

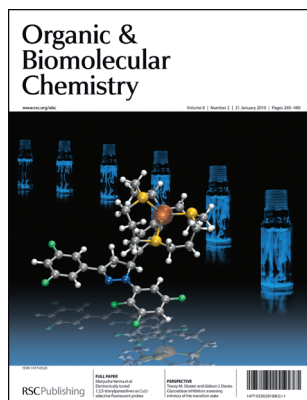
# Organic & Biomolecular Chemistry

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

ISSN 1477-0520 CODEN OBCRAK 8(2) 285–480 (2010)



### Cover

See Manjusha Verma *et al.*,  
pp. 363–370.

The fluorescence contrast of a Cu(I)-responsive probe was optimized through electronic tuning with varying numbers of fluorine substituents. The cuvettes show the time evolution of fluorescence titrations formed upon addition of the analyte.

Image reproduced by permission of Christoph J. Fahrni from *Organic & Biomolecular Chemistry*, 2010, **8**, 363.

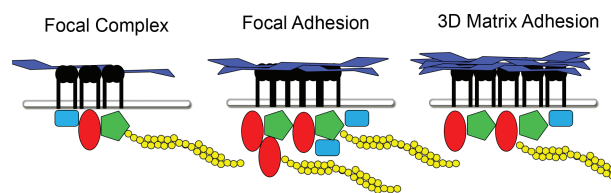
## EMERGING AREA

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### Matrix mechanics and receptor–ligand interactions in cell adhesion

Dewi Harjanto and Muhammad H. Zaman\*

This Emerging Area explores how forces and the mechanics of the extracellular matrix affect the formation and maturation of cell–matrix adhesions.



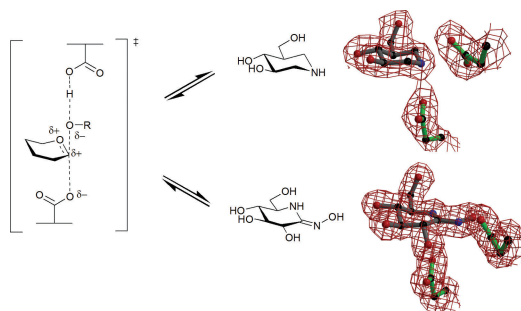
## PERSPECTIVE

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### Glycosidase inhibition: assessing mimicry of the transition state

Tracey M. Gloster\* and Gideon J. Davies\*

A review of the progress made in tackling glycosidase inhibition, which has widespread application in a number of diseases, in particular by examining those inhibitors that may mimic the transition state.



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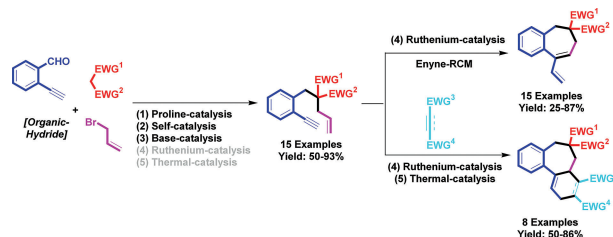
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### Rapid two-step synthesis of drug-like polycyclic substances by sequential multi-catalysis cascade reactions

Dhevalapally B. Ramachary,\* Rumpa Mondal and Chintalapudi Venkaiah

An efficient multi-catalysis cascade process for the two-step synthesis of highly substituted drug-like carbocycles was achieved through combinations of cascade TCRA/*C*-allylation/enyne-RCM/Diels–Alder reactions as key steps starting from simple acyclic substrates *via* amino acid-/self-/base-/ruthenium-/thermal-catalysis.

