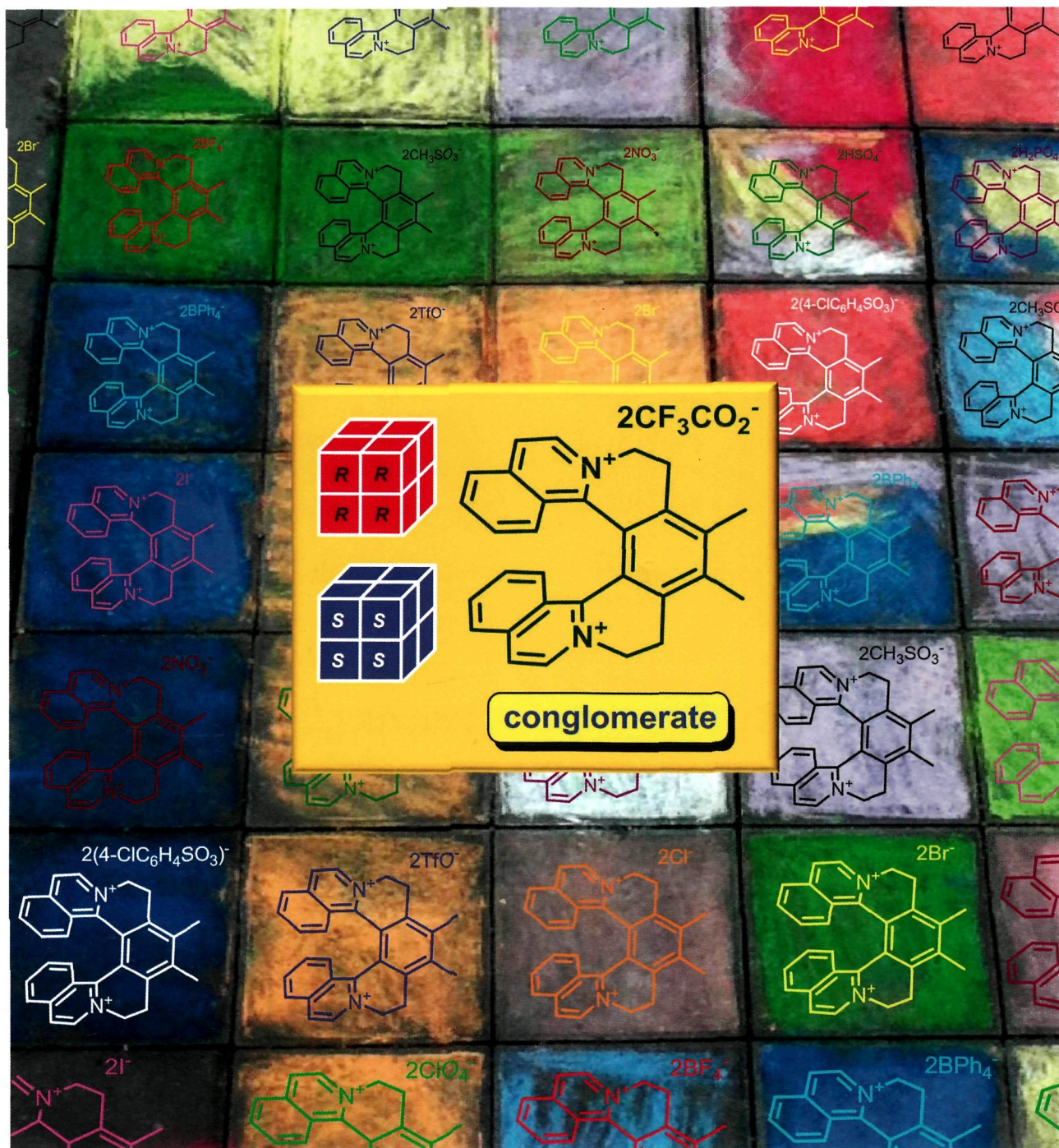


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# JOCS

*The Journal of Organic Chemistry*

FEBRUARY 15, 2013 VOLUME 78, NUMBER 4 [pubs.acs.org/joc](http://pubs.acs.org/joc)



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**ON THE COVER:** Conglomerates are substances that form two separate enantiomeric solid phases upon crystallization of their racemates. They are pivotal to a high mass-throughput resolution process called preferential crystallization. A set of [7]helquat salts with twelve distinct anions was investigated by X-ray crystal diffraction, which led to identification of conglomerate behavior in bis(trifluoroacetate) [7]helquat salt. This study demonstrates that a systematic search for conglomerates can be performed for a given helicenoid enabling straightforward multigram resolution via preferential crystallization. See Těplý and co-workers, p 1329.

