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### Contents

### Regular papers

# Michael Ardon, Graeme Hogarth, Daniel T.W. Oscroft

J. Organomet. Chem. 689 (2004) 2429

Organometallic chemistry in a conventional microwave oven: the facile synthesis of group 6 carbonyl complexes

A range of microwave-enhanced reactions of group 6 metal carbonyls is presented. Molybdenum hexacarbonyl is particularly well suited to this procedure, with reactions generally proceeding cleanly and without need for an inert atmosphere. Key examples include the high yield syntheses of cis-[Mo(CO)<sub>4</sub>(pip)<sub>2</sub>], cis-[Mo(CO)<sub>4</sub>(diphopshine)] (diphosphine = dppm, dppe), [CpMo(CO)<sub>3</sub>]<sub>2</sub> and [Mo<sub>2</sub>(µ-O<sub>2</sub>CMe)<sub>4</sub>]. Reactions of chromium and tungsten hexacarbonyls are less impressive, nevertheless, [Cr(CO)<sub>5</sub>Cl][NEt<sub>4</sub>], [( $\eta^6$ -C<sub>6</sub>H<sub>5</sub>OMe)-Cr(CO)<sub>3</sub>] and cis-[W(CO)<sub>4</sub>(pip)<sub>2</sub>] have all been prepared via this method.

# Mingzhong Cai, Yizheng Huang, Hong Zhao, Rongli Zhang

J. Organomet. Chem. 689 (2004) 2436

Carbonyl allylation of aldehydes catalyzed by a silica-supported poly-γ-diphenylarsinopropylsiloxane palladium(0) complex The silica-supported poly- $\gamma$ -diphenylarsino-propylsiloxane palladium(0) complex ('Si'– As–Pd(0)) shows high catalytic activity for the allylation of aldehydes with allylic chlorides via the formation of  $\pi$ -allylpalladium complexes, using SnCl<sub>2</sub> as reducing agent. This polymeric palladium complex can be recovered and reused.

#### Wolfgang A. Herrmann, Sabine K. Schneider, Karl Ofele, Masato Sakamoto, Eberhardt Herdtweck

J. Organomet. Chem. 689 (2004) 2441

First silver complexes of tetrahydropyrimid-2-ylidenes

The syntheses and characterizations of homoleptic silver(I) carbene complexes of the type LAgCl (3) and  $[L_2Ag]^+_2[Pd_2Cl_6]^{2-}$  (4) (L = dimesityltetrahydropyrimid-2-ylidene) are described. Unlike other silver–carbene complexes, 3 does not undergo ligand exchange and can thus not be used for carbene transfer to other metals.

iv Contents

#### Mark O. Liu, Andrew Teh Hu

J. Organomet. Chem. 689 (2004) 2450

Microwave-assisted synthesis of phthalocyanine–porphyrin complex and its photoelectric conversion properties A soluble phthalocyanine–porphyrin complex (Lu(TBPor)Pc) has been quickly prepared by microwave irradiation and Lu(TBPor)Pc has been demonstrated to have better photoelectric conversion properties than porphyrin (Lu(TBPor)OAc), phthalocyanine (H<sub>2</sub>(TBPc)), and Lu(TBPor)OAc/H<sub>2</sub>(TBPc) blend. Moreover, a n-type photoconductor (PDHEP) and TiO<sub>2</sub> have been blended into lab-made photoelectric cell with Lu(TBPor)Pc to exhibit the best photoelectric conversion effect with a short-circuit photocurrent (691.3  $\mu$ A/cm²) among all lab-made cells under illumination of white light (1.2 mW/cm²).

### Bruno Therrien, Ludovic Vieille-Petit, Julie Jeanneret-Gris, Petr Štěpnička, Georg Suss-Fink

J. Organomet. Chem. 689 (2004) 2456

Ruthenium(II) complexes with ferrocenemodified arene ligands: synthesis and electrochemistry A series of arene–ruthenium complexes of the general formula [RuCl<sub>2</sub>{ $\eta^6$ -C<sub>6</sub>H<sub>5</sub>(CH<sub>2</sub>)<sub>2</sub>R}L] with R = OH, CH<sub>2</sub>OH, OC(O)Fc, CH<sub>2</sub>O-C(O)Fc (Fc = ferrocenyl) and L = PPh<sub>3</sub>, (diphenylphosphino)ferrocene, or bridging 1,1′-bis(diphenylphosphino)ferrocene, have been synthesized. The electrochemical behavior of selected representative compounds has been studied. Complexes with ferrocenylated side arms display two *independent* reversible one-electron waves whereas, introduction of a ferrocenylphosphine onto the ruthenium is reflected by an additional reversible one-electron wave.

