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Chemistry

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Contents

Special Issue: Palladium Chemistry in 2003: Recent Developments

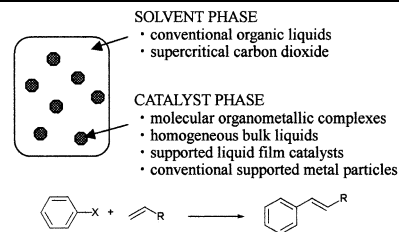
Preface 209

Bhalchandra M. Bhanage, Shin-ichiro Fujita, Masahiko Arai

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Heck reactions with various types of palladium complex catalysts: application of multiphase catalysis and supercritical carbon dioxide

Homogeneous and multiphase organo- and metal-palladium catalysts have been applied for Heck coupling reaction and separation using different types of catalysts and solvents including supercritical carbon dioxide.



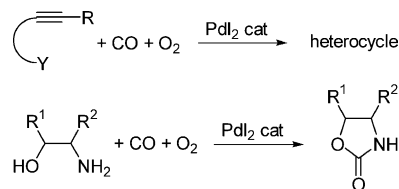
Accounts

Bartolo Gabriele, Giuseppe Salerno, Mirco Costa, Gian Paolo Chiusoli

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Recent developments in the synthesis of heterocyclic derivatives by PdI₂-catalyzed oxidative carbonylation reactions

In this short account we review the application of our PdI₂/KI-catalyzed oxidative carbonylation methodology to different acetylenic substrates bearing a suitable nucleophilic function as well as to β-amino alcohols, which has allowed the one-step synthesis of a variety of functionalized heterocyclic derivatives.

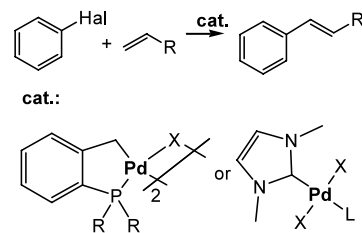


Wolfgang A. Herrmann, Karl Öfele, Denise v. Preysing, Sabine K. Schneider

J. Organomet. Chem. 687 (2003) 229

Phospha-palladacycles and N-heterocyclic carbene palladium complexes: efficient catalysts for CC-coupling reactions

This article presents a summary of recent developments in the application of tri-*o*-ligands as catalysts in Heck type reactions. During the last decade both types of palladium complexes have proven to be very efficient catalyst precursors in C-C and C-N coupling tolylphosphine bound palladacycles and palladium complexes with N-heterocyclic carbene reactions even with deactivated aryl halides.

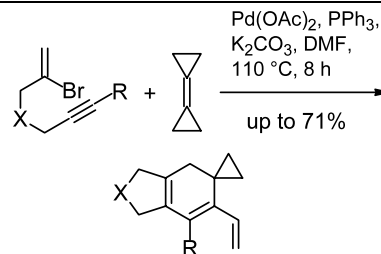


**Armin de Meijere, Michael Schelper,
Mario Knoke, Baris Yucel,
Hans Wolf Sünemann,
René Peter Scheurich, Lars Arve**

J. Organomet. Chem. 687 (2003) 249

Palladium-catalyzed cross-coupling reactions and electrocyclizations—efficient combinations for new cascade reactions

Palladium-catalyzed cross-coupling reactions can be efficiently combined with subsequent thermal reactions (such as 6π -electrocyclizations and Diels–Alder reactions) to build up complex molecules from rather simple precursors in single or two step operations. With the appropriate building blocks at hand, products range from oligo-substituted spiro[2.5]octene derivatives over spirocyclopropanated oligocycles to novel steroid analogues.



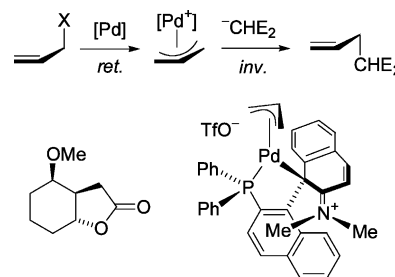
X = C(CO₂Me)₂, O, NTs
R = alkyl, alkenyl, aryl, SiR'₃, CO₂Me

Pavel Kočovský

J. Organomet. Chem. 687 (2003) 256

Reactivity control in palladium-catalyzed reactions: a personal account

The author's contribution to several classes of palladium-catalyzed reactions are summarized: allylic oxidation, diastereo- and enantio-controlled allylic substitution, transmetalation of organomercurials, stereoselective rearrangements, and development of new ligands for amination of aryl halides and for asymmetric Heck addition.

