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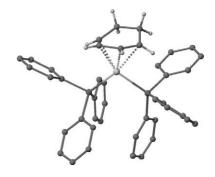
Note

Adem Rifat, Mary F. Mahon, Andrew S. Weller

J. of Organomet. Chem. 667 (2003) 1

Dehydrogenation of cyclohexenes to cyclohexadienes by $[(PPh_3)_2Rh]^+$. The isolation of an intermediate in the dehydrogenation of cyclohexane to benzene: crystal structure of $[(\eta^4-C_6H_8)Rh(PPh_3)_2][closo-CB_{11}H_6Br_6]$

Treatment of the arene bridged dimer $[(PPh_3)_2Rh]_2[closo-CB_{11}H_6Br_6]_2$ with cyclohexene affords $[(\eta^4-C_6H_8)Rh(PPh_3)_2][closo-CB_{11}H_6Br_6]$, an intermediate in the catalytic dehydrogenation of cyclohexane to benzene.



Short Communication

Chunlin Ma, Feng Li, Daqi Wang, Handong Yin

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Synthesis and crystal structure of a novel pentanuclear complex of tin (IV) with heterocyclic sulfur and nitrogen donor ligands A novel macrocyclic complex of organotin was synthesized by the reaction of dibutyltin dichloride with 2,5-dimercapto-1,3,4-thiodiazole. The title pentanuclear complex of tin is a 35-membered macrocycle. All five Sn atoms are six-coordinate, and have distorted octahedron geometries due to intramolecular Sn-N interactions. The title complex is characterized by Elemental analysis, IR, ¹H-NMR spectroscopy and X-ray diffraction.

$$(\begin{array}{c|c} Bu\text{-}n & N & N \\ Sn & C & C-S-)_{5^-} & (I) \\ Bu\text{-}n & S & S & C \\ \end{array}$$

Regular Papers

Martin A. Bennett, Mike R. Kopp, Eric Wenger, Anthony C. Willis

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Generation of nickel(0)–aryne and nickel(II)–biphenyldiyl complexes via in situ dehydrohalogenation of arenes. Molecular structures of [Ni(2,2'-C₆H₄C₆H₄)(dcpe)] and C_2 -hexabenzotriphenylene

A series of η^2 -aryne complexes [Ni(η^2 -aryne)(dcpe)] was generated *in situ* by arene dehydrohalogenations in the presence of [Ni(η^2 -C₂H₄)(dcpe)]. These reactions proved difficult to control as further reactions of free aryne with the desired aryne complexes were observed, leading to biphenyldiyl complexes or polyaromatic systems.

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Birgit Schwenzer, Helmut Fischer

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Alkynylcarbyne(bipyridine)dicarbonyl(triflouroacetato) complexes—synthesis and detection of reversible association in solution The alkynylcarbyne(bipyridine)(dicarbonyl)-trifluoroacetato complexes, [CF₃COO(L–L)(CO)₂M=C–C=C–R] (M=W, Mo; L–L=2,2'-bipyridine, 4,4'-dimethyl 2,2'-bipyridine; R=SiMe₃; Ph; p-Tol, t-Bu) exhibit an unusual feature in solution: two sets of ν (CO) absorptions (A₁/B₁ and A₁*/B₁*) are observed at high concentration instead of one. The ratio of the two sets is concentration-dependent.

$$\begin{array}{c|c}
\hline
 & N \\
\hline
 & N \\
\hline
 & N \\
\hline
 & C \\
 & C \\
\hline
 & C \\
 &$$

Clemens Reiche, Susanne Kliem, Uwe Klingebiel, Mathias Noltemeyer, Claudia Voit, Regine Herbst-Irmer, Stefan Schmatz

J. of Organomet. Chem. 667 (2003) 24

Aminosilanolates as precursors of four- and eight-membered (SiNSiO)-rings

The only hitherto known aminosilanol crystallizes under formation of a dimeric ladder structure. Its sodium and potassium derivatives form hexagonal prisms (2, 3). The potassium salt includes two H_2O and two THF molecules. The reactivity of 2 and 3 leads to the formation of so far unknown (SiOSiN)- and (SiOSiNLiF)-ring systems. Quantum chemical calculations on (SiOSiN)-rings confirm the experimental observations.

$$\begin{pmatrix} R_2Si \longrightarrow OK \\ | NH_2 \end{pmatrix} = 2 H_2O * 2 thf$$

$$R = CMe_3$$

Wolfgang Malisch, Heinrich Jehle, Dirk Schumacher, Michael Binnewies, Nicola Söger

