

# Organic & Biomolecular Chemistry

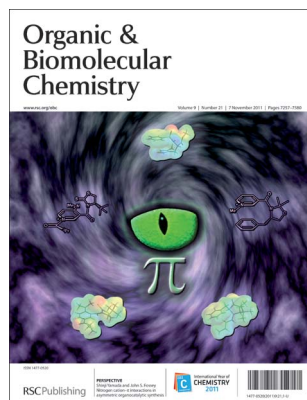
An international journal of synthetic, physical and biomolecular organic chemistry

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## IN THIS ISSUE

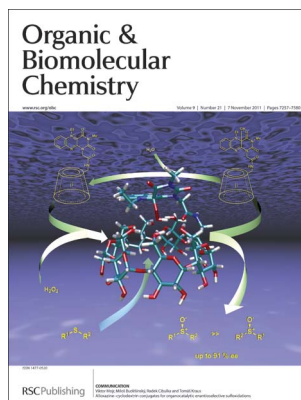
ISSN 1477-0520 CODEN OBCRAK 9(21) 7257–7580 (2011)



### Cover

See Shinji Yamada and John S. Fossey, pp. 7275–7281.

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### Inside cover

See Viktor Mojr *et al.*, pp. 7318–7326.

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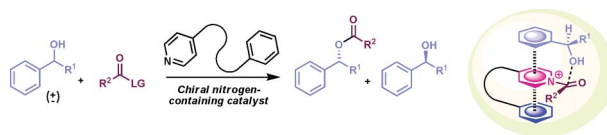
## PERSPECTIVE

7275

### Nitrogen cation– $\pi$ interactions in asymmetric organocatalytic synthesis

Shinji Yamada\* and John S. Fossey\*

This article summarises cases where cationic nitrogen– $\pi$  interactions may play an important role in asymmetric organocatalysis, and highlights the potential wealth of investigations to be had in this area.



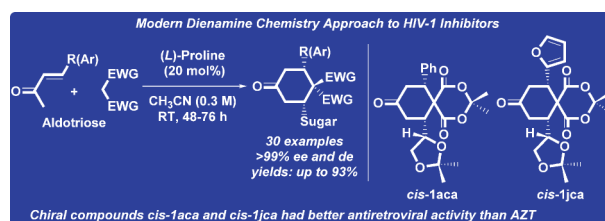
## COMMUNICATIONS

7282

### Design, synthesis and biological evaluation of optically pure functionalized spiro[5,5]undecane-1,5,9-triones as HIV-1 inhibitors

Dhevalapally B. Ramachary,\* Y. Vijayendar Reddy, Atoshi Banerjee and Sharmistha Banerjee\*

**Cascade approach to molecular therapeutics!** Preliminary biological cell culture-based in vivo screening on designed/synthesized DTCCA molecules revealed that *cis*-**1aca** and *cis*-**1jca** are better lead compounds for HIV-1 treatment than the known antiretroviral drug azidothymidine (AZT).



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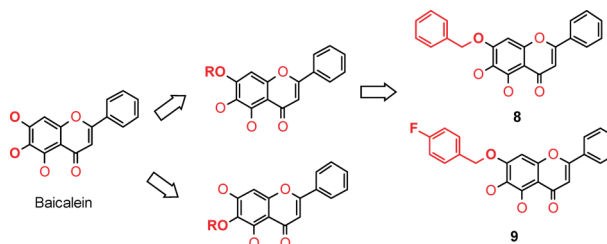
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7287

### Novel synthetic baicalein derivatives caused apoptosis and activated AMP-activated protein kinase in human tumor cells

Derong Ding, Baozi Zhang, Tao Meng, Ying Ma, Xin Wang, Hongli Peng\* and Jingkang Shen\*

Studies on the anti-proliferative activities of twelve baicalein derivatives demonstrated that compounds **8** and **9** showed more potent anti-proliferative effects than baicalein and enhanced AMPK $\alpha$  phosphorylation in A431, SK-OV-3, DU 145 and HeLa cells, suggesting an alternative therapeutic approach for benzyl baicalein in cancer therapy.



7292

### Graphite oxide: a selective and highly efficient oxidant of thiols and sulfides

Daniel R. Dreyer, Hong-Peng Jia, Alexander D. Todd, Jianxin Geng and Christopher W. Bielawski\*

The selective oxidation of thiols to disulfides and sulfides to sulfoxides was achieved using graphite oxide; the reactions were found to proceed rapidly (as short as 10 min in some cases) and in good yield (51–100%) (19 examples).

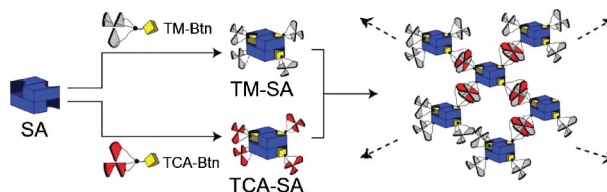


7296

### Protein assembly directed by synthetic molecular recognition motifs

Mingming Ma and Dennis Bong\*

We describe biotin derivatives of cyanuric acid and melamine that permit discrete functionalization of streptavidin protein at the tetrahedrally symmetric biotin ligand binding sites. Cyanuric acid and melamine recognition induces selective heteromeric protein assembly upon mixing streptavidin derivatives.

