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# Organic & Biomolecular Chemistry

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PAPER

Isabelle Leray *et al.*

Aggregation-induced emission enhancement upon Al<sup>3+</sup> complexation with a tetrasulfonated calix[4]bisazacrown fluorescent molecular sensor



# Organic & Biomolecular Chemistry

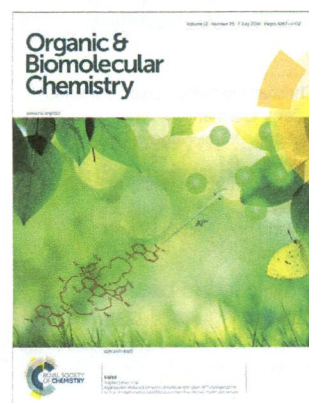
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## IN THIS ISSUE

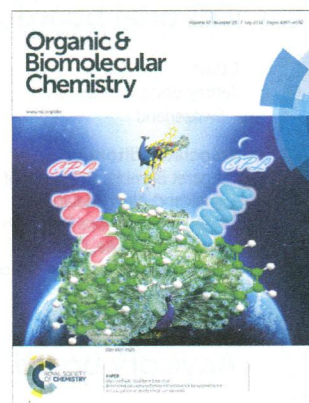
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### Cover

See Isabelle Leray *et al.*, pp. 4335–4341.

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### Inside cover

See Michiya Fujiki, Yoshitane Imai *et al.*, pp. 4342–4346.

Image reproduced by permission of Yoshitane Imai from *Org. Biomol. Chem.*, 2014, **12**, 4342.

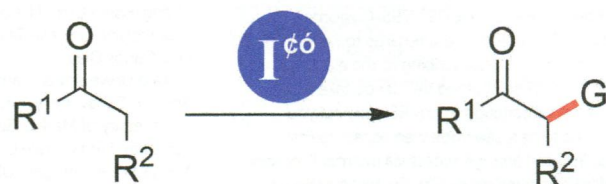
## REVIEW

4278

### Hypervalent iodine: a powerful electrophile for asymmetric $\alpha$ -functionalization of carbonyl compounds

Dao-Qing Dong, Shuang-Hong Hao, Zu-Li Wang\* and Chao Chen\*

Environmentally friendly hypervalent iodine reagents are unusually effective promoters of asymmetric  $\alpha$ -functionalization of carbonyl compounds.



moderate to high yields and ee

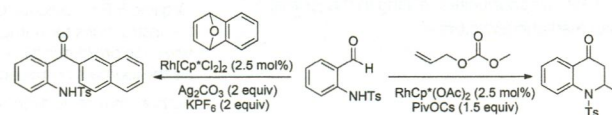
## COMMUNICATIONS

4290

### Rhodium(III)-catalyzed coupling of *N*-sulfonyl-2-aminobenzaldehydes with oxygenated allylic olefins through C–H activation

Tingting Yang, Tao Zhang, Shangdong Yang, Shanshan Chen\* and Xingwei Li\*

*N*-Sulfonyl-2-aminobenzaldehyde undergoes C–H activation and coupling with oxygenated allylic olefins under redox-neutral conditions with high efficiency.



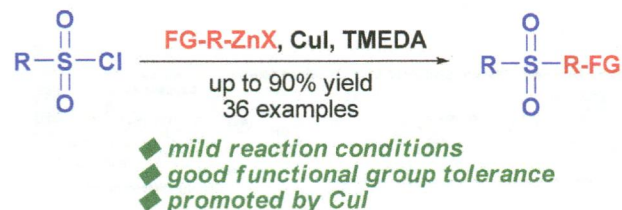
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Центральная научная библиотека  
Уральского отделения  
Российской академии наук (ЦНБ УрО РАН)

4295

### CuI catalyzed sulfonylation of organozinc reagents with sulfonyl halides

Ying Fu,\* Wenbo Zhu, Xingling Zhao, Helmut Hügel, Zhouqiang Wu, Yuhu Su, Zhengyin Du, Danfeng Huang and Yulai Hu

A general and efficient CuI/TMEDA catalyzed nucleophilic addition of functionalized organozinc reagents to organic sulfonyl chlorides has been developed for both aromatic and aliphatic sulfone synthesis.

