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Contents

Communication

SEVIER

Hélène Lebel, Chehla Ladjel, Francine Bélanger-Gariépy, **Frank Schaper**

I. Organomet. Chem. 693 (2008) 2645

Redetermination of the O–O bond length in the dioxygen-adduct of Vaska's complex

Solid state structural studies were performed with $(Ph_3P)_2IrCl(CO)(O_2)$ and the 0-0 length redetermined to be 1.47 (1) Å in contrast to results reported earlier.



Regular Papers

Sucharita Basak, Deepak Chopra, Kajal Krishna Rajak

J. Organomet. Chem. 693 (2008) 2649

Synthesis, structure and characterization of *fac*-[Re(CO)₃]⁺ complexes derived from hydrazone Schiff bases: DFT-TDDFT investigation on electronic structures

The syntheses, structural and spectroscopic characterization of the complexes of general formula [ReL(CO)₃Cl] bearing bifunctional hydrazone Schiff base ligand L are presented in this paper. The solid-state structure of the compound is involved in a secondary interaction in lattice forming a supramolecular array. The complexes were characterized by different spectroscopic studies. The physiochemical properties of the complexes were studied using density functional theory.



Shariff E. Kabir, Faruque Ahmed, Shishir Ghosh, Mohammad R. Hassan, Muhammad S. Islam, Ayesha Sharmin, Derek A. Tocher, Daniel T. Haworth, Sergev V. Lindeman. Tasneem A. Siddiquee, Dennis W. Bennett, Kenneth I. Hardcastle

I. Organomet. Chem. 693 (2008) 2657

Reactions of rhenium and manganese carbonyl complexes with 1,8-bis(diphenylphosphino)naphthalene: Ligand chelation, C-H and C-P bond-cleavage reactions

Reactions of $[Re_2(CO)_8(MeCN)_2]$ and [Mn₂(CO)₈(MeCN)₂] with 1,8-bis (diphenylphosphino)naphthalene (dppn) are described. Treatment of [Re₂(CO)₈(MeCN)₂] with dppn furnished three mono-rhenium complexes fac-[Re(CO)₃(κ^1 : η^1 -PPh₂C₁₀H₆)-(PPh₂H)] (1), fac-[Re(CO)₃{ κ^1 : κ^1 : η^1 -(O)PPh₂- $C_{10}H_6(O)PPh(C_6H_4)$] (2) and fac-[ReCl(CO)₃-(PPh₂C₁₀H₆PPh₂)] whereas two dinuclear compounds $[Mn_2(CO)_8(\kappa-PPh_2)_2]$ (4) and $[Mn_2(CO)_9[\kappa^1-PPh_2(C_{10}H_7)]]$ (6) and the mononuclear fac-[MnCl(CO)₃(PPh₂C₁₀H₆-PPh₂)] (5) were obtained from the [Mn₂- $(CO)_8(MeCN)_2$] with dppn.





Renquan Zeng, Xiangkai Fu, Yan Sui, Xinbin Yang, Meidan Sun, Jingrong Chen

J. Organomet. Chem. 693 (2008) 2666

Synthesis, characterization and intercalation property of layered zirconium benzylamino-*N*,*N*-dimethylphosphonate phosphate materials Layered zirconium benzylamino-*N*,*N*-dimethylphosphonate phosphate (ZBMPA) was prepared by the reaction of zirconyl chloride with benzylamino-*N*,*N*-dimethylphosphonic acid (H₂BMPA) and H₃PO₄ in the presence of HF. The intercalation of ZBMPA with *n*-alkylamines was primarily investigated. These materials were characterized by elemental analysis, ICP, XRD, SEM, FT-IR, Raman spectra, TG and DSC.

 $\begin{array}{c} 1\\ C_{6}H_{5}CH_{2}N(CH_{2}PO_{3}H_{2})_{2} & \overbrace{}\\ H_{2}BMPA\\ Zr(HPO_{4})_{2-2x}(C_{6}H_{5}CH_{2}N(CH_{2}PO_{3})_{2})_{x} \cdot n H_{2}O & \overbrace{}\\ ZBMPA\\ ZBMPA & ZBMPA - Alkylamine \end{array}$

Federica Bertolotti, Giuliana Gervasio, Domenica Marabello, Enrico Sappa, Andrea Secco

