

Available online at www.sciencedirect.com



Volume 691, issue 16, 1 August 2006



www.elsevier.com/locate/jorganchem

Contents

Communication

Kris Randell, Matthew J. Stanford, Guy J. Clarkson, Jonathan P. Rourke

J. Organomet. Chem. 691 (2006) 3411

N-heterocyclic carbenes: Reaction to give anilines

The attempted synthesis of bis(NHC)palladium complexes via the direct reaction of an imidazolium salt with palladium acetate results in the formation of a mixed NHC/aniline complex. The aniline originally derives from an imidazolium salt.



Regular papers

Tushar S. Basu Baul, Archana Mizar, Antonin Lyčka, Eleonora Rivarola, Robert Jirásko, Michal Holčapek, Dick de Vos, Ulli Englert

J. Organomet. Chem. 691 (2006) 3416

Diphenyltin(IV) complexes of the 5-[(E)-2-(aryl)-1-diazenyl]quinolin-8-olates: Synthesis and multinuclear NMR, ¹¹⁹Sn Mössbauer, electrospray ionization MS, X-ray characterization and assessment of in vitro cytotoxicity A series of *cis*-bis{5-[(*E*)-2-(aryl)-1-diazenyl]quinolinolato}diphenyltin(IV) complexes have been synthesized and characterized by ¹H, ¹³C, ¹¹⁹Sn NMR, ESI-MS, IR and ^{119m}Sn Mössbauer spectroscopic techniques in combination with elemental analysis. The structures of a ligand L⁶H (i.e., 5-[(*E*)-2-(4ethoxyphenyl)-1-diazenyl]quinolin-8-ol) and three diphenyltin(IV) complexes were determined by single crystal X-ray diffraction. X-ray crystallography reveals that complexes adopt a distorted *cis*-octahedral arrangement around the tin atom. The in vitro cytotoxicity of diphenyltin(IV) complex is reported and compared with Ph₂Sn(Ox)₂.



Philip C. Andrews, Peter C. Junk, Iryna Nuzhnaya, Leone Spiccia, Nafty Vanderhoek

J. Organomet. Chem. 691 (2006) 3426

Complications in metathesis reactions involving Grignard reagents: Effect of solvent on products obtained from the interaction of PhMgBr with GaCl₃ or InBr₃ The important role of supporting solvent in transmetallation reactions involving Grignard reagents is highlighted in the formation and crystallisation of the Group 13 'ate' species, $[Mg_3Br_3Cl_2(Et_2O)_6][GaPh_2Br_2]$ (1), $[Mg_3-Br_5(Et_2O)_6][InPh_2Br_2]$ (2), $[MgBr(THF)_5]-[GaPh_3Br]$ (3), $[MgBr(THF)_5][InPh_3Br]$ (4), $[Mg(THF)_6][GaPh_2Br_2]_2$ (5) obtained by reaction of PhMgBr with gallium and indium halides. The compounds have been characterised by ¹H NMR, elemental analyses, and single-crystal X-ray diffraction.



Luis A. García-Escudero, Daniel Miguel, José A. Turiel

J. Organomet. Chem. 691 (2006) 3434

Carbonyl complexes of manganese, rhenium and molybdenum with ethynyliminopyridine ligands

Complexes with ethynyliminopyridine ligands can be prepared by a facile, general method, starting from *m*- or *p*-ethynylaniline in a "onepot" reaction. The terminal ethynyl group in these complexes reacts with dicobalt octacarbonyl to afford tetrahedrane trimetallic complexes.



Adam J. Davenport, David L. Davies, John Fawcett, David R. Russell

J. Organomet. Chem. 691 (2006) 3445

Chiral pyridine imidazolines from C_1 symmetric diamines: Synthesis, arene ruthenium complexes and application as asymmetric catalysis for Diels-Alder reactions New chiral pyridine imidazolines (L) have been synthesised and reacted with $[RuCl_2-(mes)]_2$ (mes = 1,3,5-trimethyl benzene) to form $[RuCl(L)(mes)][SbF_6]$ (**5a**-**c**) which after treatment with AgSbF₆ are enantioselective catalysts for the Diels-Alder reaction of acroleins and cyclopentadiene. The NR¹ substituent affects the enantioselectivity.