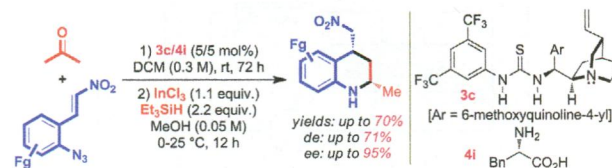


4300

### Asymmetric synthesis of tetrahydroquinolines through supramolecular organocatalysis

Dhevalapally B. Ramachary\* and Kodambahalli S. Shruthi

Functionalized chiral tetrahydroquinolines were synthesized through supramolecular organocatalysis using quinidine-*NH*-thiourea **3c**/L-phenylalanine **4i** followed by reductive amination from the simple substrates.

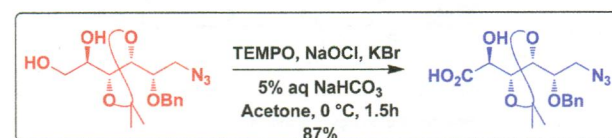


4305

### A chemoselective oxidation of monosubstituted ethylene glycol: facile synthesis of optically active $\alpha$ -hydroxy acids

Kiran Chinthapally and Sundarababu Baskaran\*

A mild and efficient method for the chemoselective oxidation of monosubstituted ethylene glycols to optically active  $\alpha$ -hydroxy acids has been achieved using the TEMPO–NaOCl reagent system.

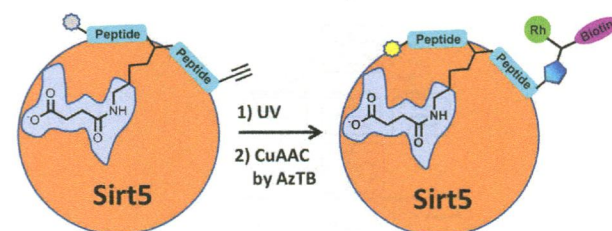


4310

### A succinyl lysine-based photo-cross-linking peptide probe for Sirtuin 5

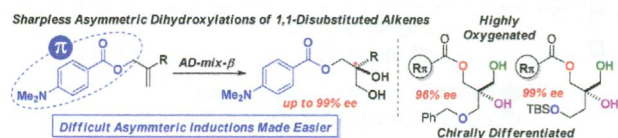
Karunakaran A. Kalesh and Edward W. Tate\*

A succinylation-specific photo-cross-linking peptide probe has been developed for the NAD<sup>+</sup>-dependent hydrolase Sirtuin 5.





4314

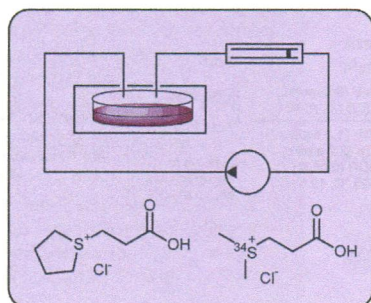


### *N,N*-Dimethylaminobenzoates enable highly enantioselective Sharpless dihydroxylations of 1,1-disubstituted alkenes

Yaohong Zhao, Xiangyou Xing, Shaolong Zhang and David Zhigang Wang\*

Exploration of beneficial catalyst–substrate interactions in the classical Sharpless asymmetric dihydroxylations (SAD) led to the identification of allylic *N,N*-dimethylaminobenzoate as an efficient auxiliary for inducing high levels of enantioselectivity in 1,1-disubstituted aliphatic alkenes.

4318



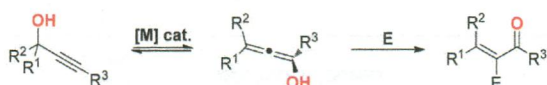
### Marine bacteria from the *Roseobacter* clade produce sulfur volatiles via amino acid and dimethylsulfoniopropionate catabolism

Nelson L. Brock, Markus Menke, Tim A. Klapschinski and Jeroen S. Dickschat\*

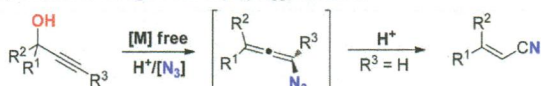
The biosynthesis of sulfur volatiles in marine bacteria was studied by feeding of  $^{34}\text{S}$ -labelled DMSP and some unnatural derivatives.

