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Volume 689, issue 22, 1 November 2004

Journal of Organometallic Chemistry

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Contents

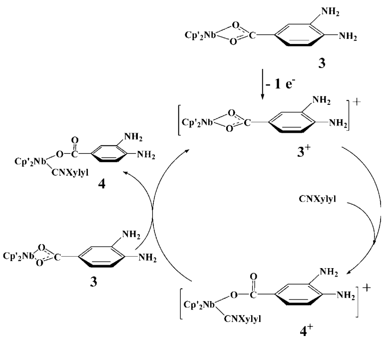
Regular papers

Laurent Salvi, Alain Vallat, Hélène Cattey, Yves Mugnier, Antonio Antinolo, Antonio Otero, Santiago García-Yuste

J. Organomet. Chem. 689 (2004) 3473

Electron-transfer-catalyzed ligand substitution of carboxylato niobocene complex induced by electrochemical oxidation

Two electron reduction of $[(\eta^5\text{-}C_5H_4SiMe_3)_2\text{-}NbCl_2]$ in the presence of 3,4-diaminobenzoic acid yield to $[Nb(\eta^5\text{-}C_5H_4SiMe_3)_2(\kappa^2\text{-}O,O\text{-}OOC(C_6H_3)(NH_2)_2)]$ **3**. An electron-transfer-catalyzed ligand substitution was induced by electrochemical oxidation of **3** with CNXylyl, leading in-stantaneously and quantitatively to $[Nb(\eta^5\text{-}C_5H_4SiMe_3)_2(\kappa^1\text{-}O\text{-}OOC(C_6H_3)(NH_2)_2)\text{-}(CN\text{-}(2,6\text{-}Me_2C_6H_3))]$ **4**. Complexes structure, their formation mechanism are discussed above electrochemical (experimental and simulated) and spectroscopy data.

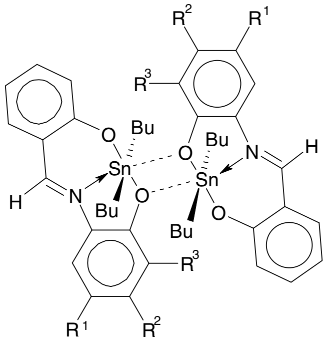


Norberto Farfán, Teresa Mancilla, Rosa Santillan, Atilano Gutiérrez, Luis S. Zamudio-Rivera, Hiram I. Beltrán

J. Organomet. Chem. 689 (2004) 3481

Preference of di-*n*-butyltin compounds to build O···Sn bonds in fused rings with five-six members

Six salicylic di-*n*-butyltin^{IV} compounds were prepared in good yields through one step reactions. The title compounds were specifically designed to show the preference for Sn···O intermolecular bond formation in five–six-membered rings containing oxygen atoms. The substituents in the salicylic fragment induce specific solution and solid state changes. Correlations of $\sigma_{Hammett}$ vs. ^{119}Sn and ^{15}N NMR indicate that the substituents serve as modulators of the O···Sn supramolecular interaction which is unique for the five-membered ring oxygen atom.

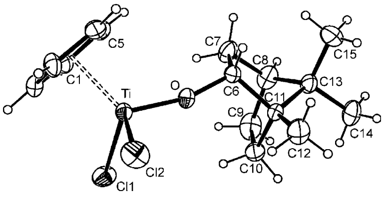


Yolanda Pérez, Sonia Morante-Zarcero, Isabel del Hierro, Isabel Sierra, Isabel López-Solera, Magda Monari, Mariano Fajardo, Antonio Otero

J. Organomet. Chem. 689 (2004) 3492

Synthesis and characterization of cyclopentadienyl/alkoxo titanium dichlorides: structural analysis of monocyclopentadienyl titanium dichlorides with ligands derived from menthol and borneol

A variety of monocyclopentadienyl alkoxide titanium dichloride and bisalkoxide titanium dichloride complexes have been prepared. The molecular structure of two half sandwich titanocene dichlorides with ligands derived from menthol and borneol, have been determined by single crystal X-ray diffraction studies.



