

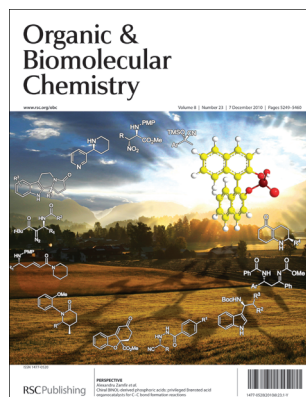
# Organic & Biomolecular Chemistry

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

ISSN 1477-0520 CODEN OBCRAK 8(23) 5249–5460 (2010)



### Cover

See Alexandru Zamfir *et al.*,  
pp. 5262–5276.

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*Organic & Biomolecular  
Chemistry*, 2010, **8**, 5262.

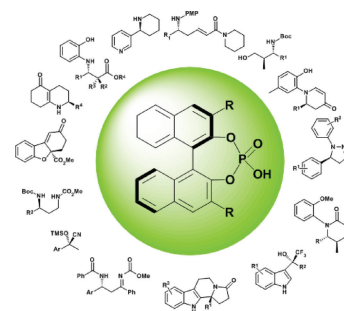
## PERSPECTIVE

5262

### Chiral BINOL-derived phosphoric acids: privileged Brønsted acid organocatalysts for C–C bond formation reactions

Alexandru Zamfir, Sebastian Schenker, Matthias Freund  
and Svetlana B. Tsogoeva\*

Chiral BINOL-derived phosphoric acids are powerful Brønsted acid catalysts in many enantioselective processes. The most successful transformations carried out with chiral BINOL-phosphates include C–C bond formation reactions, which are summarized in this review article.



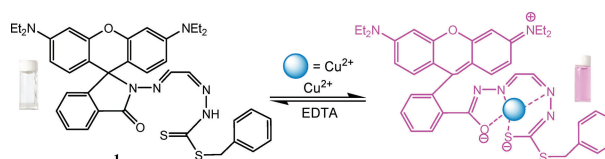
## COMMUNICATIONS

5277

### Highly sensitive and selective colorimetric and off-on fluorescent probe for Cu<sup>2+</sup> based on rhodamine derivative

Chunwei Yu, Jun Zhang, Rui Wang and Lingxin Chen\*

A new probe for Cu<sup>2+</sup> based on the Cu<sup>2+</sup>-induced reversible ring-opening mechanism of the rhodamine spirolactam was described.



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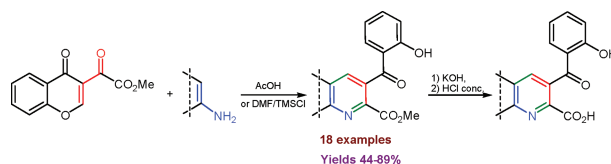
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5280

### 3-Methoxyalychromone—a novel versatile reagent for the regioselective purine isostere synthesis

Satenik Mkrtchyan, Viktor O. Iaroshenko,\* Sergii Dudkin, Ashot Gevorgyan, Marcelo Vilches-Herrera, Gagik Ghazaryan, Dmitriy M. Volochnyuk, Dmytro Ostrovskiy, Zeeshan Ahmed, Alexander Villinger, Vyacheslav Ya. Sosnovskikh and Peter Langer\*

The first synthesis of 3-methoxyalychromone was described. The regioselective reaction with aminoheterocycles provided a set of heteroannulated pyridines.



