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Display:

- 1, 2,5-Bis{N-(2,6-diisopropylphenyl)iminomethyl}pyrrolyl Complexes of the Heavy Alkaline Earth Metals: Synthesis, Structures, and Hydroamination Catalysis**

By Jenter, Jelena; Koeppel, Ralf; Roesky, Peter W.  
From Organometallics (2011), 30(6), 1404-1413. | Language: English, Database: CAPLUS

The heteroleptic iodo complexes [(DIP2pyr)MI(THF)<sub>n</sub>] (M = Ca, Sr (n = 3); Ba (n = 4); (DIP2pyr)<sup>-</sup> = 2,5-bis(N-(2,6-diisopropylphenyl)iminomethyl)pyrrolyl) were synthesized by reaction of [(DIP2pyr)K] with anhyd. alk. earth metal diiodides. All complexes are monomeric in the solid state. A κ<sup>3</sup>-coordination mode of the (DIP2pyr)<sup>-</sup> ligand was obsd. for the Sr and the Ba compds., while the analogous Ca deriv. is κ<sup>2</sup>-coordinated in the solid state. However, VT-1H NMR studies of [(DIP2pyr)CaI(THF)<sub>3</sub>] indicate a sym. coordinated (DIP2pyr)<sup>-</sup> ligand in soln. Computational studies confirm the different coordination modes in soln. and in the solid state. The preferred κ<sup>2</sup>-coordination mode obsd. in the solid state might be a result of temp. or/and crystal-packing effects. Also, the Ca and Sr amido complexes [(DIP2pyr)M{N(SiMe<sub>3</sub>)<sub>2</sub>}(THF)<sub>2</sub>] (M = Ca, Sr) were prepd. by reaction of [(DIP2pyr)MI(THF)<sub>n</sub>] (M = Ca, Sr (n = 3)) with [K{N(SiMe<sub>3</sub>)<sub>2</sub>}]. Both compds. were studied for the **intramol. hydroamination of aminoalkenes**. Both catalysts showed a good activity, and the best results were obtained for the Ca complex [(DIP2pyr)Ca{N(SiMe<sub>3</sub>)<sub>2</sub>}(THF)<sub>2</sub>].
- 2. Aminotroponimate zinc complexes as catalysts for the intramolecular hydroamination**

By Jenter, Jelena; Luehl, Anja; Roesky, Peter W.; Blechert, Siegfried  
From Journal of Organometallic Chemistry (2010), Volume Date2011, 696(1), 406-418. | Language: English, Database: CAPLUS

A review. This review deals with the synthesis of aminotroponimate and aminotroponate Zn complexes. The main focus is on their application as catalysts for the **intramol. hydroamination**, in which good activity, particularly high functional group tolerance and a relatively high stability towards moisture and air were obsd. A heterogeneous Zn catalyst is reported to increase the stability and the recyclability of the catalytic system.
- 3. Rare-Earth Metal Alkyl, Amido, and Cyclopentadienyl Complexes Supported by Imidazolin-2-iminato Ligands: Synthesis, Structural Characterization, and Catalytic Application**

By Trambitas, Alexandra G.; Panda, Tarun K.; Jenter, Jelena; Roesky, Peter W.; Daniliuc, Constantin; Hrib, Cristian G.; Jones, Peter G.; Tamm, Matthias  
From Inorganic Chemistry (Washington, DC, United States) (2010), 49(5), 2435-2446. | Language: English, Database: CAPLUS

The rare earth metal dichlorides [(1)MCl<sub>2</sub>(THF)<sub>3</sub>] (2a, M = Sc; 2b, M = Y; 2c, M = Lu) and the Gd complex [(1)GdCl<sub>2</sub>(THF)<sub>2</sub>].[LiCl(THF)<sub>2</sub>] (2d), contg. the 1,3-bis(2,6-diisopropylphenyl)imidazolin-2-