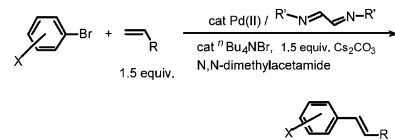


**Gabriela A. Grasa, Rohit Singh,
Edwin D. Stevens, Steven P. Nolan**

J. Organomet. Chem. 687 (2003) 269

Catalytic activity of Pd(II) and Pd(II)/DAB-R systems for the Heck arylation of olefins

Palladium-catalyzed reactions of aryl bromides with various olefins involving Pd(II)/diazabutadiene (DAB-R) systems have been investigated. The Pd(OAc)₂/DAB-Cy (**1**, DAB-Cy = 1,4-dicyclohexyl-diazabutadiene) system presents the highest activity with respect to electron-neutral and electron-deficient aryl bromides in coupling with electron rich olefins. The Pd(acac)₂ and Pd(acac)₂/DAB-Cy precatalysts were found to be very effective for the arylation of various olefins with aryl bromides with respect to reaction rate, catalyst loadings, and functional group tolerance.

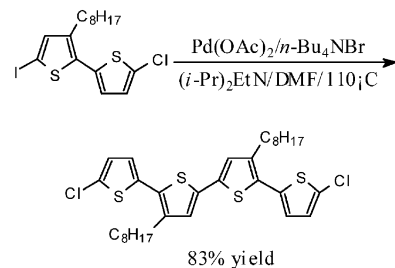


**Jwanro Hassan, Christel Gozzi,
Emmanuelle Schulz, Marc Lemaire**

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Palladium-catalyzed coupling reactions towards the synthesis of well-defined thiophene-oligomers

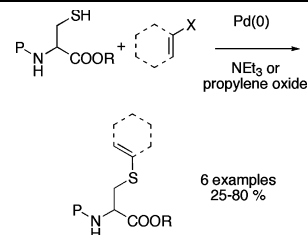
New methodologies were developed for the synthesis of regioregular oligothiophenes with good yields.



Xavier Moreau, Jean-Marc Campagne*J. Organomet. Chem.* 687 (2003) 322

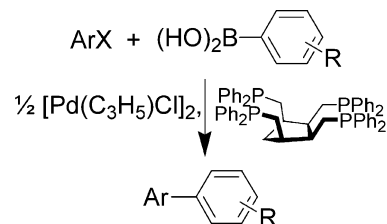
Palladium catalyzed thiol cross-coupling of cystein derivatives with aryl and alkenyl halides

Palladium catalyzed cystein thiol cross-coupling reactions with aryl and vinyl halides have been investigated: Pd₂dba₃-CHCl₃ and dppf are the key choice in these reactions. The role of the base in these reactions was also questioned: it has been shown that base can be replaced by an HX-scavenger such as propylene oxide.

**Marie Feuerstein, Henri Doucet, Maurice Santelli***J. Organomet. Chem.* 687 (2003) 327

Efficient coupling of heteroaryl halides with arylboronic acids in the presence of a palladium-tetraphosphine catalyst

Cis,cis,cis-1,2,3,4-tetrakis(diphenylphosphinomethyl)cyclopentane: 1/2[PdCl(C₃H₅)₂]₂ system catalyses the Suzuki cross-coupling of heteroaryl halides with a range of arylboronic acids with very high ratio substrate/catalyst in good yields. Substrates such as pyridines, quinolines, thiophenes, an indole, pyrimidines or a furane have been used successfully.

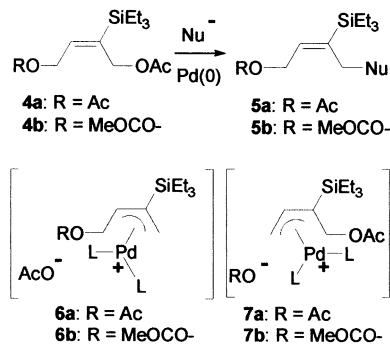


Ar: pyridine, quinoline, indole, pyrimidine, thiazole, thiophene, furane
X = Cl, Br, I
R = H, 4-F, 4-MeO, 3-CF₃, 2-Me