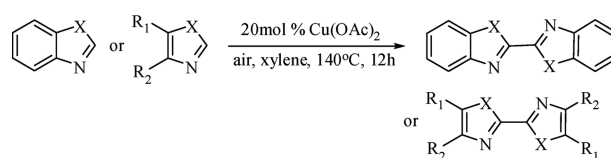


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### An efficient and convenient Cu(OAc)<sub>2</sub>/air mediated oxidative coupling of azoles *via* C–H activation

Yan Li, Jun Jin, Weixing Qian\* and Weiliang Bao\*

An efficient and convenient approach to construct C–C bonds at the 2-position of azoles *via* Cu(OAc)<sub>2</sub>/air mediated oxidative homo- and cross-coupling reaction was reported. The corresponding products were obtained in good to excellent yield.

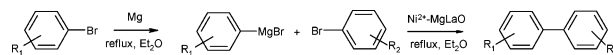


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### Nickel/magnesium–lanthanum mixed oxide catalyst in the Kumada-coupling

Árpád Kiss, Zoltán Hell\* and Mária Bálint

A new, heterogeneous, magnesium–lanthanum mixed oxide solid base-supported nickel(II) catalyst was developed. The catalyst was successfully used in the Kumada coupling of aryl halides, especially aryl bromides. The optimal reaction conditions of the coupling were determined.

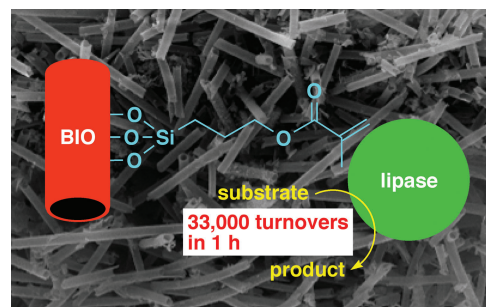


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### Chemical modification of biogenous iron oxide to create an excellent enzyme scaffold

Takashi Sakai,\* Yuki Miyazaki, Ai Murakami, Noriko Sakamoto, Tadashi Ema,\* Hideki Hashimoto, Mitsuaki Furutani, Makoto Nakanishi, Tatsuo Fujii and Jun Takada\*

The biogenous iron oxide (BIO) from *Leptothrix ochracea* was transformed to an organic–inorganic hybrid support to prepare an excellent immobilized enzyme showing high catalytic performance.



## Dalton Discussion 12: Catalytic C-H and C-X Bond Activation

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Canada*

**William D. Jones**  
*University of Rochester, USA*

**Aiwen Lei**  
*Wuhan University, China*

**Zhang-jie Shi**  
*Peking University, China*

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*University of Bristol, UK*

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*University of Oxford, UK*

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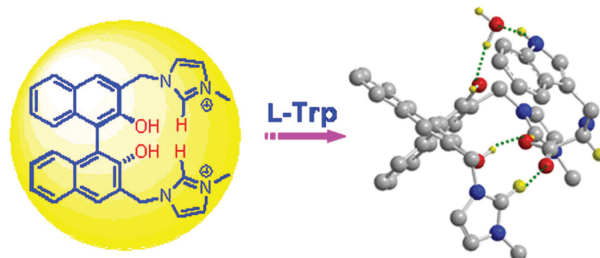


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### 1,1'-Binaphthyl-based imidazolium chemosensors for highly selective recognition of tryptophan in aqueous solutions

Li Yang, Song Qin, Xiaoyu Su, Fei Yang, Jingsong You,\*  
Changwei Hu, Rugang Xie and Jingbo Lan\*

A type of 1,1'-binaphthyl-based imidazolium chemosensor module has been synthesized for the highly selective recognition of tryptophan (Trp) in aqueous solutions *via* synergistic effects of multiple hydrogen bonding and electrostatic interactions.

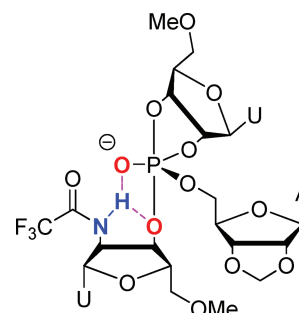


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### Phosphorane intermediate *vs.* leaving group stabilization by intramolecular hydrogen bonding in the cleavage of trinucleoside monophosphates: implications for understanding catalysis by the large ribozymes

Tuomas Lönnberg\* and Maarit Laine

Hydrolysis of a trinucleoside monophosphate model with a 2'-trifluoroacetamido-modified 3'-leaving group has been followed by HPLC over a wide pH range to study the effects of potential hydrogen bonding interactions of the 2'-trifluoroacetamido function on the rate and product distribution of the reaction.