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1. **Dianion and Monoanion Ligation of 1,4-Diaza-1,3-butadiene to Barium, Strontium, and Calcium**

By Panda, Tarun K.; Kaneko, Hiroshi; Michel, Olaf; Pal, Kuntal; Tsurugi, Hayato; Tornroos, Karl W.; Anwender, Reiner; Mashima, Kazushi  
From *Organometallics*, Ahead of Print. | Language: English, Database: CAPLUS

Two synthetic protocols, a salt metathesis reaction and a direct metalation, were developed for prepg. 1,4-diaza-1,3-butadiene complexes of barium, strontium, and calcium, in which 1,4-diaza-1,3-butadiene serves as a dianionic or monoanionic ligand. A salt metathesis reaction of BaI<sub>2</sub> with the dipotassium salt of N,N'-bis(2,6-diisopropylphenyl)-1,4-diaza-1,3-butadiene (1; abbreviated (Dip)<sub>2</sub>DAD) afforded the iodide-bridged dinuclear complex  $[[K((Dip)_2DAD)(THF)_2][Ba(\mu-I)(THF)_2]]_2$  (2) bearing a dianionic ene-diamide ligand, while the reaction of MI<sub>2</sub> (M = Sr, Ca) with the dipotassium salt of 1 gave the mononuclear complexes  $[M((Dip)_2DAD)(THF)_4]$  (4, M = Sr; 5, M = Ca). A direct metalation reaction of barium powder with (Dip)<sub>2</sub>DAD in the presence of iodine (10 mol%) afforded an iodide-bridged dinuclear complex,  $[Ba((Dip)_2DAD)(\mu-I)(THF)_2]_2$  (3), in which (Dip)<sub>2</sub>DAD coordinates as a monoanionic ligand to the barium center, as was evident from the X-ray anal. and the EPR spectral data. The products from the direct metalation reaction of Sr and Ca powders with 1 in the presence of a catalytic amt. of iodine (1 mol%) resulted in the formation of mononuclear complexes 4 and 5 bearing the dianionic ene-diamide DAD ligand.

2. **From a Cycloheptatrienylzirconium Allyl Complex to a Cycloheptatrienylzirconium Imidazolin-2-iminato "Pogo Stick" Complex with Imido-Type Reactivity**

By Gloeckner, Andreas; Bannenberg, Thomas; Daniliuc, Constantin G.; Jones, Peter G.; Tamm, Matthias  
From *Inorganic Chemistry* (Washington, DC, United States), Ahead of Print. | Language: English, Database: CAPLUS

The reaction of the cycloheptatrienylzirconium half-sandwich complex  $[(\eta^7-C_7H_7)ZrCl(tmeda)]$  (1) (tmeda = N,N,N',N'-tetramethylethylenediamine) with Li(ImDippN), generated from bis(2,6-diisopropylphenyl)imidazolin-2-imine (ImDippNH) with methyllithium, yields the imidazolin-2-iminato complex  $[(\eta^7-C_7H_7)Zr(ImDippN)(tmeda)]$  (2). The corresponding tmeda-free complex  $[(\eta^7-C_7H_7)Zr(ImDippN)]$  (5) can be synthesized via the 1,3-bis(trimethylsilyl)allyl complex  $[(\eta^7-C_7H_7)Zr\{\eta^3-C_3H_3(TMS)_2\}(THF)]$  (3; TMS = SiMe<sub>3</sub>), which undergoes an acid-base reaction with ImDippNH to form 5 and 1,3-bis(trimethylsilyl)propene. 5 exhibits an unusual one-legged piano stool ("pogo stick") geometry with a particularly short Zr-N bond of 1.997(2) Å. Addn. of 2,6-dimethylphenyl or tert-Bu isocyanide affords the complexes  $[(\eta^7-C_7H_7)Zr(ImDippN)(CNR)]$  (R = o-Xy, 6; R = t-Bu, 7), while the reaction with 2,6-dimethylphenyl isocyanate results in a [2 + 2] cycloaddn. to form the ureato(1-) complex  $[(\eta^7-C_7H_7)Zr\{ImDippN(C=O)N-o-Xy\}]$  (8). 5 can also act as an initiator for the ring-opening polymn. of ε-caprolactone. These reactivity patterns together with d. functional theory calcns. reveal a marked similarity of the bonding in imidazolin-2-iminato and conventional imido transition-metal complexes.