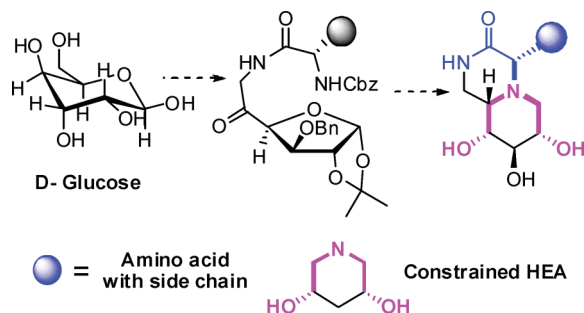


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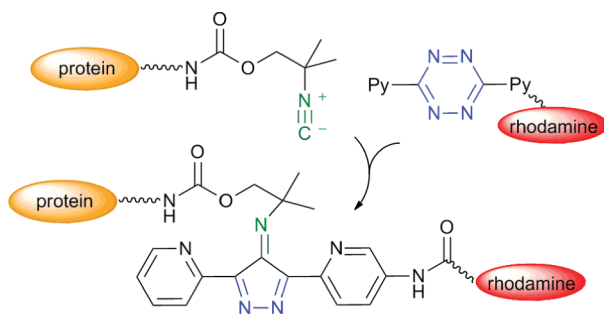
### Bicyclic amino acid-carbohydrate-conjugates as conformationally restricted hydroxyethylamine (HEA) transition-state isosteres

Sangram S. Kale, Sanjay T. Chavan, Sushma G. Sabharwal, Vedavati G. Puranik and Gangadhar J. Sanjayan\*

This communication describes a general synthetic route to bicyclic amino acid-carbohydrate-conjugates, which would be useful as constrained hydroxyethylamine (HEA) transition-state isosteres.



7303

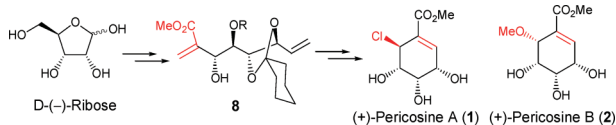


### Exploring isonitrile-based click chemistry for ligation with biomolecules

Henning Stöckmann, André A. Neves, Shaun Stairs, Kevin M. Brindle and Finian J. Leeper\*

Isonitriles can be used for biocompatible ligation reactions, reacting with dipyrildytetrazines at rates that match azide-cyclooctyne ligations.

7306

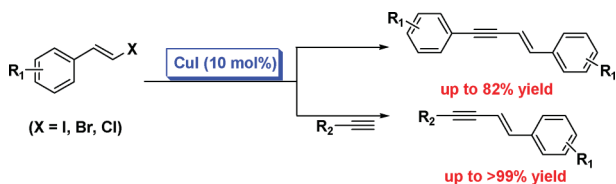


### Facile carbohydrate-based stereocontrolled divergent synthesis of (+)-pericosines A and B

Subhankar Tripathi, Ajam Chand Shaikh and Chinpiao Chen\*

A synthesis of pericosines A and B starting from D-ribose derived ene-diol in 35% and 41% overall yields respectively.

7309

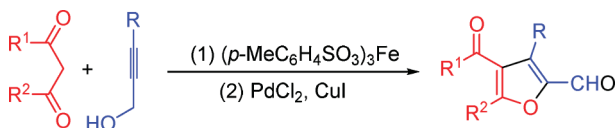


### Copper(I)-catalyzed synthesis of 1,3-enynes *via* coupling between vinyl halides and alkynes or domino coupling of vinyl halides

Yan Zhu, Tingyi Li, Xiaoming Qu, Peng Sun, Hailong Yang and Jincheng Mao\*

1,3-Enynes were easily prepared from coupling between vinyl halides and alkynes or domino coupling of vinyl halides in the presence of copper iodide. It is noteworthy that the double-bond geometry of the vinyl halides was retained during the reaction.

7313



### Pd-Catalyzed cyclization reaction: a convenient domino process for synthesis of $\alpha$ -carbonyl furan derivatives

Hua Cao, Huan-Feng Jiang,\* Hua-Wen Huang and Jin-Wu Zhao

An efficient PdCl<sub>2</sub>/CuI co-catalyzed cyclization reaction synthesizes  $\alpha$ -carbonyl furan derivatives which are useful synthetic intermediates for bioactive and natural compounds.

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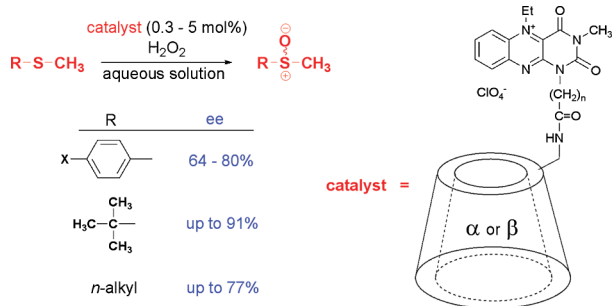
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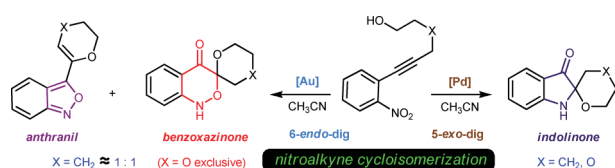


### Alloxazine–cyclodextrin conjugates for organocatalytic enantioselective sulfoxidations

Viktor Mojr, Miloš Buděšínský, Radek Cibulka\* and Tomáš Kraus\*

Alloxazine–cyclodextrin conjugates catalyze enantioselective oxidations of prochiral aromatic and aliphatic sulfides to sulfoxides by hydrogen peroxide in aqueous solution.