J. of Organomet. Chem. 667 (2003) 35

Polychlorinated metallo-siloxanes of iron, molybdenum and tungsten - structure of 1-[dicarbonyl(η^5 -cyclopentadienyl)ferrio]-1,1,3,3,3-pentachlorodisiloxane

Reaction of the metalates $Na[Fe(CO)_2C_5R_5]$ [R = H (1a); Me (1b)] and $Li[M(CO)_2(P-Me_3)Cp]$ [M = Mo (1c); W (1d)] with the chlorosiloxanes $Cl_3SiOSiCl_3$ (2a) and

Cl₃SiOSiCl₂OSiCl₃ (**2b**) leads to the metallo-siloxanes L_n M-SiCl₂OSiCl₃ $[L_n$ M = Cp(OC)₂Fe (**3a**), Cp(OC)₂(Me₃P)Mo (**4a**), Cp(OC)₂(Me₃P)W (**4b**)] and C₃R₃(OC)₂Fe-SiCl₂OSiCl₂OSiCl₃ $[R = H \ (3b), Me \ (3c)],$ respectively. Cp(OC)₂Fe-SiCl₂OSiCl₂OSiCl₃ (**3b**) has been converted to the bis-metalated polychlorinated siloxane Cp(OC)₂Fe-SiCl₂OSiCl₂OSiCl₂Fe(CO)₂Cp (**8**) by reaction with a second equivalent of Na[Fe(CO)₂Cp] (**1a**). The structure of Cp(OC)₂Fe-SiCl₂OSiCl₃ (**3a**) was obtained by X-ray diffraction.

Yoshinori Satoh, Noriko Ikitake, Yuushou Nakayama, Shingo Okuno, Hajime Yasuda

J. of Organomet. Chem. 667 (2003) 42

Syntheses of bis- and tetra(trimethylsilyl) substituted lanthanocene methyl complexes and their catalyses for polymerizations of methyl methacrylate, ϵ -caprolactone and L-lactide

Bis- and tetra(trimethylsilyl) substituted lanthanocene methyl complexes, [(Me_3-SiC_5H_4)_2SmMe]_2 (2) {[1,3-(Me_3Si)_2C_5H_3]_2-SmMe}_2 (4) and {[1,3-(Me_3Si)_2C_5H_3]_2-NdMe}_2 (5) were prepared from trimethylsilyl- or bis(trimethylsilyl)-cyclopentadienyllithium with LnCl_3(THF)_2 followed by the reaction with MeLi. Their catalyses for polymerization of methyl methacrylate and block copolymerization of L-lactide with ϵ -caprolactone were examined.

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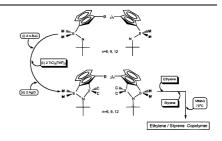
Seok Kyun Noh, Jungmin Lee, Dong-ho Lee

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Syntheses of dinuclear titanium constrained geometry complexes with polymethylene bridges and their copolymerization properties

The polymethylene bridged dinuclear half-sandwich CGC (constrained geometry catalyst) [$\text{Ti}(\eta^5:\eta^1\text{-C}_0\text{H}_3\text{SiMe}_2\text{NCMe}_3)$]₂ [$(\text{CH}_2)_n$] (n=6 (10), n=9 (11), n=12 (12)) have been prepared. ^1H - and $^{13}\text{C-NMR}$ spectra of the synthesized complexes provide firm evidence for the antici-

pated dinuclear structure. The important feature associated with the chemical shift of Carbon-13 NMR is the downfield chemical shift of 63.0 ppm due to the *ipso*-carbon of the *t*-butyl group bonded at the coordinated nitrogen. Activity of the catalyst in ethylene/styrene copolymerization increases in the order of 10 < 11 < 12 which indicates the presence of a longer bridge between two active sites contributes to facilitate the polymerization activity of the dinuclear CGC. The melting points of the generated copolymers decrease as the styrene portion in feed increases. Styrene content in copolymers formed by the dinuclear catalysts is actually bigger than that in copolymer formed by the mononuclear CGC.