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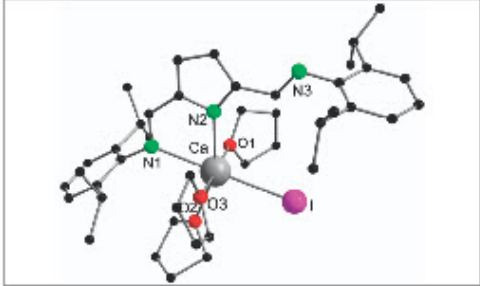
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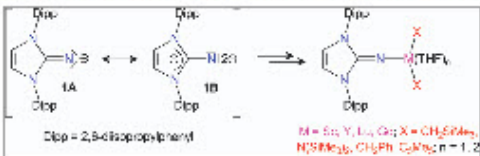
The heteroleptic iodo complexes [(DIP2pyr)MI(THF)<sub>n</sub>] (M = Ca, Sr (n = 3); Ba (n = 4); (DIP2pyr)<sup>-</sup> = 2,5-bis-[N-(2,6-diisopropylphenyl)iminomethyl]pyrrolyl) were synthesized by reaction of [(DIP2pyr)K] with anhyd. alk. earth metal diiodides. All complexes are monomeric in the solid state. A κ<sub>3</sub>-coordination mode of the (DIP2pyr)<sup>-</sup> ligand was obsd. for the Sr and the Ba compds., while the analogous Ca deriv. is κ<sub>2</sub>-coordinated in the solid state. However, VT-1H NMR studies of [(DIP2pyr)CaI(THF)<sub>3</sub>] indicate a sym. coordinated (DIP2pyr)<sup>-</sup> ligand in soln. Computational studies confirm the different coordination modes in soln. and in the solid state. The preferred κ<sub>2</sub>-coordination mode obsd. in the solid state might be a result of temp. or/and crystal-packing effects. Also, the Ca and Sr amido complexes [(DIP2pyr)M{N(SiMe<sub>3</sub>)<sub>2</sub>}(THF)<sub>2</sub>] (M = Ca, Sr) were prepd. by reaction of [(DIP2pyr)MI(THF)<sub>n</sub>] (M = Ca, Sr (n = 3)) with [K{N(SiMe<sub>3</sub>)<sub>2</sub>}]<sub>2</sub>. Both compds. were studied for the **intramol. hydroamination of aminoalkenes**. Both catalysts showed a good activity, and the best results were obtained for the Ca complex [(DIP2pyr)Ca{N(SiMe<sub>3</sub>)<sub>2</sub>}(THF)<sub>2</sub>].

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3. **Rare-Earth Metal Alkyl, Amido, and Cyclopentadienyl Complexes Supported by Imidazolin-2-iminato Ligands: Synthesis, Structural Characterization, and Catalytic Application** Full Text



By Trambitas, Alexandra G.; Panda, Tarun K.; Jenter, Jelena; Roesky, Peter W.; Daniluc, Constantin; Hrib, Cristian G.; Jones, Peter G.; Tamm, Matthias  
From Inorganic Chemistry (Washington, DC, United States) (2010), 49(5), 2435-2446. | Language: English, Database: CAPLUS

The rare earth metal dichlorides [(1)MCl<sub>2</sub>(THF)<sub>3</sub>] (2a, M = Sc; 2b, M = Y; 2c, M = Lu) and the Gd complex [(1)GdCl<sub>2</sub>(THF)<sub>2</sub>]·[LiCl(THF)<sub>2</sub>] (2d) contain the 1,2-bis(2,6-diisopropylphenyl)imidazolin-2-iminato ligand.