4324

a) Transition-metal promoted Meyer-Schuster rearrangement with  $\text{E}^+$



b) *This work*: Nitrogenation of propargylic alcohols



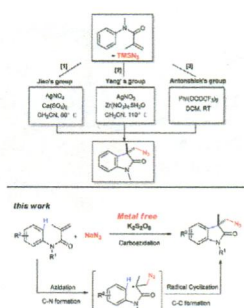
\* Incorporation of N-atom; \* Transition-metal-free; \* Efficient and practical.

### Brønsted acid mediated nitrogenation of propargylic alcohols: an efficient approach to alkenyl nitriles

Xiaoqiang Huang and Ning Jiao\*

A novel and efficient approach to alkenyl nitriles from readily available propargylic alcohols has been developed.

4329



### Transition-metal-free oxidative carboamidation of acrylamides via cascade C–N and C–C bond-forming reactions

Jun Qiu and Ronghua Zhang\*

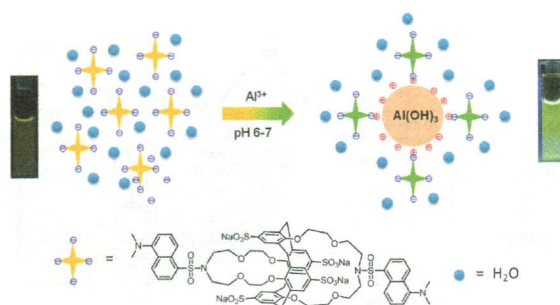
A novel transition-metal-free oxidative carboamidation of acrylamides using inexpensive  $\text{NaN}_3$  and  $\text{K}_2\text{S}_2\text{O}_8$  was achieved, which not only provided an efficient method to prepare various  $\text{N}_3$ -substituted oxindoles, but also represented a novel strategy for C–N and C–C bond formation via a free-radical cascade process.

4335

### Aggregation-induced emission enhancement upon $\text{Al}^{3+}$ complexation with a tetrasulfonated calix[4]bisazacrown fluorescent molecular sensor

Yi-Bin Ruan, Alexis Depauw and Isabelle Leray\*

A new water-soluble ligand bearing a tetrasulfonated calix[4]arene was constructed for ratiometric detection of  $\text{Al}^{3+}$  based on an aggregation-induced fluorescence enhancement mechanism.

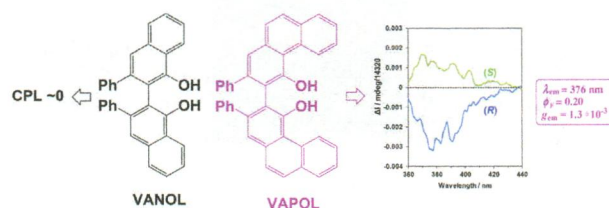


4342

### Enhancing circularly polarised luminescence by extending the $\pi$ -conjugation of axially chiral compounds

Yoko Kitayama, Tomoyuki Amako, Nozomu Suzuki, Michiya Fujiki\* and Yoshitane Imai\*

$\pi$ -Conjugated (*R*)- and (*S*)-2,2'-diphenyl-4-biphenanthrol (VAPOL) exhibited an efficient circularly polarised luminescence (CPL) ( $\sim 1.3 \times 10^{-3}$ ) at 376 nm. By comparison, (*R*)- and (*S*)-3,3-diphenyl-2,2-bi-1-naphthol (VANOL) exhibited no CPL.

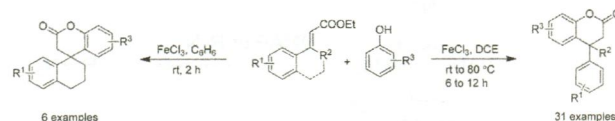


4347

### Lewis acid promoted dual bond formation: facile synthesis of dihydrocoumarins and spiro-tetracyclic dihydrocoumarins

Pedireddi Niharika, Bokka Venkat Ramulu and Gedu Satyanarayana\*

Lewis acid ( $\text{FeCl}_3$ ) mediated dual bond (C–C and C–O) formation for synthesis of 3,4-dihydrocoumarins is presented.

