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# IN THIS ISSUE

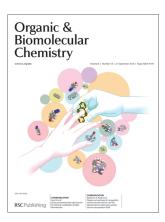
ISSN 1477-0520 CODEN OBCRAK 8(18) 4029-4184 (2010)



# Cover

See Thomas C. Nugent et al., pp. 4085-4089 Archimedes' screw, an ingenious device synonymous with water transport, is depicted here as the water active 1,2-diamine catalyst converting low value starting materials into high value multifunctional aldol products. Graphic Art Credit: Michael Holt (Bremen, Germany).

Image reproduced by permission of Thomas C. Nugent from Org. Biomol. Chem., 2010, 8, 4085.



# Inside cover

See Isao Kii et al., pp. 4051-4055. The wedding ring. He is putting the Sondheimer diyne on her finger and they are covalently joined together. This wedding ring provides a novel convergent method, "strain-promoted double-click reaction" enabling us to modify azido-biomolecules with a small reporter azide.

Image reproduced by permission of Takamitsu Hosoya from Org. Biomol. Chem., 2010, 8, 4051.

# **PERSPECTIVE**

# 4043

In water, on water, and by water: mimicking nature's aldolases with organocatalysis and water

Nobuyuki Mase\* and Carlos F. Barbas, III\*

Organocatalysis and water combine two environmentally friendly concepts in a single system. The explosive development of organocatalytic direct transformations in aqueous media without added organic solvent is reviewed.



# **COMMUNICATIONS**

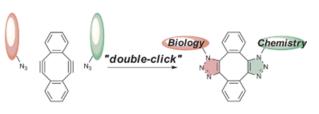
# 4051



# Strain-promoted double-click reaction for chemical modification of azido-biomolecules

Isao Kii,\* Akira Shiraishi, Toshiyuki Hiramatsu, Takeshi Matsushita, Hidehiro Uekusa, Suguru Yoshida, Makoto Yamamoto, Akira Kudo, Masatoshi Hagiwara and Takamitsu Hosoya\*

The strain-promoted "double-click" (SPDC) reaction using Sondheimer diyne, a novel convergent method conjugating three molecules spontaneously, has enabled us to readily modify an azido-biomolecule with a small reporter azido-molecule.



The Sondheimer diyne acts as a matchmaker between chemistry and biology.

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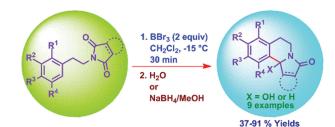
# **COMMUNICATIONS**

# 4056

# An unusual reactivity of BBr<sub>3</sub>: Accessing tetrahydroisoquinoline units from N-phenethylimides

Jayaraman Selvakumar, Alexandros Makriyannis and Chinnasamy Ramaraj Ramanathan\*

A simple methodology to construct the tetrahydroisoquinoline containing skeleton through electrophilic activation of imide carbonyl group of N-phenethylimides using BBr<sub>3</sub> as Lewis acid is developed.



4059

# Efforts toward elucidating Thalidomide's molecular target: an expedient synthesis of the first Thalidomide biotin analogue

Scott G. Stewart,\* Carlos J. Braun, Marta E. Polomska, Mahdad Karimi, Lawrence J. Abraham and Keith A. Stubbs

The synthesis of the first Thalidomide-biotin analogue is described via a Huisgen 1,3-dipolar cycloaddition (click reaction) as the key synthetic step. This compound will help unlock investigations into the as yet unknown molecular mode of action of Thalidomide.

4063



# Bifunctional thiourea-promoted cascade aza-Michael-Henry-dehydration reactions: asymmetric preparation of 3-nitro-1,2-dihydroquinolines

Xiaoqian Liu and Yixin Lu\*

A cascade aza-Michael-Henry-dehydration reaction led to a one-step preparation of 3-nitro-1,2-dihydroquinolines in high yields and up to 90% ee.

