

Organic & Biomolecular Chemistry

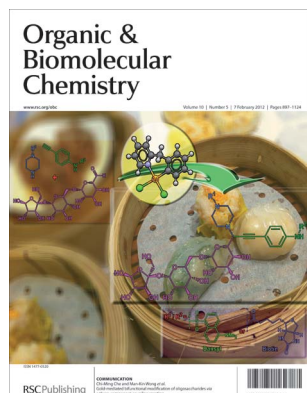
An international journal of synthetic, physical and biomolecular organic chemistry

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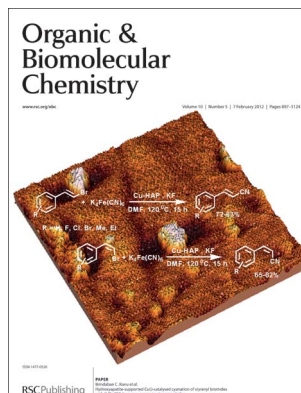
ISSN 1477-0520 CODEN OBCRAK 10(5) 897–1124 (2012)



Cover

See Karen Ka-Yan Kung *et al.*, pp. 925–930.

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Inside cover

See Debasree Saha *et al.*, pp. 952–957.

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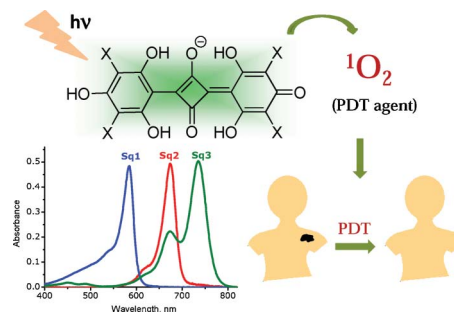
PERSPECTIVE

911

Squaraine dyes in PDT: from basic design to *in vivo* demonstration

Rekha R. Avirah, Dhanya T. Jayaram, Nagappanpillai Adarsh and Danaboyina Ramaiah*

This perspective article highlights the recent developments of squaraines as PDT sensitizers including design, tuning of photophysical and photobiological properties.



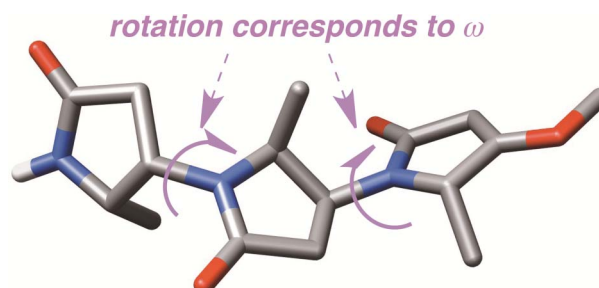
COMMUNICATIONS

921

Omegetides: constrained analogs of peptide primary sequence

Dmytro Fedoseyenko, Arjun Raghuraman, Eunhwa Ko and Kevin Burgess*

Only one twist is allowed by joining tetramic acid units into “omegetide” peptidomimetics. This new design is complementary to all the existing ways of to mimic peptide primary sequences.



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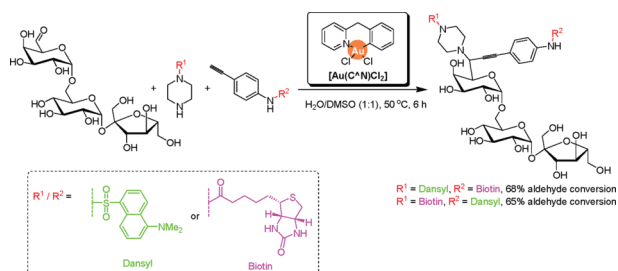
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925

Gold-mediated bifunctional modification of oligosaccharides *via* a three-component coupling reaction

Karen Ka-Yan Kung, Gai-Li Li, Lan Zou, Hiu-Chi Chong, Yun-Chung Leung, Ka-Hing Wong, Vanessa Kar-Yan Lo, Chi-Ming Che* and Man-Kin Wong*

Single-site incorporation of two independent functionalities (amines and alkynes) into aldehyde-containing oligosaccharides by using a one-pot gold-mediated three-component coupling reaction.



