Available online at www.sciencedirect.com
SCIENCE DIRECT®

ELSEVIER

Volume 689, issue 16, 15 August 2004

Journal of Organometallic Chemistry

www.elsevier.com/locate/jorganchem

Contents

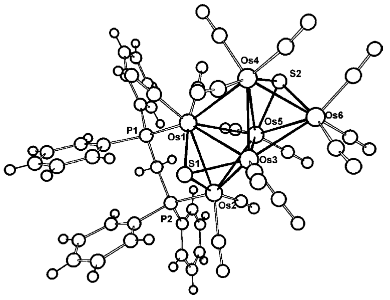
Regular papers

Tahmina Akter, Noorjahan Begum, Daniel T. Haworth, Dennis W. Bennett, Shariff E. Kabir, Md. Arzu Miah, Nitai C. Sarker, Tasneem A. Siddiquee, Edward Rosenberg

J. Organomet. Chem. 689 (2004) 2571

Hexa- and triosmium carbonyl clusters bearing bridging dppm and capping sulfido ligands

$[Os_3(CO)_7(\mu_3\text{-}S)_2(\mu\text{-dppm})]$ (**1**) reacts with Me_3NO at 80 °C to yield $[Os_3(\mu_3\text{-}S)_2(CO)_6(Me_3N)(\mu\text{-dppm})]$ (**2**) and $[Os_6(CO)_{12}(\mu_3\text{-}S)_4(\mu\text{-dppm})_2]$ (**3**) while it combines with $[Os_3(CO)_{10}(MeCN)_2]$ at 80 °C to give $[Os_6(CO)_{14}(\mu_3\text{-}S)_2(\mu\text{-dppm})]$ (**4**). Compound **2** reacts with CO, PPh_3, $P(OMe)_3$ to give **1**, $[Os_3(CO)_6(\mu_3\text{-}S)_2(\mu\text{-dppm})(PPh_3)]$ (**5**), and $[Os_3(CO)_6(\mu_3\text{-}S)_2(\mu\text{-dppm})\{P(OMe)_3\}]$ (**6**) respectively, demonstrating that the NMe_3 ligand in **2** is labile. Treatment of **1** with PPh_3 and $P(OMe)_3$ in presence of Me_3NO also gives **5** and **6**.

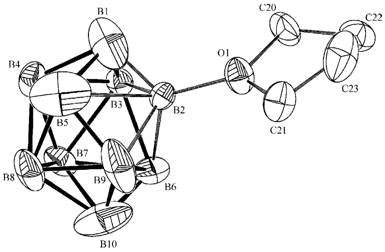


R. Bernard, D. Cornu, M. Perrin, J.-P. Scharff, P. Miele

J. Organomet. Chem. 689 (2004) 2581

Synthesis and X-ray structural characterisation of the tetramethylene oxonium derivative of the hydrodecaborate anion. A versatile route for derivative chemistry of $[B_{10}H_{10}]^{2-}$

The oxonium derivative $P(C_6H_5)_4[2\text{-}B_{10}H_9O(CH_2)_4]$ (**1**) has been prepared from $[B_{10}H_{10}]^{2-}$ by a solvent-addition reaction route, promoted by $Et_2O \cdot BF_3$. Its structure has been confirmed by single crystal X-ray analysis.

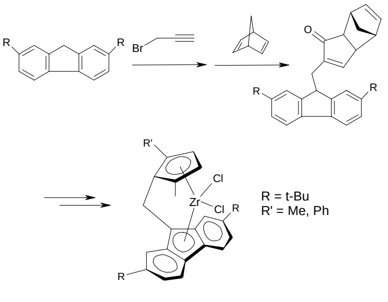


Si-Geun Lee, Sung-Don Hong, Young-Whan Park, Boong-Goon Jeong, Dae-Woo Nam, Hye Young Jung, Hyosun Lee, Kwang Ho Song

J. Organomet. Chem. 689 (2004) 2586

Syntheses of methylene-bridged *ansa*-zirconocene complexes and copolymerization studies of ethylene and norbornene

Two new *ansa*-zirconocene dichlorides with substituents α- to the *ansa*-bridge, have synthesized to investigate their ethylene–norbornene copolymerization in the presence of MAO. The ligand synthesis includes of 3-bromo-1-propyne which affords the methylene-bridging unit by way of an intermolecular Pauson–Khand reaction in which norbornadiene and a pendant alkyne cyclize to form a ring that later becomes a substituted cyclopentadienyl group.