Vicenç Branchadell, Marcial Moreno-Mañas, Roser Pleixats, Serge Thorimbert, Claude Commandeur, Cécile Boglio, Max Malacria*J. Organomet. Chem.* 687 (2003) 337

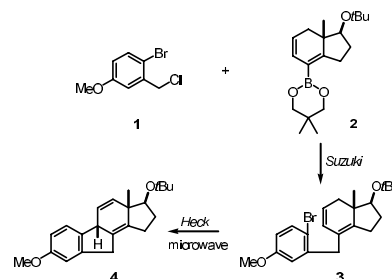
The silicon effect on the regioselectivity of the Tsuji-Trost reaction. Experimental and theoretical approaches

Compounds **4** afford regioselectively **5** under Pd(0)-catalysis. Calculation show that the higher stability of cations **6** overcompensate the higher reactivity of isomers **7**.

**Lutz F. Tietze, J. Matthias Wiegand, Carsten Vock***J. Organomet. Chem.* 687 (2003) 346

Synthesis of enantiopure B-nor-steroids by multiple Pd-catalyzed transformations

The novel B-nor-estradiol analogue **4** was synthesized using two subsequent Pd-catalyzed reactions as the key steps. Suzuki-coupling of the benzylchloride **1** and the boronic ester **2** led to the *seco*-B-nor-steroid **3**, which was transformed into **4** by an intramolecular Heck-reaction under microwave irradiation.

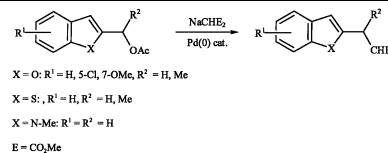


Gaëlle Primault, Jean-Yves Legros,
Jean-Claude Fiaud

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Palladium-catalyzed benzylic-like nucleophilic substitution of benzofuran-, benzothiophene- and indole-based substrates by dimethyl malonate anion

Benzylic acetates derived from benzofuran, benzothiophene and indole were prepared and their palladium-catalyzed benzylic nucleophilic substitution by sodium dimethyl malonate was investigated.

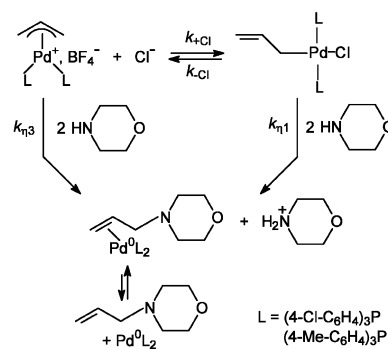


Thibault Cantat, Emilie Génin,
Claire Giroud, Gilbert Meyer,
Anny Jutand

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Structural and kinetic effects of chloride ions in the palladium-catalyzed allylic substitutions

Addition of ligands to [Pd(η³-allyl)(μ-Cl)]₂ or Cl⁻ to [(η³-allyl)PdL₂]⁺ results in the formation of neutral complexes η¹-allyl-PdClL₂. The reaction of morpholine with [(η³-allyl)PdL₂]⁺ is slower in the presence of Cl⁻ due to the formation of η¹-allyl-PdClL₂. [(η³-allyl)PdL₂]⁺ is more reactive than η¹-allyl-PdClL₂. However, η¹-allyl-PdClL₂ may become the major reactive species at high chloride concentrations.

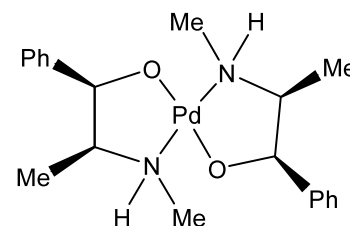


Sandrine Bouquillon, Stéphane Humbel,
Ulla Létinois-Halbes, Françoise Hélin,
Jacques Muzart

J. Organomet. Chem. 687 (2003) 377

trans-Bis-[(−)ephedrinat]e-palladium^{II} complex: synthesis, molecular modeling and use as catalyst

The reaction between palladium acetate, (−)-ephedrine and potassium acetate led to this bis-chelate complex whose the *trans*-structure has been established from calculations. This complex has been tested as catalyst for hydrogenation and Heck reaction.

