

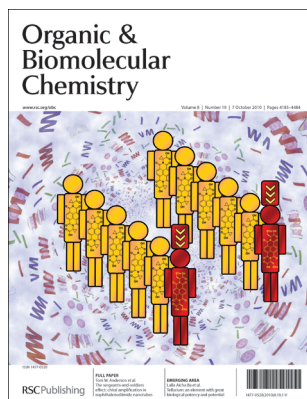
# Organic & Biomolecular Chemistry

An international journal of synthetic, physical and biomolecular organic chemistry  
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## IN THIS ISSUE

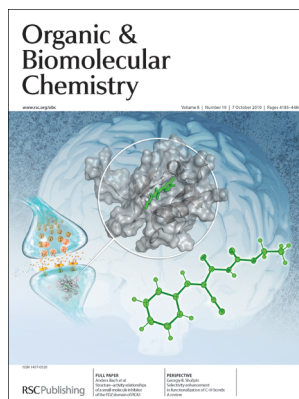
ISSN 1477-0520 CODEN OBCRAK 8(19) 4185–4484 (2010)



### Cover

See Anderson *et al.*, pp. 4274–4280.  
 Chiral naphthalenediimide-based monomers impose a helical supramolecular structure upon achiral monomers much like sergeants organising soldiers in rank and file.

Image reproduced by permission of G. Dan Pantoş from *Org. Biomol. Chem.*, 2010, **8**, 4274.



### Inside cover

See Bach *et al.*, pp. 4281–4288.  
 Structure-activity relationships of the first small-molecule inhibitor of the PDZ domain of PICK1, an important protein in the brain, have been explored.

Image reproduced by permission of Ulrik Gether and Kristian Strømgaard from *Org. Biomol. Chem.*, 2010, **8**, 4281.

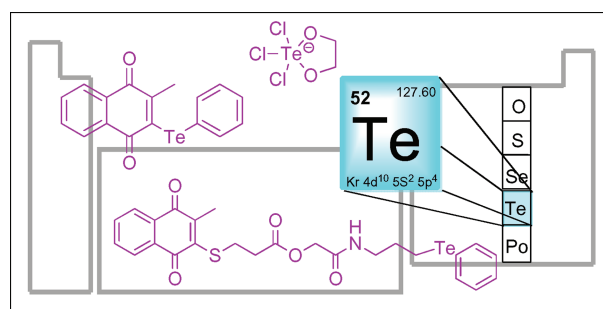
## EMERGING AREA

4203

### Tellurium: an element with great biological potency and potential

Lalla Aicha Ba, Mandy Döring, Vincent Jamier and Claus Jacob\*

Although their synthesis is not always straightforward, organotellurium compounds have recently attracted considerable interest among biological chemists, in particular in areas such as imaging, antibiotics and anticancer drug design.



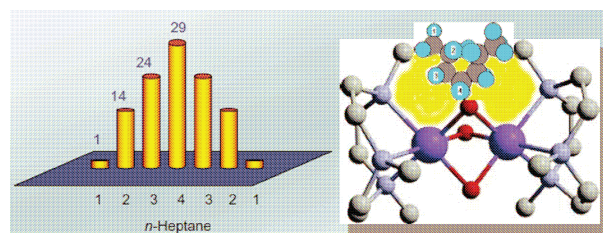
## PERSPECTIVE

4217

### Selectivity enhancement in functionalization of C–H bonds: A review

Georgiy B. Shul'pin\*

By applying the ideas of biochemistry, chemists can create new catalysts which selectively functionalize various C–H compounds.



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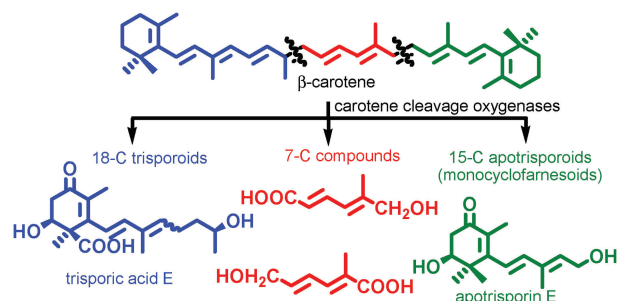
## COMMUNICATIONS

4229

### Splitting of $\beta$ -carotene in the sexual interaction of *Phycomyces*

Silvia Polaino, M. Mar Herrador, Enrique Cerdá-Olmedo and Alejandro F. Barrero\*

The new C7 natural products **1–2** are the result of enzymatic carotene degradation during the sexual interaction.