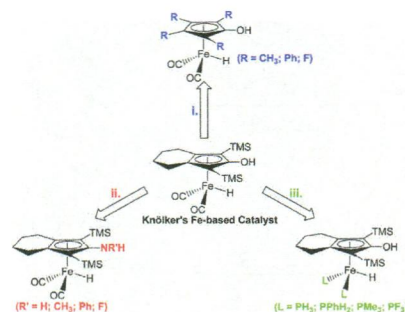


4361

### Using computational methods to explore improvements to Knölker's iron catalyst

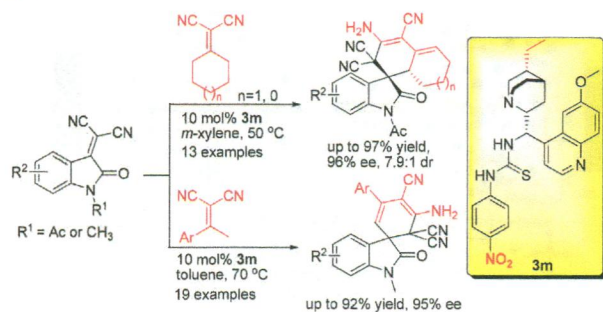
Xi Lu, Yawei Zhang, Nicholas Turner, Mingtao Zhang\* and Tonglei Li\*

Some improvements were predicted for Knölker's catalyst via density functional method.





4372

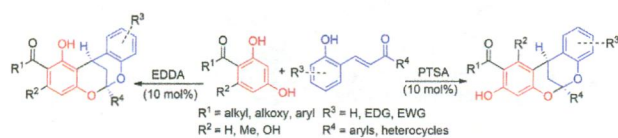


### Organocatalytic enantioselective construction of multi-functionalized spiro oxindole dienes

Xiao-Fei Huang, Ya-Fei Zhang, Zheng-Hang Qi, Nai-Kai Li, Zhi-Cong Geng, Kun Li and Xing-Wang Wang\*

A highly stereoselective Michael-cyclization–tautomerization reaction of isatylidene malononitriles with  $\alpha,\alpha$ -dicyanoalkenes was developed *via* organocatalysis. An anomalous temperature effect on the enantioselectivity is disclosed for this transformation.

4386

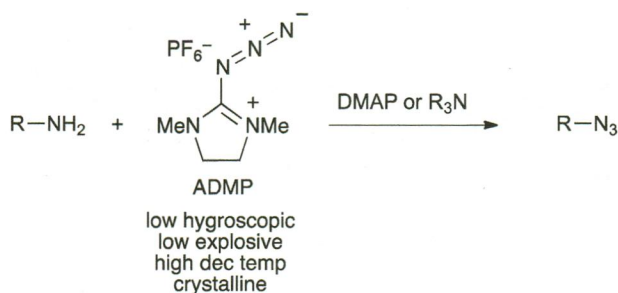


### Catalyst-controlled regio- and stereoselective synthesis of diverse 12H-6,12-methanodibenzo[d,g][1,3]dioxocines

Likai Xia, Hongyun Cai and Yong Rok Lee\*

Regio- and stereoselective synthesis of 12H-6,12-methanodibenzo[d,g][1,3]dioxocines has been accomplished by the EDDA and PTSA-catalyzed cascade reactions of resorcinols and 2-hydroxychalcones.