4066



# Design and synthesis of nonpeptidic, small molecule inhibitors for the Mycobacterium tuberculosis protein tyrosine phosphatase PtpB

Katherine A. Rawls, Christoph Grundner and Jonathan A. Ellman\*

The design and synthesis of small molecules with low micromolar inhibition of *Mycobacterium tuberculosis* protein tyrosine phosphatase PtpB is described.

# **COMMUNICATIONS**

4071

# Q.

# Direct oxidative coupling of benzenes with acrylonitriles to cinnamonitriles catalyzed by $Pd(OAc)_2/HPMoV/O_2$ system

Yasushi Obora,\* Yoshihisa Okabe and Yasutaka Ishii\*

A facile direct synthesis of cinnamonitriles from acrylonitriles and benzenes is successfully achieved by using Pd(OAc)<sub>2</sub>/HPMoV/O<sub>2</sub> catalyst system *via* the direct C–H bond activation of benzenes using molecular oxygen as a terminal oxidant.

# 4074



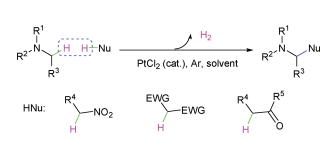
# Stereoselective synthesis of vinyl-substituted (Z)-stilbenes by rhodium-catalysed addition of arylboronic acids to allenic alcohols

Tomoya Miura, Hiroshi Shimizu, Tomohiro Igarashi and Masahiro Murakami\*

The reaction of 4-arylbuta-2,3-dien-1-ols with arylboronic acids in the presence of a  $\operatorname{rhodium}(I)$  catalyst gave  $\operatorname{vinylsubstituted}(Z)$ -stilbenes stereoselectively.

# 4077





# Platinum-catalyzed cross-dehydrogenative coupling reaction in the absence of oxidant

Xing-Zhong Shu, Yan-Fang Yang, Xiao-Feng Xia, Ke-Gong Ji, Xue-Yuan Liu and Yong-Min Liang\*

A third strategy for cross-dehydrogenative coupling reaction has been reported *via* platinum catalysis in the absence of oxidant.

# **PAPERS**

4080





# The gas phase Smiles rearrangement of anions $PhO(CH_2)_nO^-$ (n=2-4). A joint theoretical and experimental approach

Tianfang Wang, Nico M. M. Nibbering and John H. Bowie\*

A combination of theory and experiment shows that energized PhOCH<sub>2</sub>CH<sub>2</sub>O<sup>-</sup> exclusively undergoes the Smiles rearrangement.

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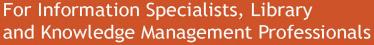
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# 4085

# Picolylamine as an organocatalyst template for highly diastereo- and enantioselective aqueous aldol reactions

Thomas C. Nugent,\* M. Naveed Umar and Ahtaram Bibi

A pyridine based 1,2-diamine containing only one stereogenic center has been identified for fast aldol reactions. Using 2-5 mol% of (R)- or (S)-PicAm-2, cyclohexanone (3.3 equiv) readily undergoes aldol reactions with o-, m-, and p-substituted aromatic aldehyde partners. Furthermore, the new catalyst excels when examining functionalized cyclic ketone addition to aldehydes (Scheme 2).

4090

# Gold(I)-catalysed alcohol additions to cyclopropenes

Maximillian S. Hadfield, Jürgen T. Bauer, Pauline E. Glen and Ai-Lan Lee\*

Gold(I)-catalysed addition of alcohols to 3,3-disubstituted cyclopropenes occurs in a highly regioselective and facile manner to produce alkyl tert-allylic ethers in good yields. The reaction is tolerant of sterically hindered substituents on the cyclopropene as well as primary and secondary alcohols as nucleophiles.