Srećko I. Kirin, Ulrich Schatzschneider, Xavier de Hatten, Thomas Weyhermüller, Nils Metzler-Nolte

J. Organomet. Chem. 691 (2006) 3451

1,*n*'-Disubstituted ferrocenoyl amino acids and dipeptides: Conformational analysis by CD spectroscopy, X-ray crystallography, and DFT calculations

An experimental and computational study (DFT, B3LYP/LanL2DZ) on ferrocenoyl peptides $Fe[C_3H_4$ -CO-Aaa-R]₂ (R = NH₂ or OCH₃ revealed a significant difference in stability of its conformers, depending on the number of hydrogen bonds. One hydrogen bond stabilizes the system by about 30 kJ mol⁻¹.

 $\frac{1}{2} = \frac{1}{2} \frac{1}{33.9 \text{ kJ·mol}^{1}} = \frac{1}{2} \frac{1}{2$

Fabio Marchetti, Guido Pampaloni, Calogero Pinzino

J. Organomet. Chem. 691 (2006) 3458

Some new results on the Fischer-Hafner synthesis of vanadium arenes

The biphasic liquid system formed in the Fisher–Hafner reduction of VCl₃ in toluene contains the $[V(\eta^6-MeC_6H_5)_2]^+$ cation, which has been isolated in the solid state as the tetraphenylborato-, $[Al_2Cl_7]^-$, $[Al_4O_2Cl_{10}]^{2-}$ and $[catena-Al_4O_2Cl_9]^-$ salts. The $[V(\eta^6-MeC_6H_5)_2]^+$ cation shows two different conformations of the toluene rings: a *trans*-eclipsed conformation in $[V(\eta^6-MeC_6H_5)_2]_2$. [Al_4O_2Cl_{10}] (2), and an almost *cis*-eclipsed one in $[V(\eta^6-MeC_6H_5)_2]$ [*catena*-Al_4O_2Cl_9] (3).



Marino Basato, Andrea Biffis, Gianluca Martinati, Cristina Tubaro, Claudia Graiff, Antonio Tiripicchio, Laura A. Aronica, Anna M. Caporusso

J. Organomet. Chem. 691 (2006) 3464

Cationic complexes of dirhodium(II) with 1,8naphthyridine: Catalysis of reactions involving silanes Cationic dirhodium(II) complexes with 1,8naphthyridine exhibit promising catalytic activity in reactions involving silanes, in particular in the silylformylation of alkynes.



José Giner Planas, Clara Viñas, Francesc Teixidor, Mark E. Light, Michael B. Hursthouse

J. Organomet. Chem. 691 (2006) 3472

A boron–boron linked large metallacarborane cluster: Characterization and X-ray structure of 8.9'-[closo-{3-Co(η^{5} -C₅H₅)-1,2-C₂B₉H₁₀}]₂

The first example of a boron–boron linked large metallacarborane cluster, 8,9'-[*closo*-{3-Co(η^5 -C₅H₅)-1,2-C₂B₉H₁₀}]₂ (1), has been characterized by single crystal X-ray diffraction analysis and NMR spectroscopy. The solid state conformation of 1 seems to be the result of a pair of intramolecular C–H···H–B dihydrogen bonds between the protonic H atoms of the C₅H₅ fragment of a sub-cluster and the hydridic H atoms of the C₂B₉H₁₀ fragment in the other sub-cluster in 1.