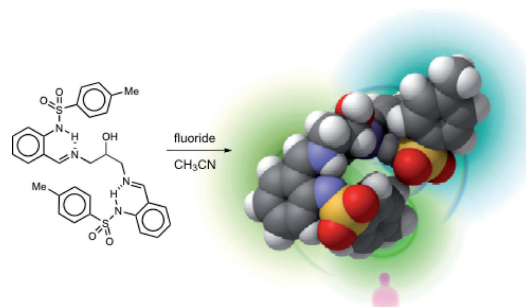


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### Sulfonamide-imines as selective fluorescent chemosensors for the fluoride anion

Miguel Vázquez López,\* Manuel R. Bermejo,\*  
M. Eugenio Vázquez, Angelo Taglietti, Guillermo Zaragoza,  
Rosa Pedrido and Miguel Martínez-Calvo

A new class of sulfonamide fluorescent chemosensor in organic media is reported. This system displayed marked changes in the fluorescence emission intensities and showed selectivity for fluoride over other inorganic anions, such as acetate or dihydrogenphosphate.

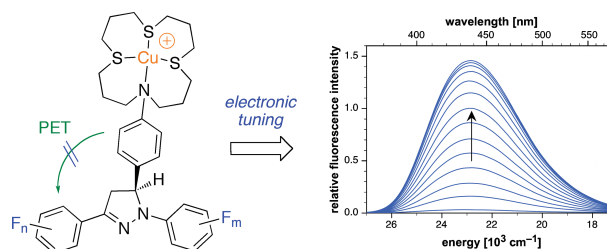


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### Electronically tuned 1,3,5-triarylpyrazolines as Cu(I)-selective fluorescent probes

Manjusha Verma, Aneese F. Chaudhry, M. Thomas Morgan  
and Christoph J. Fahrni\*

A Cu(I)-responsive fluorescent probe, constructed using a large tetradentate, 16-membered thiazacrown ligand ([16]aneNS<sub>3</sub>) and 1,3,5-triaryl-substituted pyrazoline fluorophores has been prepared and characterized.



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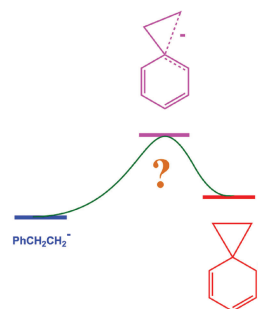
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### Diagnostic fragmentations of adducts formed between carbanions and carbon disulfide in the gas phase. A joint experimental and theoretical study

Micheal J. Maclean, Scott Walker, Tianfang Wang, Peter C. H. Eichinger, Patrick J. Sherman and John H. Bowie\*

The structures of selected carbanions have been identified by collisionally activated decomposition reactions of the corresponding adducts with carbon disulfide in a modified LCQ ion trap mass spectrometer.

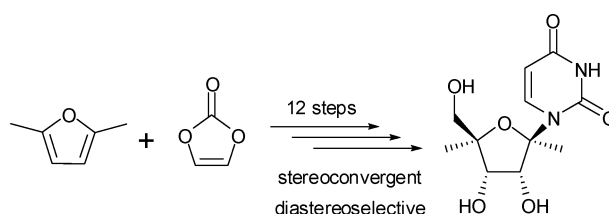


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### Diastereoselective synthesis of ( $\pm$ )-1',4'-dimethyluridine

Guillaume Sautrey, Damien Bourgeois\* and Christian Périgaud

The diastereoselective total synthesis of the title compound is described. It is based on a new methodology involving the stereoconvergent preparation of a suitable sugar ring, followed by a selective Vorbrüggen glycosylation.