César Herrera-Álvarez, Valente Gómez-Benítez, Rocío Redón, Juventino J. García, Simón Hernández-Ortega, Rubén A. Toscano, David Morales-Morales

J. Organomet. Chem. 689 (2004) 2464

[1,1'-Bis(diphenylphosphino)] ferrocene] palladium(II) complexes with fluorinated benzenethiolate ligands: examination of the electronic effects in the solid state, solution and in the Pd-catalyzed Heck reaction with the catalytic system  $[Pd(dppf)(SR_F)_2]$ 

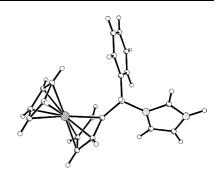
A series of palladium complexes of the type  $[Pd(dppf)(SR_F)_2]$  have been synthesized and the effect of the fluorinated thiolates in the properties of these complexes examined in both the solid state and in solution. Preliminary Heck coupling reaction experiments indicate that the presence of electron-with-drawing substituents may favor higher yields in the Pd catalyzed Heck reaction using  $[Pd(dppf)(SR_F)_2]$  as catalysts.

Lubov' V. Snegur, Alexander A. Simenel, Yury S. Nekrasov, Elena A. Morozova, Zoya A. Starikova, Svetlana M. Peregudova, Yuliya V. Kuzmenko, Valery N. Babin, Larissa A. Ostrovskaya, Natalia V. Bluchterova, Margarita M. Fomina

J. Organomet. Chem. 689 (2004) 2473

Synthesis, structure and redox potentials of biologically active ferrocenylalkyl azoles

The syntheses, structures, electrochemical properties of the series of ferrocenylalkyl azoles have been studied. Compounds were investigated by the method of cyclic voltametry. All of them exhibited a reversible one-electron oxidation–reduction wave owing to the ferrocene–ferrocenium redox couple. The X-ray determination of molecular structures of three compounds was carried out. Antitumor activity of 1-(ferrocenylmethyl)benzimidazole against solid tumors was studied.



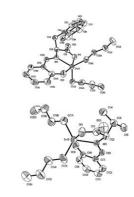
Contents

#### Han-Dong Yin, Qi-Bao Wang, Sheng-Cai Xue

J. Organomet. Chem. 689 (2004) 2480

Synthesis and structural characterization of diorganotin(IV) esters of salicylidene-amino acids

Eight diorganotin esters of salicylidene-L-tryptophan(Sal-T) and salicylidene-L-valine(Sal-V), [ $(n\text{-Bu})_2\text{Sn}(\text{Sal-T})$ ] (1), [ $(n\text{-Bu})_2\text{Sn}(\text{Sal-T})$ ] (2), [ $(n\text{-Bu})_2\text{Sn}(\text{Sal-T})$ ] (3), [ $(n\text{-Bu})_2\text{Sn}(\text{Sal-V})$ ] (4), [ $(n\text{-Bu})_2\text{Sn}(\text{Sal-T})$ ] (5), [ $(n\text{-Bu})_2\text{Sn}(\text{Sal-V})$ ] (6), [ $(n\text{-Cl})_2\text{Sn}(\text{Sal-V})$ ] (7) and [ $(n\text{-Cl})_2\text{Sn}(\text{Sal-V})$ ] (8) have been synthesized and characterized by elemental analysis, IR and  $^1\text{H}$ ,  $^1\text{C}$  and  $^1\text{H}$ Sn NMR. The crystal structures of compounds 1 and 2 have been determined by X-ray single crystal diffraction.