931

The total synthesis of a ganglioside Hp-s1 analogue possessing neuritogenic activity by chemoselective activation glycosylation

Yow-Fu Tsai,* Cheng-Hua Shih, Yu-Ting Su, Chun-Hsu Yao, Jang-Feng Lian, Chun-Chen Liao, Ching-Wu Hsia, Hao-Ai Shui and Rashmi Rani

The total synthesis of a ganglioside Hp-s1 analogue was accomplished by a chemoselective-activation glycosylation strategy; the analogue exhibited neuritogenic activity toward the human neuroblastoma cell line SH-SY5Y without the presence of NGF.



935

Thiolation of symmetrical and unsymmetrical diketopiperazines

Bettina M. Ruff, Sabilla Zhong, Martin Nieger and Stefan Bräse*

Treatment of diketopiperazines with base and electrophilic sulfur reagents yielded symmetrical and unsymmetrical monomethylthio-, bis(methylthio)- and epithiodiketopiperazines.

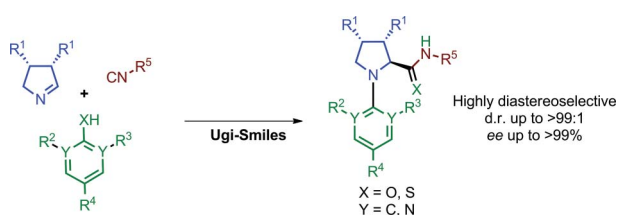


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Stereoselective synthesis of *N*-aryl proline amides by biotransformation–Ugi–Smiles sequence

Anass Znabet, Sara Blanken, Elwin Janssen, Frans J. J. de Kanter, Madeleine Helliwell, Nicholas J. Turner, Eelco Ruijter* and Romano V. A. Orru*

The combination of biocatalytic desymmetrization of cyclic *meso*-amines with Ugi–Smiles multicomponent chemistry produced pharmaceutically relevant, optically pure *N*-aryl proline amides.



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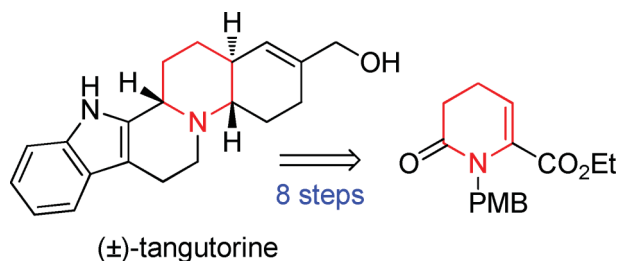
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Total synthesis of the monoterpene indole alkaloid (\pm)-tangutorine

Sebastiaan (Bas) A. M. W. van den Broek,
Jaap G. H. Lemmers, Floris L. van Delft and
Floris P. J. T. Rutjes*

An efficient formal synthesis of the monoterpene indole alkaloid (\pm)-tangutorine was realized starting from a cyclic dehydroamino ester.

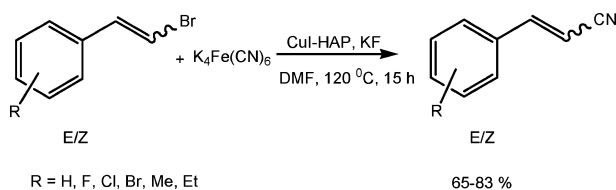


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Hydroxyapatite-supported Cu(I)-catalysed cyanation of styrenyl bromides with $K_4[Fe(CN)_6]$: an easy access to cinnamonitriles

Debasree Saha, Laksmikanta Adak, M. Mukherjee and
Brindaban C. Ranu*

An efficient cyanation of styrenyl bromides by $K_4[Fe(CN)_6]$ has been achieved under the catalysis of hydroxyapatite-supported copper(I) producing a variety of functionalized cinnamonitriles in high yields. The stereochemistry of the styrenyl double bond is preserved during the process providing only one stereoisomer.