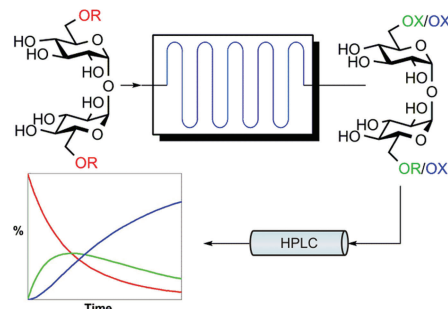


4232

### Flow chemistry kinetic studies reveal reaction conditions for ready access to unsymmetrical trehalose analogues

Mitul K. Patel and Benjamin G. Davis\*

Trehalose functionalization was studied in a microreactor, furnishing kinetic data that allowed the development of a synthetic route for large scale desymmetrizations.

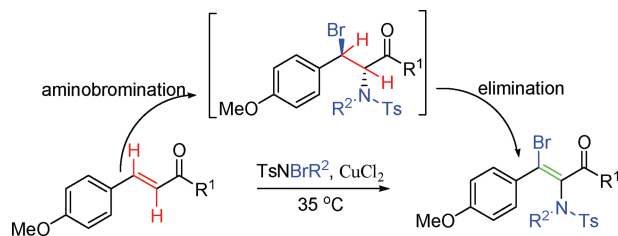


4236

### Copper-catalyzed aminobromination/elimination process: an efficient access to $\alpha,\beta$ -unsaturated vicinal haloamino ketones and esters

Hao Sun, Guangqian Zhang, Sanjun Zhi, Jianlin Han,\* Guigen Li and Yi Pan\*

A novel copper-catalyzed aminobromination-elimination process has been developed, which provides an easy access to  $\alpha,\beta$ -unsaturated vicinal haloamindes derivatives.

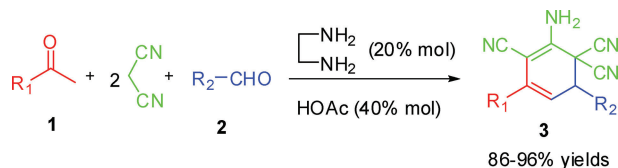


4240

### Primary 1,2-diamine catalysis III: an unexpected domino reaction for the synthesis of multisubstituted cyclohexa-1,3-dienamines

Junfeng Wang, Qin Li, Chao Qi, Yi Liu, Zemei Ge\* and Runtao Li\*

Primary 1,2-diamine was employed in the first organocatalyzed multicomponent domino reactions of aryl ketones, aldehydes and malononitrile, affording multisubstituted cyclohexa-1,3-dienamines **3** in satisfactory results.



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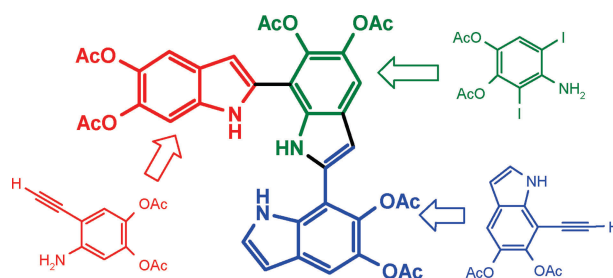
## COMMUNICATIONS

4243

**First synthetic entry to the trimer stage of 5,6-dihydroxyindole polymerization: *ortho*-alkynylaniline-based access to the missing 2,7':2',7''-triindole**

Luigia Capelli, Paola Manini,\* Alessandro Pezzella and Marco d'Ischia

An *ortho*-alkynylaniline-based strategy allowed the first access to a 5,6-dihydroxyindole trimer, 5,5',5'',6,6',6''-hexaacetoxy-2,7':2',7''-triindole, via a sequence of Sonogashira coupling and metal cation catalyzed intramolecular cyclization.

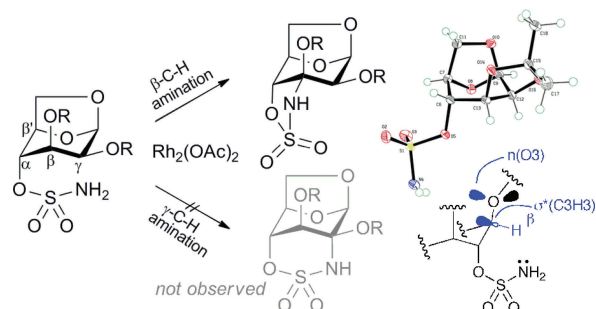


4246

**Inverted regioselectivity of C–H amination: Unexpected oxidation at  $\beta$ - rather than  $\gamma$ -C–H**

Filip J. Wyszynski, Amber L. Thompson and Benjamin G. Davis\*

From derivatives of 1,6-anhydro- $\beta$ -D-mannopyranose, five-membered sulfamidates were formed in preference to the typical six-membered oxathiazinane intramolecular insertion products.

