was attainable in a very high optical yield by seeding with a slight amount of seed crystals.

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(4) (a) The methyl derivative gave a linear polymer on photoirradiation: Hasegawa, M.; Harashina, H.; Kato, S.; Saigo, K. *Macromolecules* **1986**, *19*(4), 1276-1278. (b) ^1H NMR: δ 0.95 (t, 3 H), 1.29 (t, 3 H), 3.91 (m, 3 H), 4.21 (q, 2 H), 4.40 (q, 1 H), 4.46 (t, 1 H), 4.73 (t, 1 H), 6.30 (d, 1 H), 6.93 (d, 1 H), 6.96 (d, 2 H), 7.09 (d, 2 H), 7.18 (d, 1 H), 7.23-7.36 (m, 8 H), 7.53 (d, 1 H), 8.54-8.56 (m, 4 H); mass spectrum, *m/e* 513 (M − C_2H_5O), 381 (asymmetric cleavage of cyclobutane ring), 279 (M/2), 250 (M/2 − C_2H_5), 234 (M/2 − C_2H_5O), 206 (M/2 − $COOC_2H_5$), 177 (asymmetric cleavage of cyclobutane ring).

(5) The column used was Chiralcel OC of Daicel Chemical Industries, Ltd., Himeji, Japan.

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