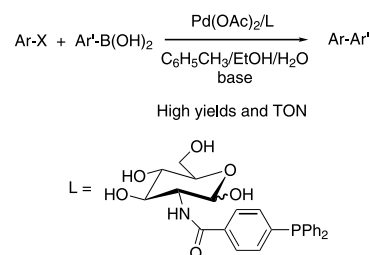


Robert Kolodziuk, Alexandra Penciu,
Mustapha Tollabi, Eric Framery,
Catherine Goux-Henry,
Alexander Iourtchenko, Denis Sinou

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Palladium-catalyzed Suzuki cross-coupling of aryl halides with aryl boronic acids in the presence of glucosamine-based phosphines

Coupling of D-glucosamine or of the corresponding acetate with various diphenylphosphino acids afforded carbohydrate-substituted phosphines in quite good yields. The catalysts obtained by the association of these ligands with Pd(OAc)₂ are very effective in the Suzuki cross-coupling of various iodo- and bromoaryl derivatives with aryl boronic acids in a mixture toluene-ethanol-water. The highest activities were obtained using the polyhydroxy phosphines, turnovers of up to 97 000 being obtained.

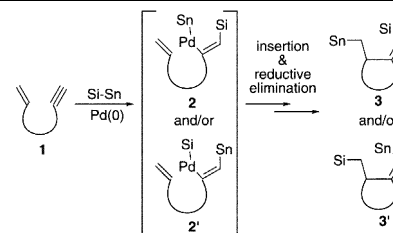


**Yoshihiro Sato, Noriko Imakuni,
Tomohiro Hirose, Hideaki Wakamatsu,
Miwako Mori**

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Further studies on palladium-catalyzed bismetallative cyclization of enynes in the presence of $\text{Bu}_3\text{SnSiMe}_3$

Bismetallative cyclization of enynes with $\text{Bu}_3\text{SnSiMe}_3$ catalyzed by Pd(0) complex was realized for the first time, which gives cyclized products containing a vinylsilane moiety and a homoallyl tin moiety in good yield. In this cyclization, $\text{Pd}_2(\text{dba})_3 \cdot \text{CHCl}_3$ or $\text{Pd}(\text{OH})_2$ on charcoal is effective as a Pd(0) catalyst and the addition of a phosphine ligand increased the formation of alkyne bismetallation by-product.

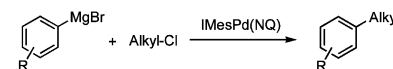


**Anja C. Frisch, Franck Rataboul,
Alexander Zapf, Matthias Beller**

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First Kumada reaction of alkyl chlorides using *N*-heterocyclic carbene/palladium catalyst systems

For the first time it is shown that palladium-carbene complexes catalyze the Kumada coupling of arylmagnesium bromides with primary alkyl chlorides.

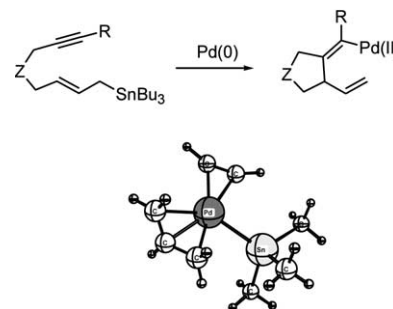


**Belén Martín-Matute, Elena Buñuel,
María Méndez, Cristina Nieto-Oberhuber,
Diego J. Cárdenas, Antonio M. Echavarren**

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Allyl stannanes as electrophiles or nucleophiles in the palladium-catalyzed reactions with alkynes

The reaction of allyl stannanes with Pd(0) proceeds by oxidative addition to form $(\eta^3\text{-allyl})\text{palladium}$ complexes. Subsequent formal *syn* addition to the alkyne gives intermediate alkenyl palladium(II) complexes that are diastereomeric to those formed in the Pd(II)-catalyzed cyclization of allyl stannanes with alkynes.

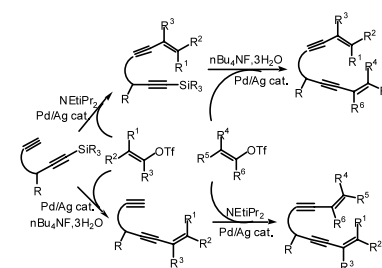


Ulla Halbes-Létinois, Patrick Pale

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Pd-Ag catalyzed selective dicoupling of α -trialkylsilyl α,ω -diynes; the first one-pot synthesis of dienediynes

Using $\text{Pd}(\text{PPh}_3)_4$ and AgI as catalysts, the first one-pot synthesis of dienediynes has been achieved. Sequential dicoupling reactions afforded a regioselective access to dienediynes starting from any and especially non-symmetrical α,ω -diynes. Good to excellent yields were obtained in dimethylformamide at room temperature.