J. Organomet. Chem. 693 (2008) 2673

Reactions of $Fe_3(CO)_{12}$ with 2-methyl-3butyn-1-ol and 3-pentyn-1-ol under basic methanolic conditions: The crystal structures of $Fe_2(CO)_6(\mu$ -CO)(μ - η^2 -[HCC{=C(CH_3)_2}C(=O)(OCH_3)]) and of (μ -H)Fe_3(CO)₉(μ_3 - η^3 -[H₃CCC(CH₂)(CH₂)CO]) The reactions of $Fe_3(CO)_{12}$ with 2-methyl-3-butyn-1-ol and 3-pentyn-1-ol in CH₃OH/ KOH solution lead, respectively, to the binuclear $Fe_2(CO)_6(\mu$ -CO)[HCC(=CMe_2)-C(=O)OMe] and to the trinuclear hydridic complex (μ -H)Fe₃(CO)₉[MeCC(CH₂)(CH₂)-CO]. The ligands of the complexes are formed respectively by coupling of a coordinated CO with a methoxy group and by coupling of CO with a deoxygenated alkynic residual.



Aichen Wang, Hongjian Sun, Liang Wang, Xiaoyan Li

J. Organomet. Chem. 693 (2008) 2679

Formation of niobium and tantalum ylide complexes

The hexa-coordinate ylide adducts complexes 5 (CpNbCl₄(CH₂= $P(NEt_2)_3$)), 6 (Cp^{*}NbCl₄(CH₂= $P(NEt_2)_3$)) and 8 (Cp^{*}TaCl₄(CH₂= $P(NEt_2)_3$)) with a pseudo-octahedral geometry were structurally characterized by X-ray diffraction. Compound 4 (Cp^{*}TaCl₄) reacted with three molar equivalent of the phosphorus ylide to form an ionic complex 9 ([H₃C-P-(NEt₂)₃][Cp^{*}TaCl₅]). The possible formation mechanism of compound 9 has been discussed.



Vladimir B. Golovko, Martin J. Mays, Gregory A. Solan

J. Organomet. Chem. 693 (2008) 2683

Study of ferrocenyl-substituted $Co_2(CO)_6$ bispropargylic alcohol complexes as substrates for the formation of chains and macrocycles A series of ferrocenyl-containing but-2yne-1,4-diols have been prepared and their alkynic moieties complexed with dicobalt hexacarbonyl units; acid-catalysed substitution of the hydroxyl groups in the resultant complexes by thiol-based nucleophiles leads to chain, macrocyclic and dehydration products.



Nermin Biricik, Feyyaz Durap, Cezmi Kayan, Bahattin Gümgüm, Nevin Gürbüz, İsmail Özdemir, Wee Han Ang, Zhaofu Fei, Rosario Scopelliti

J. Organomet. Chem. 693 (2008) 2693

Synthesis of new aminophosphine complexes and their catalytic activities in C-C coupling reactions

Two new aminophosphines, benzyl-N(Ph₂P)₂ and 2-picolyl-N(Ph₂P)₂, have been synthesized. Complexes [benzyl-N(Ph₂P)₂]-MCl₂ and [2-picolyl-N(Ph₂P)₂]MCl₂, where M = Pd, Pt, were obtained by the reaction of the aminophosphines with MCl₂(cod). With a view to develop useful catalysts for C–C coupling reactions, the palladium complexes were further investigated as potential catalyst.



John R. Moss, Siyabonga Ngubane, Akella Sivaramakrishna, Banothile C.E. Makhubela, John E. Bercaw, Jay A. Labinger, Michael W. Day, Lawrence M. Henling, Hong Su

J. Organomet. Chem. 693 (2008) 2700

C-H activation of benzene with $Cp^*Ru(CO)_2CH_3$

The complex Cp^{*}Ru(CO)₂CH₃ can activate the C–H bond of benzene to give the new Ru–phenyl complex Cp^{*}Ru(CO)₂C₆H₅ and methane. The Ru–phenyl complex has also been prepared by another route and successfully characterized.

David C. Graham, Michael I. Bruce, Gregory F. Metha, John H. Bowie, Mark A. Buntine

J. Organomet. Chem. 693 (2008) 2703

Regioselective control of the nickelmediated coupling of acetylene and carbon dioxide – A DFT study The regioselectivity of the nickel-mediated coupling of various asymmetric alkynes with carbon dioxide has been investigated using density functional theory (DFT). In general, the kinetically favoured 2-R-substituted nickelacycles are predicted to dominate due to the unfavourable steric interaction in the transition structure on the pathway leading to the thermodynamically preferred 3-R-substituted product.