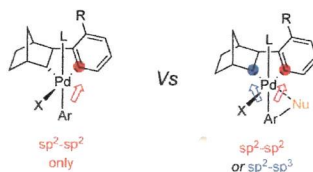
## JOCSynopsis

1323

dx.doi.org/10.1021/jo302581c

**Palladium/Norbornene Catalytic System: Chelation as a Tool To Control Regioselectivity of Pd(IV) Reductive Elimination**

Max Malacria\* and Giovanni Maestri



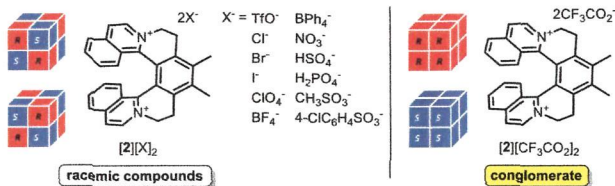
## Featured Articles

1329 S

dx.doi.org/10.1021/jo301615x

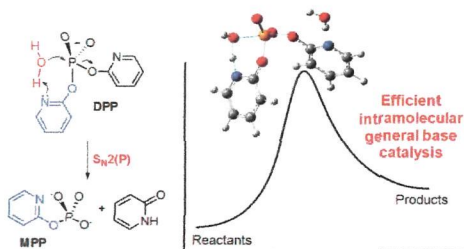
**Search for Conglomerate in Set of [7]Helquat Salts: Multigram Resolution of Helicene–Viologen Hybrid by Preferential Crystallization**

Jan Vávra, Lukáš Severa, Ivana Čiřarová, Blanka Klepetářová, David Šaman, Dušan Koval, Václav Kašička, and Filip Těplý\*



### Intramolecular General Base Catalysis in the Hydrolysis of a Phosphate Diester. Computational Guidance to a Choice of Mechanism

Anthony J. Kirby, Michelle Medeiros, José R. Mora, Pedro S. M. Oliveira, Almahdi Amer, Nicholas H. Williams, and Faruk Nome\*



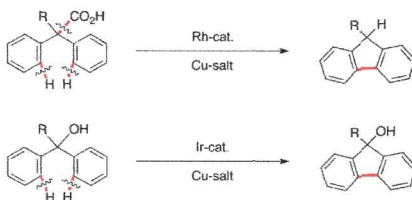
### 3-(Dialkoxyporphoryl)-N-confused Porphyrin. Synthesis, Stereochemistry, and Coordination Properties

Bin Liu, Xiaofang Li,\* Xin Xu, Marcin Stepień, and Piotr J. Chmielewski\*



### Rhodium- and Iridium-Catalyzed Dehydrogenative Cyclization through Double C–H Bond Cleavages To Produce Fluorene Derivatives

Masaki Itoh, Koji Hirano, Tetsuya Satoh,\* Yu Shibata, Ken Tanaka, and Masahiro Miura\*

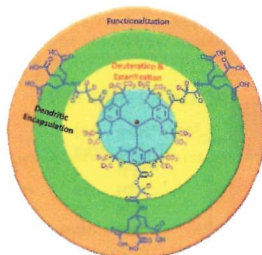


1371 **S**

dx.doi.org/10.1021/jo301849k

**Esterified Dendritic TAM Radicals with Very High Stability and Enhanced Oxygen Sensitivity**

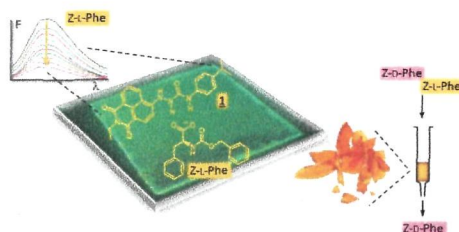
Yuguang Song, Yangping Liu,\* Craig Hemann, Frederick A. Villamena, and Jay L. Zweier\*