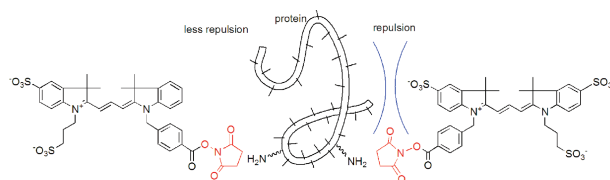


4249

**Asymmetric trimethine 3*H*-indocyanine dyes: efficient synthesis and protein labeling**

Fengling Song, Li Wang, Xiaoqiang Qiao, Bingshuai Wang, Shiguo Sun, Jiangli Fan, Lihua Zhang and Xiaojun Peng\*

To achieve better protein labeling performance, sulfo groups should be away from the NHS ester end.

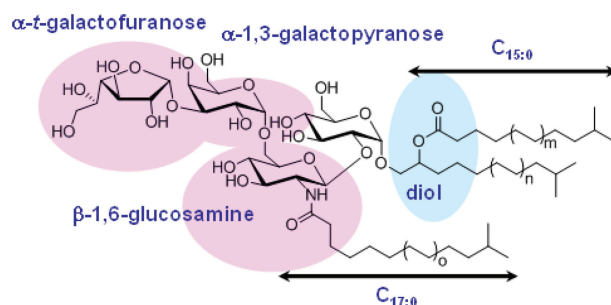


4252

**Structural variation of glycolipids from *Meiothermus taiwanensis* ATCC BAA-400 under different growth temperatures**

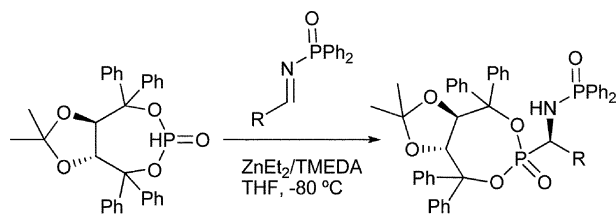
Yu-Liang Yang, Feng-Ling Yang, Zih-You Huang, Yu-Hsuan Tsai, Wei Zou and Shih-Hsiung Wu\*

Structural variation of membrane glycolipids (including lipid and carbohydrate) under different growth temperature was revealed in thermophilic bacteria. The variation is assumed to stabilize membrane structure for survival in harsh environment.



## COMMUNICATIONS

4255



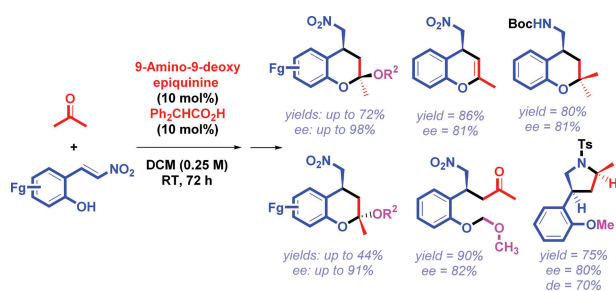
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### Diastereoselective hydrophosphonylation of imines using (*R,R*)-TADDOL phosphite. Asymmetric synthesis of $\alpha$ -aminophosphonic acid derivatives

Francisco Palacios,\* Tomasz K. Olszewski and Javier Vicario

Efficient synthesis of  $\alpha$ -aminophosphonic acids is achieved the key step being a diastereoselective hydrophosphonylation of *N*-diphenylphosphinyl imines using a chiral phosphonate derived from inexpensive natural tartaric acid.

4259



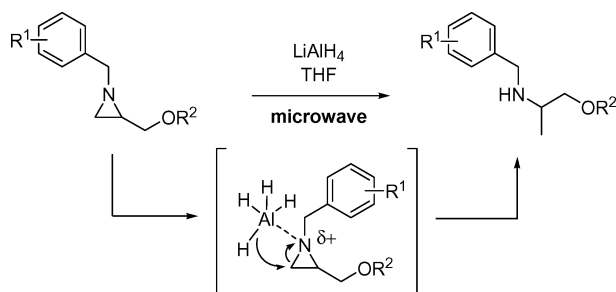
### Sequential combination of Michael and acetalization reactions: direct catalytic asymmetric synthesis of functionalized 4-nitromethyl-chromans as drug intermediates

Dhevalapally B. Ramachary\* and Rajasekar Sakthidevi

Functionalized chiral 4-nitromethyl-chromans as drug intermediates were achieved for the first time through sequential combination of Michael and acetalization reactions on 2-(2-nitrovinyl)-phenols with acetone and alcohols under organocatalysis.