7327

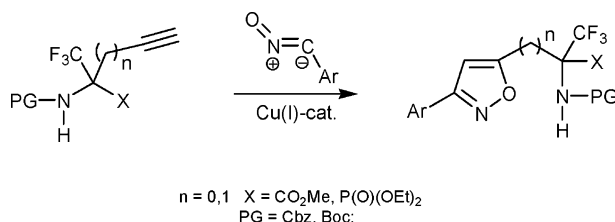


### Divergent Pd(II) and Au(III) mediated nitroalkynol cycloisomerizations

Pitambar Patel and Chepuri V. Ramana\*

A new cycloisomerization reaction comprising the simultaneous addition of nitro and alcohol groups across C=C leading to skeletally diverse small molecules is reported.

7335

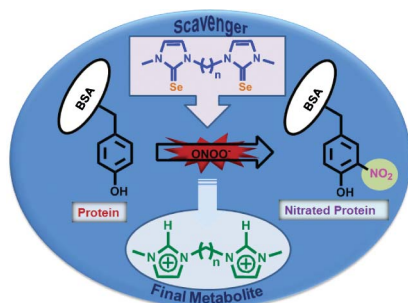


### Click-chemistry approach to isoxazole-containing $\alpha$ -CF<sub>3</sub>-substituted $\alpha$ -aminocarboxylates and $\alpha$ -aminophosphonates

Daria V. Vorobyeva,\* Natalya M. Karimova, Irina L. Odinets, Gerd-Volker Rösenthaler\* and Sergey N. Osipov

A convenient strategy for the synthesis of novel  $\alpha$ -CF<sub>3</sub>- $\alpha$ -aminocarboxylates and  $\alpha$ -aminophosphonates bearing the isoxazole pharmacophore have been developed.

7343



### Inhibition of peroxynitrite- and peroxidase-mediated protein tyrosine nitration by imidazole-based thiourea and selenourea derivatives

Krishna P. Bhabak, Kandhan Satheeshkumar, Subramaniam Jayavelu and Govindasamy Mugesh\*

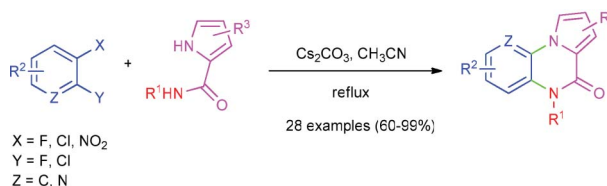
Studies on a series of *N*-methylimidazole-based di-thioureas/di-selenoureas (**14–21**) linked by a  $-(\text{CH}_2)_n-$  chain indicate that the di-selenoureas are more zwitterionic than the corresponding di-thioureas and all the selenoureas effectively inhibit the nitration of bovine serum albumin.

7351

**One-pot synthesis of pyrrolo[1,2-*a*]quinoxalines**

Aiping Huang, Feng Liu, Chunjing Zhan, Yanli Liu and Chen Ma\*

A transition metal-free process for the regioselective synthesis of pyrrolo[1,2-*a*]quinoxalines under mild conditions in one-pot is described.

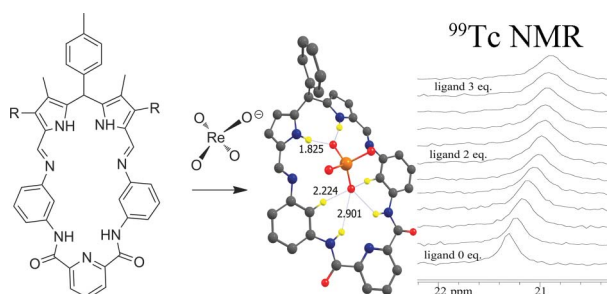


7358

**Macrocyclic receptor for pertechnetate and perrhenate anions**

Grigory V. Kolesnikov, Konstantin E. German, Gayane Kirakosyan, Ivan G. Tananaev, Yuri A. Ustynyuk, Victor N. Khrustalev and Evgeny A. Katayev\*

The design and synthesis of a neutral macrocyclic host that is capable of perrhenate and pertechnetate recognition is described.



7365

**One pot synthesis of amino acid derived chiral disubstituted morpholines and 1,4-oxazepanes via tandem aziridine/epoxide ring opening sequences**

Krishnananda Samanta and Gautam Panda\*

A new one-pot synthetic strategy is described for the synthesis of *cis*-3,5-disubstituted morpholines and 3,6-disubstituted 1,4-oxazepanes via tandem aziridine/epoxide ring opening sequences. This new strategy describes how epoxy alcohols could act as both a nucleophile and an electrophile in a tandem fashion and undergo intermolecular regioselective ring opening of chiral aziridines for the first time.