1377 **S**

dx.doi.org/10.1021/jo3019522

**Synthesis, Spectroscopic, and Analyte-Responsive Behavior of a Polymerizable Naphthalimide-Based Carboxylate Probe and Molecularly Imprinted Polymers Prepared Thereof**

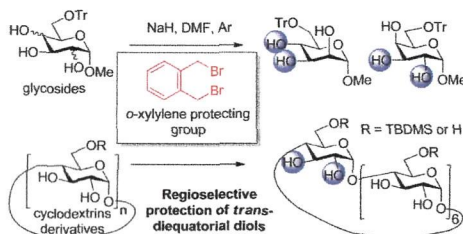
Ricarda Wagner, Wei Wan, Mustafa Biyikal, Elena Benito-Peña, María Cruz Moreno-Bondi, Issam Lazraq, Knut Rurack,\* and Börje Sellergren\*

1390 **S**

dx.doi.org/10.1021/jo302178f

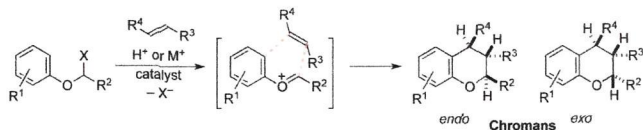
***o*-Xylylene Protecting Group in Carbohydrate Chemistry: Application to the Regioselective Protection of a Single *vic*-Diol Segment in Cyclodextrins**

Patricia Balbuena, Rita Gonçalves-Pereira, José L. Jiménez Blanco,\* M. Isabel García-Moreno, David Lesur, Carmen Ortiz Mellet, and José M. García Fernández\*



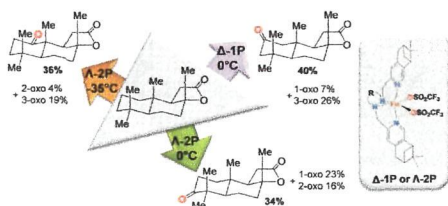
### A Hetero Diels–Alder Approach to the Synthesis of Chromans (3,4-Dihydrobenzopyrans) Using Oxonium Ion Chemistry: The Oxa-Povarov Reaction

Rivka R. R. Taylor and Robert A. Batey\*



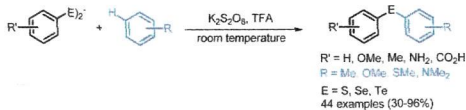
### Regioselective Oxidation of Nonactivated Alkyl C–H Groups Using Highly Structured Non-Heme Iron Catalysts

Laura Gómez, Mercè Canta, David Font, Irene Prat, Xavi Ribas, and Miquel Costas\*



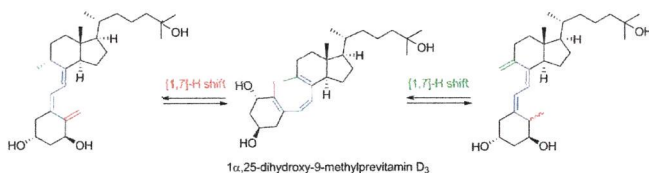
### Transition-Metal-Free Synthesis of Unsymmetrical Diaryl Chalcogenides from Arenes and Diaryl Dichalcogenides

Ch Durga Prasad, Shah Jaimin Balkrishna, Amit Kumar, Bhagat Singh Bhakuni, Kaustubh Shrimali, Soumava Biswas, and Sangit Kumar\*



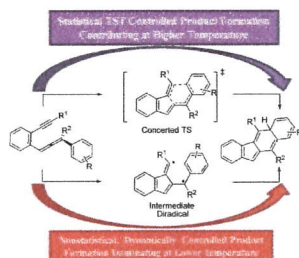
### Synthesis of 9-Alkylated Calcitriol and Two 1 $\alpha$ ,25-Dihydroxy-9-methylene-10,19-dihydrovitamin D<sub>3</sub> Analogues with a Non-natural Triene System by Thermal Sigmatropic Rearrangements