## PAPERS

4266

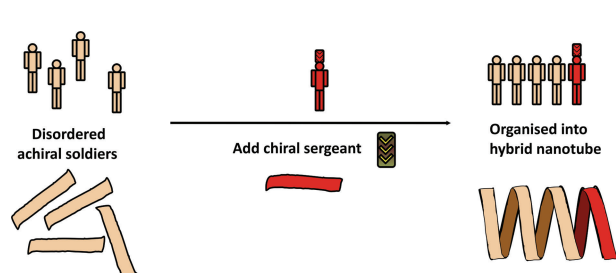


### Microwave-assisted regioselective ring opening of non-activated aziridines by lithium aluminium hydride

Sonja Stanković, Matthias D'hooghe and Norbert De Kimpe\*

A new protocol for the  $\text{LiAlH}_4$ -promoted regioselective ring opening of non-activated aziridines towards 2-aminopropanes under microwave irradiation was developed.

4274



### The sergeants-and-soldiers effect: chiral amplification in naphthalenediimide nanotubes

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

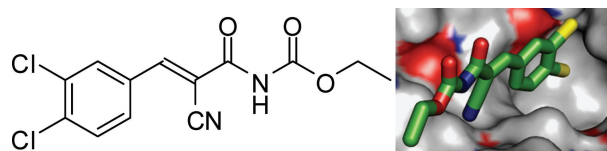
Self-assembling naphthalenediimide-based helical organic nanotubes display sergeants-and-soldiers behaviour, chiral monomers imposing a supramolecular structure upon achiral monomers. Nanotubes containing predominantly the most effective soldier are effective hosts for  $\text{C}_{60}$ .

4281

### Structure–activity relationships of a small-molecule inhibitor of the PDZ domain of PICK1

Anders Bach, Nicolai Stuhr-Hansen, Thor S. Thorsen, Nicolai Bork, Irina S. Moreira, Karla Frydenvang, Shahrokh Padrah, S. Brøgger Christensen, Kenneth L. Madsen, Harel Weinstein, Ulrik Gether\* and Kristian Strømgaard\*

Structure–activity studies of the first small-molecule inhibitor of the PICK1 PDZ domain leading to improved affinity.

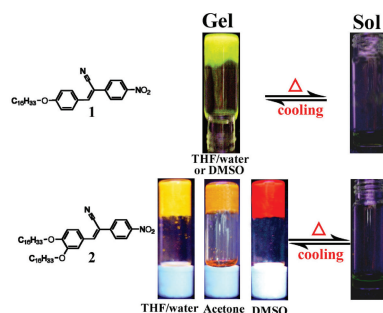


4289

### Multicolor fluorescent switches in gel systems controlled by alkoxy chain and solvent

Yue Xu, Pengchong Xue,\* Defang Xu, Xiaofei Zhang, Xingliang Liu, Huipeng Zhou, Junhui Jia, Xinchun Yang, Fengyong Wang and Ran Lu\*

All gels of **1** and **2** in different solvents possess aggregation-induced emission (AIE) characteristics. Moreover, the gels of **1** formed in THF–water and DMSO show the same packing model and similar AIE properties. However, the self-assembly and fluorescent characteristics of molecule **2** strongly depend on the solvent.

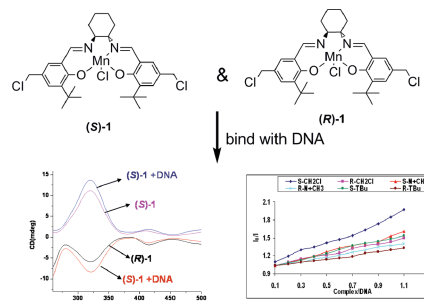


4297

### Influence of chirality using Mn(III) salen complexes on DNA binding and antioxidant activity

Noor-ul H. Khan,\* Nirali Pandya, Manoj Kumar, Prasanta Kumar Bera, Rukhsana I. Kureshy, Sayed H. R. Abdi and Hari C. Bajaj

Interaction of the synthesized chiral Mn(III) salen complexes *viz.* **S-1**, **R-1**, **S-2**, **R-2**, **S-3** and **R-3** with Calf Thymus DNA was studied by various physico-chemical methods. Among all the complexes used the best result in terms of binding constants ( $130.4 \times 10^4 \text{ M}^{-1}$ ) and strong antioxidant activity was achieved for (**S-1**).

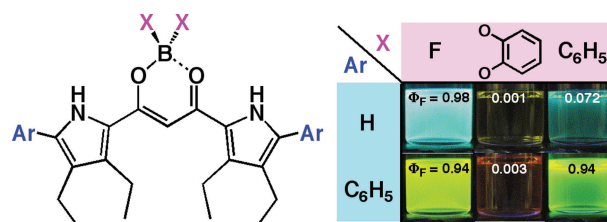


4308

### Modification at a boron unit: tuning electronic and optical properties of $\pi$ -conjugated acyclic anion receptors

Hiromitsu Maeda,\* Mayumi Takayama, Kazuki Kobayashi and Hideyuki Shinmori

Substituents at the boron unit of dipyrrolyldiketone boron complexes as  $\pi$ -conjugated acyclic anion receptors play crucial roles for tuning of solid-state molecular assemblies, anion-binding behaviour and electronic and optical properties.