4397

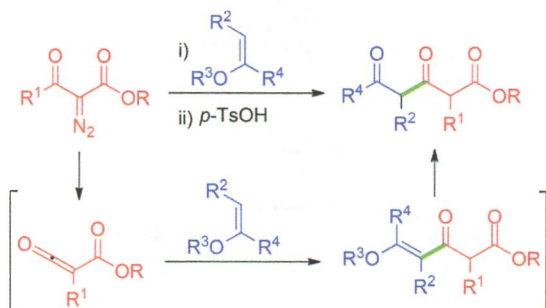


### A reagent for safe and efficient diazo-transfer to primary amines: 2-azido-1,3-dimethylimidazolinium hexafluorophosphate

Mitsuru Kitamura,\* So Kato, Masakazu Yano, Norifumi Tashiro, Yuichiro Shiratake, Mitsuyoshi Sando and Tatsuo Okauchi

Organic azides were synthesized from primary amines by diazo-transfer with a safe and stable crystalline reagent, 2-azido-1,3-dimethylimidazolinium hexafluorophosphate (ADMP).

4407



### Novel one-pot synthesis of diverse $\gamma,\delta$ -unsaturated $\beta$ -ketoesters by thermal cascade reactions of diazodicarbonyl compounds and enol ethers: transformation into substituted 3,5-diketoesters

Rameshwar Prasad Pandit and Yong Rok Lee\*

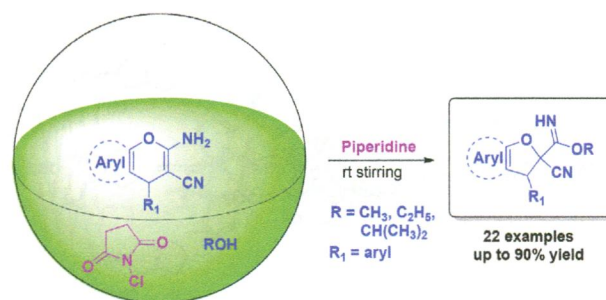
Novel and efficient synthesis of diverse  $\gamma,\delta$ -unsaturated  $\beta$ -ketoesters was accomplished by thermal cascade reactions of diazodicarbonyl compounds with enol ethers and the synthesized compounds were further transformed into the corresponding 3,5-diketoesters.

4412

### "One-pot" access to dihydrofurans via tandem oxidative difunctionalization and ring contraction of aminopyrans

Santhosh Reddy Mandha, Manjula Alla\* and Jagadeesh Babu Nanubolu

A new strategy for the construction of dihydrofuran derivatives by one-pot tandem oxidative difunctionalization and base catalysed ring contraction of aminopyrans.

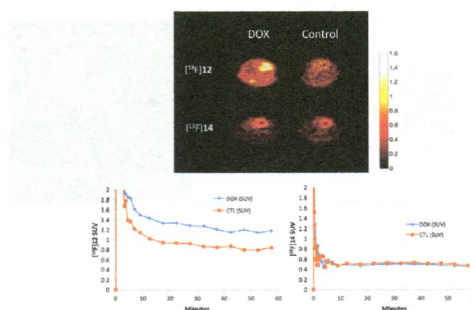


4421

### Development of a PET radiotracer for non-invasive imaging of the reactive oxygen species, superoxide, *in vivo*

Wenhua Chu, Andre Chepetan, Dong Zhou, Koresh I. Shoghi, Jinbin Xu, Laura L. Dugan, Robert J. Gropler, Mark A. Mintun and Robert H. Mach\*

Non-invasive imaging of reactive oxygen species (ROS) *in vivo* was investigated using a dihydroethidium analog [<sup>18</sup>F]**12** as a PET radiotracer. The data shown indicates that [<sup>18</sup>F]**12** is a promising PET tracer for non-invasive imaging of ROS *in vivo*.

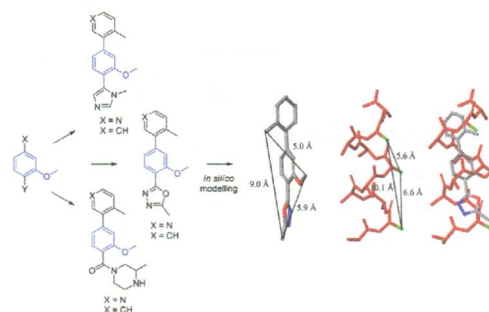


4432

### An iterative *in silico* and modular synthetic approach to aqueous soluble tercyclic $\alpha$ -helix mimetics

Zelong Lim, Peter J. Duggan, Adam G. Meyer\* and Kellie L. Tuck\*

Tercyclic scaffolds, designed to have improved synthetic accessibility and aqueous solubility, were evaluated as structural  $\alpha$ -helix mimetics by using an iterative *in silico* approach.