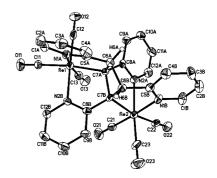


### Rubén A. Machado, David Rivillo, Alejandro J. Arce, Lindora D'Ornelas, Ysaura De Sanctis, Reinaldo Atencio, Teresa González, Esperanza Galarza

J. Organomet. Chem. 689 (2004) 2486

Synthesis and molecular structure of  $[Re_2(\mu:\eta^6-C_{24}H_{18}N_4)(CO)_6]$  containing the *rtct*-tetrakis(2-pyridyl)cyclobutandiyl ligand, derived from the reaction of  $[Re_2(CO)_8-(MeCN)_7]$  and 1,2-bis(2-pyridyl)ethene

The facile [2+2] cycloaddition of 1,2-bis(2-pyridyl)ethene mediated by  $[Re_2(CO)_8-(CH_3CN)_2]$  leads to the complex  $[Re_2(\mu;\eta^6-C_24H_{18}N_4)(CO)_6]$  where the new ligand acts as a 10-electron donor. The new derivative was fully characterised by spectroscopic assignments and X-ray diffraction studies.



#### Ali Naghipour, Seyyed Javad Sabounchei, David Morales-Morales, Simón Hernández-Ortega, Craig M. Jensen

J. Organomet. Chem. 689 (2004) 2494

Synthesis of a new class of unsymmetrical PCP' pincer ligands and their palladium (II) complexes: X-ray structure determination of PdCl{C<sub>6</sub>H<sub>3</sub>-2-CH<sub>2</sub>PPh<sub>2</sub>-6-CH<sub>2</sub>PBu<sup>1</sup><sub>2</sub>}

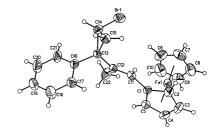
The unsymmetrical PCP' pincer ligands  $\{C_6H_4\text{-}1\text{-}CH_2PPh_2\text{-}3\text{-}CH_2PBu_2^t\}$  and  $\{C_6\text{-}H_4\text{-}1\text{-}CH_2PPh_2\text{-}3\text{-}CH_2PPr_2^t\}$  and their palladium complexes  $PdCl\{C_6H_3\text{-}2\text{-}CH_2PPh_2\text{-}6\text{-}CH_2PBu_2^t\}$  and  $PdCl\{C_6H_3\text{-}2\text{-}CH_2PPh_2\text{-}6\text{-}CH_2PPr_2^t\}$  have been synthesized in good yields. Preliminary results concerning the application of these complexes in the coupling reactions of halo-benzenes with styrene (Heck reaction) are reported.

### Méndez I. Daniel, Klimova Tatiana, Klimova Elena, Hernández O. Simon, Perez F. Javier, G. Marcos Martnez

J. Organomet. Chem. 689 (2004) 2503

Synthesis of di- and monobromo(ferrocenylvinyl)cyclopropanes

The synthesis of vinyl *gem*-dibromo-(ferrocenyl)cyclopropane compounds was carried out from 1,3-dienes. Vinyl monobromocyclopropanes were obtained selectively by a reductive debromination with ethyl magnesium bromide.



vi Contents

#### Wai-Him Kwok, Guo-Liang Lu, Clifton E.F. Rickard, Warren R. Roper, L. James Wright

J. Organomet. Chem. 689 (2004) 2511

Nucleophilic substitution reactions at the Si–Cl bonds of the dichloro(methyl)silyl ligand in five- and six-coordinate complexes of ruthenium(II) and osmium(II)

The dichloromethylsilyl ligand in  $M(SiCl_2-Me)Cl(CO)(PPh_3)_2$  (M=Ru or Os) undergoes nucleophilic substitution reactions with the OH functions of  $H_2O$  or EtOH, and with the NH function of 2-aminopyridine. The last reaction produces a complex with a tridentate "NSiN" ligand.

#### Wade J. Mace, Lyndsay Main, Brian K. Nicholson, Daniel J. van de Pas

J. Organomet. Chem. 689 (2004) 2523

Manganese carbonyl-mediated reactions of azabutadienes with phenylacetylene, methyl acrylate and other unsaturated molecules PhCH<sub>2</sub>Mn(CO)<sub>5</sub> reacts with 1,4-di-aryl-1-aza-1,3-butadienes in the presence of unsaturated substrates to give products based on a cyclomanganated intermediate.

$$\begin{array}{c|c} PhCH_2Mn(CO)_5 \\ \hline \\ PhC = CH \\ \hline \\ PhNCO \\ \hline \end{array}$$

#### Mingzhong Cai, Jun Xia, Guiqin Chen

J. Organomet. Chem. 689 (2004) 2531

A facile stereoselective synthesis of (*E*)-1,2-disubstituted vinylstannanes via hydromagnesiation of alkylarylacetylenes

Hydromagnesiation of alkylarylacetylenes 1 in diethyl ether gave (E)- $\alpha$ -arylvinyl Grignard reagents 2, which reacted with trialkylstannyl chlorides 3 in diethyl ether to afford stereoselectively (E)-1,2-disubstituted vinylstannanes 4 in high yields.