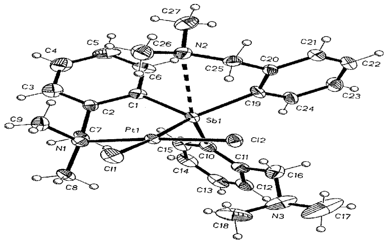
Elsevier B.V.
doi:10.1016/S0022-328X(04)00478-4

P. Sharma, D. Castillo, N. Rosas, A. Cabrera, E. Gomez, A. Toscano, F. Lara, S. Hernández, G. Espinosa

J. Organomet. Chem. 689 (2004) 2593

Synthesis and structures of organoantimony compounds containing intramolecular Sb–N interactions

Stibines R'_3Sb containing the pendant arm [2-$(Me_2NCHR)C_6H_4$] (where R = H **1** or Me **2**) and their derivatives {[2-$(Me_2NCHR)C_6H_4$][2-$(Me_3N^+CHR)C_6H_4$]}Sb 2[I]$^-$ (where R = H **3**; Me **4**) and {2-[$(Me_2HN^+CH_2)$-$C_6H_4]_3$}Sb 3[Br]$^-$ **5** respectively were synthesized. A novel platinum complex **6** [$PtCl_2 \cdot$ **1**] containing stibine **1** as a bidentate ligand has also been prepared.

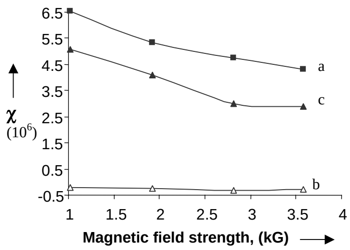


B.N. Achar, K.S. Lokesh

J. Organomet. Chem. 689 (2004) 2601

Studies on phthalocyanine sheet polymers

Cobalt, nickel and copper phthalocyanine sheet polymers are synthesized by heating their respective metal (II) phthalocyanine tetracarboxylic acids at 400 °C in nitrogen atmosphere. These polymers are characterized using UV–Visible spectra, IR spectra, magnetic susceptibility, X-ray powder diffraction and thermogravimetric analysis.

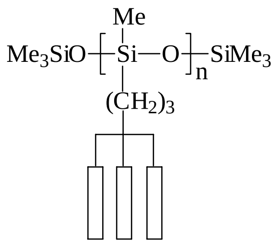


Tomasz Ganicz, Wodzimierz A. Stańczyk

J. Organomet. Chem. 689 (2004) 2606

Synthesis of novel tri-podal mesogenic alkenes and side-chain polysiloxanes

Two synthetic routes for novel tri-podal mesogenic alkenes and side-chain polysiloxanes have been developed starting from the precursors $HC(SiMe_2H)_3$ and $HC(SiMe_2Vi)_3$. Polysiloxanes with high mesogenic density may be obtained in four to six simple synthetic steps. Such novel and simple mesogenic groups should be easily attached to polymer systems (e.g., dendritic polysiloxanes or polycarbosilanes) in which the number of available sites is limited.



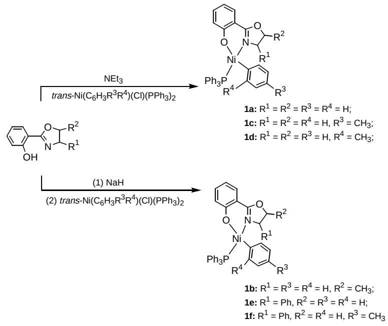
$$Me_3SiO\left[\overset{Me}{\underset{(CH_2)_3}{Si}}-O\right]_n SiMe_3$$

Wei Zhao, Yanlong Qian, Jiling Huang, Jianjun Duan

J. Organomet. Chem. 689 (2004) 2614

Novel neutral arylnickel(II) phosphine catalysts containing 2-oxazolinylphenolato N–O chelate ligands for ethylene oligomerization and propylene dimerization

A series of new neutral arylnickel(II) phosphine complexes **1** bearing 2-oxazolinylphenolato ligands [2-(4-R^1-5-R^2-C_3H_2NO)–C_6H_4O]Ni(2-R^4-4-R^3-C_6H_3)(PPh_3) were synthesized by reactions of sodium salts of 2-(4,5-dihydro-2-oxazolyl)phenol derivatives with *trans*-Ni(Ar)(Cl)$(PPh_3)_2$ or by direct reactions of the ligands with *trans*-Ni(Ar)(Cl)$(PPh_3)_2$ in the presence of NEt_3.