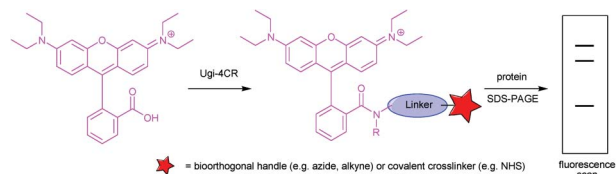


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Fast and efficient MCR-based synthesis of clickable rhodamine tags for protein profiling

Sebastian Brauch, Michael Henze, Bianca Osswald,
Kai Naumann, Ludger A. Wessjohann, Sander S. van Berkel
and Bernhard Westermann*

A one-pot protocol for the synthesis of activated fluorescent labels (*i.e.* azide, alkynyl or NHS), based on the Ugi-4-component reaction, is presented.

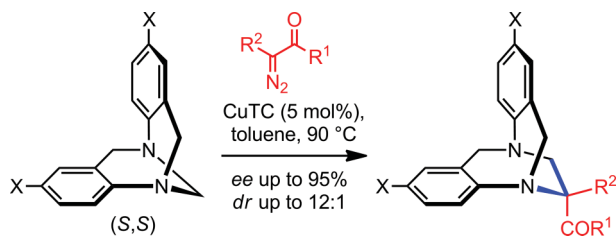


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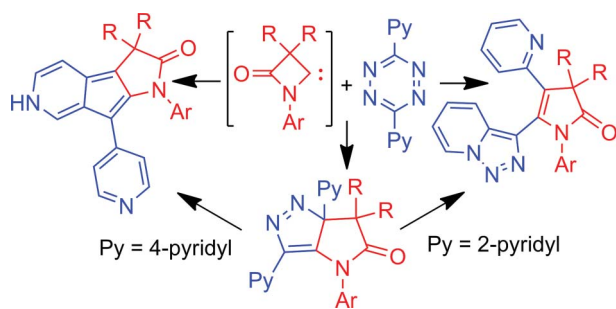
Asymmetric synthesis of ethano-Tröger bases using CuTC-catalyzed diazo decomposition reactions

Ankit Sharma, Céline Besnard, Laure Guénée and
Jérôme Lacour*

Ethano-bridged Tröger bases are readily prepared using diazo decomposition reactions. Under CuTC catalysis excellent levels of diastereo- and enantio-control (*dr* and *ee* up to 12:1 and 95% respectively) are obtained with aryl diazoketone precursors.



970

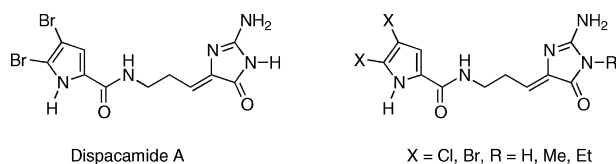


The reaction of β -lactam carbenes with 3,6-dipyridyltetrazines: switch of reaction pathways by 2-pyridyl and 4-pyridyl substituents of tetrazines

Xiao-Rong Wang, Juan Xing, Cai-Xia Yan and Ying Cheng*

The reactions between β -lactam carbenes and 3,6-dipyridyltetrazines were switched by the 2- and 4-pyridyl substituents to produce 5-triazolo[1,5-*a*]pyridylpyrrol-2-ones or pyrido[*c*]cyclopenta[*b*]pyrrol-2-ones.

978

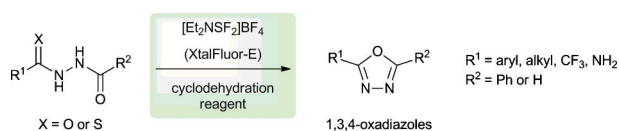


An efficient approach to dispacamide A and its derivatives

Solène Guihéneuf, Ludovic Paquin, François Carreaux, Emilie Durieu, Laurent Meijer and Jean Pierre Bazureau*

Dispacamide A and new analogs of this marine alkaloid were prepared in seven steps with a good overall yield (12–33%). The six synthetic products have been obtained with a *Z* geometry and the key step of the strategy was a stereocontrolled Knoevenagel condensation under microwave dielectric heating in the last step.

