

# Green Chemistry

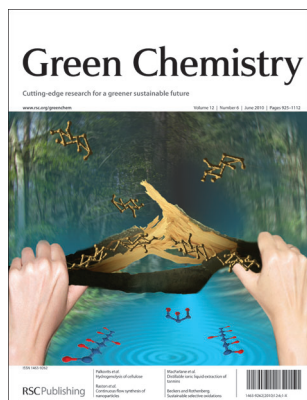
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## IN THIS ISSUE

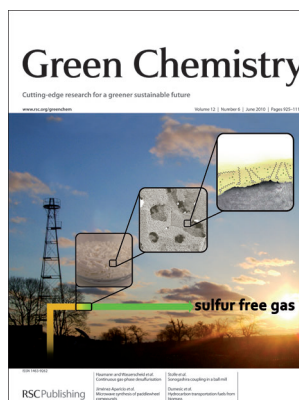
ISSN 1463-9262 CODEN GRCHFJ 12(6) 925–1112 (2010)



### Cover

See Palkovits *et al.*, pp. 972–978. Combining hydrolysis and hydrogenation/hydrogenolysis of cellulose and even wood opens the way to a multitude of valuable chemicals.

Image reproduced by permission of Regina Palkovits from *Green Chemistry*, 2010, **12**, 972.



### Inside cover

See Wasserscheid and Haumann *et al.*, pp. 979–984. Novel supported ionic liquid phase (SILP) materials for efficient, continuous gas cleaning processes.

Image reproduced by permission of Marco Haumann and Florian Kohler from *Green Chemistry*, 2010, **12**, 979.

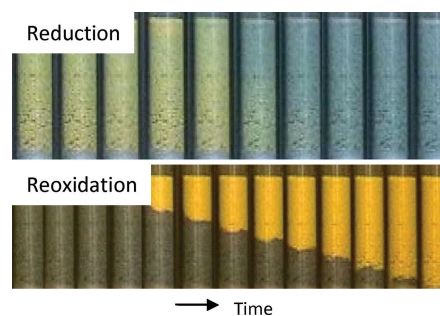
## PERSPECTIVE

939

### Sustainable selective oxidations using ceria-based materials

Jurriaan Beckers and Gadi Rothenberg\*

We review several types of catalytic oxidation processes using ceria-based materials, and discuss their possibilities for sustainable chemistry and sustainable energy applications.



## COMMUNICATIONS

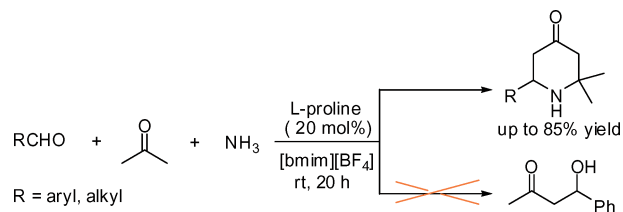
949



### Highly efficient chemoselective construction of 2,2-dimethyl-6-substituted 4-piperidones via multi-component tandem Mannich reaction in ionic liquids

Li-Chun Feng, Ya-Wei Sun, Wei-Jun Tang, Li-Jin Xu,\* Kim-Lung Lam, Zhongyuan Zhou and Albert S. C. Chan

The room temperature ionic liquid [bmim][PF<sub>6</sub>] has been demonstrated to be an efficient and recyclable medium for highly chemoselective synthesis of 2,2-dimethyl-6-substituted 4-piperidones via a L-proline catalyzed tandem Mannich reaction of ammonia, aldehydes and acetone, and good yields were achieved for aryl and alkyl aldehydes.



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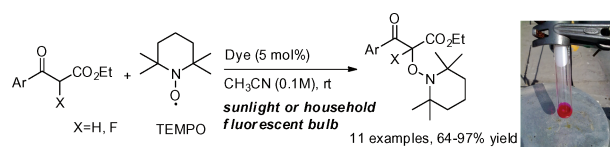
## COMMUNICATIONS

953

### Organic dye photocatalyzed $\alpha$ -oxyamination through irradiation with visible light

Hongjun Liu, Wei Feng, Choon Wee Kee, Yujun Zhao, Dasheng Leow, Yuanhang Pan and Choon-Hong Tan\*

Rose Bengal, an organic dye, was used as a visible light photocatalyst to catalyze novel  $\alpha$ -oxyamination reactions between 1,3-dicarbonyl compounds and a free radical (TEMPO) in sunlight or in the presence of an inexpensive and convenient source of photons – household fluorescent bulbs. Compounds that are difficult to obtain such as quaternary fluorinated compounds were prepared using this method.