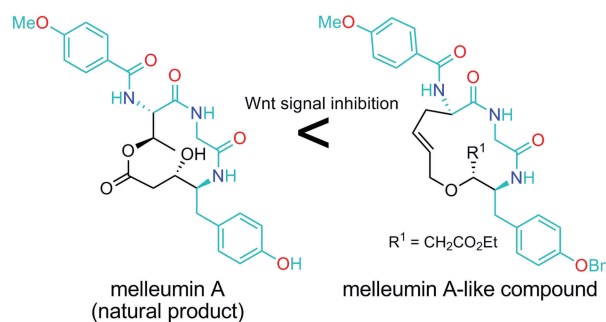
## PAPERS

5285

### Total synthesis and evaluation of Wnt signal inhibition of melleumin A and B, and their derivatives

Midori A. Arai,\* Shuwa Hanazawa, Yujiro Uchino, Xiaofan Li and Masami Ishibashi\*

The total synthesis of melleumin A, a novel cyclic depsipeptide isolated from the myxomycete *Physarum melleum*, 3-*epi*-melleumin A and designed melleumin A-like compounds was achieved. Comparison of the Wnt signal inhibitory activity of synthesized melleumin derivatives led to further investigation of the structural conformation of the active molecules.

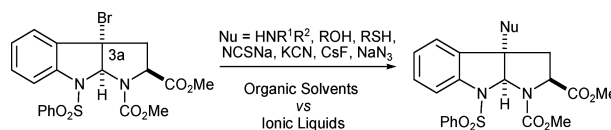


5294

### Facile nucleophilic substitution at the C3a tertiary carbon of the 3a-bromohexahydropyrrolo[2,3-*b*]indole scaffold

Isabel Villanueva-Margalef, David E. Thurston and Giovanna Zinzalla\*

The synthesis of 3a-substituted hexahydropyrrolo[2,3-*b*]indole derivatives *via* nucleophilic substitution at the C3a position is reported using both conventional organic solvents and ionic liquids.

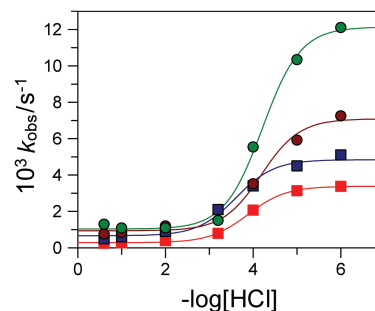


5304

### Butanolysis of 4-methylbenzenediazonium ions in binary *n*-BuOH/H<sub>2</sub>O mixtures and in *n*-BuOH/SDS/H<sub>2</sub>O reverse micelles. Effects of solvent composition, acidity and temperature on the switch between heterolytic and homolytic dediazoniatio mechanisms

Alejandra Fernández-Alonso, M<sup>a</sup> José Pastoriza Gallego and Carlos Bravo-Díaz\*

Heterolytic and homolytic dediazoniatio mechanisms can be modulated in solvolytic dediazoniatio allowing determination of relevant thermodynamic parameters.



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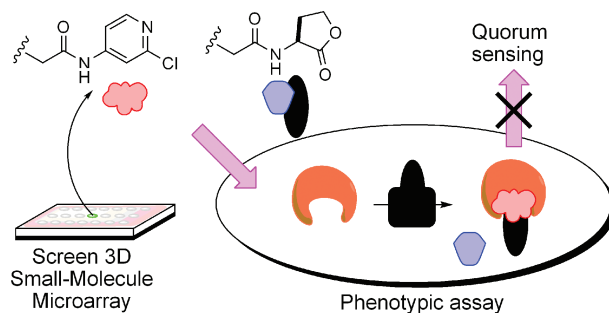


5313

### Discovery of a quorum sensing modulator pharmacophore by 3D small-molecule microarray screening

David M. Marsden, Rebecca L. Nicholson, Mette E. Skindersoe, Warren R. J. D. Galloway, Hannah F. Sore, Michael Givskov, George P. C. Salmond, Mark Ladlow, Martin Welch and David R. Spring\*

A 3D microarray platform was used to discover the biologically active chloro-pyridine pharmacophore, which was found to be able to inhibit N acyl-homoserine-lactone (AHL) mediated quorum sensing phenotypes in *Serratia* and *Pseudomonas aeruginosa*.