Urszula Kulesza, Rita Sigüeiro, Antonio Mourino,\* and Rafal R. Siciński\*



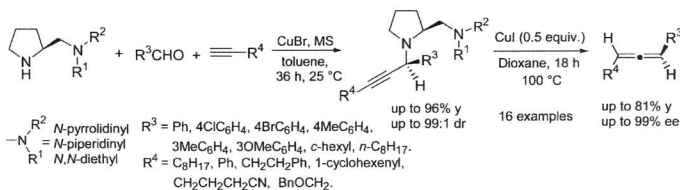
### Nonstatistical Dynamic Effects in the Thermal C<sup>2</sup>–C<sup>6</sup> Diels–Alder Cyclization of Enyne–Allenes

Debabrata Samanta, Mehmet Emin Cinar, Kalpataru Das, and Michael Schmittel\*



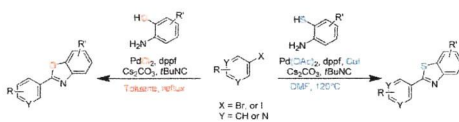
### Copper(I) Halide Promoted Diastereoselective Synthesis of Chiral Propargylamines and Chiral Allenes using 2-Dialkylaminoethylpyrrolidine, Aldehydes, and 1-Alkynes

Ramani Gurubrahmam and Mariappan Periasamy\*



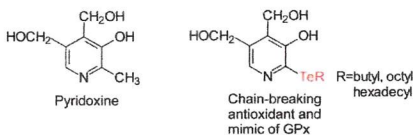
### Mechanistic Exploration of the Palladium-catalyzed Process for the Synthesis of Benzoxazoles and Benzothiazoles

Valentin N. Bochatay, Patrick J. Boissarie, John A. Murphy, Colin J. Suckling, and Stuart Lang\*



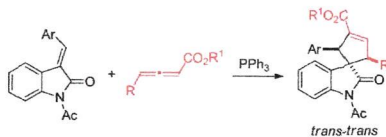
### Turning Pyridoxine into a Catalytic Chain-Breaking and Hydroperoxide-Decomposing Antioxidant

Vijay P. Singh, Jia-fei Poon, and Lars Engman\*



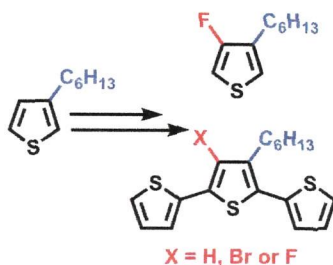
### Phosphine-Catalyzed Synthesis of 3,3-Spirocyclopenteneoxindoles from $\gamma$ -Substituted Allenoates: Systematic Studies and Targeted Applications

Catherine Gomez, Maxime Gicquel, Jean-Christophe Carry, Laurent Schio, Pascal Retailleau, Arnaud Voituriez,\* and Angela Marinetti\*



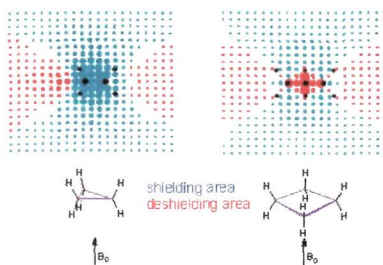
### 3-Fluoro-4-hexylthiophene as a Building Block for Tuning the Electronic Properties of Conjugated Polythiophenes

Frédéric Gohier, Pierre Frère,\* and Jean Roncali



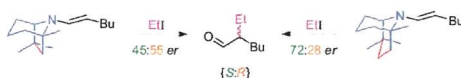
### $^1\text{H}$ NMR Chemical Shifts of Cyclopropane and Cyclobutane: A Theoretical Study

Marija Baranac-Stojanović\* and Milovan Stojanović



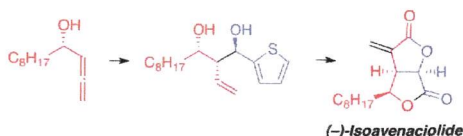
### C-Alkylation of Chiral Tropane- and Homotropane-Derived Enamines

David M. Hodgson,\* Andrew Charlton, Robert S. Paton,\* and Amber L. Thompson



**Total Synthesis of (–)-Isoavenaciolide**

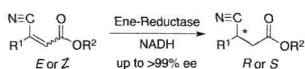
David Santos, Xavier Ariza,\* Jordi Garcia,\* Paul Lloyd-Williams, Agustin Martínez-Laporta, and Carolina Sánchez

**1525 **S****

dx.doi.org/10.1021/jo302484p

**Chemoenzymatic Asymmetric Synthesis of Pregabalin Precursors via Asymmetric Bioreduction of  $\beta$ -Cyanoacrylate Esters Using Ene-Reductases**