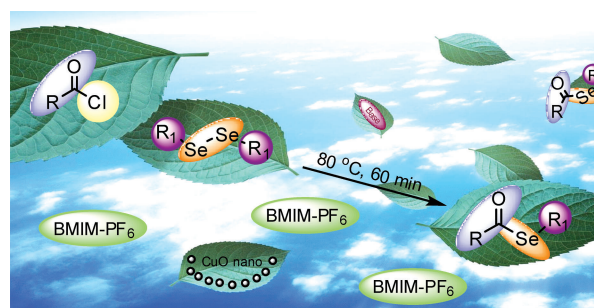


957

### Efficient synthesis of selenoesters from acyl chlorides mediated by CuO nanopowder in ionic liquid

Devender Singh, Senthil Narayanaperumal, Kashif Gul, Marcelo Godoi, Oscar Endrigo Dorneles Rodrigues\* and Antonio Luiz Braga\*

We report an eco-friendly synthesis of selenoesters from acyl chlorides catalyzed by recyclable CuO nanopowder in ionic liquid as a recyclable solvent in good to excellent yields. This protocol shows high efficiency in catalyzing this transformation in a greener fashion.

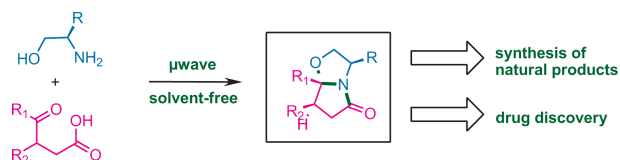


961

### Solvent-free microwave-assisted Meyers' lactamization

Mouhamad Jida, Rebecca Deprez-Poulain,\* Sandra Malaquin, Pascal Roussel, Francine Agbossou-Niedercorn, Benoit Deprez and Guillaume Laconde\*

Solvent-free microwave-activation allows the efficient synthesis of bicyclic or tricyclic lactams *via* the Meyers' reaction in excellent yields and good diastereoselectivities in very short times. This is the first greener alternative for this reaction that is a keystone for syntheses of natural products derivatives and bioactive compounds in drug discovery.

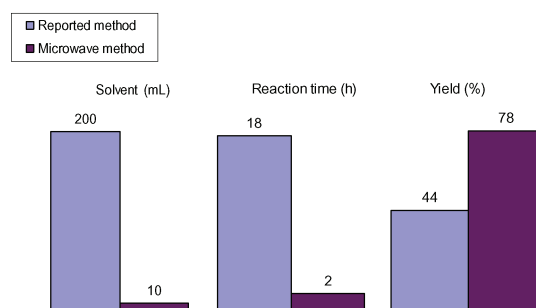


965

### First microwave synthesis of multiple metal-metal bond paddlewheel compounds

Santiago Herrero, Reyes Jiménez-Aparicio,\* Josefina Perles, José L. Priego and Francisco A. Urbanos

A green and efficient microwave assisted synthesis of elusive tetrakis(diaryltriazenido)diruthenium complexes was carried out under microwave irradiation. It is a simple, clean, and fast method that proceeds with good yield.



The aim of the conference is to highlight innovative concepts for the substitution of volatile organic solvents in solution phase synthesis. Emphasis will be laid on the development and application of alternative reaction media based on advanced fluids such as aqueous phases, ionic liquids, supercritical phases, green organic solvents, or soluble polymers, but includes also phase-separable reagents and related separation strategies in all areas of chemical synthesis.



**October 10 – 13, 2010**  
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# Green Solvents Conference

[www.dechema.de/gsfs2010](http://www.dechema.de/gsfs2010)



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The submission of abstracts proceeds via file upload at the website effective until **April 30, 2010**.

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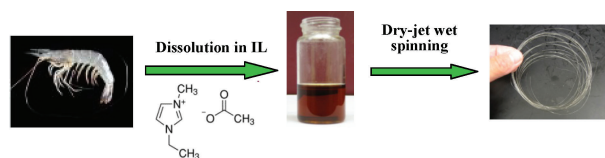
## COMMUNICATIONS

968

**Dissolution or extraction of crustacean shells using ionic liquids to obtain high molecular weight purified chitin and direct production of chitin films and fibers**

Ying Qin, Xingmei Lu, Ning Sun and Robin D. Rogers\*

1-Ethyl-3-methyl-imidazolium acetate can completely dissolve raw crustacean shells, leading to recovery of a high purity, high molecular weight chitin powder and to fibers and films which can be spun directly from the extract solution.



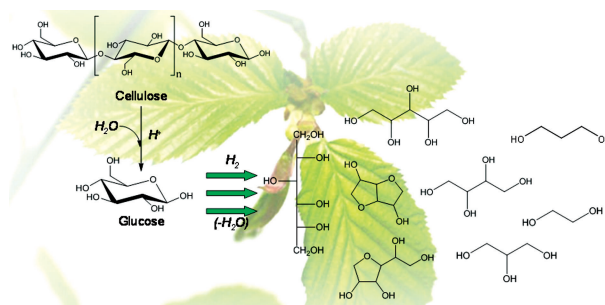
## PAPERS

972

**Hydrogenolysis of cellulose combining mineral acids and hydrogenation catalysts**

Regina Palkovits,\* Kameh Tajvidi, Joanna Procelewska, Roberto Rinaldi and Agnieszka Ruppert

Cellulose and even wood can be converted efficiently combining mineral acids and hydrogenation catalysts. Therein, supported metal catalysts significantly facilitate the conversion of cellulose and determine the product distribution.