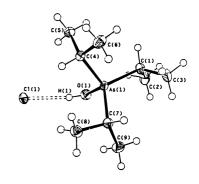
Anjali Gupta, Rajnish K. Sharma, Rakesh Bohra, Vimal K. Jain, John E. Drake, Michael B. Hursthouse, Mark E. Light

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Triorganoarsenic(V) compounds with internally functionalized oximes: synthetic and spectroscopic aspects of $[R_3As(Cl)L]$, $[R_3As(OH)L]$ and $[R_3AsL_2]$: crystal and molecular structure of $[Pr_3^iAsOH]^+Cl^-$

Triorganoarsenic(V) oximates of the type $[R_3As\{ON=C(Me)Ar\}_2]$ (1) $(R=Pr^i, Bu^i;$

 $\rm Ar=C_5H_4N\text{-}2,\ C_4H_3O\text{-}2)$ are formed by the reactions of R_3AsCl_2 with the sodium salts of internally functionalized oximes in 1:2 molar ratio in anhydrous benzene. The redistribution products $[R_3As(X)\{ON\text{=}C(Me)Ar\}]$ (2) (X = Cl, Br, OH) are obtained by treatment of 1 with equimolar R_3AsX_2 . All of these complexes are characterized by IR and NMR (1H and ^{13}C) spectroscopy and elemental analyses. Controlled hydrolysis of a representative monochloro-complex $[Pr_3^iAs(Cl)\{ON\text{=}C(Me)\text{-}C_4H_3O\text{-}2\}]$ yields crystals of $Pr_3^iAs(OH)Cl$ in which single crystal X-ray diffraction indicates that there is a distorted tetrahedral environment around arsenic.



Alan R. Bassindale, David J. Parker, Peter G. Taylor, Norbert Auner, Bernhard Herrschaft

J. of Organomet. Chem. 667 (2003) 66

Modelling $S_{\rm N}2$ nucleophilic substitution at silicon by structural correlation with X-ray crystallography and NMR spectroscopy

Excellent agreement is obtained between structural correlations of four 1-(halodimethylsilylmethyl)-2-quinolinones using X-ray crystallography and NMR spectroscopy in solution to map substitution at silicon. Comparison of calculations from both techniques confirms the validity of the NMR method for structural correlation in solution

Peter B. Hitchcock, G. Jeffery Leigh, Maria Togrou

J. of Organomet. Chem. 667 (2003) 73

Synthesis of titanium(IV) ferrocenylaminate complexes

The ligand [CpFe $\{(1,2-C_5H_3)(CH_2)_nNR_2\}$] $^-$ (n=1 or 2, R=Me or Et) is monodentate when complexed to a Cp₂Ti^{IV} moiety, and also when two are complexed to a CpTi^{IV} group. Only when steric pressures are minimal does it act as a bidentate ligand.

CpFe
$$Ti^{|V|}$$
 OR

CpFe $Ti^{|V|}$
 $n = 1 \text{ or } 2, R = \text{Me or Et}$

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Martin N. Ackermann, Keara B. Moore, Amanda S. Colligan, Jennifer A. Thomas-Wohlever, Kirk J. Warren

J. of Organomet. Chem. 667 (2003) 81

Tetracarbonylmolybdenum complexes of 2-(phenylhydrazino)pyridine ligands. Correlations of spectroscopic data with pyridyl substituent effects A series of 2-(phenylhydrazino)pyridine complexes cis-Mo(CO)₄(X–2-(phenylhydrazino)pyridine) have been synthesized and characterized. The complexes show a number of good correlations among the Hammett sigma parameter; the sum of the carbonyl-stretching frequencies, and $\delta(^{95}\text{Mo})$ and $\delta(^{13}\text{CO})$ of the complexes. Comparisons are made to the analogous 2-(phenylazo)pyridine complexes.

X = 4-CH₃O, 4-CH₃, H, 4-Cl, 5-Br, 6-CH₃, 4,6-(CH₃)₂

Arihiro Iwata, Yutaka Toyoshima, Tsuyoshi Hayashida, Takahiko Ochi, Atsutaka Kunai, Joji Ohshita

J. of Organomet. Chem. 667 (2003) 90

PdCl₂ and NiCl₂-catalyzed hydrogen-halogen exchange for the convenient preparation of bromo- and iodosilanes and germanes

Treatment of hydrosilanes with an excess of alkyl bromides in the presence of a PdCl₂ or NiCl₂ catalyst gave bromosilanes in good to high yield. Using propyl iodide as the iodine source, similar iodination of hydrosilanes occurred. Halogenation of hydrogermanes also proceeded by similar treatment with PdCl₂ as the catalyst.