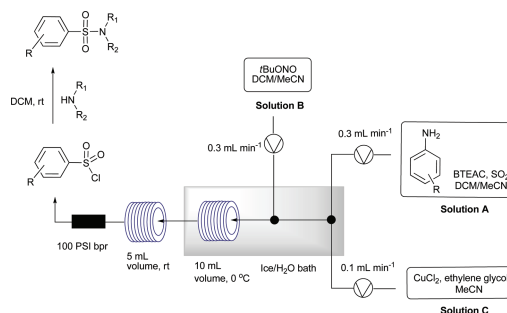


5324

### Preparation of arylsulfonyl chlorides by chlorosulfonylation of *in situ* generated diazonium salts using a continuous flow reactor

Laia Malet-Sanz,\* Julia Madrzak, Steven V. Ley and Ian R. Baxendale

First homogeneous and acid free method for the synthesis of sulfonyl chlorides from anilines, amenable to flow, safe and scalable.

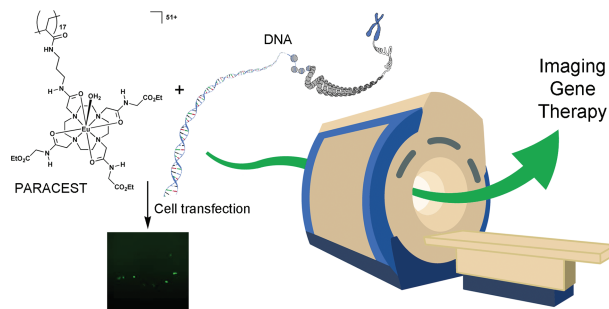


5333

### Polymeric PARACEST MRI contrast agents as potential reporters for gene therapy

Yunkou Wu, Christiane E. Carney, Michael Denton, Elaine Hart, Piyu Zhao, Daniel N. Streblov, A. Dean Sherry and Mark Woods\*

Polymeric MR agents suggest a radical new approach gene therapy; the agent could both mediate transfection and report location and transfection by MRI.

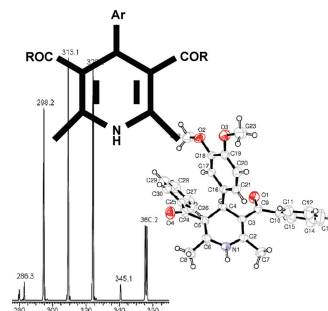


5339

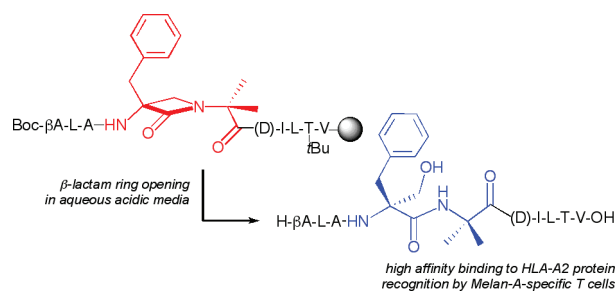
### Synthesis, structural and conformational properties, and gas phase reactivity of 1,4-dihydropyridine ester and ketone derivatives

Gianluca Giorgi,\* Mauro F. A. Adamo, Fabio Ponticelli and Antonio Ventura

A series of 4-aryl-2,6-dimethyl-1,4-dihydropyridines has been synthesized. Their structural and conformational properties have been studied by X-ray crystallography and nuclear magnetic resonance. The gas phase ion chemistry of their protonated and deprotonated species have been investigated by electrospray and CID-MS<sup>n</sup>.