1a: $R^1 = R^2 = R^3 = R^4 = H$;
1c: $R^1 = R^2 = R^4 = H, R^3 = CH_3$;
1d: $R^1 = R^2 = R^3 = H, R^4 = CH_3$;

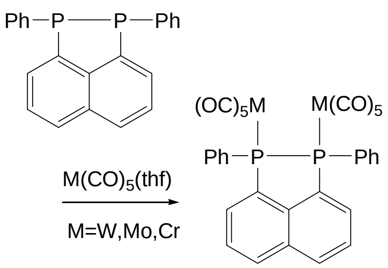
1b: $R^1 = R^3 = R^4 = H, R^2 = CH_3$;
1e: $R^1 = Ph, R^2 = R^3 = R^4 = H$;
1f: $R^1 = Ph, R^2 = R^4 = H, R^3 = CH_3$

Tsutomu Mizuta, Satoru Kunikata, Katsuhiko Miyoshi

J. Organomet. Chem. 689 (2004) 2624

Synthesis and molecular structures of dinuclear complexes with 1,2-dihydro-1,2-diphenyl-naphtho[1,8-c,d]1,2-diphosphole as a bridging ligand

A bisphosphine in which a PhP–PPh bond bridges 1,8-positions of naphthalene, 1,2-dihydro-1,2-diphenyl-naphtho[1,8-cd]-1,2-diphosphole (**1**), was used as a bridging ligand for the preparation of dinuclear group 6 metal complexes $(OC)_5M(\mu\text{-}\mathbf{1})M(CO)_5$ (M = W, Mo, Cr).

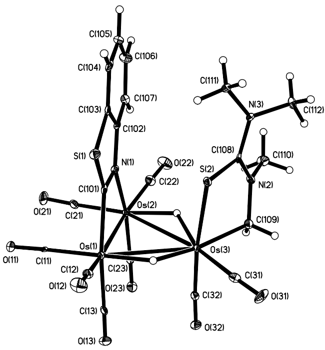


Noorjahan Begum, Antony Deeming, Mohammad Islam, Shariff Kabir, Dalia Rokhsana, Edward Rosenberg

J. Organomet. Chem. 689 (2004) 2633

Reactions of benzothiazolide triosmium clusters with tetramethylthiourea

The valence saturated 1,2-coordinated benzothiazolide triosmium cluster $[Os_3(CO)_{10}(\mu\text{-}\eta^2\text{-}C_7H_4NS)(\mu\text{-}H)]$ (**1**) and the electron deficient 1,7-coordinated complex $[Os_3(CO)_9(\mu_3\text{-}\eta^2\text{-}C_7H_4NS)(\mu\text{-}H)]$ (**2**) react with tetramethylthiourea to give $[Os_3(CO)_8(\mu\text{-}\eta^2\text{-}C_7H_4NS)(\eta^2\text{-}SCNMe_2NMeCH_2)(\mu\text{-}H)_2]$ (**5**) and $[Os_3(CO)_8(\mu\text{-}\eta^2\text{-}C_7H_4NS)(\eta^2\text{-}SCNMe_2NMeCH_2)(\mu\text{-}H)_2]$ (**6**), respectively which both contain an unusual chelate ring formed by sulfur coordination and C–H activation of one methyl group coordinated to the osmium atom that is not bound to the benzothiazole ring. In contrast, the reaction of $[Os_3(CO)_9(\mu_3\text{-}\eta^2\text{-}C_7H_3(2\text{-}CH_3)NS)(\mu\text{-}H)]$ (**3**) gives only $[Os_3(CO)_9(\mu\text{-}\eta^2\text{-}C_7H_3(2\text{-}CH_3)NS)(\eta^1\text{-}SC(NMe_2)_2)(\mu\text{-}H)]$ (**8**).

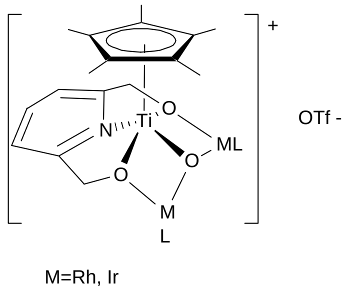


Rosa Fandos, Antonio Otero, Ana Rodríguez, María José Ruiz, Pilar Terreros

J. Organomet. Chem. 689 (2004) 2641

Heterobimetallic complexes as oxygen donor bidentate ligands: synthesis of early–late trinuclear complexes

The ability of the heterobimetallic complexes $[TiCp((OCH_2)_2Py)(\mu_2\text{-}O)\,M(COD)](M$ = Rh, Ir) to behave as bidentate ligands to yield early–late trinuclear complexes complexes $[TiCp((OCH_2)_2Py)(\mu_3\text{-}O)\{M(COD)\}_2]^+$ has been studied.