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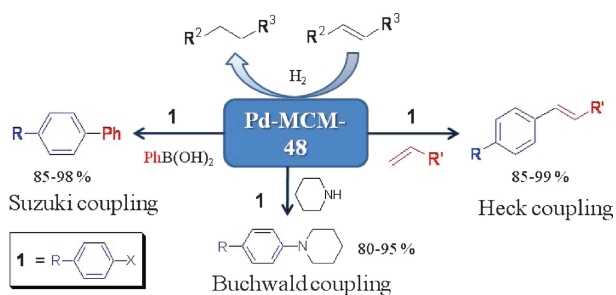


4316

### Pd-MCM-48: a novel recyclable heterogeneous catalyst for chemo- and regioselective hydrogenation of olefins and coupling reactions

Subhash Banerjee,\* Vagulejan Balasanthiran, Ranjit T. Koodali\* and Grigoriy A. Sereda\*

A novel, heterogeneous Pd-MCM-48 catalyst has been developed by encapsulating palladium nanoparticles into the cubic phase of mesoporous MCM-48 matrix for chemo- and regioselective hydrogenation of olefins and coupling reactions under ligand-free and aerobic conditions.

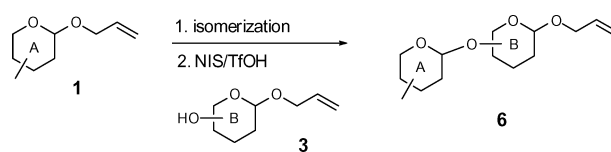


4322

### Facile glycosylation strategy with two-stage activation of allyl glycosyl donors. Application to concise synthesis of *Shigella flexneri* serotype Y O-antigen

Yun Wang, Xin Zhang and Pengfei Wang\*

A glycosylation method employing only allyl glycoside building blocks has been developed. The donor's glycosylation reactivity is turned on *via* isomerization of its anomeric allyl group into the corresponding prop-1-enyl moiety.

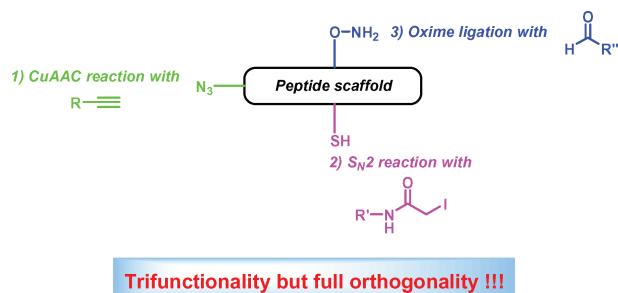


4329

### A universal and ready-to-use heterotrifunctional cross-linking reagent for facile synthetic access to sophisticated bioconjugates

Guillaume Clavé, Hervé Volland, Mélanie Flaender, Didier Gasparutto, Anthony Romieu\* and Pierre-Yves Renard\*

For the first time, three different bioconjugable functions namely aminoxy, azido and thiol are associated within the same peptidyl architecture to get a new generation of cross-linking reagents which offer unprecedented possibilities for the construction of highly sophisticated bioconjugates.

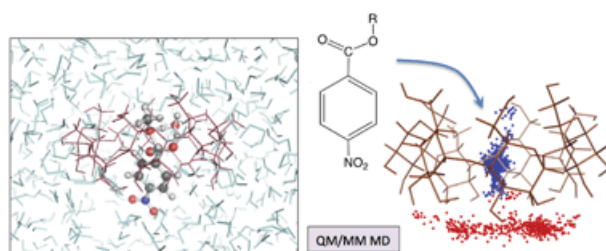


4346

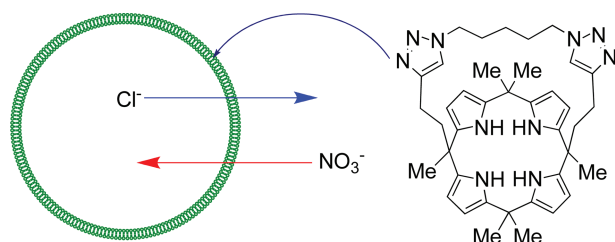
### Computer simulation of reactions in $\beta$ -cyclodextrin molecular reactors: transition state recognition

Violeta Yeguas, Ramón López, Alexandrine Lambert, Gérald Monard and Manuel F. Ruiz-López\*

Combined quantum mechanics and molecular dynamics calculations highlight the role of transition state shape in the mechanism and kinetics of ester hydrolysis mediated by  $\beta$ -cyclodextrins.