### Vladimir P. Kirin, Vladimir A. Maksakov, Alexandr V. Virovets, Sergey A. Popov, Alexey V. Tkachev

J. Organomet. Chem. 689 (2004) 2535

Triosmium clusters with bridging terpenic thioureato-like ligands

Bridging coordination of (3aR 3bR 4aR 5aS)-5a-hydroxy-3,4,4-trimethyl-3a,3b,4,4a, 5,5a-hexahydrocyclopropa[3,4]cyclopenta [1, 2-c]pyrazole-1-carbothioic acid amide on a triosmium cluster results in tautomeric rearrangement of the organic ligand followed by the cleavage of the pyrazolinol cycle. An unusual silica gel induced oxidative cleavage of the cyclopropane ring and epoxidation of cycloalcane C–C bond occurs after chromatography of the open chain derivative.

Contents vii

### Timothy N. Danks, Gabriele Wagner

J. Organomet. Chem. 689 (2004) 2543

The chemistry of (1-azabuta-1,3-diene)-tricarbonyliron(0) complexes

The synthesis, structure and reactivity of (1-azabuta-1,3-diene)tricarbonyliron(0) complexes, with respect to inorganic, organometallic and organic aspects of their chemistry is reviewed.

#### Notes

#### Michael I. Bruce, Paul A. Humphrey, Brian W. Skelton, Allan H. White

J. Organomet. Chem. 689 (2004) 2558

Two gold–ruthenium clusters derived from  $Ru_3(\mu\text{-}H)_3(\mu_3\text{-}CBr)(CO)_9$ 

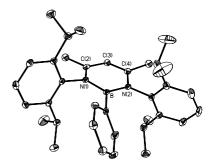
The reaction between AuMe(PPh<sub>3</sub>) and Ru<sub>3</sub>( $\mu$ -H)<sub>3</sub>( $\mu$ <sub>3</sub>-CBr)(CO)<sub>9</sub> affords the novel heptanuclear cluster Au<sub>4</sub>Ru<sub>3</sub>( $\mu$ <sub>3</sub>-CMe)(Br)-(CO)<sub>9</sub>(PPh<sub>3</sub>)<sub>3</sub>, containing an Au/Ru<sub>3</sub>/Au trigonal pyramidal cluster face-capped by two Au(PPh<sub>3</sub>) groups and a CMe ligand, together with Au<sub>2</sub>Ru<sub>3</sub>( $\mu$ -H)( $\mu$ <sub>3</sub>-CMe)(CO)<sub>9</sub>(PPh<sub>3</sub>)<sub>2</sub> which co-crystallises with the analogous  $\mu$ <sub>3</sub>-CH complex.

# Alan H. Cowley, Zheng Lu, Jamie N. Jones, Jennifer A. Moore

J. Organomet. Chem. 689 (2004) 2562

Synthesis and structure of a boron cation supported by a -diketiminate ligand

Boron cation  $3^+$  has been isolated as its  $[Al_2Cl_7]^-$  salt. An X-ray crystallographic study revealed that the  $BN_2C_3$  ring of  $3^+$  is planar and that each atom of this six-membered ring adopts a trigonal planar geometry. A DFT calculation showed  $\pi$ -type interactions in the HOMO-6 and HOMO-7 orbitals.



## J.W. Faller, Roman G. Kultyshev, Jonathan Parr

J. Organomet. Chem. 689 (2004) 2565

New synthetic routes to allylic germatranes

Two complementary routes allow control of regiochemistry and stereochemistry in the synthesis of allylic germatranes. The first method utilizes a transmetallation reaction between germanium(IV) chloride and the corresponding allylic tributylstannanes. The second route is via a palladium-catalyzed hydrogermylation of conjugated dienes by germatrane, N(CH<sub>2</sub>CH<sub>2</sub>O)<sub>3</sub>GeH, which gives predominantly Z products.

$$"HGeO_3N"$$

$$R \longrightarrow GeO_3N$$

$$R \longrightarrow$$

Contents viii

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