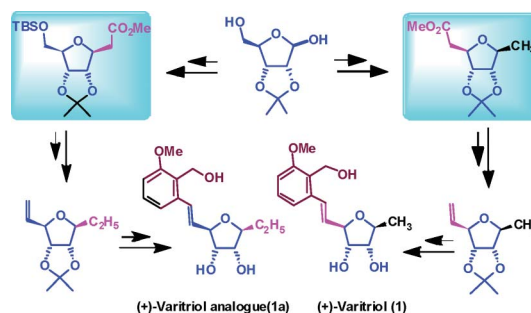


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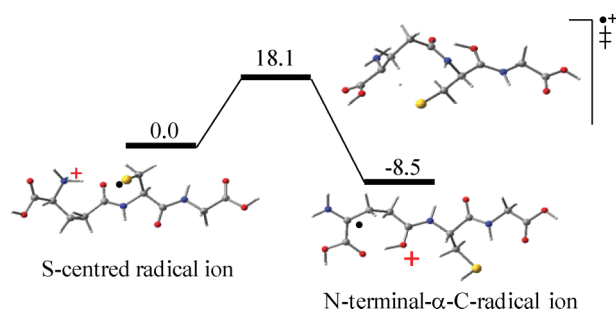
**Diastereoselective one-pot Wittig olefination–Michael addition and olefin cross metathesis strategy for total synthesis of cytotoxic natural product (+)-varitriol and its higher analogues**

Partha Ghosal, Deepty Sharma, Brijesh Kumar, Sanjeev Meena, Sudhir Sinha and Arun K. Shaw\*

A stereoselective route for the total synthesis of (+)-varitriol (**1**) and some higher analogues (**1a–j**) of this molecule is detailed herein.



7384

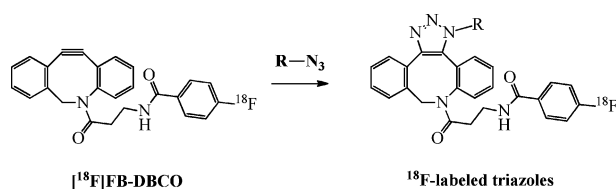


### Glutathione radical cation in the gas phase; generation, structure and fragmentation

Junfang Zhao, K. W. Michael Siu and Alan C. Hopkinson\*

Glutathione radical cations ( $\text{GSH}^{+\bullet}$ ) have been formed by two chemical methods. Mass selections and subsequent CIDs of the two  $\text{GSH}^{+\bullet}$  are presented. DFT calculations probe interconversion and fragmentation mechanisms.

7393

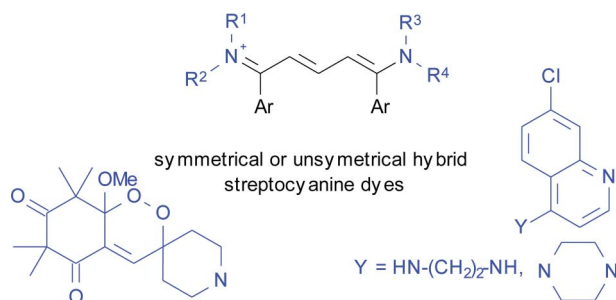


### Copper-free click chemistry with the short-lived positron emitter fluorine-18

Vincent Bouvet, Melinda Wuest and Frank Wuest\*

The copper-free strain-promoted click chemistry between  $^{18}\text{F}$ -labeled aza-dibenzocyclooctyne [ $^{18}\text{F}$ ]FB-DBCO and various azides is described. [ $^{18}\text{F}$ ]FB-DBCO was prepared in 85% isolated radiochemical yield (decay-corrected) through acylation of amino aza-dibenzocyclooctyne 1 with *N*-succinimidyl 4- $^{18}\text{F}$ fluorobenzoate ([ $^{18}\text{F}$ ]SFB). Copper-free click chemistry was performed with various azides at low concentrations (1–2  $\mu\text{M}$ ). Reaction proceeded best in methanol (>95% yield after 15 min at room temperature), whereas reaction in BSA required longer reaction times of 60 min and 40 °C upon completion.

7400

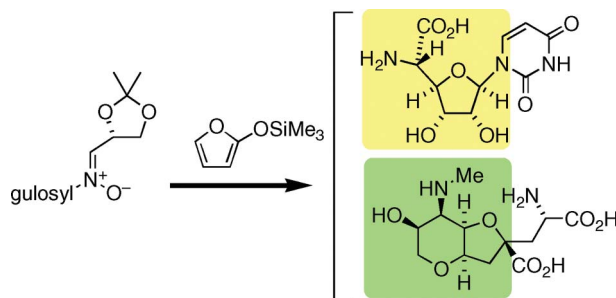


### Synthesis and antiplasmodial activity of streptocyanine/peroxide and streptocyanine/4-aminoquinoline hybrid dyes

Marie-Pierre Maether, Virginie Bernat, Marie Maturano, Christiane André-Barrès, Sonia Ladeira, Alexis Valentin, Henri Vial and Corinne Payrastré\*

Design, synthesis and antiplasmodial activities of two series of streptocyanine dyes incorporating cyclic peroxide or 4-aminoquinoline moieties were reported.