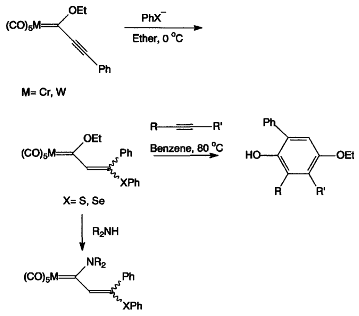
Elsevier B.V.
doi:10.1016/S0022-328X(04)00740-5

Dilip K. Sinha-Mahapatra, Debasis Hazra, Vedavati G. Puranik, Amitabha Sarkar

J. Organomet. Chem. 689 (2004) 3501

Synthesis, structure and benzannulation of chalcogen-tethered Fischer carbene complexes

Addition of PhSH-NEt$_3$ or PhSeNa to Ph—C≡C—C(OC$_2$H$_5$)=M(CO)$_5$ [M = Cr or W] afforded stable, β-chalcogenide tethered conjugated carbene complexes **3–6** as a mixture of *E*,*Z*-isomers. Only *E*-isomers of the S or Se-tethered ethoxy complexes undergo benzannulation reaction with alkynes, with loss of chalcogenide atom. Aminolysis of complexes **3–6** yielded a variety of amino carbene complexes.

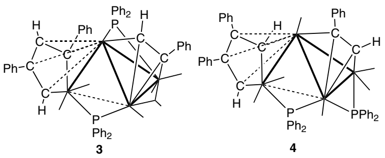


Maria Helena Araujo, Maria D. Vargas, Anthony G. Avent, Dario Braga, Fabrizia Grepioni

J. Organomet. Chem. 689 (2004) 3513

Carbon–carbon coupling on tetrahedral iridium clusters: X-ray molecular structures and multinuclear NMR studies of the two isomeric forms of [Ir$_4$(CO)$_6$(μ$_3$-η^2-HCCPh)(μ$_2$-η^4-C$_4$H$_2$Ph$_2$)(μ-PPh$_2$)$_2$]

Clusters [HIr$_4$(CO)$_9$(Ph$_2$PC≡CPh)(μ-PPh$_2$)] (**1**) and [Ir$_4$(CO)$_8$(μ$_3$-η^2-HCCPh)(μ-PPh$_2$)$_2$] (**2**) react with HC≡CPh gave two isomeric forms of [Ir$_4$(CO)$_6$(μ$_3$-η^2-HCCPh)(μ$_2$-η^4-C$_4$H$_2$Ph$_2$)(μ-PPh$_2$)$_2$] (**3** and **4**) in good yields as the only products, whose structures were established by X-ray diffraction studies.

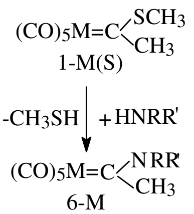


Mahammad Ali, Dipankar Maiti

J. Organomet. Chem. 689 (2004) 3520

Transition metal carbene chemistry 2: kinetic studies on the nucleophilic substitution reactions of (CO)$_5$M=C(SCH$_3$)CH$_3$ (M = Cr and W) with morpholine in aqueous acetonitrile

A kinetic study of the aminolysis of (CO)$_5$M=C(CH$_3$)(SCH$_3$) (M = Cr and W) with morpholine in aqueous acetonitrle (50%, v/v) is reported. The reaction proceeds through three steps mechanism and general base catalysis is apparent.



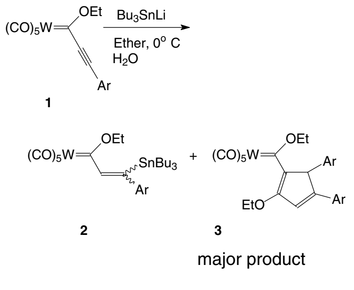
$$(CO)_5M{=}C\begin{cases}SCH_3\\CH_3\end{cases}\ \text{1-M(S)}\ \xrightarrow[-CH_3SH]{+HNRR'}\ (CO)_5M{=}C\begin{cases}NRR'\\CH_3\end{cases}\ \text{6-M}$$

Dilip K. Sinha-Mahapatra, Vedavati G. Puranik, Amitabha Sarkar

J. Organomet. Chem. 689 (2004) 3528

SET pathway in tributylstannyllithium addition to alkynyl Fischer carbene complexes

Addition of tributyltin lithium with Fischer type alkynyl carbene complexes *via* a nucleophilic pathway to produce the conjugate additon product, as well as a single electron transfer pathway to afford cyclopentannulated derivatives derived from two units of the alkyne complex. Structure of the latter was confirmed by crystal structure determination of a representative complex.