Mahammad Ali, Claude F. Bernasconi, Supriya Biswas

J. Organomet. Chem. 691 (2006) 3477

Transition metal carbene chemistry 6: Kinetic studies of the reactions of hydroxide ion with $(CO)_5MO=C(XCH_2CH_2OH)(C_6H_5)$ (X = O and S) and $(CO)_5W=C(OCH_2CH_2OH)-(C_6H_4-Z)$

A kinetic study of the reaction of hydroxide ion with $(CO)_5Mo=C(XCH_2CH_2OH)(C_6H_5)$ (X = O for Mo-OR, and X = S for Mo-SR), and $(CO)_5W=C(OCH_2CH_2OH)(C_6H_4-Z)$ (W-OR(Z)) shows a pathway in basic solutions that involves rapid deprotonation of the OH group followed by rate-limiting cyclization. The much higher ($k_1 K^{OH}$) (2.19) value for W-OR(Z) over W-SR(Z) (0.96) arises due to the stabilization of the reactant carbene complex by the stronger π -donor effect of oxygen over sulfur.



Mark R. Burgess, Su Jing, Christopher P. Morley

J. Organomet. Chem. 691 (2006) 3484

Electrochemical and NMR spectroscopic studies of selenium- and tellurium-substituted ferrocenes II: Diferrocenyl chalcogenides, Fc₂E, diferrocenyl dichalcogenides, Fc₂E₂, and bis(ferrocenylchalcogeno)alkanes, FcE(CH₂)_{*n*}-E'Fc (E, E' = Se: $n = 1, 2, 3; E, E' = Te: n = 1, 3; E = Se, E' = Te: <math>n = 3; Fc = [Fe(\eta^5-C_5H_5)-(\eta^5-C_5H_4)])$ Cyclic and differential pulse voltammetry of compounds containing two ferrocenes linked by a chalcogen-containing bridge show that there is electronic communication between the two ferrocene units when the bridge is short (as in Fc₂E), but that the interaction rapidly becomes weaker with increasing $Fe\cdots Fe$ distance, and is undetectable for the 1,3-bis-(ferrocenylchalcogeno)propanes.



Antonio Rodriguez-Delgado,

Wesley R. Mariott, Eugene Y.-X. Chen

J. Organomet. Chem. 691 (2006) 3490

Synthesis and MMA polymerization of chiral ansa-zirconocene ester enolate complexes with C_2 - and C_s -ligation

While the zirconocene methyl cation with $C_{\rm s}$ -ligation {[Me₂C(Cp)(Flu)ZrMe]⁺} and the neutral methyl zirconocene ester enolate with C_2 -ligation {rac-(EBI)ZrMe[OC(O'Pr)=CMe₂]} are inactive for MMA polymerization, their respective cationic ester enolate and triflate derivatives are active for polymerization of MMA affording high molecular weight polymers.



Alexandre Flahaut, Jean-Pierre Baltaze, Sylvain Roland, Pierre Mangeney

J. Organomet. Chem. 691 (2006) 3498

Synthesis of chiral imino- and aminoimidazolium salts and of chelating amino-*N*heterocyclic carbene palladium(II) complexes A new preparation of chiral imino-imidazolium salts has been developed. Amino-imidazolium salts with *de* >95% could be obtained by reduction of these imino salts with sodium borohydride and recrystallisation. Chelating amino-NHC Pd^{II} complexes were obtained in two steps via formation of the AgI complexes and reaction with PdCl₂(MeCN)₂. Crystal structure details of a Pd^{II} *cis*-dichloro amino-NHC complex are presented.



Keisham Sarjit Singh, Kevin A. Kreisel, Glenn P.A. Yap, Mohan Rao Kollipara

J. Organomet. Chem. 691 (2006) 3509

Synthesis of arene ruthenium triazolato complexes by cycloaddition of the corresponding arene ruthenium azido complexes with activated alkynes or with fumaronitrile Arene ruthenium(II) β -diketonate complexes [(η^6 -*p*-cymene)Ru(LL)CI] readily react with NaN₃ to afford the corresponding azido complexes of the type [(η^6 -*p*-cymene)Ru(LL)(N₃)]; where LL = β -diketonate viz., *acac*, *bzac*, *dbzm*. These neutral azido complexes undergo [3+2] cycloaddition reactions with activated alkynes and fumaronitrile to produce the corresponding arene ruthenium triazolato complexes.