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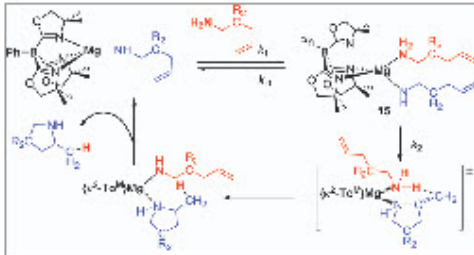
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1. **Optically active, bulky tris(oxazoliny)borato magnesium and calcium compounds for asymmetric hydroamination/cyclization** Full Text  
By Neal, Steven R.; Ellern, Arkady; Sadow, Aaron D.  
From Journal of Organometallic Chemistry (2010), Volume Date2011, 696(1), 228-234. | Language: English, Database: CAPLUS  
The synthesis of the new chiral, pseudo C3-sym., monoanionic ligand tris(4S-tert-butyl-2-oxazoliny) phenylborate [ToT]- is reported. The steric bulk, tridentate coordination, and anionic charge of [ToT]- are suitable for formation of complexes of the type ToTMX, where one valence is available for reactivity. With this point in mind, we prepd. magnesium and calcium ToT complexes that resist redistribution to (ToT)2M compds. Both ToTMgMe and ToTCaC(SiHMe2)3 contain tridentate ToT-coordination to the metal center, as shown by NMR spectroscopy, IR spectroscopy, and x-ray crystallog. These compds. are active catalysts for the cyclization of three aminoalkenes to pyrrolidines, and provide non-racemic mixts. of pyrrolidines in enantiomeric excesses up to 36%.

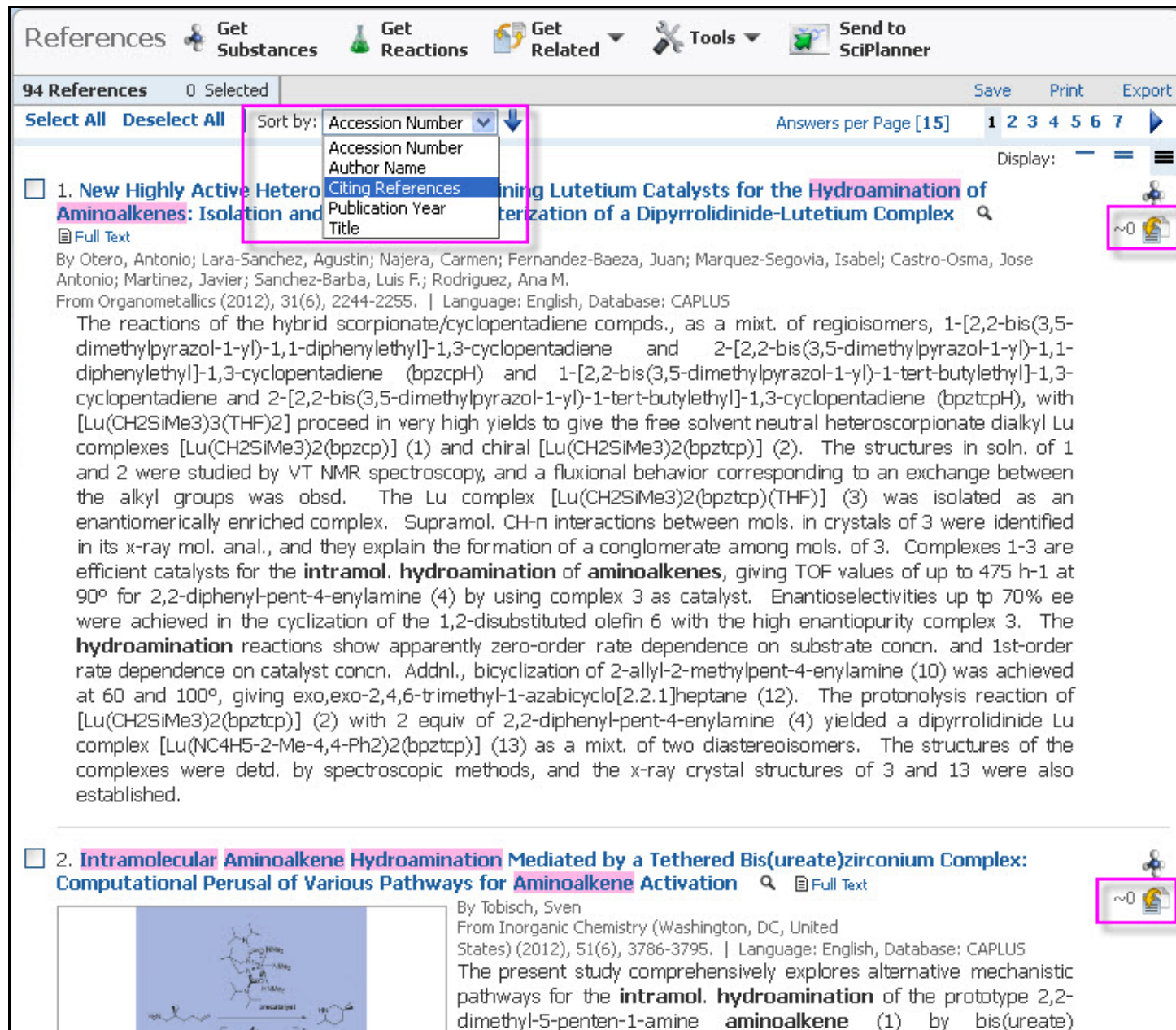
2. **Concerted C-N and C-H Bond Formation in a Magnesium-Catalyzed Hydroamination** Full Text  
By Dunne, James F.; Fulton, D. Bruce; Ellern, Arkady; Sadow, Aaron D.  
From Journal of the American Chemical Society (2010), 132(50), 17680-17683.  
| Language: English, Database: CAPLUS  
  