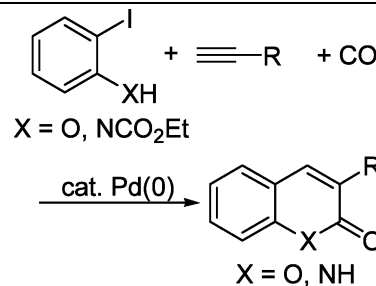


Dmitry V. Kadnikov, Richard C. Larock

J. Organomet. Chem. 687 (2003) 425

Palladium-catalyzed carbonylative annulation of terminal alkynes: synthesis of coumarins and 2-quinolones

Terminal alkynes react with *o*-iodophenols or *N*-ethoxycarbonyl-*o*-iodoanilines and carbon monoxide in the presence of pyridine and a catalytic amount of Pd(OAc)₂ to produce coumarins or 2-quinolones, respectively. The scope and limitations of these processes and isotope-labeling experiments to probe the mechanism are described.

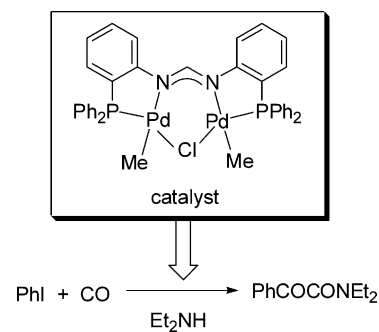


Naofumi Tsukada, Yoichi Ohba, Yoshio Inoue

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Double carbonylation of aryl iodides with diethylamine catalyzed by dinuclear palladium complexes

Dinuclear palladium complexes bridged by a novel PNNP ligand were found to be very efficient and selective catalysts for the double carbonylation of iodobenzene with diethylamine using K₃PO₄ as base and 1,4-dioxane as solvent with a TON up to 10⁵ and selectivity of 96%.



Carsten Bolm, Hiroaki Okamura, Marinella Verrucci

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Palladium-catalyzed intramolecular α -arylation of sulfoximines

Palladium-catalyzed intramolecular α -arylations of sulfoximines bearing 2-bromobenzyl and 2-bromobenzoyl groups at the imine nitrogen result in the formation of heterocycles in moderate to good yields. A catalytic protocol based on the use Pd(OAc)₂ as metal source, *rac*-BINAP as ligand and K₂CO₃ or Cs₂CO₃ as base has been developed.

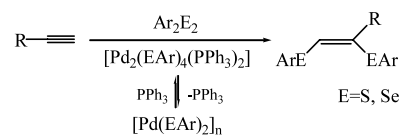


Valentine P. Ananikov, Michael A. Kabeshov, Irina P. Beletskaya, Grigory G. Aleksandrov, Igor L. Eremenko

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Mechanistic study of palladium catalyzed S–S and Se–Se bonds addition to alkynes

An unusual phosphine ligand effect increasing the yield of the Ar₂E₂ addition reaction to alkynes was found. The catalytic reaction involves intermediate formation of dinuclear palladium complexes, which may be a subject of further polymerization.

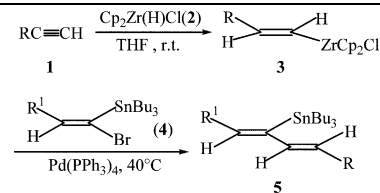


Mingzhong Cai, Hongde Ye, Hong Zhao, Caisheng Song

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Stereoselective synthesis of 1,3-dienylstannanes by palladium catalyzed cross-coupling reactions

Hydrozirconation of 1-alkynes (**1**) gives (*E*)-alkenylzirconium complexes (**3**), which are cross-coupled with (*Z*)- α -bromovinylstannanes (**4**) in the presence of Pd(PPh₃)₄ catalyst to afford stereoselectively 1,3-dienylstannanes (**5**) in good yields.

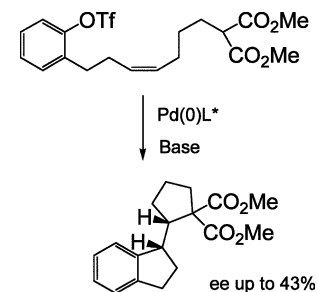


Didier Bruyère, Nuno Monteiro, Didier Bouysi, Geneviève Balme

J. Organomet. Chem. 687 (2003) 466

New developments in the Pd-catalysed cyclisation-coupling reaction of alkene-containing carbonucleophiles with organic halides (and triflates). The first examples of asymmetric catalysis.