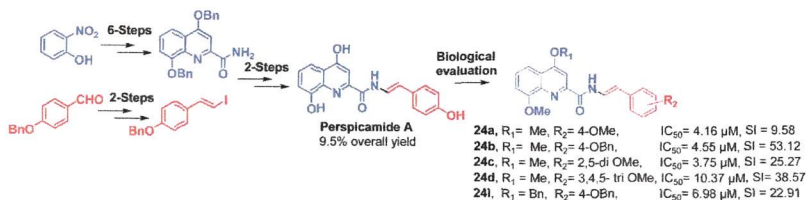
Christoph K. Winkler, Dorina Clay, Simon Davies, Pat O'Neill, Paul McDaid, Sebastien Debarge, Jeremy Steffik, Mike Karmilowicz, John W. Wong,\* and Kurt Faber\*

**1534 **S****

dx.doi.org/10.1021/jo3025626

**Synthesis of Perspicamide A and Related Diverse Analogues: Their Bioevaluation as Potent Antileishmanial Agents**

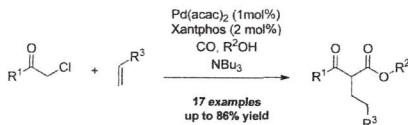
Anand Kumar Pandey, Rashmi Sharma, Rahul Shivahare, Ashish Arora, Neeraj Rastogi, Suman Gupta, and Prem M. S. Chauhan\*

**1547 **S****

dx.doi.org/10.1021/jo3026143

**Synthesis of  $\alpha$ -Alkylated  $\beta$ -Ketoesters by Alkoxyacylation/Michael Addition Domino Reaction**

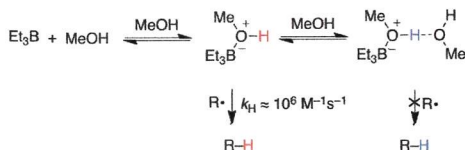
Benoit Wahl, Yann Philipson, Hélène Bonin, André Mortreux, and Mathieu Sauthier\*





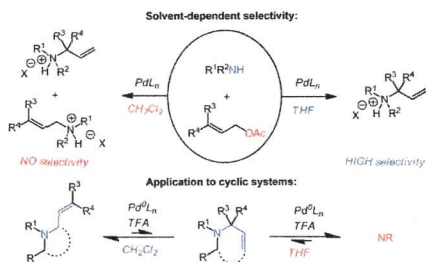
### Role of Equilibrium Associations on the Hydrogen Atom Transfer from the Triethylborane–Methanol Complex

Guillaume Povie, Mattia Marzorati, Peter Bigler, and Philippe Renaud\*



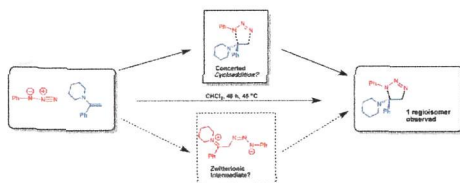
### Achieving Control over the Branched/Linear Selectivity in Palladium-Catalyzed Allylic Amination

Igor Dubovyk, Iain D. G. Watson, and Andrei K. Yudin\*



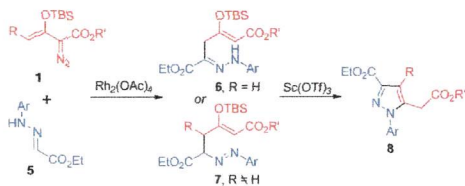
### Mechanisms and Transition States of 1,3-Dipolar Cycloadditions of Phenyl Azide with Enamines: A Computational Analysis

Steven A. Lopez, Morton E. Munk, and K. N. Houk\*



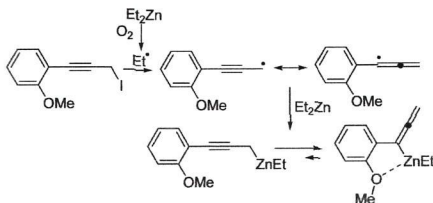
### Vinylogous Reactivity of Enol Diazoacetates with Donor–Acceptor Substituted Hydrazones. Synthesis of Substituted Pyrazole Derivatives