Coordinatively satd. ToMMgMe (1; ToM = tris(4,4-dimethyl-2-oxazoliny)phenylborate) is an active precatalyst for intramol. hydroamination/cyclization at 50 °C. The empirical rate law of  $-d[\text{substrate}]/dt = k'_{\text{obs}}[\text{Mg}]^1[\text{substrate}]^1$  and Michalis-Menton-type kinetics are consistent with a mechanism involving reversible catalyst-substrate assocn. prior to cyclization. The resting state of the catalyst, ToMMgNHCH2CR2CH2CH-CH2 [R = Ph, Me, -(CH2)5-], is isolable, but isolated magnesium amidoalkene does not undergo unimol. cyclization at 50 °C. However, addn. of trace amts. of substrate allows cyclization to occur. Therefore, we propose a two-substrate, six-center transition state involving concerted C-N bond formation and N-H bond cleavage as the turnover-limiting step of the catalytic cycle.






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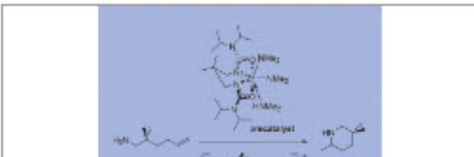

1. **New Highly Active Hetero** **Aminoalkenes: Isolation and** **Characterization of a Dipyrrolidinide-Lutetium Complex** 

 Full Text  ~0

By Otero, Antonio; Lara-Sanchez, Agustin; Najera, Carmen; Fernandez-Baeza, Juan; Marquez-Segovia, Isabel; Castro-Osma, Jose Antonio; Martinez, Javier; Sanchez-Barba, Luis F.; Rodriguez, Ana M.  
From Organometallics (2012), 31(6), 2244-2255. | Language: English, Database: CAPLUS