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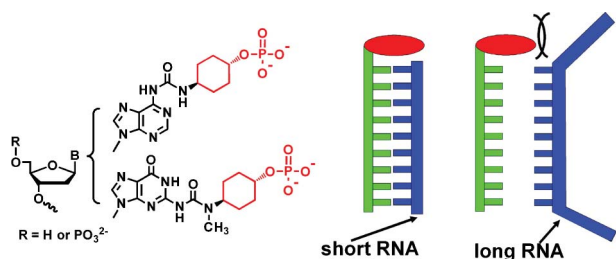


Synthesis of 1,3,4-oxadiazoles from 1,2-diacylhydrazines using [Et₂NSF₂]BF₄ as a practical cyclodehydration agent

Marie-France Pouliot, Laetitia Angers, Jean-Denys Hamel and Jean-François Paquin*

The preparation of 1,3,4-oxadiazoles from 1,2-diacylhydrazines using XtalFluor-E ([Et₂NSF₂]BF₄) as cyclodehydration reagent is described. Various functionalized 1,3,4-oxadiazoles were synthesized and it was found that the use of acetic acid as an additive generally improved the yields.

994



Short-RNA selective binding of oligonucleotides modified using adenosine and guanosine derivatives that possess cyclohexyl phosphates as substituents

Kohji Seio,* Sayako Kurohagi, Erika Kodama, Yoshiaki Masaki, Hirosuke Tsunoda, Akihiro Ohkubo and Mitsuo Sekine*

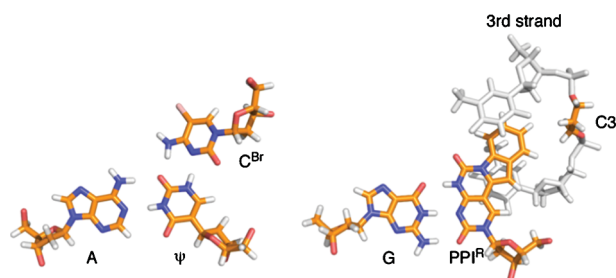
The oligonucleotides whose 5'-terminal residues were modified with bulky and negatively charged groups were developed and their short RNA selective binding were demonstrated.

1007

DNA duplexes and triplex-forming oligodeoxynucleotides incorporating modified nucleosides forming stable and selective triplexes

Takashi Kanamori, Yoshiaki Masaki, Masahiro Mizuta, Hirosuke Tsunoda, Akihiro Ohkubo, Mitsuo Sekine* and Kohji Seio*

Selective triplex formation between DNA duplexes incorporating artificial Watson–Crick base pairs and appropriately designed artificial TFOs was achieved.

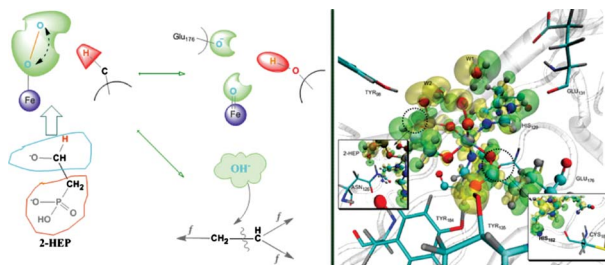


1014

The reaction mechanism of hydroxyethylphosphonate dioxygenase: a QM/MM study

Likai Du, Jun Gao,* Yongjun Liu, Dongju Zhang and Chengbu Liu*

The hydroxyethylphosphonate dioxygenase (HEPD) catalytic reaction mechanism with concatenated bifurcations provides further evidence against the proposed hydroperoxylation and hydroxylation mechanism.