4356

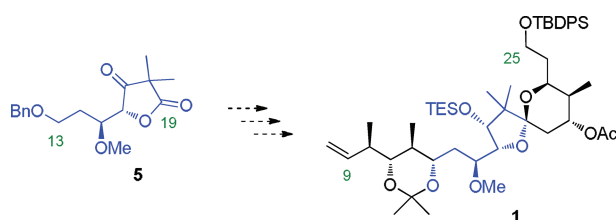


### Calix[4]pyrrole-based anion transporters with tuneable transport properties

Masafumi Yano, Christine C. Tong, Mark E. Light, Franz P. Schmidtchen and Philip A. Gale\*

Bilayer transport efficiency for transmembrane chloride transport was found to directly depend on the length of the alkyl chain present in the bis-triazole strap in a series of calix[4]pyrrole macrocycles.

4364

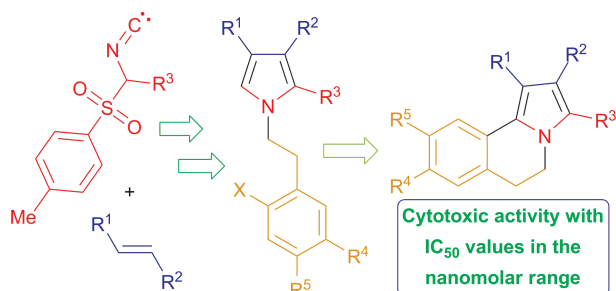


### Towards the total synthesis of calyculin C: preparation of the C<sub>9</sub>–C<sub>25</sub> spiroketal-dipropionate unit

Damien Habrant and Ari M. P. Koskinen\*

An asymmetric synthesis of the C<sub>9</sub>–C<sub>25</sub> spiroketal fragment of calyculin C is described. Key steps include two crotylation reactions, ynone formation by a Pd-catalyzed coupling of a thiol ester with a terminal alkyne and a double intramolecular hetero-Michael addition for the stereoselective construction of the spiroketal framework.

4374

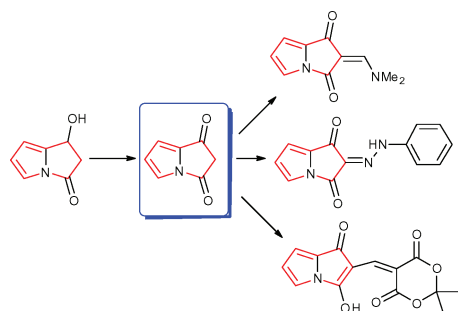


### Synthesis of 5,6-dihydropyrrolo[2,1-*a*]isoquinolines featuring an intramolecular radical-oxidative cyclization of polysubstituted pyrroles, and evaluation of their cytotoxic activity

Paul E. Reyes-Gutiérrez, José R. Camacho, Ma. Teresa Ramírez-Apan, Yazmin M. Osornio and Roberto Martínez\*

A three-step protocol for the synthesis of 5,6-dihydropyrrolo[2,1-*a*]isoquinolines is described. Structure–activity studies revealed the importance of the C-2 substituent for cytotoxicity.

4383



### Pyrrolizine-1,3-dione

Hamish McNab,\* James Montgomery, Simon Parsons and David G. Tredgett

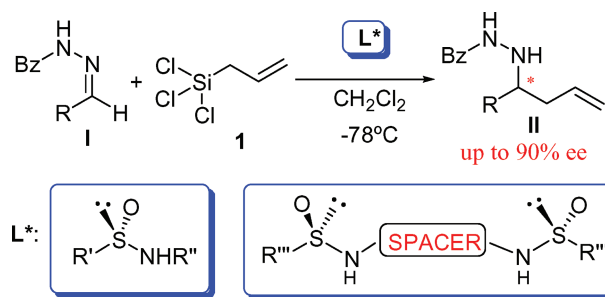
Pyrrolizine-1,3-dione is a unique system whose carbonyl groups show unusual physical properties; the active methylene unit couples with electrophiles.

4388

### Chiral sulfur derivatives in the allylation of acyl hydrazones: C<sub>2</sub>-symmetric bis-sulfinamides as enhanced chiral organic promoters.

Inmaculada Fernández,\* Ana Alcudia, Beatrice Gori, Victoria Valdivia, Rocio Recio, María Victoria García and Noureddine Khair\*

Monosulfinamides and C<sub>2</sub>-symmetric bis-sulfinamides have been found to be convenient neutral chiral promoters in the allylation of acyl hydrazones, the nature of the spacer and the substituent at the sulfinyl sulfur are key elements for the enantioselectivity of the process.

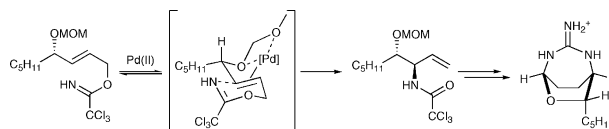


4394

### Stereoselective synthesis of the bicyclic guanidine alkaloid (+)-monanchorin

Ahmed M. Zaed and Andrew Sutherland\*

A new approach for the stereoselective synthesis of the bicyclic guanidine alkaloid (+)-monanchorin has been developed.