The reactions of the hybrid scorpionate/cyclopentadiene compds., as a mixt. of regioisomers, 1-[2,2-bis(3,5-dimethylpyrazol-1-yl)-1,1-diphenylethyl]-1,3-cyclopentadiene and 2-[2,2-bis(3,5-dimethylpyrazol-1-yl)-1,1-diphenylethyl]-1,3-cyclopentadiene (bpzcpH) and 1-[2,2-bis(3,5-dimethylpyrazol-1-yl)-1-tert-butylethyl]-1,3-cyclopentadiene and 2-[2,2-bis(3,5-dimethylpyrazol-1-yl)-1-tert-butylethyl]-1,3-cyclopentadiene (bpztcpH), with [Lu(CH<sub>2</sub>SiMe<sub>3</sub>)<sub>3</sub>(THF)<sub>2</sub>] proceed in very high yields to give the free solvent neutral heteroscorpionate dialkyl Lu complexes [Lu(CH<sub>2</sub>SiMe<sub>3</sub>)<sub>2</sub>(bpzcp)] (1) and chiral [Lu(CH<sub>2</sub>SiMe<sub>3</sub>)<sub>2</sub>(bpztcp)] (2). The structures in soln. of 1 and 2 were studied by VT NMR spectroscopy, and a fluxional behavior corresponding to an exchange between the alkyl groups was obsd. The Lu complex [Lu(CH<sub>2</sub>SiMe<sub>3</sub>)<sub>2</sub>(bpztcp)(THF)] (3) was isolated as an enantiomerically enriched complex. Supramol. CH-π interactions between mols. in crystals of 3 were identified in its x-ray mol. anal., and they explain the formation of a conglomerate among mols. of 3. Complexes 1-3 are efficient catalysts for the **intramol. hydroamination of aminoalkenes**, giving TOF values of up to 475 h<sup>-1</sup> at 90° for 2,2-diphenyl-pent-4-enylamine (4) by using complex 3 as catalyst. Enantioselectivities up to 70% ee were achieved in the cyclization of the 1,2-disubstituted olefin 6 with the high enantiopurity complex 3. The **hydroamination** reactions show apparently zero-order rate dependence on substrate concn. and 1st-order rate dependence on catalyst concn. Addnl., bicyclization of 2-allyl-2-methylpent-4-enylamine (10) was achieved at 60 and 100°, giving *exo,exo*-2,4,6-trimethyl-1-azabicyclo[2.2.1]heptane (12). The protonolysis reaction of [Lu(CH<sub>2</sub>SiMe<sub>3</sub>)<sub>2</sub>(bpztcp)] (2) with 2 equiv of 2,2-diphenyl-pent-4-enylamine (4) yielded a dipyrrolidinide Lu complex [Lu(NC<sub>4</sub>H<sub>5</sub>-2-Me-4,4-Ph<sub>2</sub>)<sub>2</sub>(bpztcp)] (13) as a mixt. of two diastereoisomers. The structures of the complexes were detd. by spectroscopic methods, and the x-ray crystal structures of 3 and 13 were also established.

2. **Intramolecular Aminoalkene Hydroamination Mediated by a Tethered Bis(ureate)zirconium Complex: Computational Perusal of Various Pathways for Aminoalkene Activation**  

  ~0

By Tobisch, Sven  
From Inorganic Chemistry (Washington, DC, United States) (2012), 51(6), 3786-3795. | Language: English, Database: CAPLUS

The present study comprehensively explores alternative mechanistic pathways for the **intramol. hydroamination** of the prototype 2,2-dimethyl-5-penten-1-amine **aminoalkene** (1) by bis(ureate)