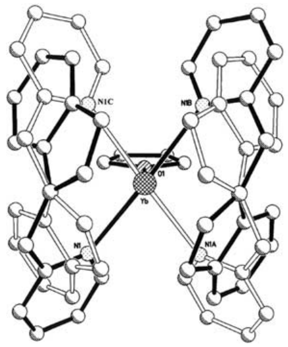


Jianhua Cheng, Dongmei Cui, Wenqi Chen, Ninghai Hu, Tao Tang, Baotong Huang

J. Organomet. Chem. 689 (2004) 2646

Organolanthanides with 3-(2-pyridylmethyl) indenyl ligands: synthesis, crystal structures and catalytic activities of divalent complexes for ε-caprolactone polymerization

A series of complexes of divalent and trivalent lanthanides with pyridyl-functionalized indenyl ligand were synthesized by metathesis reaction of Ln(II) iodides or LN(III) chlorides with the alkyllithium of the ligand. Molecular structures of divalent lanthanides show high symmetry, significantly different from previous reports on divalent bis-indenyl lanthanides.

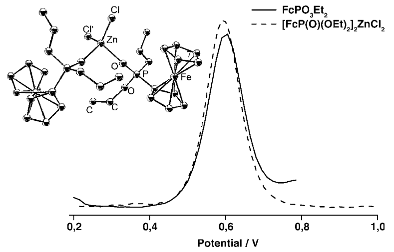


Olivier Oms, Frédéric Maurel, Francis Carré, Jean Le Bideau, André Vioux, Dominique Leclercq

J. Organomet. Chem. 689 (2004) 2654

Improved synthesis of diethyl ferrocenylphosphonate, crystal structure of $(FcPO_3Et_2)_2 \cdot ZnCl_2$, and electrochemistry of ferrocenylphosphonates, FcP(O)$(OR)_2$, $FcCH_2P(O)(OR)_2$, 1,1′-fc$[P(O)(OR)_2]_2$ and $[FcP(O)(OEt)_2]_2 \cdot ZnCl_2$ (Fc = $(\eta^5C_5H_5)Fe(\eta^5C_5H_4)$, fc = $(\eta^5C_5H_4)Fe(\eta^5C_5H_4)$, R = Et, H)

An improved synthesis of diethyl ferrocenylphosphonate using the ^{t}BuLi/^{t}BuOK system at low temperature is reported and the structure of $[FcPO_3Et_2]_2 \cdot ZnCl_2$ complex is described. The electrochemical behaviour of FcP(O)$(OEt)_2$, 1,1′-fc$[P(O)(OEt)_2]_2$, $FcCH_2$-P(O)$(OEt)_2$, and their corresponding acids were compared. Each of them shows a reversible one-electron transfer reaction. Ferrocenylbisphosphonate is more difficult to oxidize than ferrocenylphosphonate due to the presence of two electron-withdrawing substituents.

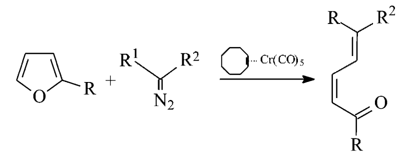


Norbert D. Hahn, Martin Nieger, Karl Heinz Dötz

J. Organomet. Chem. 689 (2004) 2662

Efficient and regioselective chromium(0)-catalyzed reaction of 2-substituted furans with diazo compounds: stereoselective synthesis of (2*E*,4*Z*)-2-aryl-hexadienedioic acid diesters

Pentacarbonyl(η^2-*cis*-cyclooctene)chromium(0) is an effective catalyst for the regio- and stereoselective cyclopropanation/ring-opening reaction of electron-rich furans with diazo compounds under mild conditions. The reaction of 2-methoxyfuran with alkyl α-diazoarylacetate provides synthetically useful (2*E*,4*Z*)-2-aryl-hexadienedioic acid diesters in excellent yields after ring opening of the primary cyclopropanation products.