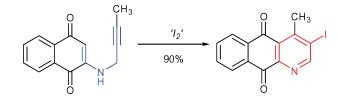
4096



# Azaanthraquinone assembly from N-propargylamino quinone via iodine-induced 6-endo-dig electrophilic cyclization

Na Fei, Qiwen Hou, Shaozhong Wang,\* Huaqin Wang and Zhu-Jun Yao

A metal-free protocol taking advantage of the excellent nucleophilicity of aminoquinone to assemble the azaanthraquinone framework was developed by employing an iodine-induced 6-endo-dig electrophilic cyclization.



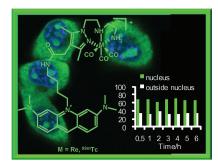
4104



# Tricarbonyl M(I) (M = Re, $^{99m}$ Tc) complexes bearing acridine fluorophores: synthesis, characterization, DNA interaction studies and nuclear targeting

Teresa Esteves, Catarina Xavier, Sofia Gama, Filipa Mendes, Paula D. Raposinho, Fernanda Marques, António Paulo, João Costa Pessoa, José Rino, Giampietro Viola and Isabel Santos\*

Nuclear-targeting and DNA intercalation of Re and 99mTc-tricarbonyl complexes.



# 4117

# alcohol dehydrogenases Y up to 99% (S)/(R) up to 99:1 OH

# Chemoenzymatic synthesis of (2S)-2-arylpropanols through a dynamic kinetic resolution of 2-arylpropanals with alcohol dehydrogenases

Paola Galletti,\* Enrico Emer, Gabriele Gucciardo, Arianna Quintavalla, Matteo Pori and Daria Giacomini\*

(2S)-2-Arylpropanols, useful intermediates in the synthesis of Profens, were obtained by means of a dynamic kinetic resolution (DKR) of racemic 2-arylpropanals. The DKR process combines an enzymatic reduction with a chemical base-racemization of the unreacted aldehyde.

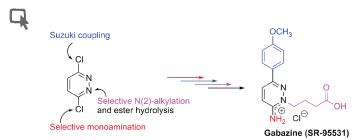
$$R^3 - O - N$$
 $R^2$ 
 $R^1 = Me, n-Bu$ 
 $R^2 = H, Me, F$ 
 $R^3 = H, Me$ 

# Acyclic triaryl olefins possessing a sulfohydroxamic acid pharmacophore: synthesis, nitric oxide/nitroxyl release, cyclooxygenase inhibition, and anti-inflammatory studies

Zhangjian Huang, Carlos Velázquez, Khaled Abdellatif, Morshed Chowdhury, Sarthak Jain, Julie Reisz, Jenna DuMond, S. Bruce King and Edward Knaus\*

A group of sulfohydroxamic acids were synthesized and evaluated as COX isozyme inhibitors, NO and HNO donors, and anti-inflammatory

# 4131

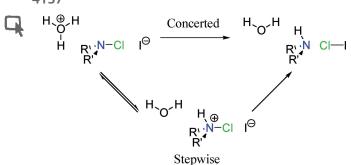


# Microwave-enhanced synthesis of 2,3,6-trisubstituted pyridazines: application to four-step synthesis of gabazine (SR-95531)

Navnath Gavande, Graham A. R. Johnston, Jane R. Hanrahan\* and Mary Chebib\*

Microwave-enhanced, highly efficient protocols for the synthesis of synthetically and biologically important 2,3,6-trisubstituted pyridazine architectures have been developed by sequential amination/Suzuki coupling/alkylation reactions. The synthesis of gabazine (SR-95531) has been achieved in four steps and 73% overall yield.

# 4137



# Acid-catalysed chlorine transfer from N-chloramines to iodide ion: experimental evidence for a predicted change in mechanism

Paula Calvo, Juan Crugeiras\* and Ana Ríos

A change from a stepwise to a concerted mechanism for acid catalysis of chlorine transfer to iodide ion is observed upon decreasing the basicity of the leaving amine. This change appears to occur when the protonated chloramine ceases to have a significant lifetime in the presence of the nucleophile.