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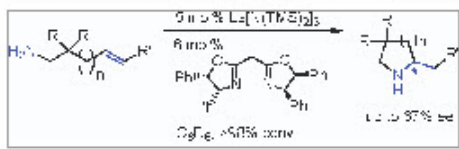
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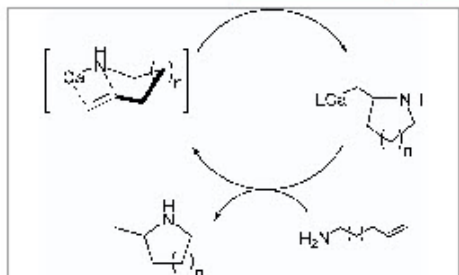
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1. **C2-Symmetric Bis(oxazolinato)lanthanide Catalysts for Enantioselective Intramolecular Hydroamination/Cyclization** ~197



By Hong, Sukwon; Tian, Shun; Metz, Matthew V.; Marks, Tobin J.  
From Journal of the American Chemical Society (2003), 125(48), 14768-14783.  
| Language: English, Database: CAPLUS  
C2-sym. bis(oxazolinato)lanthanide complexes of the type [(4*R*,5*S*)-Ph2Box]La[N(TMS)2]2, [(4*S*,5*R*)-Ar2Box]La[N(TMS)2]2, and [(4*S*)-Ph-5,5-Me2Box]La[N(TMS)2]2 (Box = 2,2'-bis(2-oxazoline)methylenyl; Ar = 4-*tert*-butylphenyl, 1-naphthyl; TMS = SiMe3) serve as precatalysts for the efficient enantioselective **intramol. hydroamination/cyclization** of **aminoalkenes** and aminodienes. These new catalyst systems are conveniently generated in situ from the known metal precursors Ln[N(TMS)2]3 or Ln[CH(TMS)2]3 (Ln = La, Nd, Sm, Y, Lu) and 1.2 equiv of com. available or readily prepd. bis(oxazoline) ligands such as (4*R*,5*S*)-Ph2BoxH, (4*S*,5*R*)-Ar2BoxH, and (4*S*)-Ph-5,5-Me2BoxH. The X-ray crystal structure of [(4*S*)-*t*BuBox]Lu[CH(TMS)2]2 provides insight into the structure of the in situ generated precatalyst species. Lanthanides having the largest ionic radii exhibit the highest turnover frequencies as well as enantioselectivities. Reaction rates maximize near 1:1 BoxH:Ln ratio (ligand acceleration); however, increasing the ratio to 2:1 BoxH:Ln decreases the reaction rate, while affording enantiomeric excesses similar to the 1:1 BoxH:Ln case. A screening study of bis(oxazoline) ligands reveals that aryl stereodirecting groups at the oxazoline ring 4 position and addnl. substitution (geminal di-Me or aryl) at the 5 position are crucial for high turnover frequencies and good enantioselectivities. The optimized precatalyst, in situ generated [(4*R*,5*S*)-Ph2Box]La[N(TMS)2]2, exhibits good rates and enantioselectivities, comparable to or greater than those achieved with chiral C1-sym. organolanthanocene catalysts, even for poorly responsive substrates (up to 67% ee at 23 °C). Kinetic studies reveal that **hydroamination** rates are zero order in [amine substrate] and first order in [catalyst], implicating the same general mechanism for organolanthanide-catalyzed **hydroamination/cyclizations** (**intramol.** turnover-limiting olefin insertion followed by the rapid protonolysis of an Ln-C bond by amine substrate) and implying that the active catalytic species is monomeric.

2. **Calcium-mediated intramolecular hydroamination catalysis** ~161



By Crimmin, Mark R.; Casely, Ian J.; Hill, Michael S.  
From Journal of the American Chemical Society (2005), 127(7), 2042-2043.  
| Language: English, Database: CAPLUS  
The calcium-catalyzed **intramol. hydroamination** of alkenes and alkynes is reported. The  $\beta$ -diketiminato complex [(HC(CMe)2N-2,6-*i*Pr2C6H3)2]-Ca{N(SiMe3)2}(THF)] affected the catalytic cyclization of a range of **aminoalkenes** and aminoalkynes with activities that were broadly commensurate to those of established rare earth catalysts.

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