7411



### Stereoselective vinylogous Mannich reaction of 2-trimethylsilyloxyfuran with *N*-gulosyl nitrones

Osamu Tamura,\* Kodai Takeda, Naka Mita, Masanori Sakamoto, Iwao Okamoto, Nobuyoshi Morita and Hiroyuki Ishibashi

Stereoselective vinylogous Mannich reaction of 2-trimethylsilyloxyfuran with *L*-gulose-derived nitrones was employed for synthetic studies on polyoxin C and dysiherbaine.



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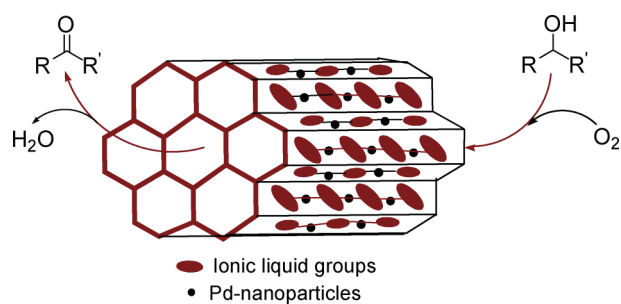
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7420

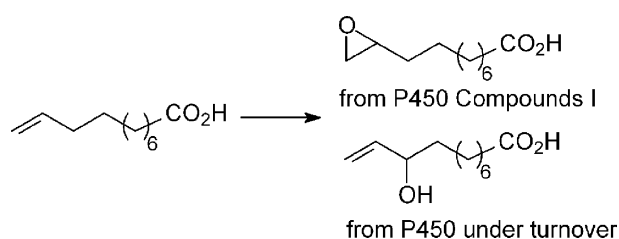


### Palladium containing periodic mesoporous organosilica with imidazolium framework (Pd@PMO-IL): an efficient and recyclable catalyst for the aerobic oxidation of alcohols

Babak Karimi,\* Dawood Elhamifar, James H. Clark and Andrew J. Hunt

The application of a novel palladium containing ionic liquid based periodic mesoporous organosilica catalyst in the aerobic oxidation of primary and secondary alcohols under molecular oxygen and air atmospheres is investigated.

7427

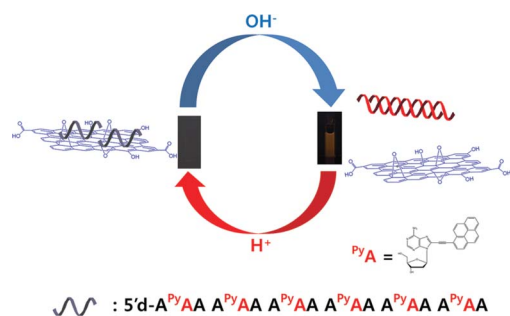


### Oxidation of 10-undecenoic acid by cytochrome P450<sub>BM-3</sub> and its Compound I transient

Xiaohong Chen, Zhi Su, John H. Horner and Martin Newcomb\*

Products and kinetics from oxidations of 10-undecenoic acid by cytochrome P450 Compounds I and a P450 enzyme are reported.

7434

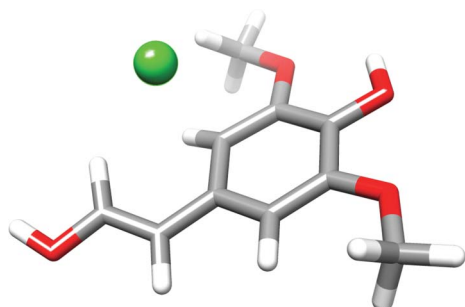


### pH-Responsive self-duplex of <sup>Py</sup>A-substituted oligodeoxyadenylate in graphene oxide solution as a molecular switch

Jeong Wu Yi, Jaesung Park, Kwang S. Kim and Byeang Hyeon Kim\*

We demonstrated a highly discriminated and reliable molecular switch based on the interaction between the self-duplex of <sup>Py</sup>A-substituted oligodeoxyadenylate and graphene oxide.

7439



### Controlling the action of chlorine radical: from lab to environment

A. K. Croft,\* H. M. Howard-Jones, C. E. Skates and C. C. Wood

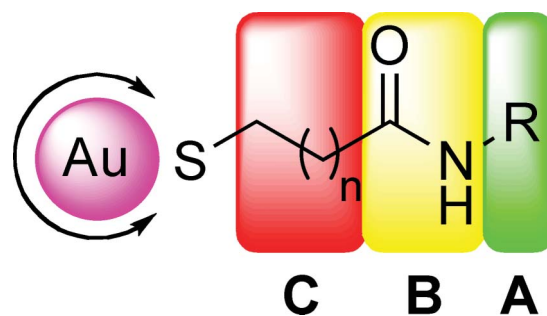
Stable chlorine atom complexes with lignin derivatives may mediate reaction of chlorine atom in the environment and in bleaching facilities.

7448

### Multivalent interaction and selectivities in selectin binding of functionalized gold colloids decorated with carbohydrate mimetics

Meike Roskamp, Sven Enders, Fabian Pfrengle, Shahla Yekta, Vjekoslav Dekaris, Jens Dervede, Hans-Ulrich Reissig and Sabine Schlecht\*

Gold colloids decorated with sulfated carbohydrate mimetics bind exceptionally strongly to selectins. Depending on the molecular structure of the multivalently presented epitopes **A**, the presence of amide **B**, and the length of linker **C**, selectivity was observed.