Biscyclisation of the (*Z*)-2-[7-(2-trifluoromethanesulfonyloxy-phenyl)-hept-4-enyl]-malonic acid dimethyl ester with PdCl₂[*S*-(−)-TolBINAP] as chiral catalyst and K₂CO₃ as base leads to a cyclopentylindane as a single diastereomer in 54% chemical yield and 43% ee.

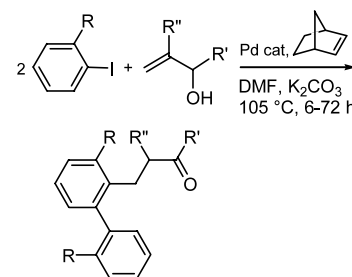


Marta Catellani, Sara Deledda, Benjamin Ganchehui, François Hénin, Elena Motti, Jacques Muzart

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A new catalytic method for the synthesis of selectively substituted biphenyls containing an oxoalkyl chain

Selectively substituted oxoalkylbiphenyl derivatives are obtained by reaction of ortho-substituted aryl iodides with allylic alcohols in the presence of palladium and norbornene as catalysts.



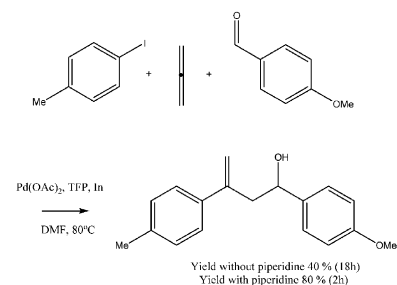
Laura A.T. Cleghorn, Ian R. Cooper, Colin W.G. Fishwick, Ronald Grigg, William S. MacLachlan, Marcello Rasparini, Visuvanathar Sridharan

J. Organomet. Chem. 687 (2003) 483

Three-component bimetallic (Pd/In) mediated cascade allylation of C = X functionality

Part 1. Scope and Class 1 examples with aldehydes and ketones

A new general three-component Pd/In bimetallic cascade reaction with four synthetic variants involving aryl iodides, allenes and C = X compounds affording homoallylic alcohols/amines as products is described and exemplified for Class 1 processes (intermolecular Pd–intermolecular In steps). Remarkable increases in yield and reaction rates were observed in the presence of amine additives.

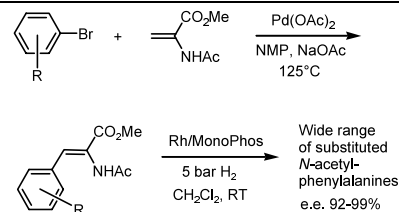


**Charlotte E. Willans, Jan M.C.A. Mulders,
Johannes G. de Vries, André H.M. de Vries**

J. Organomet. Chem. 687 (2003) 494

Ligand-free palladium catalysed Heck reaction of methyl 2-acetamido acrylate and aryl bromides as key step in the synthesis of enantiopure substituted phenylalanines

A variety of substituted *N*-acetyl phenylalanine esters were produced using a cost-effective sequence of 2 homogeneously catalysed steps. A ligand-free palladium catalysed arylation of methyl 2-acetamido acrylate using aryl bromides followed by a Rh/MonoPhos catalysed hydrogenation of the acetamido-cinnamate esters gave good yields of the various phenylalanine derivatives in very high enantioselectivities.

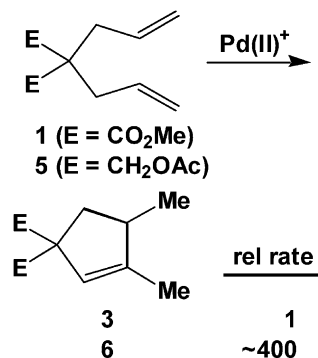


**Laurel A. Goj, G. Andrés Cisneros,
Weitao Yang, Ross A. Widenhofer**

J. Organomet. Chem. 687 (2003) 498

Dramatic effect of homoallylic substitution on the rate of palladium-catalyzed diene cycloisomerization

Palladium-catalyzed cycloisomerization of 4,4-bis(acetoxymethyl)-1,6-heptadiene (**5**) was ~400 times faster than was the cycloisomerization of dimethyl diallylmalonate (**1**) under identical conditions. Described is a combined mechanistic and computational study directed toward elucidating the effect of the homoallylic group of the diene on the rate of palladium-catalyzed cycloisomerization.