5345

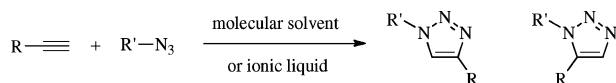


### Design, synthesis and evaluation of $\beta$ -lactam antigenic peptide hybrids; unusual opening of the $\beta$ -lactam ring in acidic media

M. Tarbe, I. Azcune, E. Balentová, J. J. Miles, E. E. Edwards, K. M. Miles, P. Do, B. M. Baker, A. K. Sewell, J. M. Aizpurua, C. Douat-Casassus\* and S. Quideau\*

An unusual  $\beta$ -lactam ring opening occurred during aqueous TFA-mediated release of  $\beta$ -lactam-peptide hybrids from solid support, leading to the formation of a pseudopeptide that expresses a high HLA-A2 binding affinity and stimulates Melan-A-specific T cells.

5354

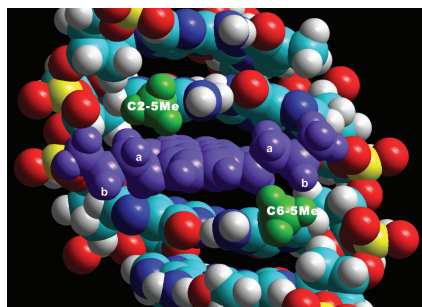


### The effects of ionic liquids on azide-alkyne cycloaddition reactions

Stephen R. D. George, Gavin L. Edwards and Jason B. Harper\*

Ionic liquids increase the regioselectivity whilst effects on activation parameters are intermediate between coordinating and non-coordinating salts. The presence of small amounts of water grossly affects activation parameters.

5359

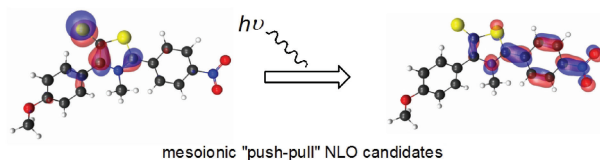


### DNA binding by pixantrone

Najia Adnan, Damian P. Buck, Benny J. Evison, Suzanne M. Cutts,\* Don R. Phillips and J. Grant Collins\*

The anticancer drug pixantrone intercalates at the 5'-<sup>5</sup>MeCpG sites of the octanucleotide  $d(A^{5Me}CGAT^{5Me}CGT)_2$  from the major groove, with the methyl groups not presenting a steric barrier to intercalation.

5367



### Push-pull 1,3-thiazolium-5-thiolates. Formation via concerted and stepwise pathways, and theoretical evaluation of NLO properties

David Cantillo,\* Martín Ávalos, Reyes Babiano, Pedro Cintas, José L. Jiménez, Mark E. Light, Juan C. Palacios and Valentín Rodríguez

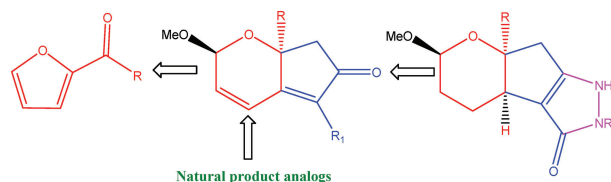
Mesoionic rings possessing a 1,3-thiazolium-5-thiolate unit show promising perspectives in non-linear optics. Full synthetic studies combined with DFT calculations also provide a plausible mechanistic picture.

5375

### Tandem Achmatowicz-Knoevenagel protocol: diastereoselective synthesis and anticancer evaluation of cyclopenta[b]pyrane derivatives

Taleb H. Al-Tel,\* Mohammad H. Semreen and Wolfgang Voelter

Tandem synthesis and biological evaluation of novel cyclopenta[b]pyrane derivatives.