Xinfang Xu, Peter Y. Zavalij, Wenhao Hu, and Michael P. Doyle\*

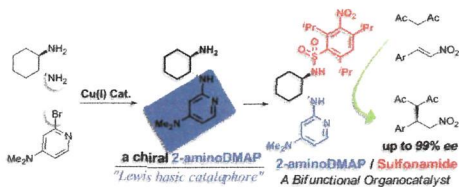


**Theoretical Support for the Involvement of a Radical Pathway in the Formation of Allenylzincs from Propargyl Iodides and Dialkylzincs: Influence of Zinc Coordination**

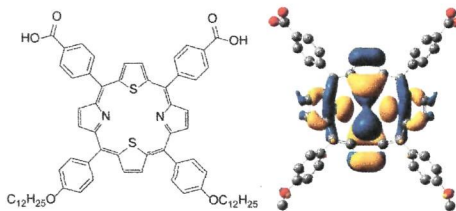
Surribabu Jammi, Dominique Mouysset, Didier Siri,\* Michèle P. Bertrand,\* and Laurence Feray\*

**Cu-Catalyzed Selective Mono-N-pyridylation: Direct Access to 2-AminoDMAP/Sulfonamides as Bifunctional Organocatalysts**

Murat Isik and Cihangir Tanyeli\*

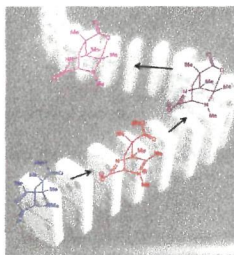
**Synthesis and Optical and Electronic Properties of Core-Modified 21,23-Dithiaporphyrins**

Ashley D. Bromby, Ryan P. Jansonius, and Todd C. Sutherland\*



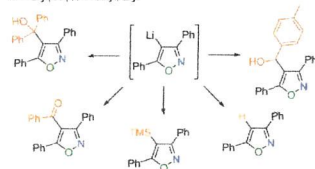
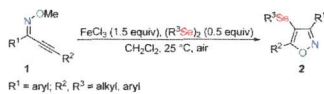
### A DFT Study of the [3 + 2] versus [4 + 2] Cycloaddition Reactions of 1,5,6-Trimethylpyrazinium-3-olate with Methyl Methacrylate

Luis R. Domingo,\* Jose A. Sáez, John A. Joule, Lydia Rhyman, and Ponnadurai Ramasami



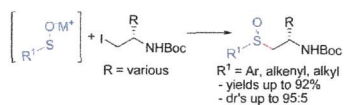
### Iron(III) Chloride/Diorganyl Diselenides: A Tool for Intramolecular Cyclization of Alkynone O-Methyloximes

Adriane Sperança, Benhur Godoi, and Gilson Zeni\*



### Sulfonate Substitution as a Complement and Alternative to Sulfoxidation in the Diastereoselective Preparation of Chiral $\beta$ -Substituted $\beta$ -Amino Sulfoxides

Stefan C. Söderman and Adrian L. Schwan\*



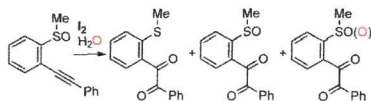
# Notes

1650 **S**

dx.doi.org/10.1021/jo3023186

**$\alpha$ -Diketone Formation Accompanied by Oxidation of Sulfur Functional Group by the Reaction of *o*-Alkynylarenesulfoxide with Iodine**

Shoji Matsumoto,\* Hiroyuki Shibata, and Motohiro Akazome

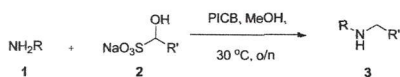


1655 **S**

dx.doi.org/10.1021/jo3023878

**Direct Reductive Amination of Aldehyde Bisulfite Adducts Induced by 2-Picoline Borane: Application to the Synthesis of a DPP-IV Inhibitor**

Margaret Faul, Rob Larsen, Adam Levinson, Jason Tedrow, and Filisaty Vounatsos\*

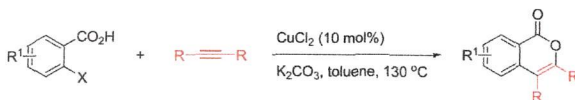


1660 **S**

dx.doi.org/10.1021/jo302499k

**Synthesis of Isocoumarin Derivatives by Copper-Catalyzed Addition of *o*-Halobenzoic Acids to Active Internal Alkynes**