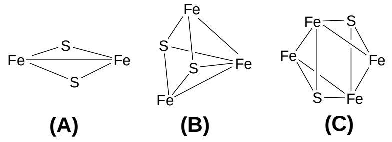


Botao Zhuang, Jun Chen, Lingjie He, Jiutong Chen, Zhangfeng Zhou, Kechen Wu

J. Organomet. Chem. 689 (2004) 2674

Synthesis, structure and formation pathways of new Fe–S complexes containing $[Fe_2S_2]$-units in different valences, $[Fe_2S_2(CO)_4(PPh_3)_2]$, $[Fe_3S_2(CO)_6(PPh_3)_3]$ and $[Fe_4S_2(CO)_{10}]^{2-}$ and the origin of the $[Fe_2S_2]$-unit in metal–$[Fe_2S_2(CO)_6]$ complexes

$[Fe_2S_2(CO)_4(PPh_3)_2]$ (**1**), $[Fe_3S_2(CO)_6(PPh_3)_3]$ (**2**), and $[Ph_4P]_2[Fe_4S_2(CO)_{10}]$ (**3**) have been isolated from the reaction involving with $[Fe_2S_2(CO)_6]$ and **1**, **2** and **3** contained $[Fe_2S_2]^0$ core (**A**) with $[Fe_2S_2]^0$-unit, $[Fe_3S_2]^0$ core (**B**) with $[Fe_2S_2]^{2-}$-unit and $[Fe_4S_2]^{2-}$ core (**C**) with $[Fe_2S_2]^{4-}$-unit, respectively, of which the $[Fe_2S_2]^{0,\ 2-,\ 4-}$-units just correspond to that in the disproportionation products of $[Fe_2S_2(CO)_6]^{2-}$ evidencing that a disproportionation of $[Fe_2S_2(CO)_6]^{2-}$ occurs in the synthetic reaction system.The formation pathways of **1**, **2** and **3** via unit construction was figured out and the origin of $[Fe_2S_2]$-units in M–$[Fe_2S_2(CO)_6]$ was discussed.



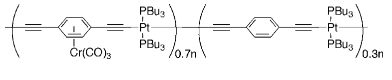
(A) **(B)** **(C)**

Yasuhiro Morisaki, Hui Chen, Yoshiki Chujo

J. Organomet. Chem. 689 (2004) 2684

Synthesis and characterization of organometallic conjugated polymers containing tricarbonyl(arene)chromium unit and platinum

Novel π-conjugated polymer containing (η^6-arene)$Cr(CO)_3$ and Pt in the main chain was prepared. The polymer obtained was soluble in common organic solvents and characterized by NMR and FT-IR spectra. Optical, electrochemical, thermal properties, and reactivity of the polymer are discussed.

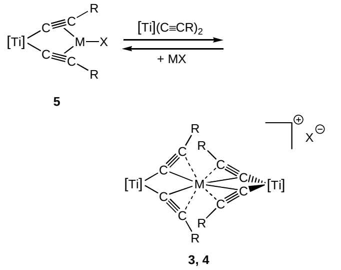


H. Lang, T. Stein, S. Back, G. Rheinwald

J. Organomet. Chem. 689 (2004) 2690

Titanocene-based group-11 metal ions; solid-state structure of $\{[(\eta^5\text{-}C_5H_4SiMe_3)_2Ti\text{-}(C{\equiv}CPh)_2]_2Ag\}NO_3$

The synthesis and reaction chemistry of heterobi- (TiM) (**5**) tri- (Ti_2M) (**3**) and pentanuclear (Ti_2MFe_2) (**4**) complexes (M = Cu, Ag) is described. The X-ray structure analysis as well as the electrochemical behaviour of one example is reported.



5

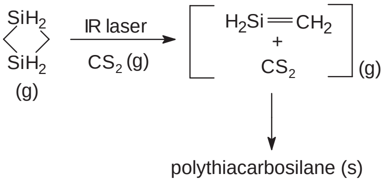
3, 4

Marketa Urbanová, Josef Pola

J. Organomet. Chem. 689 (2004) 2697

IR laser decomposition of 1,3-disilacyclobutane in presence of carbon disulfide: chemical vapour deposition of polythiacarbosilane

TEA CO_2 laser irradiation of gaseous mixtures of 1,3-disilacyclobutane – carbon disulfide affords chemical vapour deposition of solid polythiacarbosilane films that possess Si–S–X (X = Si, C), S–H and Si–H bonds and undergo slow hydrolysis in air to polyoxothiacarbosilanes containing Si–H, Si–O–Si and (C)S–H bonds. The formation of the polythiacarbosilane is proposed to take place via polymerization of transient silene and incorporation of CS_2 into growing polysilene network.