Lianqing Chen, Chuluo Yang, Jingui Qin, Jia Gao, Han You, Dongge Ma

J. Organomet. Chem. 691 (2006) 3519

Synthesis, structure, electrochemistry, photophysics and electroluminescence of 1,3,4oxadiazole-based *ortho*-metalated iridium(III) complexes Synthesis, characterization, crystal structure, electrochemical, photophysical and electroluminescent properties of four new iridium(III) complexes with 1,3,4-oxadiazole derivative as cyclometalated ligand have been reported.



Hai-Xia Yu, Jian-Fang Ma, Guo-Hai Xu, Shun-Li Li, Jin Yang, Ying-Ying Liu, Yan-Xiang Cheng

J. Organomet. Chem. 691 (2006) 3531

Syntheses and crystal structures of four new organotin complexes with Schiff bases containing triazole

Björn Pampuch, Wolfang Saak, Manfred Weidenbruch

J. Organomet. Chem. 691 (2006) 3540

New compounds with Ge/Ge and Ge/C multiple bonds

Complexes 1–3 show similar structures containing a Sn_4O_4 ladder skeleton in which each of the *exo* tin atoms is bonded to the N atom of a corresponding thione-form deprotonated ligand. Complex 4 shows a mononuclear structure in which the tin atom of triphenyltin group is coordinated by the S atom of a thiolform L4⁻ anion.





Haritosh Mishra, Rabindranath Mukherjee

J. Organomet. Chem. 691 (2006) 3545

Half-sandwich η^6 -benzene Ru(II) complexes of pyridylpyrazole and pyridylimidazole ligands: Synthesis, spectra, and structure

The reaction of the Grignard compound RMgBr, $R = 2,5-tBu_2C_6H_3$, with GeCl₂ · dioxane furnishes the digermene **4** with a long Ge=Ge double bond length and the largest *trans*-bent angles observed so far for digermenes. In solution, the digermene $R_2Ge=GeR_2$, $R = 2-tBu-4,5,6-Me_3C_6H$ dissociates into two molecules of the germylene R_2Ge : which react with hexa-2,4-diyne to give the acetylene-linked bis(germaethene) **7**.

Systematic comparative studies (X-ray and ¹H NMR) have been made on "piano-stool" complexes of Ru(II) with bidentate ligands 2-[3-(4-chlorophenyl)pyrazol-1-ylmethyl]pyridine, 3-(2-pyridyl)pyrazole, 1-benzyl-[3-(2'-pyridyl)]pyrazole, and 2-(1-imidazol-2-yl)pyridine]. Structural studies reveal extensive non-covalent interactions.

PF₆

Alwyn G. Davies, Andrea Sella, Rajaveen Sivasubramaniam

J. Organomet. Chem. 691 (2006) 3556

Can mono- or di-butyltin chlorides produce tributyltin chloride at elevated temperatures? Implications for applications in chemical vapour deposition The decomposition and potential redistribution reaction of Bu_3SnCl , Bu_2SnCl_2 , and $BuSnCl_3$ at 200–300 °C have been investigated by NMR spectroscopy. In particular, no toxic Bu_3SnCl was formed from $BuSnCl_3$. The principles behind the distribution reactions are discussed.

$Does BuSnCl_3 \longrightarrow Bu_3SnCl + SnCl_4 ?$
BuSnCl ₃ 200 °C
Bu ₂ SnCl ₂ no reaction
Bu ₃ SnCl 200 °C Bu ₂ SnCl ₂ + Bu ₄ Sn
Bu₄Sn 200 °C → → → → → → → + Sn



Full text of this journal is available, on-line from ScienceDirect. Visit www.sciencedirect.com for more information.