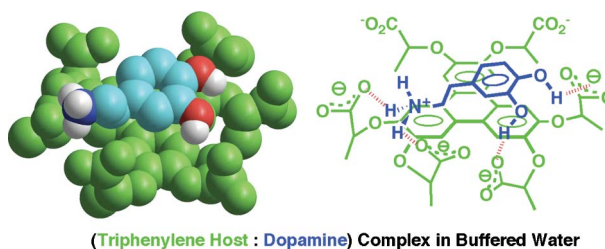


7457

### A simple ionic triphenylene receptor for catecholamines, serotonin and D-glucosamine in buffered water

Cécile Givelet and Brigitte Bibal\*

An ionic triphenylene was exploited as a receptor for biological amines and D-glucosamine in phosphate buffered water.

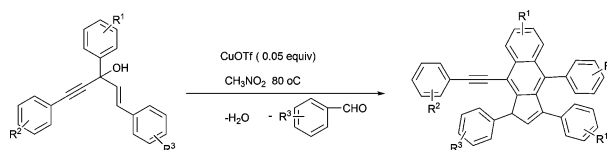


7461

### Copper-catalyzed dimerization fragmentation cyclization reactions of (*E*)-1-en-4-yn-3-ols as a versatile approach for the synthesis of multisubstituted 1*H*-cyclopenta[*b*]naphthalenes

Xiang-Chuan Wang, Jie Hu, Peng-Shuai Sun, Mei-Jin Zhong, Shaukat Ali and Yong-Min Liang\*

An intermolecular condensation reaction of 1,3,5-triarylenynols catalyzed by copper is developed, a straightforward method for the synthesis of highly conjugated 1*H*-cyclopenta[*b*]naphthalene.

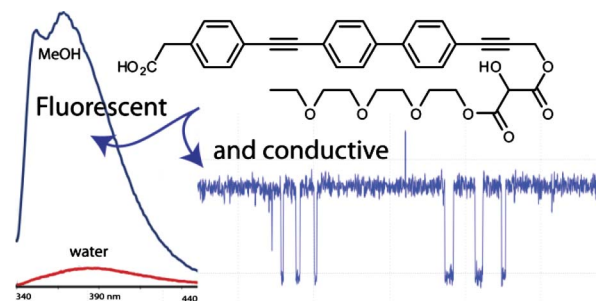


7468

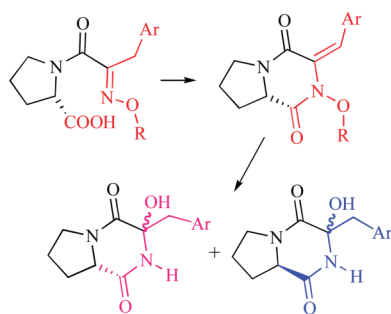
### Synthesis and ion transport activity of oligoesters containing an environment-sensitive fluorophore

Joanne M. Moszynski and Thomas M. Fyles\*

A soluble rigid fluorophore has environment-sensitive emission that is used as a mechanistic probe of ion-channel activity.



7476

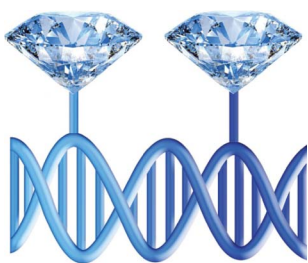


### Stereoselective synthesis and rearrangement-fragmentation of arylidene *N*-alkoxydiketopiperazines

Shouxin Liu,\* Yun Mu, Jianrong Han, Xiaoli Zhen, Yihua Yang, Xia Tian and Andrew Whiting\*

We describe a stereoselective synthesis of arylidene *N*-alkoxydiketopiperazines and rearrangement to new diketopiperazine containing hemiaminal systems under acidic conditions, plus biological activity vs. tumour cells.

7482

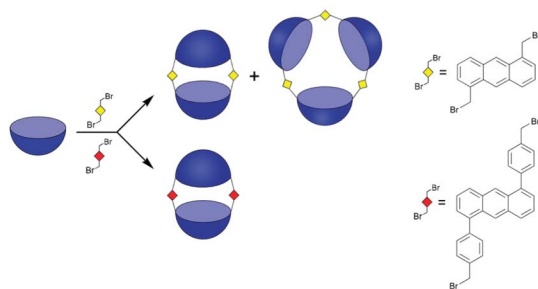


### Diamondoid-modified DNA

Yan Wang, Boryslav A. Tkachenko, Peter R. Schreiner\* and Andreas Marx\*

The manuscript describes the synthesis of diamondoid-modified DNA by chemical and enzymatic means.

7491

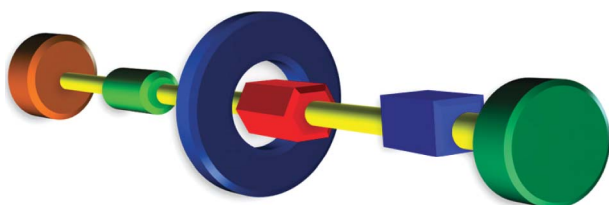


### Anthracene-resorcin[4]arene-based capsules: Synthesis and photoswitchable features

Sebastian Bringmann, Ralf Brodbeck, Ramona Hartmann, Christian Schäfer and Jochen Mattay\*

In this work we present three new hemicarcerands containing resorcinarenes linked by anthracenes. The reversibility of the anthracene cycloaddition could firstly be shown in the case of one dimer.