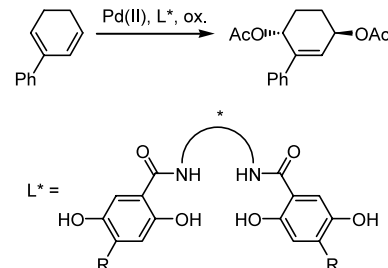


**Renzo C. Verboom, Bernd J. Plietker,
Jan-E. Bäckvall**

J. Organomet. Chem. 687 (2003) 508

New chiral diamide ligands containing redox-active hydroquinone groups. Synthesis and results in the palladium(II)-catalyzed 1,4-diacetoxylation of 1,3-dienes

Chiral C_2 -symmetric diamides containing hydroquinone groups were synthesized from different chiral diamines. By in situ oxidation of the hydroquinones, the compounds could be used as benzoquinone ligands in the asymmetric Pd(II)-catalyzed diacetoxylation reaction of 1,3-dienes, in which they gave an enantiomeric excess up to 42%.

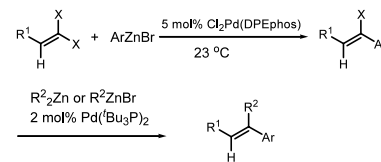


Ji-cheng Shi, Ei-ichi Negishi

J. Organomet. Chem. 687 (2003) 518

Pd-catalyzed selective tandem arylation-alkylation of 1,1-dihalo-1-alkenes with aryl- and alkylzinc derivatives to produce α -alkyl-substituted styrene derivatives

Trans-selective monoarylation of 1,1-dibromo- and 1,1-dichloro-1-alkenes can be achieved in >80% yields and in ≥ 98 –99% stereoselectivity with arylzinc bromides in the presence of a catalytic amount of $\text{Cl}_2\text{Pd}(\text{DPEphos})$. The second substitution of (*Z*)- α -bromostyrenes with alkylzincs in the presence of 2 mol% of $\text{Pd}(\text{tBu}_3\text{P})_2$ proceeds to give the corresponding trisubstituted alkenes in >90% yields and in ≥ 98 –99% stereoselectivity. Although somewhat less satisfactory, the use of $\text{Cl}_2\text{Pd}(\text{DPEphos})$ permits a one-pot tandem arylation-alkylation.

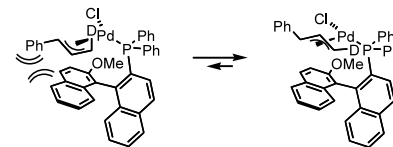


Laure Gouriou, Guy C. Lloyd-Jones,
Štěpán Vyskočil, Pavel Kočovský

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^2H -quadrupolar coupling-based analysis of stereochemical and regiochemical memory in the Pd-catalysed allylic alkylation of *iso*-cinnamyl type substrates employing the chiral monophosphine ligands 'MOP' and 'MAP'

Strategic placement of a ^2H -label at the *cis*-alkene terminus of *iso*-cinnamyl ester substrates allows detailed information to be deduced about competing regiochemical and stereochemical memory effects through ^2H -NMR analysis of the Pd-catalysed allylation product mixtures in a chiral liquid crystal matrix.

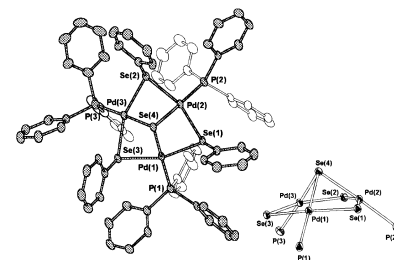


Milja S. Hannu-Kuure, Kaisa Palda'n,
Raija Oilunkaniemi, Risto S. Laitinen,
Markku Ahlgrén

J. Organomet. Chem. 687 (2003) 538

Formation and structural characterization of novel polynuclear palladium selenolato complexes $[\text{Pd}_3\text{Se}(\text{SePh})_3(\text{PPh}_3)_3]\text{Cl}$ and $[\text{Pd}_6\text{Cl}_2\text{Se}_4(\text{SePh})_2(\text{PPh}_3)_6]$

The treatment of $[\text{PdCl}_2(\text{PPh}_3)_2]$ with Na-SePh affords, in addition to well-known dinuclear main products, also trinuclear and hexanuclear complexes $[\text{Pd}_3\text{Se}(\text{SePh})_3(\text{PPh}_3)_3]\text{Cl}$ and $[\text{Pd}_6\text{Cl}_2\text{Se}_4(\text{SePh})_2(\text{PPh}_3)_6]$.