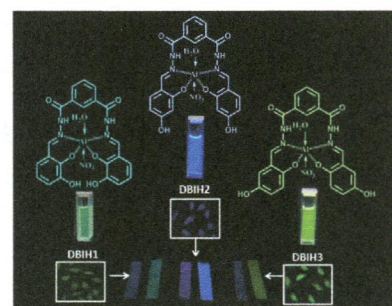


4445

### Nanomolar fluorogenic detection of Al(III) by a series of Schiff bases in an aqueous system and their application in cell imaging

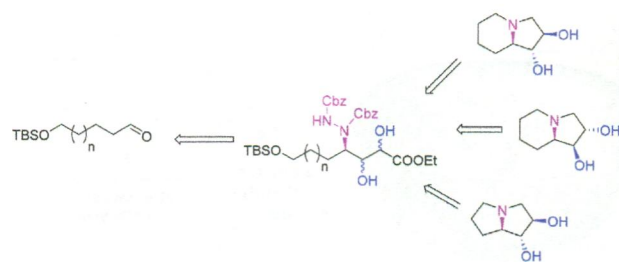
Sanyog Sharma, Maninder Singh Hundal,\* Amandeep Walia, Vanita Vanita and Geeta Hundal\*

Three Schiff bases, showing nanomolar, naked eye detection of Al(III) under UV light, in an aqueous system are reported.





4454

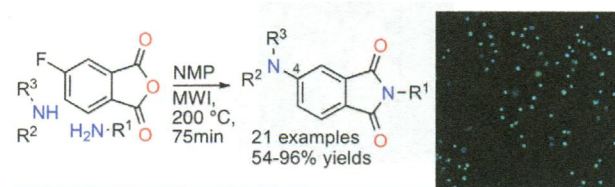


### A stereoselective approach to indolizidine and pyrrolizidine alkaloids: total synthesis of (-)-lentiginosine, (-)-epi-lentiginosine and (-)-dihydroxypyrrolizidine

Shruti Vandana Kauloorkar, Vishwajeet Jha, Ganesh Jogdand and Pradeep Kumar\*

The total synthesis of (-)-lentiginosine, *epi*-1,2-lentiginosine and dihydroxypyrrolizidine is reported from an aldehyde as a starting material using organocatalysis and asymmetric dihydroxylation as key steps.

4461

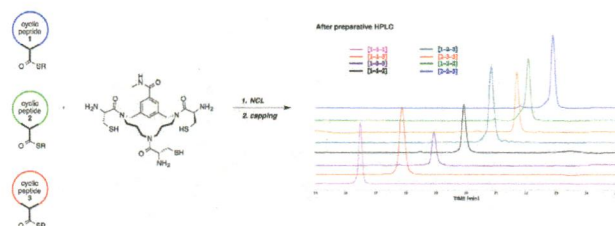


### Efficient one-step synthesis of 4-amino substituted phthalimides and evaluation of their potential as fluorescent probes

Tomas Kindahl and Erik Chorell\*

*Chemistry:* One step, high yields, general, atom efficient, no additives. *Compounds:* Highly fluorescent and solvatochromic, many display non-toxic properties.

4471

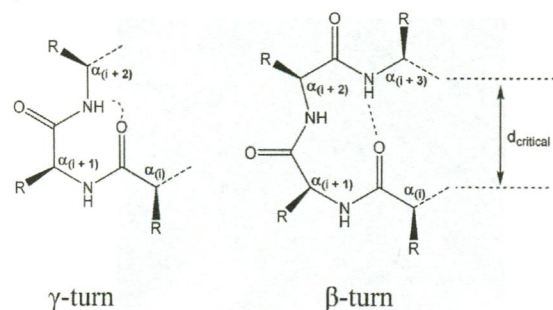


### Scaffolded multiple cyclic peptide libraries for protein mimics by native chemical ligation

H. van de Langemheen, M. van Hoeke, H. C. Quarles van Ufford, J. A. W. Kruijtzter and R. M. J. Liskamp\*

The accessibility to collections, libraries and arrays of cyclic peptides is increasingly important since cyclic peptides may provide better mimics of the loop-like structures ubiquitously present in and – especially – on the surface of proteins.