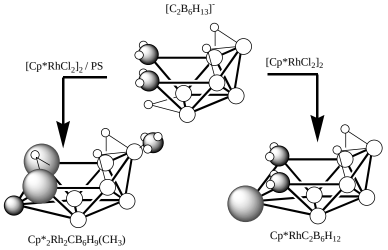


Michael G.S. Londesborough, Zbyněk Janoušek, Bohumil Štíbr, Ivana Císařová

J. Organomet. Chem. 689 (2004) 2702

Metallacarborane chemistry of the *hypho*-$[6,7\text{-}C_2B_6H_{13}]^-$ anion: the formation of uniquely structured metallacarboranes [5-Cp^*-*arachno*-5, 4, 6-$RhC_2B_6H_{12}$] and [2, 5-Cp^*_2-10-Me-nido-2, 5, 1-$Rh_2CB_6H_9$] ($Cp^* = \eta^5\text{-}C_5Me_5^-$): complete rhodium analogues of *arachno*-4,6-$C_2B_7H_{13}$ and *nido*-1-CB_8H_{12}

Reaction of the tetramethylammonium salt of the *hypho*-$[6,7\text{-}C_2B_6H_{13}]^-$ anion (**1**) with the $[Rh(\eta^5\text{-}C_5Me_5)Cl_2]_2$ dimer in THF solution, resulted in the formation of a novel nine-vertex metalladicarbaborane [5-Cp-*arachno*-5,4,6-$RhC_2B_6H_{12}$] (**2**) (Cp = η^5-C_5Me_5).

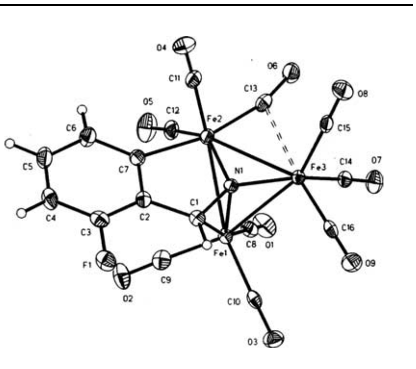


Daniel Dönnecke, Kathi Halbauer, Wolfgang Imhof

J. Organomet. Chem. 689 (2004) 2707

The reaction of *ortho*-halogenated aromatic aldazine ligands with $Fe_2(CO)_9$: symmetrical cleavage of the azine and carbon–halogen activation

Aromatic azines with halogen substituents in *ortho*-position with respect to the imine nitrogen atoms by treatment with $Fe_2(CO)_9$ show two typical reaction pathways. Either the azine is symmetrically cleaved and iron carbonyl compounds exhibiting iminato moieties are produced or one of the carbon halogen bonds is activated. The latter reactivity leads to the formation of iron carbonyl clusters in which the N–N bond of the azine is still preserved or in which an arylidenimido ligand is present in the molecule.

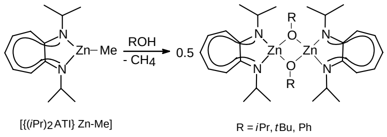


Jost-Steffen Herrmann, Gerrit A. Luinstra, Peter W. Roesky

J. Organomet. Chem. 689 (2004) 2720

Aminotroponiminate alkyl and alkoxide complexes of zinc

Reaction of $\{(i\text{Pr})_2\text{ATI}\}\text{H}$ with the dimethyl zinc afforded the methyl complex $[\{(i\text{Pr})_2\text{ATI}\}\text{Zn–Me}]$. Subsequent reaction with different alcohols gave the dimeric alkoxide complexes $[\{(i\text{Pr})_2\text{ATI}\}\text{Zn–OR}]_2$ (R = *i*Pr, *t*Bu, Ph).



Note

C. Sivasankar, J.K. Bera, M. Nethaji, A.G. Samuelson

J. Organomet. Chem. 689 (2004) 2726

Reactions of $\text{Cu}_3(\text{dppm})_3(\mu_3\text{-OH})(\text{ClO}_4)_2$ (dppm = bis-(diphenylphosphino) methane) with Soft Heterocumulenes

The reaction of $[\text{Cu}_3(\text{dppm})_3(\mu_3\text{-OH})](\text{ClO}_4)_2$ (**1**) with heterocumulenes (XCS; X = NPh, NMe and S) has been studied. The μ_3-OH ligand inserts into PhNCS and MeNCS only in the presence of methanol.