# 4143

# Rhodamine-based chemosensor for Hg<sup>2+</sup> in aqueous solution with a broad pH range and its application in live cell imaging

Yun Zhao, Yue Sun, Xin Lv, Yunlong Liu, Maliang Chen and Wei Guo\*

A rhodamine B-based chemosensor bearing an 8-hydroxyquinoline chelating group was found to exhibit dual chromo- and fluorogenic responses toward Hg2+ with high selectivity and sensitivity.



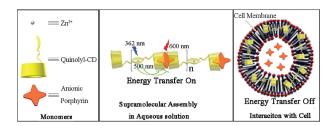
4148



# Construction and transmembrane dissociation behavior of supramolecular assembly of quinolinocyclodextrin with porphyrin

Miao Yu, Yong Chen, Ning Zhang and Yu Liu\*

A non-covalently constructed Zn<sup>2+</sup>/cyclodextrin/porphyrin supramolecule exhibited environment-sensitive association/dissociation behaviors, which referred to a controlled transmembrane release



4155



# An efficient route to xanthine based $A_{2A}$ adenosine receptor antagonists and functional derivatives

Paul LaBeaume, Ma Dong, Michail Sitkovsky, Elizabeth V. Jones, Rhiannon Thomas, Sara Sadler, Amy E. Kallmerten and Graham B. Jones\*

A one-pot route to 8-substituted xanthines has been developed from 5,6-diaminouracils and carboxaldehydes. Yields are good and the process applicable to a range of substrates including a family of  $A_{2A}$ adenosine receptor antagonists.

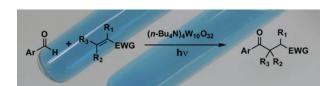
4158



# Benzoyl radicals from (hetero)aromatic aldehydes. Decatungstate photocatalyzed synthesis of substituted aromatic ketones

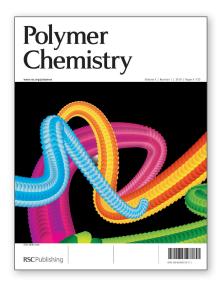
Davide Ravelli, Michele Zema, Mariella Mella, Maurizio Fagnoni\* and Angelo Albini

Benzoyl radicals are generated directly from (hetero)aromatic aldehydes upon tetrabutylammonium decatungstate ((n-Bu<sub>4</sub>N)<sub>4</sub>W<sub>10</sub>O<sub>32</sub>), TBADT photocatalysis under mild conditions. In the presence of  $\alpha,\beta$ -unsaturated esters, ketones and nitriles radical conjugate addition ensues and gives the corresponding β-functionalized aryl alkyl ketones.





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36098

# 4165



# Switching from (R)- to (S)-selective chemoenzymatic DKR of amines involving sulfanyl radical-mediated racemization

Lahssen El Blidi, Nicolas Vanthuyne, Didier Siri, Stéphane Gastaldi,\* Michèle P. Bertrand\* and Gérard Gil\*

Chemoenzymatic dynamic kinetic resolution of amines involving sulfanyl radical-induced racemization happened to be the very first switchable DKR process allowing the synthesis of either (R)- or (S)-amides.

# 4169



# Substituent effect of group 14 elements on the ring-opening reaction of cyclobutene

Munehiro Hasegawa, Ippei Usui, Soichiro Konno and Masahiro Murakami\*

A series of cyclobutenes bearing group 14 elements at the 3-position were synthesized, and their rotational behaviors in the thermal ring-opening reaction are discussed.

E = C, Si, Ge, Sn

# 4176



# 2',4'-BNA bearing a 2-pyridine nucleobase for CG base pair recognition in the parallel motif triplex DNA

Yoshiyuki Hari,\* Sachiko Matsugu, Hiroyasu Inohara, Yuri Hatanaka, Masaaki Akabane, Takeshi Imanishi and Satoshi Obika\*

2',4'-BNA bearing a 2-pyridine nucleobase (PyB, see picture) in triplex-forming oligonucleotides was able to recognize a CG base pair within dsDNA with high sequence-selectivity and strong binding affinity.

# **AUTHOR INDEX**

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