4479



### Proline *N*-oxides: modulators of the 3D conformation of linear peptides through "NO-turns"

Majid D. Farahani, Bahareh Honarparvar, Fernando Albericio, Glenn E. M. Maguire, Thavendran Govender, Per I. Arvidsson\* and Hendrik G. Kruger\*

*N*-Oxide residues enforce turn conformations in peptides.

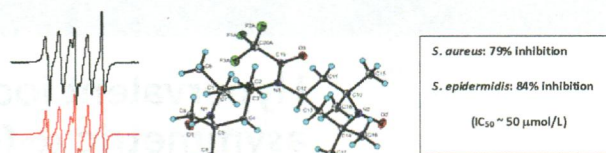


4491

### Synthesis, physicochemical properties and antimicrobial activity of mono-/dinitroxyl amides

Miroslav Kavala, Vlasta Brezová, Lubomír Švorc, Zuzana Vihonská, Petra Olejníková, Ján Moncol, Jozef Kožíšek, Peter Herich and Peter Szolcsányi\*

Novel TEMPO and PROXYL derivatives reveal (quasi)-reversible redox behavior. The EWG-substituents increase their oxidation potential in comparison to the EDG-groups. Unlike pyrrolidinyl derivatives, the piperidinyl nitroxides inhibit the growth of *Staphylococcus* sp.

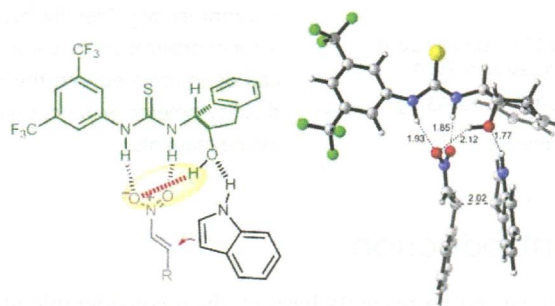


4503

### A Friedel–Crafts alkylation mechanism using an aminoindanol-derived thiourea catalyst

David Roca-López, Eugenia Marqués-López, Ana Alcaine, Pedro Merino and Raquel P. Herrera\*

Computational calculations based on experimental results shed light on the mechanistic proposal for a Friedel–Crafts alkylation reaction between indole and nitroalkenes, catalysed by a chiral aminoindanol-derived thiourea.

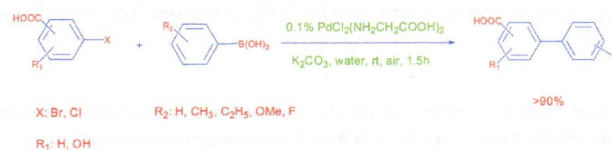


4511

### A highly efficient catalyst of a nitrogen-based ligand for the Suzuki coupling reaction at room temperature under air in neat water

Shiwen Liu, Meiyun Lv, Daoan Xiao, Xiaogang Li, Xiuling Zhou and Mengping Guo\*

Glycine is used to prepare an air-stable and water-soluble catalyst for the Suzuki–Miyaura reaction. In the presence of 0.1% [PdCl<sub>2</sub>(NH<sub>2</sub>CH<sub>2</sub>COOH)<sub>2</sub>], excellent catalytic activity is observed at room temperature under air in neat water.



4517

### Synthesis and biological evaluation of hybrids from farnesylthiosalicylic acid and hydroxycinnamic acid with dual inhibitory activities of Ras-related signaling and phosphorylated NF-κB

Yong Ling, Zhiqiang Wang, Xuemin Wang, Ying Zhao, Wei Zhang, Xinyang Wang, Li Chen, Zhangjian Huang\* and Yihua Zhang\*

Hybrid **5f** significantly inhibited both Ras-related signaling and phosphorylated NF-κB, which may synergistically contribute to its apoptosis induction and tumor growth inhibition *in vitro* and *in vivo*.