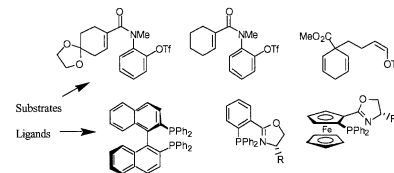


Denis Kiely, Patrick J. Guiry

J. Organomet. Chem. 687 (2003) 545

Palladium complexes of phosphinamine ligands in the intramolecular asymmetric Heck reaction

The synthesis of two novel cyclisation substrates for the asymmetric intramolecular Heck reaction is reported. Their cyclisation, in addition to a known substrate for *cis*-decalin formation, were tested with palladium complexes of BINAP and heterobidentate oxazoline-containing ligands. Poor to good chemical yields, excellent regioselectivities and moderate to high enantioselectivities (up to 85%) were obtained.

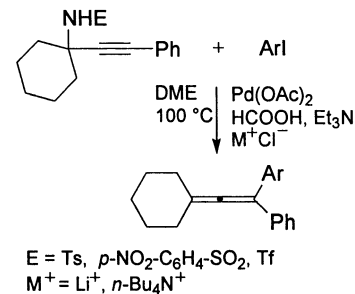


Antonio Arcadi, Sandro Cacchi,
Giancarlo Fabrizi, Fabio Marinelli,
Luca M. Parisi

J. Organomet. Chem. 687 (2003) 562

Palladium-catalyzed reaction of aryl iodides with *tertiary* propargylic amides. Highly substituted allenes through a regioselective carbopalladation/ β -N-Pd elimination reaction

The palladium-catalyzed reaction of aryl iodides with *tertiary* propargylic amides affords highly substituted allenes. Best results have been obtained by using $\text{Pd}(\text{OAc})_2$, $n\text{-Bu}_3\text{N}$, HCOOH , and $n\text{-Bu}_4\text{NCl}$ or LiCl in DME at 100°C . The reaction is highly regioselective and the carbopalladation step is controlled by the strong directing effect of the *tertiary* amide group.

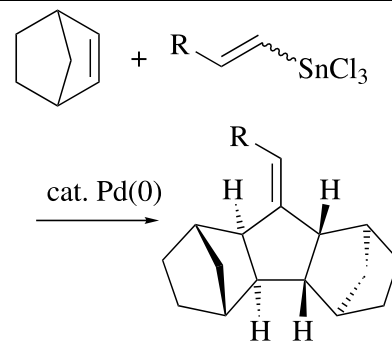


**Keigo Fugami, Yasuyuki Hirunuma,
Takashi Nishikata, Daisuke Koyama,
Masayuki Kameyama, Masanori Kosugi**

J. Organomet. Chem. 687 (2003) 567

Palladium-catalyzed novel addition–elimination reaction of alkenyltin reagents to norbornene

Palladium-catalyzed reaction between 1-alkenyltin trichlorides and norbornene resulted in stereoselective formation of 3-alkylidenepentacyclo[9.2.1.^{5,8}1.^{1,11}0.^{2,10}0^{4,9}]pentadecane instead of an expected simple alkenylstannylation product.

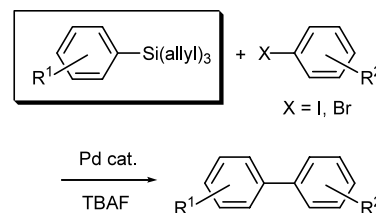


**Yoshiaki Nakao, Takuro Oda,
Akhila K. Sahoo, Tamejiro Hiyama**

J. Organomet. Chem. 687 (2003) 570

Triallyl(aryl)silanes serve as a convenient agent for silicon-based cross-coupling reaction of aryl halides

Triallyl(aryl)silanes, stable toward moisture, bases and acids, are found to react with aryl bromides and an aryl iodide in good yields in the presence of a palladium catalyst and TBAF. The reaction tolerates a diverse range of functional groups.



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