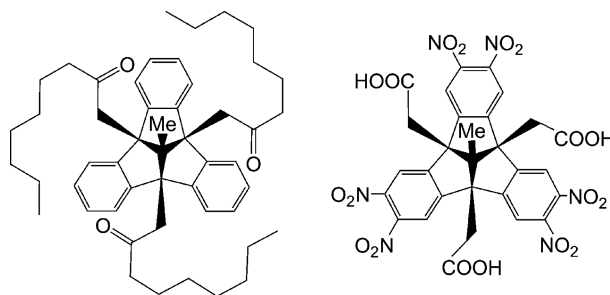


5383

### New $C_{3v}$ -symmetrical tribenzotriquinacenes bearing extended and oxy-functionalised alkyl groups at their benzhydrylic bridgeheads

Ehsan U. Mughal and Dietmar Kuck\*

The convex surface of the bowl-shaped tribenzotriquinacene framework has been furnished with various higher, mostly oxy-functionalised alkyl groups to increase the suitability of TBTQ derivatives for extension of their aromatic periphery.

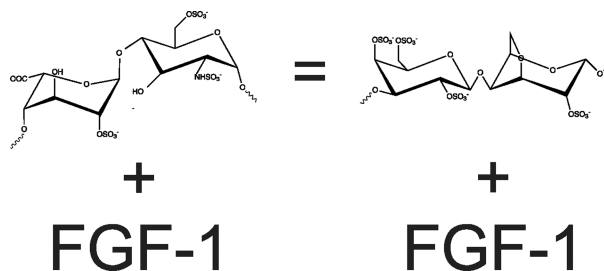


5390

### Comparable stabilisation, structural changes and activities can be induced in FGF by a variety of HS and non-GAG analogues: implications for sequence-activity relationships

Timothy R. Rudd, Katarzyna A. Uniewicz, Alessandro Ori, Scott E. Guimond, Mark A. Skidmore, Davide Gaudesi, Ruoyan Xu, Jeremy E. Turnbull, Marco Guerrini, Giangiacomo Torri, Giuliano Siligardi, Mark C. Wilkinson, David G. Fernig and Edwin A. Yates\*

Appropriate characteristics for FGF binding and activity are provided by various HS structures and non-GAG analogues.

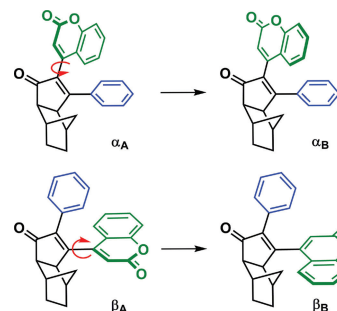


5398

### Atropisomerisation in sterically hindered $\alpha,\beta$ -disubstituted cyclopentenones derived from an intermolecular cobalt(0)-mediated Pauson–Khand reaction

Benjamin E. Moulton, Jason M. Lynam, Anne-Kathrin Duhme-Klair, Wenxu Zheng, Zhenyang Lin\* and Ian J. S. Fairlamb\*

For the first time, sterically hindered  $\alpha,\beta$ -(2,3)-disubstituted cyclopentenones, formed by a Pauson–Khand reaction, are shown to exhibit atropisomerisation. The energetic barrier to atropisomer interconversion is dependent on the relative position of the coumarin moiety.



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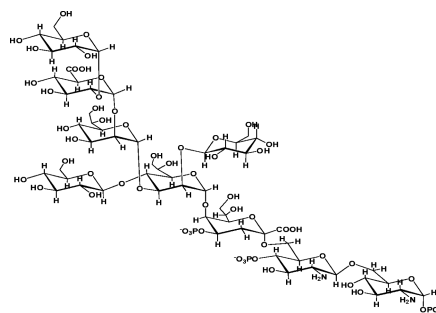
John McCarthy, UK

5404

### Structural characterization of the core region from the lipopolysaccharide of the haloalkaliphilic bacterium *Halomonas alkaliantarctica* strain CRSS

Giuseppina Pieretti, Sara Carillo, Barbara Nicolaus, Annarita Poli, Rosa Lanzetta, Michelangelo Parrilli and Maria Michela Corsaro\*

We described the core oligosaccharide structure from the LPS of the haloalkaliphile *Halomonas alkaliantarctica*, obtained after alkaline hydrolysis of the LPS, HPAEC purification, NMR spectroscopy and mass spectrometry analysis.