C₆H₆



Habib Firouzabadi, Nasser Iranpoor, Abbas Ali Jafari, Mohammad Reza Jafari

J. Organomet. Chem. 693 (2008) 2711

Iron(III) trifluoroacetate $[Fe(F_3CCO_2)_3]$ as an easily available, non-hygroscopic, noncorrosive, highly stable and a reusable Lewis Acid catalyst: Efficient *O*-silylation of α -hydroxyphosphonates, alcohols and phenols by hexamethyldisilazane (HMDS) under solvent-free conditions O-Silylation of hydroxyl groups of α -hydroxyphosphonates, primary, secondary tertiaryalcohols and phenols with HMDS was achieved in high to excellent yields using iron(III) trifluoroacetate [Fe(F₃CCO₂)₃] as an easily available and a cost effective, non-hygroscopic, non-corrosive, highly stable and an efficient catalyst under solvent-free conditions.

ROH $\xrightarrow{\text{HMDS, Fe}(F_3\text{CCO}_2)_3}$ ROSiMe₃

C. John McAdam, Stephen C. Moratti, Brian H. Robinson, Jim Simpson

J. Organomet. Chem. 693 (2008) 2715

A *neo*-pentyl-ferrocene motif for the synthesis of electroactive polyesters

The article contains synthesis and characterisation details of new ferrocenyl substituted diols suitable for polyesterification. Three crystal structure determinations are reported including a diester model of the polymer repeating unit. Electrochemistry of the ferrocene molecules is discussed, and an example given of a successful polymerisation with terephthalic acid.



Jérémie D.A. Pelletier, John Fawcett, Kuldip Singh, Gregory A. Solan

J. Organomet. Chem. 693 (2008) 2723

From symmetrical to unsymmetrical bimetallic nickel complexes bearing aryllinked iminopyridines; synthesis, structures and ethylene polymerisation studies Both symmetrical and unsymmetrical aryllinked iminopyridines have been prepared and their bimetallic nickel(II) halide complexes synthesised; crystallisation from DMF results in adduct formation (either as salts or neutral complexes). All dinickel systems on treatment with MAO are active for the polymerisation of ethylene affording low molecular weight branched polyethylene.



Richard D. Adams, Erin M. Boswell, Burjor Captain, Shaobin Miao, Chad Beddie, Charles Edwin Webster, Michael B. Hall, Naresh S. Dalal, Narpinder Kaur, David Zipse

J. Organomet. Chem. 693 (2008) 2732

Disulfido iron-manganese carbonyl cluster complexes: Synthesis, structure, bonding and properties of the radical CpFeMn₂- $(CO)_7(\mu_3-S_2)_2$

$S_2)_2$ (2) and $Cp_3Fe_3Mn(CO)_4(\mu_3-S_2)_2(\mu_3-S)$ (3) were obtained by the treatment of $[CpFeMn(CO)_5(\mu_3-S_2)]_2$ (1) with CO in the presence of room light. Compound 2 is paramagnetic. It contains two triply bridging disulfido ligands on opposite sides of an open FeMn₂ triangular cluster. The electronic structure of 2 was established by molecular orbital calculations. Compound 3 contains three iron atoms and one manganese atom with two triply bridging disulfido ligands and one triply bridging sulfido ligand.

Two new compounds $CpFeMn_2(CO)_7(\mu_3 -$



Adriana Esparza-Ruiz, Adrián Peña-Hueso, Iris Ramos-García, Angelina Flores-Parra, Rosalinda Contreras

J. Organomet. Chem. 693 (2008) 2739

Heptacoordinated diphenyllead; hexa- and pentacoordinated triphenyllead and tin compounds derived from 5H-benzimidazo[1,2-c]quinazoline-6-thione Compounds were studied by ¹¹⁹Sn and ²⁰⁷Pb NMR and X-ray diffraction. The ligand forms a four-membered ring. Hypervalent Sn and Pb atoms were obtained by Lewis bases coordination. A heptacoordinated diphenyllead compound with pentagonal bipyramidal geometry with *cis-mer* configuration and two S atoms lying very close together, was obtained.