$$R_{4-n}MH_n$$
 $\xrightarrow{R'X}$ $R_{4-n}MX_n$

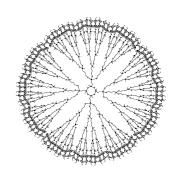
$$M = Si, Ge; n = 1-3$$

Chungkyun Kim, Hyojeong Kim, Kyungrae Park

J. of Organomet. Chem. 667 (2003) 96

Diels-Alder reaction of anthracene and *N*-ethylmaleimide on the carbosilane dendrimer

Dendrimers with bicyclo-groups on the periphery were synthesized by the DA reaction between anthracene derivatives, which have 24, 48 and 96 end-groups, and *N*-ethylmaleimide

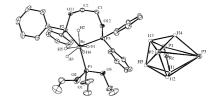


Sandra Bolaño, Jorge Bravo, Soledad García-Fontán, Jesús Castro

J. of Organomet. Chem. 667 (2003) 103

Rhenium pentahydride complexes: characterisation and protonation reactions. Crystal structure of $ReH_5L^1L^2$ ($L^1 = Ph_2PO(CH_2)_2$ -OPPh₂; $L^2 = P(OCH_3)_3$, $P(OCH_2CH_3)_3$)

Variable temperature $^1H\text{-NMR}$ studies of the classical rhenium pentahydrides $[ReH_3L^1L^2]$ show three coalescence events in the hydride region. Compounds with $L^2=P(OR)_3$ show a dodecahedral co-ordination geometry. Protonation of pentahydrides yielded the non-classical cations $[ReH_4(\eta^2\text{-}H_2)L^1L^2]^+$. Their stability increase with the number of OR groups on L^2 .



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R. Angharad Baber, Matthew L. Clarke, A. Guy Orpen, David A. Ratcliffe

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Synthesis and transition metal chemistry of 'phosphomide' ligands: a comparison of the reactivity and electronic properties of diphenyl-*P*-perfluoro-octanoyl-phosphine, *P*-acetyl-diphenylphosphine and *P*-anisoyl-diphenylphosphine. X-ray crystal structure of [RhCp*(Ph₂PC(O)CH₃)Cl₂]

The spectroscopic and catalytic properties of rhodium complexes of novel 'phosphomide' ligands, R₂PC(O)R¹, are described. Our results suggest that the nature of the group adjacent to the C=O bond, R¹ has a significant effect on the donor properties and stability of the corresponding phosphomide. The new anisoyl substituted phosphines and their metal complexes are considerably more stable to hydrolysis than acetyldiphenylphosphine or diphenyl-*P*-perfluoro-octanoylphosphine.

$$R_2$$
 P R_2 P R_2

Paloma Paredes, Marta Arroyo, Daniel Miguel, Fernando Villafañe

J. of Organomet. Chem. 667 (2003) 120

The first pyrazole molybdenum(0) complexes: cis-[Mo(CO)₄(Hdmpz)₂] crystallizes as a N-H···OC hydrogen-bonded dimer

The reactions of [Mo(CO)₄(NBD)] with two equivalents of Hpz (pyrazole) or Hdmpz (3,5-dimethylpyrazole) in hexane lead to the precipitation of *cis*-[Mo(CO)₄(Hpz)₂] or *cis*-[Mo(CO)₄(Hdmpz)₂], which are the first pyrazole complexes of molybdenum(0). In

the solid state structure of *cis*-[Mo(CO)₄(Hdmpz)₂], two molecules are self-assembled by intermolecular hydrogen bonds between the N-bound hydrogen of one Hdmpz ligand and a oxygen atom of a carbonyl group. This explains the lack of planarity observed for the nitrogen donor atom of the Hdmpz involved in the intermolecular interaction. The complexes are unstable in solution and as solids.

Glòria Esquius, Josefina Pons, Ramón Yáñez, Josep Ros, René Mathieu, Noël Lugan, Bruno Donnadieu

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Synthesis of Ru(II) complexes of the new 1-[(P-diphenyl)-2-phosphinoethyl]-3,5-dimethylpyrazole ligand and study of their reactivity toward terminal alkynes

One or two equivalents of the title ligand reacts with RuCl₂(PPh₃)₃ to give complexes **2** or **3** and the reactivity of these two complexes toward propargylic alcohols or phenyl acetylene has been studied leading to cationic allenylidene or vinylidene complexes in the case of **3**.