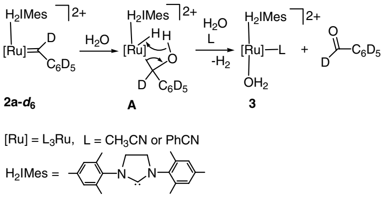


Mieock Kim, Min-Sik Eum, Moon Young Jin, Ki-Won Jun, Chul Wee Lee, Kyoung A. Kuen, Chong Hyeak Kim, Chong Shik Chin

J. Organomet. Chem. 689 (2004) 3535

Reactions of ruthenium benzylidenes with H_2O to give benzaldehyde and (aqua)-ruthenium complex

Cationic ruthenium carbene complexes, $[(RCN)_3(H_2IMes)Ru{=}CHPh](OTf)_2$ (**2**, R = CH_3 (**a**), Ph (**b**)) are obtained from the reactions of $(PCy_3)(H_2IMes)Cl_2Ru{=}CHPh$ (**1**) with CH_3CN in the presence of AgOTf. **2a** reacts with H_2O in the presence of CH_3CN to afford (aqua)ruthenium complex, $[Ru(H_2IMes)(NCCH_3)_4(H_2O)](OTf)_2$ (**3**) and benzaldehyde. Plausible reaction pathways are suggested for the degradation of ruthenium benzylidenes to give benzaldehyde on the basis of the isotope labeling experiments.

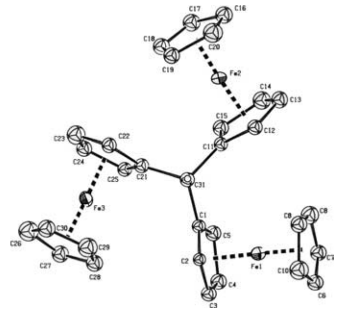


Jose Ramon Garabatos Perera, Rudolf Wartchow, Holger Butenschön

J. Organomet. Chem. 689 (2004) 3541

Synthesis and structures of some di- and triferrocenylmethane derivatives

Triferrocenylmethane derivatives were prepared including some with functionalities at the opposite cyclopentadienyl ligands. Two crystal structure analyses are presented, including that of the parent compound.

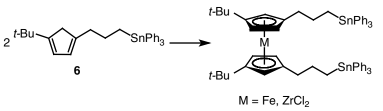


Jens Christoffers, Thomas Werner, Angelika Baro, Peter Fischer

J. Organomet. Chem. 689 (2004) 3550

Synthesis of a tin-functionalized cyclopentadiene derivative

A novel 1,3-functionalized cyclopentadienyl ligand precursor **6** is accessible by a three-step reaction including hydrostannylation. Conversion with $FeCl_2$ and $ZrCl_4 \cdot 2THF$ gave corresponding metallocenes.

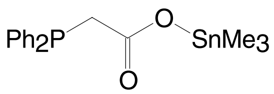


Petra Zoufalá, Róbert Gyepes, Petr Štěpnička

J. Organomet. Chem. 689 (2004) 3556

Trimethylstannyl (diphenylphosphino)acetate: a source of (diphenylphosphino)acetate ligand in the synthesis of coordination compounds

Trimethylstannyl (diphenylphosphino) acetate (**1**) reacts with $[M(cod)Cl_2]$ (M = Pd and Pt; cod = $\eta^2{:}\eta^2$-cycloocta-1,5-diene) to give $[M(Ph_2PCH_2CO_2\text{-}\kappa^2O,P)_2]$ (**2**, **3**), and with $[\{Pd(\mu\text{-}Cl)Cl(PFur_3)\}_2]$ (**4**) (Fur = 2-furyl) to afford $[SP\text{-}4\text{-}3]\text{-}[PdCl(Ph_2PCH_2CO_2\text{-}\kappa^2O,P)(PFur_3)]$ (**5**). Reactions between the dimers $[\{M(L)Cl(\mu\text{-}Cl)\}_2]$, where M/L = Rh/$\eta^5$-$C_5Me_5$ and Ru/η^6-1,4-$MeC_6H_4(CHMe_2)$, with **1** produce stable adducts: $[RhCl(\eta^5\text{-}C_5Me_5)(Ph_2PCH_2CO_2\text{-}\kappa^2O,P)] \cdot Me_3SnCl$ (**6**) and $[RuCl(\eta^6\text{-}1,4\text{-}MeC_6H_4(CHMe_2))(Ph_2PCH_2CO_2\text{-}\kappa^2O,P)] \cdot Me_3SnCl$ (**8**).