7500



### Construction of a functional [2]rotaxane with multilevel fluorescence responses

Yingjie Zhao, Yongjun Li,\* Siu-Wai Lai, Jien Yang, Chao Liu, Huibiao Liu, Chi-Ming Che and Yuliang Li\*

By the tuning of acid/base (input), a rotaxane incorporating three different stations and fluorescent states (output) was prepared.

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# Food & Function

Linking the chemistry and physics of food with health and nutrition

www.rsc.org/foodfunction

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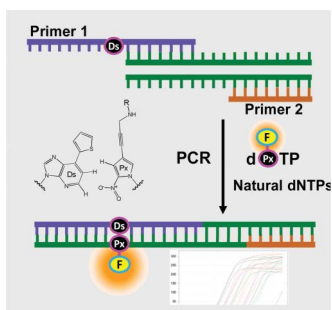
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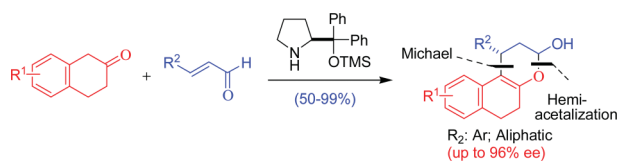


### Monitoring the site-specific incorporation of dual fluorophore-quencher base analogues for target DNA detection by an unnatural base pair system

Rie Yamashige, Michiko Kimoto, Tsuneo Mitsui, Shigeyuki Yokoyama and Ichiro Hirao\*

New fluorescent base analogues, intramolecular dual fluorophore-quencher unnatural bases, which function in replication as a third base pair with its pairing partner, enabling the site-specific enzymatic labeling of DNA molecules are reported.

7510

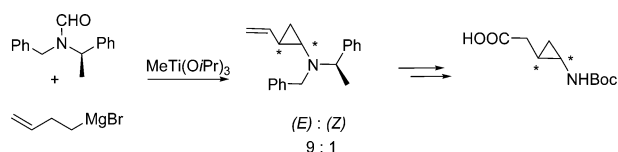


### Synthesis of 2,3,5,6-tetrahydro-1-alkyl/aryl-1H-benzo[f]chromen-3-ol derivatives from $\beta$ -tetralones and $\alpha,\beta$ -unsaturated aldehydes

Jung-Hsuan Chen, Chihliang Chang, Hui-Ju Chang and Kwunmin Chen\*

Treatment of  $\beta$ -tetralones with  $\alpha,\beta$ -unsaturated aldehydes in the presence of diphenylprolinol silyl ether gave benzo[f]chromen-3-ol derivatives with high to excellent chemical yields (50–99%) and high enantioselectivities (up to 96% ee).

7517

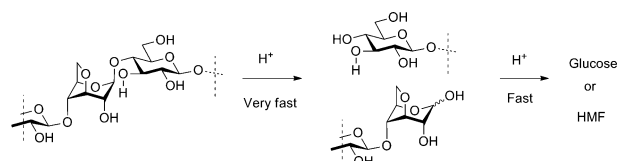


### Stereoselective preparation of $\beta,\gamma$ -methano-GABA derivatives

David J. Aitken,\* Ludovic Drouin, Sarah Goretta, Régis Guillot, Jean Ollivier\* and Marco Spiga

The Kulinkovich–de Meijere reaction provides non-racemic *trans* cyclopropylamines, which are converted into enantiomerically pure *N*-Boc protected  $\beta,\gamma$ -methano-GABA derivatives.

7525



### A study of anhydrocelluloses – Is a cellulose structure with residues in a ${}^1C_4$ -conformation more prone to hydrolysis?

Vrushali Jadhav, Christian M. Pedersen\* and Mikael Bols\*

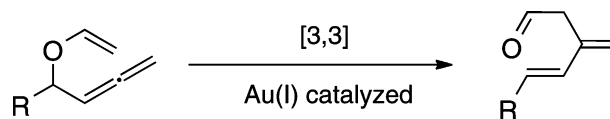
3,6-Anhydrocelluloses hydrolyse faster than unmodified cellulose due to the very reactive 3,6-anhydroglycosidic bonds they contain.

7535

**Gold(I)-catalyzed Claisen rearrangement of allenyl vinyl ethers; synthesis of substituted 1,3-dienes**

Marie E. Krafft,\* Kassem M. Hallal, Dinesh V. Vidhani and John W. Cran

Synthesis of substituted 1,3-dienes was achieved *via* gold(I)-catalyzed Claisen rearrangement of allenyl vinyl ethers. The N-heterocyclic carbene gold chloride catalyst (IPrAuCl) was superior in terms of activity and selectivity and afforded the 3,3-product in excellent yields. A proposed cation- $\pi$  inter-action played a significant role in affecting the reaction rate.