Masato Nanjo, Takaomi Sasage, Kunio Mochida

J. of Organomet. Chem. 667 (2003) 135

Synthesis and characterization of alkylger-masesquioxanes

Alkyl(chloro)ethoxygermanes, RGe(OEt) $_n$ -Cl $_{3-n}$ (R = i-Pr, n = 0; R = t-Bu, n = 0–3; R = cyclo-C $_6$ H $_{11}$, n = 0) were hydrolyzed with aqueous NaOH in xylene at 130–140 °C to give cage hexakis(alkylgermases-quioxane)s, (RGe) $_6$ O $_9$.

$$RGe(OEt)_{n}Cl_{3-n} \xrightarrow{H_{2}O} \begin{array}{c} R & \\ Q & \\ Q & \\ Q & \\ R & \\ Q & \\ R & \\ Q &$$

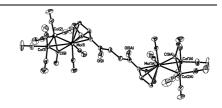
x Contents

Li-Cheng Song, Wen-Feng Zhu, Qing-Mei Hu, Hao Wu, Guang-Au Yu

J. of Organomet. Chem. 667 (2003) 143

Synthesis and characterization of organotransition metal double μ_3 -RCCo₂M (M = Mo, W) cluster complexes containing bridged dicyclopentadienyl ligands. Crystal structure of $[\mu_3$ -MeCCo₂Mo(CO)₈]₂ $[\eta^5$ -C₅H₄C(O) CH₂]₂

Three types of double cluster complexes containing bridged dicyclopentadienyl ligands $[\mu_3\text{-RCCo}_2M(CO)_8]_2[\eta^5\text{-}C_5H_4C(O)\text{-}CH_2]_2$ ($3\mathbf{a}\mathbf{-d},\ M=Mo,\ W;\ R=Me,\ Ph), \\ <math display="inline">[\mu_3\text{-RCCo}_2M(CO)_8]_2[\eta^5\text{-}C_5H_4CH_2(CH_2\ O-CH_2)_3CH_2C_5H_4\text{-}\eta^5]$ ($5\mathbf{a}\mathbf{-d},\ M=Mo,\ W;\ R=Me,\ Ph)$ and $[\mu_3\text{-MeCCo}_2M(CO)_8]_2[\eta^5\text{-}C_5H_4CH(OH)CH_2]_2$ ($6\mathbf{a},\ \mathbf{b},\ M=Mo,\ W)$ have been synthesized. The crystal structure of $3\mathbf{a}$ ($M=Mo,\ R=Me)$ is reported.

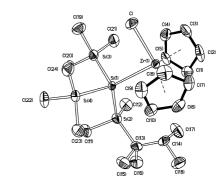


Christian Kayser, Dieter Frank, Judith Baumgartner, Christoph Marschner

J. of Organomet. Chem. 667 (2003) 149

Reactions of oligosilyl potassium compounds with Group 4 metallocene dichlorides

By the reaction of silyl anions with Group 4 metallocene dichlorides, a number of new Group 4 oligosilyl compounds have been synthesized. The silyl residues employed include the popular tris(trimethylsilyl)silyl group and sterically more encumbered derivatives. For the cases of the tris(trimethylsilyl)silyl group both the mono and the disilylated products were obtained.

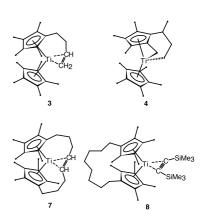


Michal Horáček, Petr Štěpnička, Jiří Kubišta, Ivana Císařová, Lidmila Petrusová, Karel Mach

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Reduction-induced double bond coordination and multiple C-H activation in fully-substituted titanocenes bearing a pendant double bond or an eight-membered hydrocarbyl *ansa*-chain