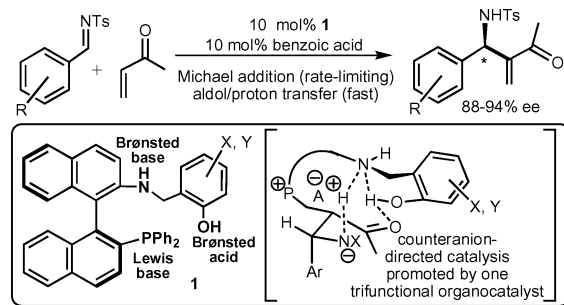


4400

### Mechanistic investigations of multidentate organocatalyst-promoted counterion catalysis for fast and enantioselective aza-Morita–Baylis–Hillman reactions at ambient temperature

Christopher Anstiss, Jean-Marc Garnier and Fei Liu\*

The first kinetic characterization of an enantioselective trifunctional organocatalyst-promoted counterion catalysis of asymmetric aza-Morita–Baylis–Hillman reactions.

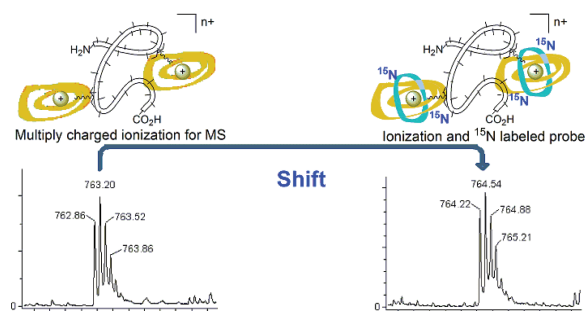


4408

### <sup>15</sup>N-Labeled ionic probes for bioanalytical mass spectrometry

Fumihiro Ito,\* Shin Ando, Masato Iuchi, Tomoko Nakamura, Satoko Yorita and Kentaro Yamaguchi\*

An ionization method that uses La-complex-based <sup>15</sup>N-labeled ionization probes containing the <sup>15</sup>N-labeled pybox, NHS-TMpybox, Mal-TMpybox and BrAc-TMpybox, was provided for isotope ratio mass spectrometry. This method was proven to effectively ionize isotope-labeled molecules.



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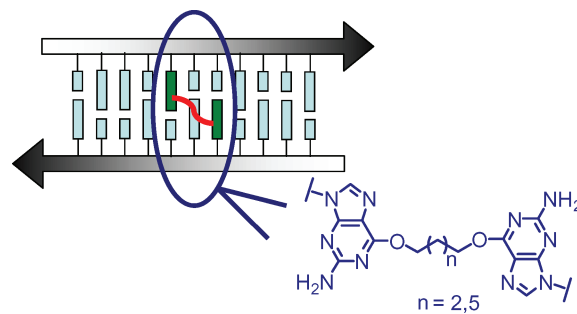
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4414

**Synthesis and characterization of an O<sup>6</sup>-2'-deoxyguanosine-alkyl-O<sup>6</sup>-2'-deoxyguanosine interstrand cross-link in a 5'-GNC motif and repair by human O<sup>6</sup>-alkylguanine-DNA alkyltransferase**

Francis P. McManus, Qingming Fang, Jason D. M. Booth, Anne M. Noronha, Anthony E. Pegg and Christopher J. Wilds\*

O<sup>6</sup>-2'-Deoxyguanosine-alkyl-O<sup>6</sup>-2'-deoxyguanosine interstrand DNA cross-links in a 5'-GNC- motif were synthesized and found to undergo repair by human O<sup>6</sup>-alkylguanine-DNA alkyltransferase.

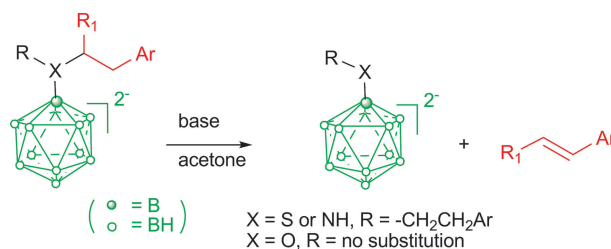


4427

**Undecahydro-*closo*-dodecaborates as good leaving groups in organic synthesis: generation of substituted styrenes *via* elimination of arylethyl dodecaborates**

Afaf R. Genady and Hiroyuki Nakamura\*

Functionalized arylethyl *closo*-dodecaborates are prepared by a simple one-step reaction. The functionalized styrene derivatives can be synthesized by treating arylethyl *closo*-dodecaborates with various bases.