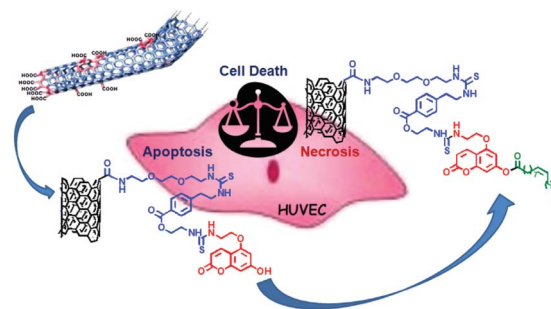


1025

Functionalization of multi-walled carbon nanotubes with coumarin derivatives and their biological evaluation

D. Iannazzo,* A. Piperno, A. Ferlazzo, A. Pistone,* C. Milone, M. Lanza, F. Cimino, A. Speciale, D. Trombetta, A. Saija and S. Galvagno

The synthesis and the biological evaluation of multi-walled carbon nanotubes linked to 5,7-coumarin and/or oleic acid residues were reported. Coumarin-conjugates are able to activate cell signaling pathways related to the apoptotic process without significantly influencing cell viability.

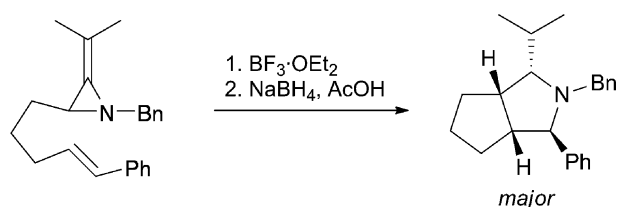


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Lewis acid promoted intramolecular (3 + 2) 'cycloadditions' of methyleneaziridines with alkene and alkyne acceptors

Karen Griffin, Cyril Montagne, Cam Thuy Hoang, Guy J. Clarkson and Michael Shipman*

Methyleneaziridines tethered to π -nucleophiles react with Lewis acids to give *cis*-octahydrocyclopenta[*c*]pyrroles in which up to four stereocentres can be created.



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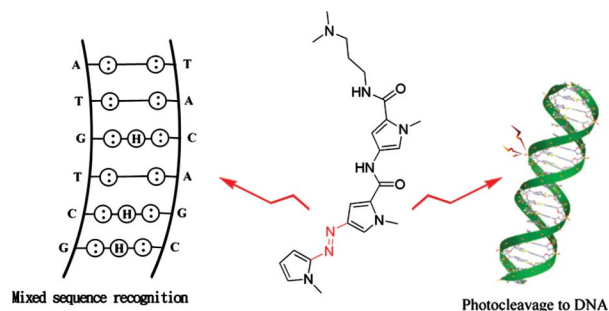
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Synthesis of directly linked diazine isosteres of pyrrole-polyamide that photochemically cleave DNA

Chi Wi Ong,* Ya-Ting Yang, Meng-Chi Liu, Keith R. Fox, Ping Hao Liu and Hung-Wei Tung

An isosteric diazine linked pyrrole-polyamide have been designed and synthesized as a strategy for mixed sequence recognition and photo-induce DNA damage.

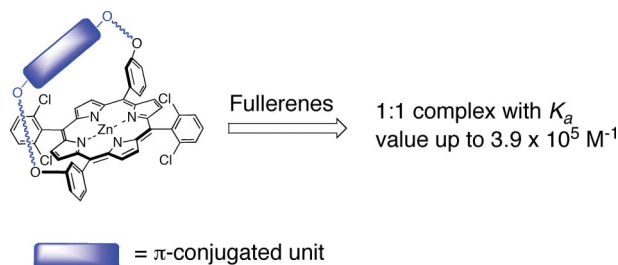


1047

New strapped porphyrins as hosts for fullerenes: synthesis and complexation study

Jean-Benoît Giguère and Jean-François Morin*

New strapped porphyrin-based hosts with different π -conjugated moieties have been prepared and their ability to complex fullerenes was studied in dilute solution. Electron-rich hosts can bind fullerenes with association constants of up to $3.9 \times 10^5 \text{ M}^{-1}$.