1

Béla Győri, István Lázár, Zoltán Berente, Róbert Király, Attila Bényei

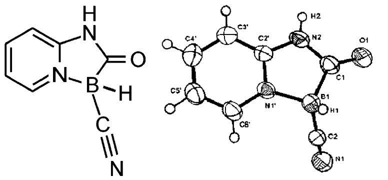
J. Organomet. Chem. 689 (2004) 3567

Synthesis and examination of amine-cyanocarboxyboranes, the boron analogues of α-cyanocarboxylic acids: X-ray structural study of the first lactam containing a boron atom in the lactam ring

Several new amine-cyanocarboxyboranes, their methyl esters (A · BH(CN)COOR) as well as three new cyanomethoxycarbonylborates having the composition of $K[BH_n(CN)_{3-n}COOMe]$ ($n = 1,2$) and $K[B(OH)(CN)_2COOMe]$ have been synthesized and their acididity and hydrolytic properties were examined. The first example of a lactam derivative containing a boron atom in the lactam ring has been synthesized and fully characterized by X-ray diffraction.

A·BH(CN)COOR (R = H, Me)
if R = H: $pK_a = 5.82 - 6.34$
hydrolysis $t_{1/2} = 0.005$ h - 400 h

$K[BH_n(CN)_{3-n}COOMe]$ (n = 1, 2)
$K[B(OH)(CN)_2COOMe]$

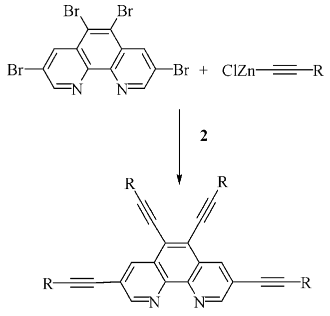


Sven Rau, Katja Lamm, Helmar Görls, Julia Schöffel, Dirk Walther

J. Organomet. Chem. 689 (2004) 3582

Bi- and trinuclear oxalamidinate complexes of palladium as catalysts in the copper-free Sonogashira reaction and in the Negishi reaction

Pd complexes with oxalamidinate bridges containing two peripheral palladium(II) centres are selective catalysts for the copper-free Sonogashira reaction. Furthermore, the complex [(acac)Pd(*oxam*)Pd(acac)] (**2**) (*oxam*: tetra(*p*-tolyl)oxalic amidinate) catalyzes the Negishi coupling of tetrabromophentanthroline with ClZn–C≡C–R resulting in the extremely selective formation of new tetra-alkynylated phenanthroline ligands.

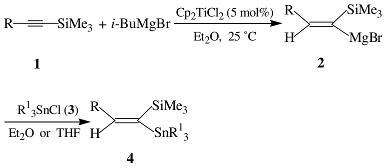


Mingzhong Cai, Wenyan Hao, Hong Zhao, Jun Xia

J. Organomet. Chem. 689 (2004) 3593

A facile stereoselective synthesis of (E)-α-silylvinylstannanes via hydromagnesiation of alkynylsilanes

(E)-α-Silylvinylstannanes have been synthesized by the hydromagnesiation reaction of alkynylsilanes, followed by the reaction with trialkylstannyl chlorides. (E)-α-Silylvinylstannanes can undergo the cross-coupling reaction with aryl iodides in the presence of a catalytic amounts of $Pd(PPh_3)_4$ and CuI to afford (Z)-1,2-disubstituted vinylsilanes in good yields.

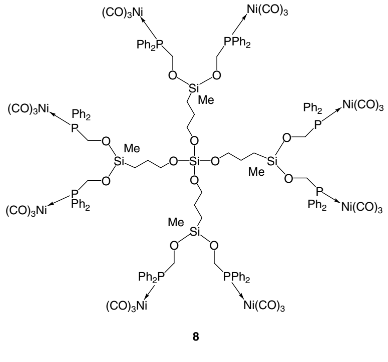


Heinrich Lang, Bettina Lühmann, Roy Buschbeck

J. Organomet. Chem. 689 (2004) 3598

Branched and dendritic metallo-carbosiloxanes with $OCH_2PPh_2ML_n$ end-grafted units

The synthesis and characterisation of branched and dendritic metallo carbosiloxanes of type $Si(OCH_2CH_2CH_2SiMe_2[OCH_2PPh_2M(CO)_n])_4$ ($n = 3$: M = Ni; $n = 4$: M = Fe; $n = 5$: M = Mo, W), $Si(OCH_2CH_2CH_2SiMe[OCH_2PPh_2Ni(CO)_3]_2)_4$ and $Me_2Si(OCH_2CH_2CH_2SiMe[OCH_2PPh_2Ni(CO)_3]_2)_2$ is described. These molecules possess phosphorus–metal carbonyl groups as end-grafted units.