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N-versus *O*-silylation in *cis*-[(^tBuHN)O=P- $(\mu$ -N^tBu)₂P=O(NH^tBu)] and [Me₂Si(μ -N^tBu)₂P=O(NHPh)]. Solid-state structures of their silylation products, of co-crystalline *cis*-[(^tBuHN)O=P(μ -N^tBu)₂P=O(NH^tBu)], and of {[Me₂Si(μ -N^tBu)₂P=O(N(SiMe₃)Ph)]VCl₃}

Despite silicon's well known affinity for oxygen, the silylations of two closely-related cyclic *N*,*N*,*N*-phosphoramidates furnished *N*- and *O*-silylated derivatives, respectively, as thermodynamic products. The syntheses and solid-state structures of these molecules and those of a vanadium(III) complex of one of them are presented.



Samar Eid, Thierry Roisnel, Dominique Lorcy

J. Organomet. Chem. 693 (2008) 2755

Titanocene complexes of the *N*-methyl and *N*-phenyl-1,3-thiazoline-2-thione-4,5-dithiolates and 4,5-diselenolate ligands

Cp₂Ti(R-thiazdt) and Cp₂Ti(R-thiazds) complexes were prepared following three synthetic approaches through the reaction of 1,3-thiazoline-2-thione-4,5-dithiolate (Rthiazdt) and -4,5- diselenolate ligand (Rthiazds) with Cp₂TiCl₂. The complexes were characterized and compared using X-ray structure determinations, cyclic voltammetry, UV–Vis spectroscopy, as well as variable-temperature ¹H NMR experiments.



 $Cp_2Ti(R-thiazdt), X = S$ $Cp_2Ti(R-thiazds), X = Se$

Leah N. Appelhans, Christopher D. Incarvito, Robert H. Crabtree

J. Organomet. Chem. 693 (2008) 2761

Synthesis of monodentate bis(N-heterocyclic carbene) complexes of iridium: Mixed complexes of abnormal NHCs, normal NHCs, and triazole NHCs A stepwise synthetic route to mixed monodentate bis-NHC complexes of Ir(I) is described, allowing the synthesis of complexes with two NHCs having different electronic and steric characteristics and applicable to normal, abnormal, and triazole-derived NHC ligands. The complexes do not disproportionate, consistent with the NHC ligands not being labile.



Xiu-Li Wang, Hong-Yan Lin, Guo-Cheng Liu, Hai-Yan Zhao, Bao-Kuan Chen

J. Organomet. Chem. 693 (2008) 2767

Synthesis, structures, electrochemistry and electrocatalysis of two novel copper(II) complexes constructed with aromatic polycarboxylates and dipyrido[3,2-d:2',3'-f]quinoxaline

Hydrothermal synthesis, crystal structures, electrochemical and electrocatalytical properties of two novel coordination polymers constructed with mixed ligands of aromatic polycarboxylate (1,3-BDC or BTC) and dipyrido[3,2-d:2',3'-f]quinoxaline (Dpq): $[Cu_3(1,3-BDC)_4(Dpq)_2]$ (1) and $[Cu_2(BTC)(OH)-(Dpq)_2] \cdot H_2O$ (2).

Two novel coordination polymers $[Cu_3(1,3-BDC)_4(Dpq)_2]$ (1) and $[Cu_2(BTC)(OH)-(Dpq)_2] \cdot H_2O$ (2) have been obtained from hydrothermal reactions. Two copper complexes exhibit one-dimensional chain for 1 and two-dimensional layer structure for 2, respectively. And the adjacent chains in 1 and the adjacent layers in 2 are further linked by π - π stacking interactions to form three-dimensional supramolecular framework. Moreover, the copper(II) complexes bulk-modified carbon paste electrodes (Cu-CPEs: 1-CPE and 2-CPE) have good electrocatalytic activities toward the reduction of nitrite and bromate in 0.1 M pH 2 phosphates bulfer solution.



Cristian Ledesma, Angel Alvarez-Larena, Joan Suades

J. Organomet. Chem. 693 (2008) 2775

Rhenium carbonyl compounds with (diphenyl)phosphinoalkynes and a sterically hindered phosphinoalkyne

Phosphinoalkynes $Ph_2PC \equiv CR$ (X = Ph; R = Ph, Tol, ^{*t*}Bu) react with $[ReBr_3(CO)_3]^{2-}$ and the hydrolyzed complex $[ReBr_{3-n^-}(CO)_3(H_2O)_n]^{n-2}$ to form anionic *fac*-[Re-Br₂(CO)₃X₂C \equiv CR]⁻ or neutral *fac*-[Re-Br(CO)₃(X₂PC \equiv CR)₂] complexes depending on the starting precursor and independently of metal/phosphine ratio used. The same behaviour has been observed using the phosphinoalkyne with bulky groups (*o*-Tol)₂PC \equiv CPh.