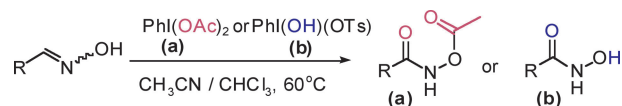


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### Hypervalent iodine(III)-mediated oxidation of aldoximes to *N*-acetoxy or *N*-hydroxy amides

Harisadhan Ghosh and Bhisma K. Patel\*

Various aldoximes were converted to either *N*-acetoxy or *N*-hydroxy amides in good yield using the hypervalent iodine(III) reagents (diacetoxyiodo)benzene (DIB) or Koser's reagent [hydroxy(tosyloxy)iodo]benzene (HTIB). A plausible mechanism for this transformation involves the attack of acetate/hydroxy on the intermediate aryl nitrile oxides, which, upon rearrangement, gave the expected *N*-acetoxy or *N*-hydroxy amides.

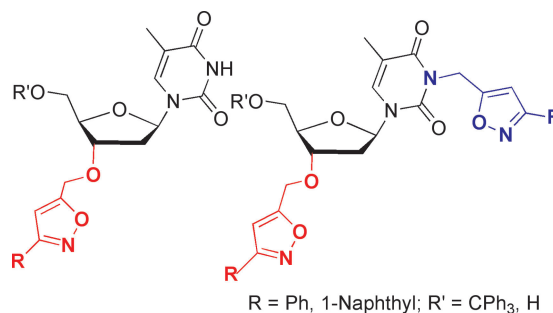


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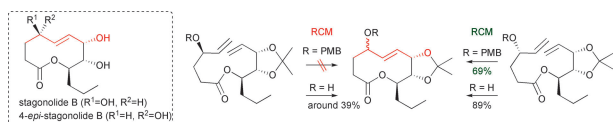
### Nucleoside and nucleotide analogues by catalyst free Huisgen nitrile oxide–alkyne 1,3-dipolar cycloaddition

Virginie Algay, Ishwar Singh and Frances Heaney\*

*C*<sup>3'</sup>-*O*-Isoxazole conjugated, and *C*<sup>3'</sup>-*O*,*N*<sup>3</sup>-bisoxazole nucleosides and nucleotides are prepared regiospecifically, rapidly and in high yield under atmospheric conditions and in an aqueous environment; the protocol identifies chloramine-T as a practical reagent for *in situ* nitrile oxide formation, and the alkyne partner may be attached to the sugar residue or the nucleobase.



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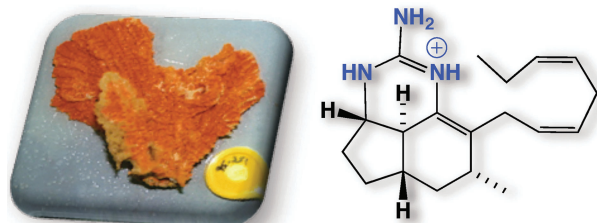


### Effect of the allylic substituents on ring closing metathesis: the total synthesis of stagonolide B and 4-*epi*-stagonolide B

Awadut G. Giri, Mohabul A. Mondal, Vedavati G. Puranik and Chepuri V. Ramana\*

The total syntheses of stagonolide B and its 4-*epi* were carried out to probe into how the relative stereochemistry of allylic hydroxy groups and their protecting groups influence the efficiency of the ring closing metathesis.

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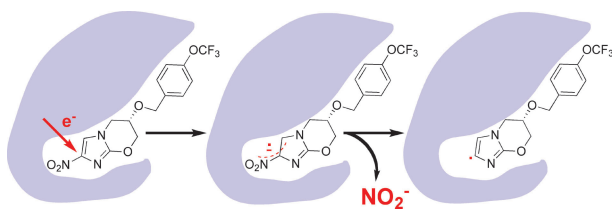


### Mirabilins revisited: polyketide alkaloids from a southern Australian marine sponge, *Clathria* sp.

Mohamed El-Naggar, Melissa Conte and Robert J. Capon\*

Chemical analysis of a southern Australian sponge, *Clathria* sp. yielded new examples of the mirabilin class of marine polyketide alkaloid.