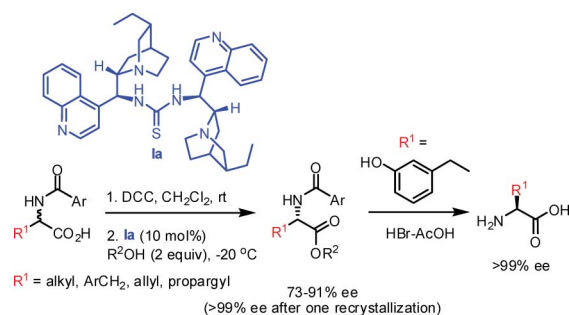


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Self-association free bifunctional thiourea organocatalysts: synthesis of chiral α -amino acids *via* dynamic kinetic resolution of racemic azlactones

Joong-Suk Oh, Ji-Woong Lee, Tae Hee Ryu, Jae Heon Lee and Choong Eui Song*

Concentration-independent high enantioselectivity in the dynamic kinetic resolution (DKR) of racemic azlactones affording chiral α -aminoesters has been achieved using self-association free thiourea-based dimeric cinchona alkaloid organocatalysts.

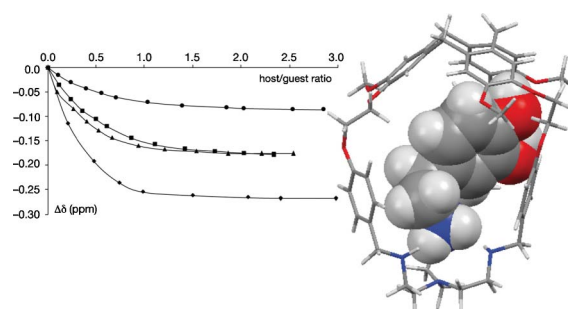


1056

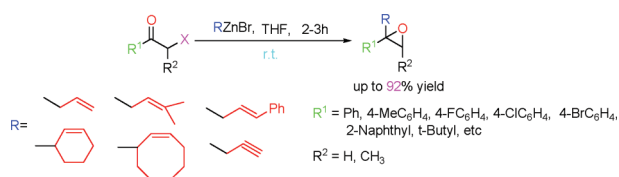
Hemicryptophane host as efficient primary alkylammonium ion receptor

Olivier Perraud, Sara Lefevre, Vincent Robert, Alexandre Martinez* and Jean-Pierre Dutasta*

The tren-hemicryptophane host is found to bind primary alkyl-ammonium efficiently and selectively, and is particularly suited for the recognition of dopamine neurotransmitter.



1060

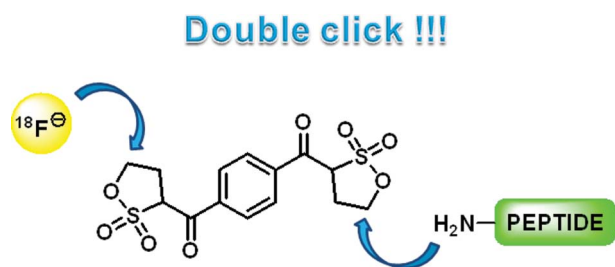


Efficient synthetic method for the preparation of allyl- and propargyl-epoxides by allylation and propargylation of α -halo ketones with organozinc reagents

Jie Pan, Min Zhang and Songlin Zhang*

The manuscript describes the synthesis of substituted allyl- and propargyl-epoxides by allylation and propargylation of α -halo ketones with organozinc reagents in mild conditions.

1068

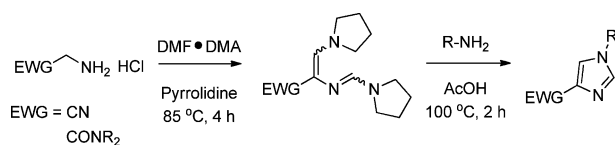


Synthesis and reactivity of a bis-sultone cross-linker for peptide conjugation and [^{18}F]-radiolabelling *via* unusual “double click” approach

Thomas Priem, Cédric Bouteiller,* David Camporese, Anthony Romieu and Pierre-Yves Renard

The synthesis, reactivity and bioconjugation ability of an original bis-propanesultone cross-linking reagent have been described for the first time. Its conversion into a prosthetic group suitable for peptide radiolabelling is also presented.