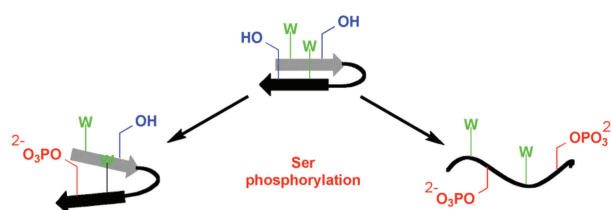


5411

### Positional effects of phosphoserine on $\beta$ -hairpin stability

Alexander J. Riemen and Marcey L. Waters\*

Phosphorylation of a  $\beta$ -hairpin demonstrates the ability of this covalent modification to alter structure in a position-dependent manner, providing insight potential mechanisms by which protein phosphorylation influences structure and function.

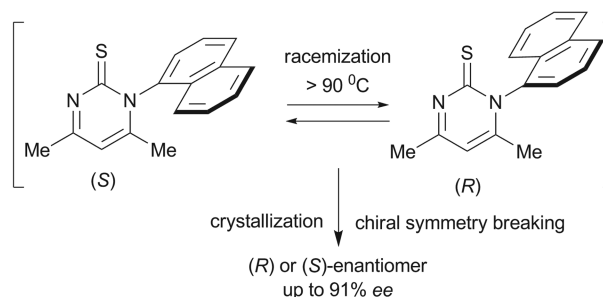


5418

### Generation and amplification of optical activity of axially chiral *N*-(1-naphthyl)-2(*1H*)-pyrimidinethione by crystallization

Masami Sakamoto,\* Fumitoshi Yagishita, Masaru Ando, Yuich Sasahara, Norifumi Kamataki, Mai Ohta, Takashi Mino, Yoshio Kasashima and Tsutomu Fujita

The crystallization of racemic axially chiral pyrimidinethione at high temperature led to the chiral breaking of symmetry up to 91% ee.

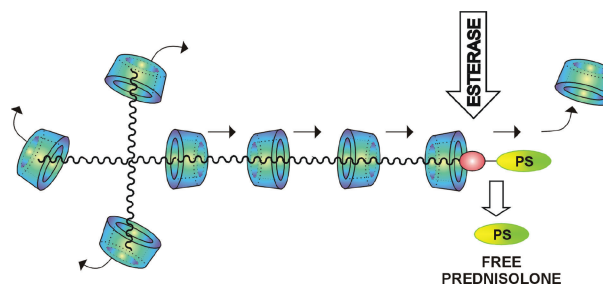


5423

### Prednisolone- $\alpha$ -cyclodextrin-star PEG polypseudorotaxanes with controlled drug delivery properties

Eliška Bílková, Miloš Sedlák,\* Bohuslav Dvořák, Karel Ventura, Petr Knotek and Ludvík Beneš

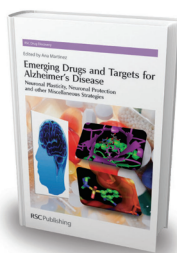
The synthesized polypseudorotaxanes were characterized by 2D NOESY NMR spectra, powder X-ray diffraction patterns, and STM. The rate of release of prednisolone from the carrier can be controlled by: character of the linker between polymeric carrier and prednisolone, molecular mass of PEG, and the kinetics of the dethreading of  $\alpha$ -CD units.





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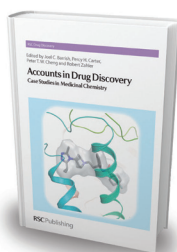
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## New for 2011

### Pharmaceutical Process Development: Current Chemical and Engineering Challenges

Price: £121.99 | ISBN 9781849731461

This book is aimed at both graduates and postgraduates interested in a career in the pharmaceutical industry and informs them about the breadth of the work carried out in chemical research and development departments. It is also of value to academics wishing to advise students on the merits of careers in chemical development versus discovery.