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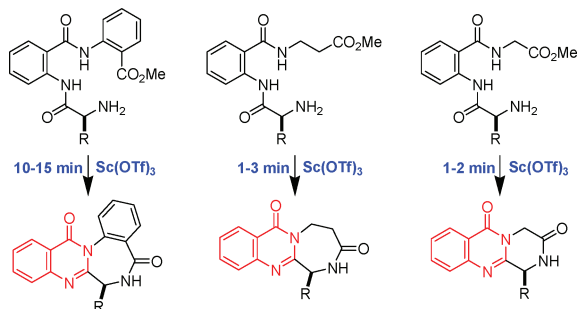


### Release of nitrite from the antitubercular nitroimidazole drug PA-824 and analogues upon one-electron reduction in protic, non-aqueous solvent

Andrej Maroz, Sujata S. Shinde, Scott G. Franzblau, Zhenkun Ma, William A. Denny, Brian D. Palmer and Robert F. Anderson\*

One-electron addition to the antituberculosis drug PA-824 in propan-2-ol leads to the release of nitrite. Such conditions may mimic the water-restricting active site of a reducing protein.

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### Total synthesis of asperlicin C, circumdatin F, demethylbenzomalvin A, demethoxycircumdatin H, sclerotigenin, and other fused quinazolinones

Ming-Chung Tseng, Huei-Yun Yang and Yen-Ho Chu\*

Using  $\text{Sc}(\text{OTf})_3$  and microwaves, direct double dehydrocyclization of anthranilate-containing tripeptides was efficiently achieved, to afford total syntheses of fused quinazolinones in short reaction times, with good overall isolated yields.

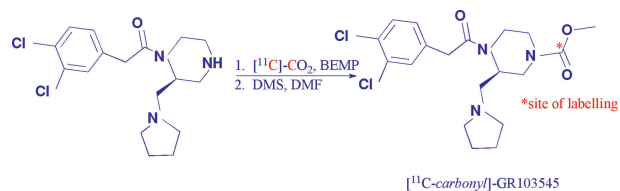


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### Direct fixation of [ $^{11}\text{C}$ ]-CO $_2$ by amines: formation of [ $^{11}\text{C}$ -carbonyl]-methylcarbamates

Alan A. Wilson,\* Armando Garcia, Sylvain Houle and Neil Vasdev

A general method for the synthesis of [ $^{11}\text{C}$ -carbonyl]-radiolabelled carbamates from cyclotron-produced [ $^{11}\text{C}$ ]-CO $_2$  and primary or secondary amines is described. Using this method the selective kappa opioid agonist, [ $^{11}\text{C}$ -carbonyl]-GR103545, was prepared in high yield and at high specific activity, suitable for human positron emission tomography studies.

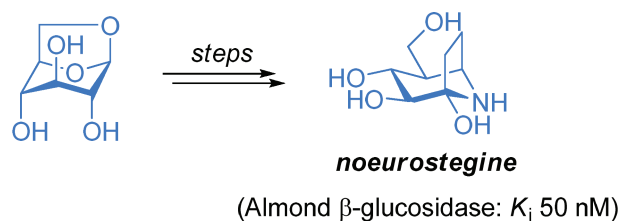


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### Synthesis and glycosidase inhibitory activity of neurostegine—a new and potent inhibitor of $\beta$ -glucoside hydrolases

Tina Secher Rasmussen and Henrik Helligsø Jensen\*

A new, stable hemi-aminal nor-tropane christened neurostegine was synthesised in 22 steps from levoglucosan and tested for inhibitory activity against glycoside hydrolases.