Xun-Xiang Guo\*

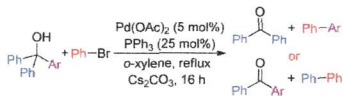


1665 **S**

dx.doi.org/10.1021/jo302592g

**Steric and Electronic Effects Influencing  $\beta$ -Aryl Elimination in the Pd-catalyzed Carbon–Carbon Single Bond Activation of Triarylmethanols**

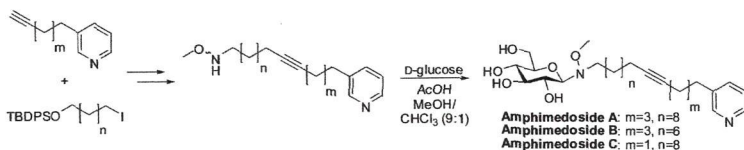
James R. Bour, Jacob C. Green, Valerie J. Winton, and Jeffrey B. Johnson\*



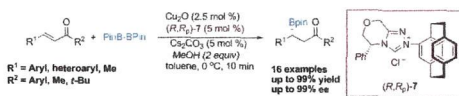
- turnover limiting  $\beta$ -aryl elimination  
-  $\beta$ -aryl elimination promoted by *o*-substitution,  
strongly electron withdrawing or donating groups

## Amphimedosides A–C: Synthesis, Chemoselective Glycosylation, And Biological Evaluation

Joseph M. Langenhan,\* Edouard Mullarky, Derek K. Rogalsky, James R. Rohlfing, Anja E. Tjaden, Halina M. Werner, Leonardo M. Rozal, and Steven A. Loskot

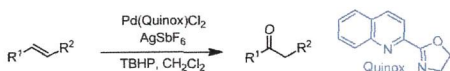
Enantioselective  $\beta$ -Boration of Acyclic Enones by a [2.2]Paracyclophane-Based N-Heterocyclic Carbene Copper(I) Catalyst

Lei Zhao, Yudao Ma,\* Fuyan He, Zengzeng Duan, Jianqiang Chen, and Chun Song\*



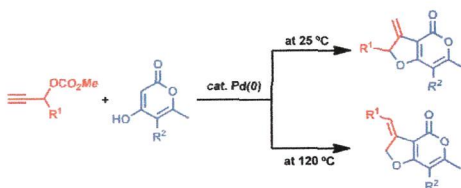
## Wacker-Type Oxidation of Internal Alkenes using Pd(Quinox) and TBHP

Ryan J. DeLuca, Jennifer L. Edwards, Laura D. Steffens, Brian W. Michel, Xiaoxiao Qiao, Chunyin Zhu, Silas P. Cook,\* and Matthew S. Sigman\*



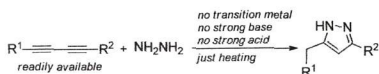
## Regiocontrolled Construction of Furo[3,2-c]pyran-4-one Derivatives by Palladium-Catalyzed Cyclization of Propargylic Carbonates with 4-Hydroxy-2-pyrones

Masahiro Yoshida,\* Tomomi Nakagawa, Kouki Kinoshita, and Koza Shishido



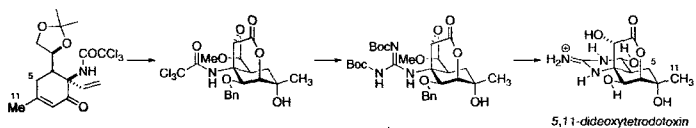
## Synthesis of 3,5-Disubstituted Pyrazoles via Cope-Type Hydroamination of 1,3-Dialkynes

Liangguang Wang, Xiaoqiang Yu,\* Xiujuan Feng, and Ming Bao\*



**An Improved Synthesis of (-)-5,11-Dideoxytetradotoxin**

Masaatsu Adachi, Takuya Imazu, Minoru Isobe, and Toshio Nishikawa\*

**Additions and Corrections****Correction to Expedient Microwave-Assisted Synthesis of 5-Alkoxyoxazoles from  $\alpha$ -Triflyloxy Esters and Nitriles**

Laurie-Anne Jouanno, Cyrille Sabot, and Pierre-Yves Renard\*