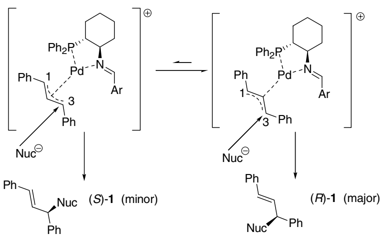
8

Shadi Dalili, Aldo Caiazzo, Andrei K. Yudin

J. Organomet. Chem. 689 (2004) 3604

Aziridine-derived iminophosphine ligands in palladium-catalyzed allylic substitution

New iminophosphines have been synthesized from (*R*,*R*)-1-amino-2-diphenylphosphino cyclohexane in good to excellent yields. The catalysts obtained from enantiomerically pure iminophosphines and $[Pd(C_3H_5)Cl]_2$ promote the enantioselective allylic substitution of 1,3-diphenyl-2-propenyl acetate with diethyl malonate as nucleophile to give (*R*)-**1** with good enantioselectivity. The air-stable complex $PdCl_2[\kappa^2\text{-}P,N\text{-}(R,R)\text{-}2\text{-}Ph_2PC_6H_{10}N{=}CHPh]$ has been prepared and structurally characterized by X-ray crystallography.



Ar = Ph, o-$OCH_3C_6H_4$, o-FC_6H_4, 2-Pyridyl, 9-anthryl, o-OHC_6H_4, o-$OH(m\text{-}OMe)C_6H_3$

Sanjay K. Singh, Manish Chandra, D.S. Pandey, M.C. Puerta, Pedro Valerga

J. Organomet. Chem. 689 (2004) 3612

Helices of ruthenium complexes involving pyridyl–azine ligands: synthesis, spectral and structural aspects

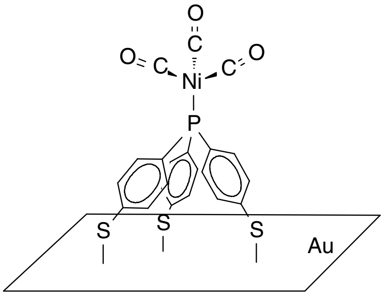
New cationic complexes $[Ru(\eta^5\text{-}C_5H_5)(EPh_3)(L)]BF_4$ [L = pyridine-2-carbaldehyde azine (paa); E = P, **1**; E = As, **2**; E = Sb, **3**] and κ^1 bonded dppm complexes $[Ru(\eta^5\text{-}C_5H_5)(\kappa^1\text{-}dppm)(L)]BF_4$ [L = paa **4;** L = *p*-phenylene-bis(picoline)aldimine (pbp) **5**] containing both group V donor and pyridyl–azine ligand are reported. Weak interaction studies revealed that inter- and intra-molecular C–H···X (X = O, F, Cl, π) and π–π interactions in the complexes **1** and **4** lead to helical structures.

Fabio Ragaini, Luca Lunardi, Diego Tomasoni, Vittoria Guglielmi

J. Organomet. Chem. 689 (2004) 3621

Synthesis of triarylphosphines having *para* –SH and –SMe groups. Preparation of their complexes and formation of a monolayer on a gold surface

Several new aromatic phosphines bearing –SH or –SMe groups directly linked to the aromatic ring and some of their complexes have been obtained. Interaction of $Ni(CO)_3(P(4\text{-}HSC_6H_4)_3)$ with a gold lamina afforded a monolayer, which was characterized by reflection–absorption IR spectroscopy.



Note

Rodrigo L.O.R. Cunha, Álvaro T. Omori, Priscila Castelani, Fabiano T. de Toledo, João V. Comasseto

J. Organomet. Chem. 689 (2004) 3631

One-pot synthesis of aryl butyl tellurides from tellurium tetrachloride and activated aromatics through a solventless step

Aryltellurium trichlorides were prepared by a solventless reaction of $TeCl_4$ with activated aromatics. A reduction/alkylation sequence transformed the crude aryltellurium trichlorides into aryl butyl tellurides.