1079

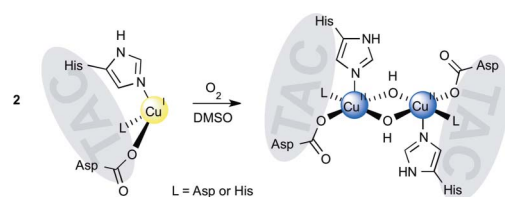


Regioselective synthesis of 1,4-disubstituted imidazoles

Michael A. Schmidt* and Martin D. Eastgate

A mild, regioselective synthesis of 1,4-disubstituted imidazoles, with a variety of nitrogen substitutions is described.

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Triazacyclophane (TAC)-scaffolded histidine and aspartic acid residues as mimics of non-heme metalloenzyme active sites

H. Bauke Albada, Fouad Soulimani, Hans J. F. Jacobs, Cees Versluis, Bert M. Weckhuysen and Rob M. J. Liskamp*

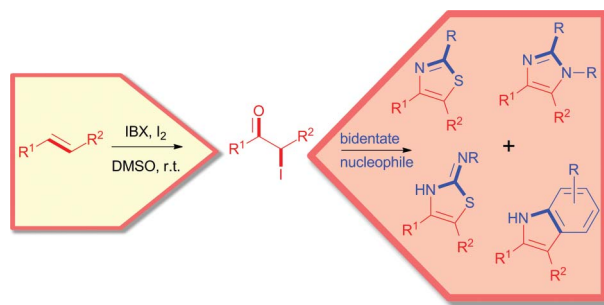
Two new structural mimics of aspartic acid and histidine based non-heme metalloenzyme active sites are described. This completes a first set of mimics of these prominent active sites.

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Direct preparation of thiazoles, imidazoles, imidazopyridines and thiazolidines from alkenes

Timothy J. Donohoe,* Mikhail A Kabeshov, Akshat H. Rathi and Ian E. D. Smith

A range of heterocycles, including thiazoles, imidazoles, imidazopyridines, thiazolidines and dimethoxyindoles, have been synthesised directly from alkenes *via* an efficient ketoidination/cyclisation protocol.

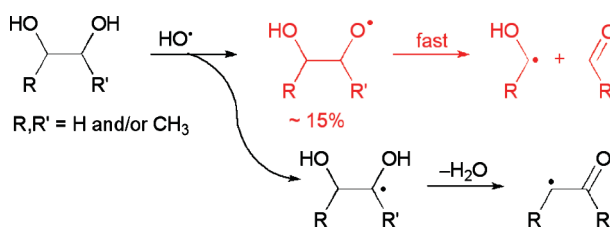


1102

Revisiting the reaction of hydroxyl radicals with vicinal diols in water

Dong Jiang, Sebastián Barata-Vallejo, Bernard T. Golding,* Carla Ferreri and Chrystostomos Chatgililoglu*

The reaction of hydroxyl radicals with 1,2-diols results in the formation of alkoxy radicals (~15%) and alkyl radicals.

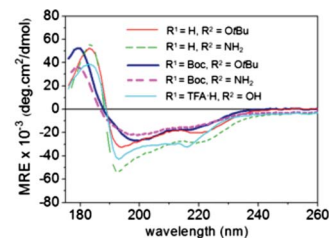
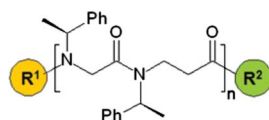


1108

Effect of capping groups at the *N*- and *C*-termini on the conformational preference of α,β -peptoids

Emiliana De Santis, Thomas Hjelmgard, Cécile Caumes, Sophie Faure, Bruce D. Alexander, Simon J. Holder, Giuliano Siligardi, Claude Taillefumier* and Alison A. Edwards*

Different capping groups have significant effects on spectral features/conformation of α,β -peptoids by CD, IR and NMR. These are most pronounced for shorter oligomers and are solvent dependent. This underlines the importance of considering capping groups for conformational studies.



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