### Animal Models for Neurodegenerative Disease

Price: £121.99 | ISBN 9781849731843

This book provides up-to-date information on the use of transgenic mouse models in the study of neurodegenerative disorders such as Alzheimer's and Huntington's disease. The editors have extensive knowledge and experience in this field and the book is aimed at undergraduates, postgraduates and academics.

### Metallostasis and Proteostasis in Neurodegeneration

Price: £121.99 | ISBN 9781849730501

This book provides up-to-date information on key developments in this fast moving field. Topics covered emphasize the importance of metals and oxidation chemistry to neuroscientists as well as providing a wider, multidisciplinary background to chemists who are attracted by these fascinating subjects.

Whilst every effort was made to ensure these prices were correct at the time of going to press, all information is subject to change without notice.

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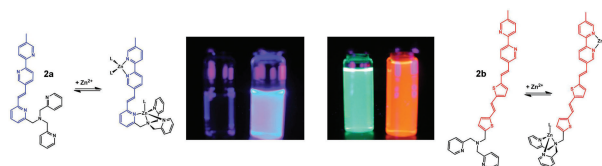
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5431

### Electronic structural dependence of the photophysical properties of fluorescent heteroditopic ligands – implications in designing molecular fluorescent indicators

Ali H. Younes, Lu Zhang, Ronald J. Clark, Michael W. Davidson\* and Lei Zhu\*

This investigation reveals the rationale and limitations of engineering a heteroditopic fluorescent indicator for zinc ion.

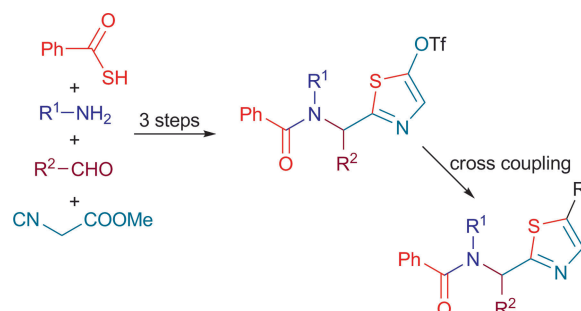


5442

### A straightforward approach towards 5-substituted thiazolylpeptides via the thio-Ugi-reaction

Uli Kazmaier\* and Andrea Persch

Activated thiazoles can easily be obtained by Ugi reactions using thioacids and subsequent cyclisations of the *endo* thiopeptides formed with triflic anhydride.

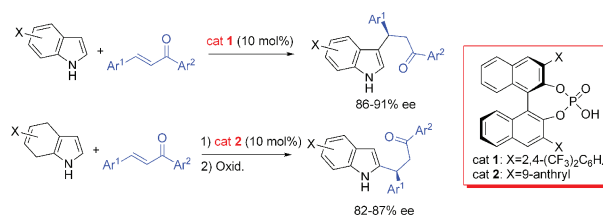


5448

### Chiral Brønsted acid catalyzed asymmetric Friedel–Crafts alkylation reaction of indoles with $\alpha,\beta$ -unsaturated ketones: short access to optically active 2- and 3-substituted indole derivatives

Tsubasa Sakamoto, Junji Itoh, Keiji Mori and Takahiko Akiyama\*

Phosphoric acid catalyzed enantioselective Friedel–Crafts alkylation of indole with  $\alpha,\beta$ -unsaturated ketones.



5455

### Converting drugs into gelators: supramolecular hydrogels from *N*-acetyl-L-cysteine and coinage-metal salts

Pablo Casuso, Pedro Carrasco, Iraida Loinaz, Hans J. Grande and Ibon Odriozola\*

A thiol-containing small drug such as *N*-acetyl-L-cysteine is easily transformed into a potent hydrogelator by the simple addition of a gold, silver or copper salt.