Ole Winkelmann, Christian Näther, Ulrich Lüning

J. Organomet. Chem. 693 (2008) 2784

Synthesis, structure and catalytic activity of a bimacrocylic NHC palladium allyl complex

The bimacrocylic NHC palladium allyl complex 4 was synthesized by transmetalation from NHC silver complex 2. Palladium complex 4 was characterized by single-crystal X-ray analysis and spectroscopic methods. The catalytic activity of 4 was proven in Mizoroki–Heck and Suzuki– Miyaura cross-coupling reactions.



Abdallah Hamze, Olivier Provot, Jean-Daniel Brion, Mouâd Alami

J. Organomet. Chem. 693 (2008) 2789

Xphos ligand and platinum catalysts: A versatile catalyst for the synthesis of functionalized β -(*E*)-vinylsilanes from terminal alkynes

Totally regioselective hydrosilylation of functionalized terminal arylalkynes was achieved using PtO_2 or $PtCl_2$ associated with the stable and bulky Xphos ligand with various silanes. Regardless of the electronic nature of the substituents on the aromatic ring, a single β -(*E*)-vinylsilanes was obtained in excellent yields. This chemo- and regioselective procedure is general as it was successfully applied to terminal aliphatic alkynes.



R = 4-CNC₆H₄, 4-MeO₂CC₆H₄, 2-MeO₂CC₆H₄, 4-MeC₆H₄, 3-MeOC₆H₄, 4-BrC₆H₄, 2-pyridyl, C₄H₁₁, HOCH₂, C₈H₁₁,CH(OH)CH₂, (EtO)₂CH...

Jiawei Hu, Guanghui Zhang, Hung-Hsin Shih, Xiaoqing Jiang, Peipei Sun, Chien-Hong Cheng

J. Organomet. Chem. 693 (2008) 2798

Synthesis of a highly phosphorescent emitting iridium(III) complex and its application in OLEDs A strong phosphorescent iridium complex $(biio)_2 Ir(acac)$ with a rigid cyclometalated ligand was designed and conveniently synthesized. The maximum brightness of the electroluminescent device based on this complex reached to 79640 cd m⁻² with an external quantum efficiency of 12.1% and a maximum current efficiency of 31.7 cd A⁻¹.





M.P. García Armada, J. Losada, F.J. López-Villanueva, H. Frey, B. Alonso, C.M. Casado

J. Organomet. Chem. 693 (2008) 2803

Electrochemical and bioelectrocatalytical properties of novel block-copolymers containing interacting ferrocenyl units In this new work, we describe the electrochemical characterization of three different polystyrene-*b*-polybutadiene block-copolymers functionalized with ferrocenyl units electronically communicated and the bioelectrocatalytical and analytical properties of electrodes modified with them in the nicotinamide dinucleotide (NADH) and glucose oxidase (GOx) oxidations. We also describe an amperometric biosensor for glucose, prepared by electrostatic immobilization of glucose oxidase onto a platinum electrode modified with one of the ferrocenyl block-copolymers as an example.



Note

John H. Brownie, Michael C. Baird

J. Organomet. Chem. 693 (2008) 2812

Synthesis, properties and structures of methyldiphenylphosphonium 1-indenylide, $1-C_9H_6PMePh_2$, and its chiral tricarbonyl-chromium(0) complex $Cr(\eta^5-1-C_9H_6-PMePh_2)(CO)_3$

The indenyl-derived ylide, methyldiphenylphosphonium 1-indenylide, 1- $C_9H_6PMePh_2$, reacts with $Cr(CO)_6$ to form the corresponding chromium(0) complex, $Cr(\eta^5-1-C_9H_6PMePh_2)(CO)_3$. Both new compounds have been characterized spectroscopically and crystallographically, and $Cr(\eta^5-1-C_9H_6PMePh_2)(CO)_3$ is shown to exhibit planar chirality resulting from coordination of the prochiral aromatic ligand.







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