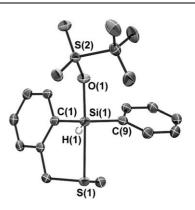
Reduction of $[TiCl_2(\eta^5-C_5Me_4R)(\eta^5-C_5Me_5)]$ $(R = CH_2CH_2CH=CH_2$ (1); $R = CH(Me)CH=CH_2$ (2)) with Mg in THF in the presence of Me₃SiC=CSiMe₃ affords different products depending on the length of the alkenyl chain: $[Ti(\eta^5-C_5Me_5)\{\eta^2:\eta^5-C_5Me_4(CH_2CH_2CH=CH_2)\}]$ (3) from 1 and $[Ti\{\eta^1:\eta^1:\eta^5-C_5Me_3(CH_2)(CH_2)]$ $(Me)CH_2CH_2$ $\{\eta^5-C_5Me_5\}$ (4) from 2. Under similar conditions but without Me₃SiC≡CSiMe₃, the saturated eight-membered chain in ansa- $[TiCl_2{\eta^5:\eta^5-C_5Me_4(CH_2)_8C_5Me_4}]$ (6) undergoes a hydrogen abstraction to give ansa-[Ti $\{\eta^2:\eta^5:\eta^5 C_5Me_4(CH_2)_3CH=CH(CH_2)_3C_5Me_4$ (7), which also results from the reduction of ansa-[Ti- $Cl_2\{\eta^5:\eta^5-C_5Me_4CH_2CH=CH(CH_2)_5C_5Me_4\}\}$ (5). The thermolysis of ansa-[Ti{η⁵:η⁵-C₅Me₄(CH₂)₈- C_5Me_4 { $(\eta^2-Me_3SiC\equiv CSiMe_3)$] (8) gives a mixture of asym and sym isomers of doubly tucked-in ansa- $[Ti\{(\eta^3:\eta^4-C_5Me_2(CH_2)_2)(CH_2)_8(\eta^5-H_2)\}$ $C_5Me_4)$] (9).



U.H. Berlekamp, A. Mix, B. Neumann, H.-G. Stammler, P. Jutzi

J. of Organomet. Chem. 667 (2003) 167

2-(Dimethylphosphinomethyl)- and 2-(methylthiomethyl)phenyl silicon compounds: higher coordination with soft donors The synthesis of silanes and of silyl triflates bearing additional aryl substituents with thiomethyl or dimethylphosphino groups in the side chain is described. The coordination behavior of the donor in these compounds has been investigated by NMR methods and by X-ray crystallography.



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Dmitry V. Moiseev, Aleksey V. Gushchin, Andrey S. Shavirin, Yury A. Kursky, Viktor A. Dodonov

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Pd-catalyzed C-arylation of unsaturated compounds with pentavalent triarylantimony dicarboxylates

 $Ar_3Sb(O_2CR)_2$ are effective and mild C-arylation reagents for the Pd-catalyzed C-arylation reaction of the unsaturated compounds under Pd catalysis. The reaction takes place at 50 °C and affords the arylated product in up to 200% yields based on $Ar_3Sb(O_2CR)_2.$

Yue-Sheng Li, Yan-Rong Li, Xiao-Fang Li

J. of Organomet. Chem. 667 (2003) 185

New neutral nickel(II) complexes bearing pyrrole-imine chelate ligands: synthesis, structure and norbornene polymerization behavior

New neutral nickel(II) complexes bearing nonsymmetric bidentate pyrrole-imine chelate ligands (4a-d), [2-(ArNCH)C₄H₃N]-Ni(PPh₃)Ph [Ar=2,6-diisopropylphenyl (a), 2-methyl-6-isopropylphenyl (b), 2,6-diethylphenyl (c), 2-tert-butylphenyl (d)], have been synthesized and used as catalysts for the vinylic polymerization of norbornene.

Joan Albert, Jaume Granell, Raquel Tavera

J. of Organomet. Chem. 667 (2003) 192

On the mechanism of the cyclopalladation reaction of benzyl-benzylidene-amine with palladium(II) acetate in acetic acid

A set of reactions that produces 2 when 1 and palladium(II) acetate react in acetic acid is reported.

Piero Frediani, Carlo Giannelli, Antonella Salvini, Sandra Ianelli

J. of Organomet. Chem. 667 (2003) 197

Ruthenium complexes with 1,1'-biisoquinoline as ligands. Synthesis and hydrogenation activity The following complexes containing the 1,1'-biisoquinoline (biisoq) ligand [Ru(biisoq)₃](PF₆)₂, [Ru₂(biisoq)₂(Cl)₄·NEt₃], [Ru₂-(biisoq)₂(CO)₄(CH₃COO)](CH₃COO and [Ru(η^6 -p-cymene)(Cl)(biisoq)]X [X: Cl, BPh₄] were synthesized and characterized. The X-ray structure of [Ru(η^6 -p-cymene)-(Cl)(biisoq)][BPh₄] was determined. Some of these complexes were catalytically active in the homogeneous hydrogenations of alkenes, alkynes and acetone in hydroalcoholic solvents.

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