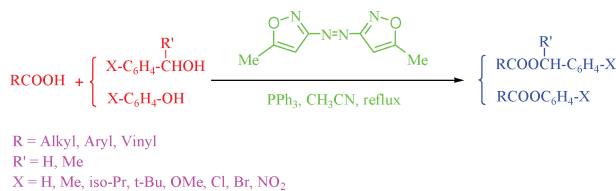


4436

**5,5'-Dimethyl-3,3'-azoisoxazole as a new heterogeneous azo reagent for esterification of phenols and selective esterification of benzylic alcohols under Mitsunobu conditions**

Nasser Iranpoor,\* Habib Firouzabadi\* and Dariush Khalili

5,5'-Dimethyl-3,3'-azoisoxazole is introduced as an efficient heterogeneous azo reagent for esterification of phenols and selective esterification of benzylic alcohols.

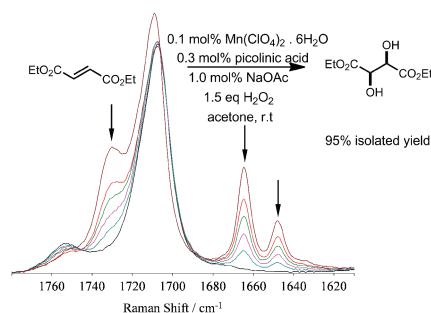


4444

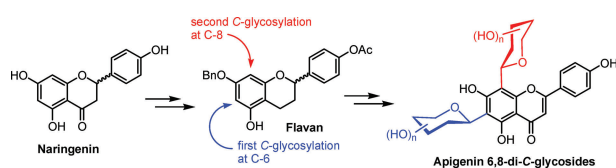
**Manganese catalyzed *cis*-dihydroxylation of electron deficient alkenes with H<sub>2</sub>O<sub>2</sub>**

Pattama Saisaha, Dirk Pijper, Ruben P. van Summeren, Rob Hoen, Christian Smit, Johannes W. de Boer, Ronald Hage, Paul L. Alsters, Ben L. Feringa and Wesley R. Browne\*

A high turnover method for the multigram scale selective *cis*-dihydroxylation of electron deficient alkenes using pyridine-2-carboxylic acid/Mn/H<sub>2</sub>O<sub>2</sub> is described.



4451

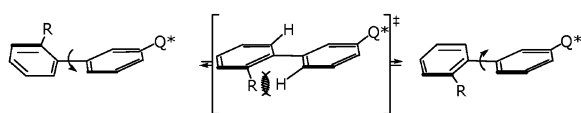


### Regioselective synthesis of di-C-glycosylflavones possessing anti-inflammation activities

Jiun-Jie Shie, Chih-An Chen, Chih-Chien Lin, Angela Fay Ku, Ting-Jen R. Cheng, Jim-Min Fang\* and Chi-Huey Wong\*

A series of 6,8-di-C-glycosylflavones bearing identical or distinct glycosyl moieties are synthesized and shown to exhibit anti-inflammation activity.

4463

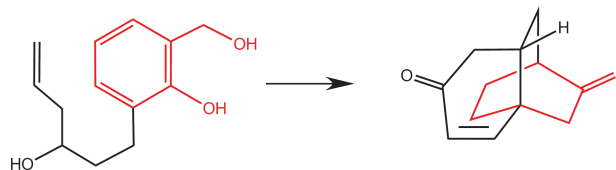


### The biphenyl-monitored effective size of unsaturated functional or fluorinated *ortho* substituents

Renzo Ruzziconi,\* Sara Spizzichino, Andrea Mazzanti,\* Lodovico Lunazzi and Manfred Schlosser\*

The effective size (*B* value) of a number of substituents has been probed by dynamic NMR measurements of the barriers to aryl-aryl rotation of the corresponding mono-*ortho* substituted biphenyls.

4472



### Intramolecular cycloaddition in 6,6-spiroepoxycyclohexa-2,4-dienone: simple aromatics to (±)-Platencin

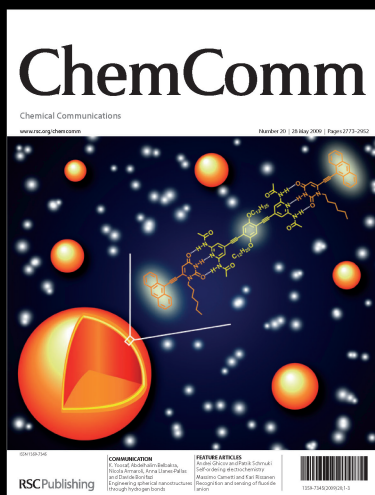
Vishwakarma Singh,\* Bharat Chandra Sahu, Varsha Bansal and Shaikh M. Mobin

A formal synthesis of (±)-platencin from a simple aromatic precursor is reported. Oxidative dearomatization and intramolecular Diels–Alder reaction are the key features of the methodology.

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