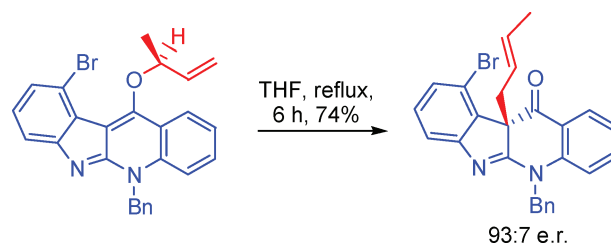


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### Studies on the Claisen rearrangements in the indolo[2,3-*b*]quinoline system

Nicholas Voûte, Douglas Philp, Alexandra M. Z. Slawin and Nicholas J. Westwood\*

A study of the effect of substrate structure on a Claisen–aza-Cope reaction is presented including a rationalisation of the reaction outcome using computational techniques. An asymmetric version of the reaction is also described.

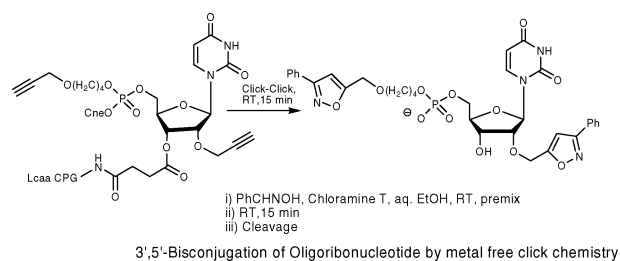


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### Metal free, “click and click–click” conjugation of ribonucleosides and 2'-OMe oligoribonucleotides on the solid phase

Ishwar Singh and Frances Heaney\*

Nitrile oxide alkyne click cycloaddition is reported as a practical, metal free approach to conjugation of ribonucleosides and 2'-OMe 4-mer oligoribonucleotides on the solid phase, the methodology is suited to modification at either, or both, the 3'- or the 5'-terminus of the oligoribonucleotide substrate.



3',5'-Bisconjugation of Oligoribonucleotide by metal free click chemistry

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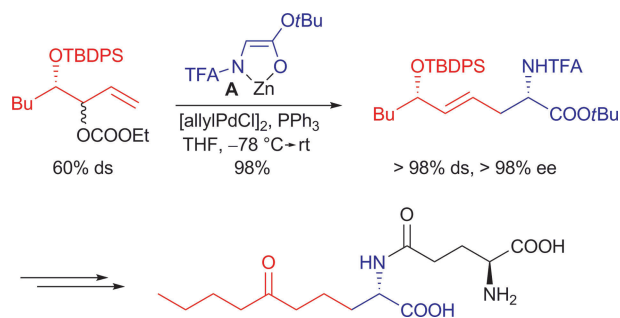
## Thank you

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### Diastereotopos-differentiating allylic alkylation as a key step in the synthesis of $\gamma$ -glutamyl boletine

Dnyaneshwar Gawas and Uli Kazmaier\*

A straightforward approach towards  $\gamma$ -glutamyl boletine is described, based on a diastereotopos-differentiating allylic alkylation of chelated amino acid ester enolates. Independent of the configuration of the leaving group in the allylic substrate, the allylation product is obtained as a single stereoisomer.



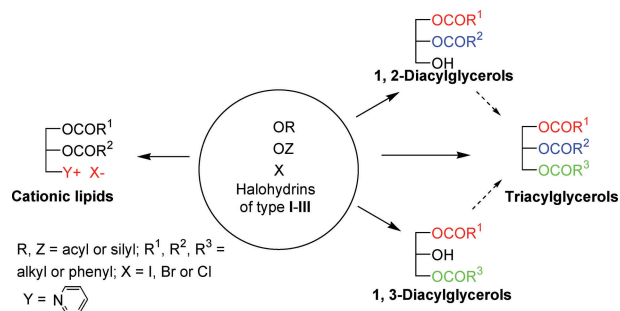
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### *O*-Silylated C3-halohydrins as a novel class of protected building blocks for total, regio- and stereocontrolled synthesis of glycerolipid frameworks

Stephan D. Stamatov\* and Jacek Stawinski\*

Halohydrins of type I–III were developed as key intermediates for construction of diglycerides, triglycerides and cation glycerolipids.



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