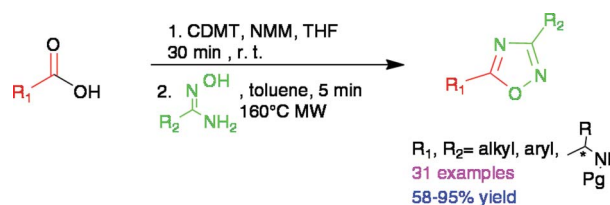


7539

**A fast and efficient one-pot microwave assisted synthesis of variously di-substituted 1,2,4-oxadiazoles**

Andrea Porcheddu, Roberta Cadoni and Lidia De Luca\*

A speedy and high-yielding one-pot microwave assisted synthesis of 1, 2, 4-oxadiazoles with both alkyl, aryl and amino acids in 3,5 positions.

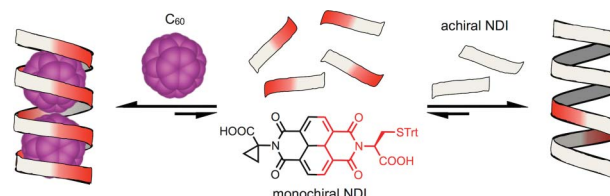


7547

**Supramolecular chemistry of monochiral naphthalenediimides**

Tom W. Anderson, G. Dan Pantoş\* and Jeremy K. M. Sanders\*

*N*-Desymmetrised naphthalenediimides (NDIs) containing one chiral and one achiral amino acid residue organise achiral NDIs into helical nanotubes and form receptors for C<sub>60</sub> and C<sub>70</sub>.

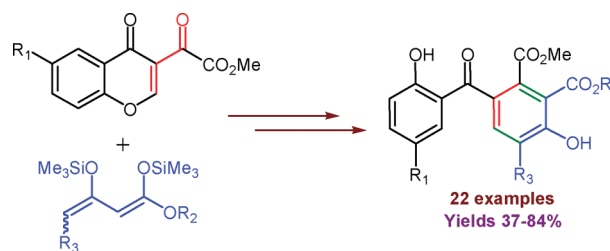


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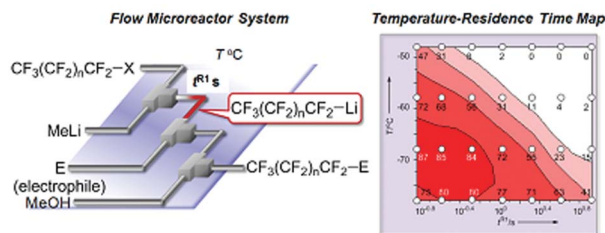
**3-Methoxallylchromones – versatile reagents for the regioselective synthesis of functionalized 2,4'-dihydroxybenzophenones, potential UV-filters**

Viktor O. Iaroshenko,\* Alina Bunescu, Anke Spannberg, Linda Supe, Maria Milyutina and Peter Langer\*

The reaction of 1,3-bis-silyl enol ethers with 3-methoxallylchromones affords a great variety of functionalised 2,4'-dihydroxybenzophenones. These products are formed by a Michael/retro-Michael/Mukaiyama-aldol reaction. These compounds are promising candidates for the synthesis of the novel UV-A/B and UV-B filters.



7559

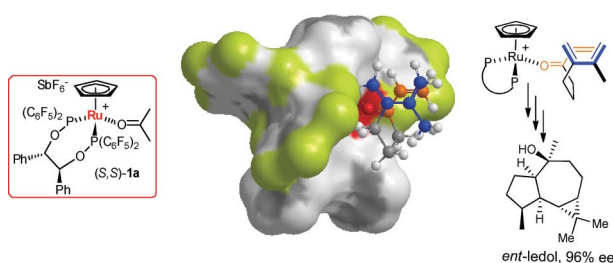


### Perfluoroalkylation in flow microreactors: generation of perfluoroalkyllithiums in the presence and absence of electrophiles

Aiichiro Nagaki, Shinya Tokuoka, Shigeyuki Yamada, Yutaka Tomida, Kojun Oshiro, Hideki Amii and Jun-ichi Yoshida\*

Perfluoroalkyllithiums were generated from perfluoroalkyl halides in the presence and absence of electrophiles using flow microreactor systems. The subsequent trapping method is effective for highly reactive electrophiles that are not compatible with the lithiation process.

7564

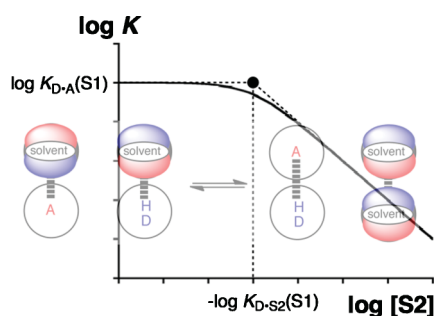


### Intramolecular Diels–Alder reactions using chiral ruthenium Lewis acids and application in the total synthesis of *ent*-ledol

Sirinporn Thamapipol and E. Peter Kündig\*

The ruthenium Lewis acid catalysed asymmetric intramolecular Diels–Alder reaction is a key step in the synthesis of *ent*-ledol.

7571



### Molecular recognition probes of solvation thermodynamics in solvent mixtures

Valeria Amenta, Joanne L. Cook, Christopher A. Hunter,\* Caroline M. R. Low and Jeremy G. Vinter

H-bonded complexes reveal the relationship between solute structure and selective solvation in mixed solvents: the more polar the solute, the more sensitive it is to the composition of the solvent.