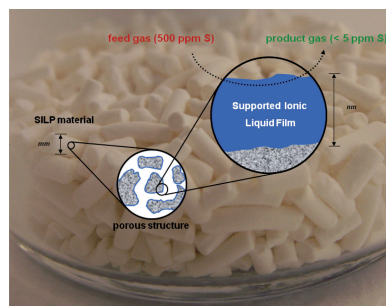


979

**Continuous gas-phase desulfurisation using supported ionic liquid phase (SILP) materials**

Florian Kohler, Daniel Roth, Esther Kuhlmann, Peter Wasserscheid\* and Marco Haumann\*

Supported ionic liquid phase (SILP) materials have been developed for a continuous gas cleaning process. A model gas stream consisting of 500 ppm<sub>w</sub> n-butyl mercaptan in n-heptane vapour to levels of below 5 ppm<sub>w</sub> mercaptan. Up to 130 h time-on-stream stability and several loading/unloading cycles were realised.



985

**Fast copper-, ligand- and solvent-free Sonogashira coupling in a ball mill**

Rico Thorwirth, Achim Stolle\* and Bernd Ondruschka

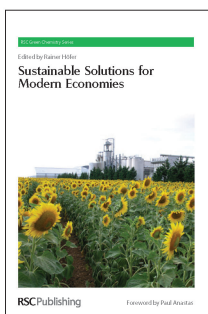
A protocol for a Pd-catalyzed Sonogashira reaction is presented employing neither copper, solvents nor ligands. Applying DABCO as the base yielded the products after 20 min of ball milling with selectivities >95%.



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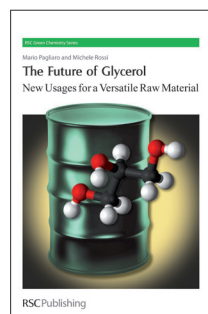
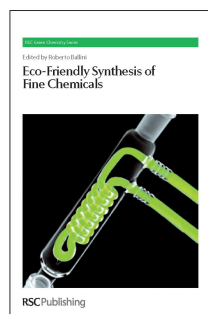
kinds of solvents including alternative greener solvent choices.

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## The Future of Glycerol

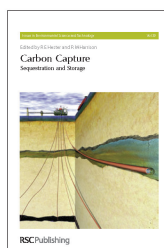
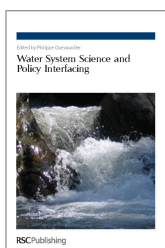
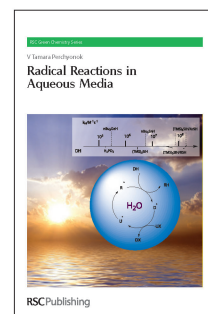
This book depicts how practical limitations posed by glycerol chemistry are solved based on the understanding of the fundamental chemistry of glycerol.

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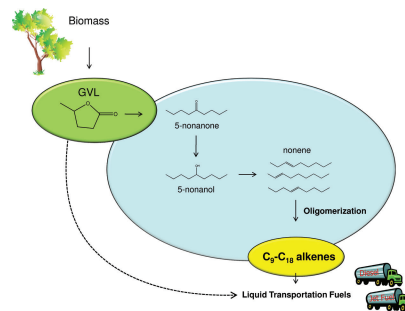
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992

### Production of liquid hydrocarbon transportation fuels by oligomerization of biomass-derived C<sub>9</sub> alkenes

David Martin Alonso, Jesse Q. Bond,  
Juan Carlos Serrano-Ruiz and James A. Dumesic\*

$\gamma$ -valerolactone, a biomass derived platform molecule, can be transformed in a cascade process to liquid transportation fuels *via* oligomerization of non-terminal C<sub>9</sub> alkenes.

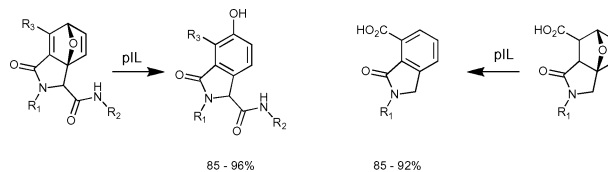


1000

### A facile, protic ionic liquid route to *N*-substituted 5-hydroxy-4-methyl-3-oxoisindoline-1-carboxamides and *N*-substituted 3-oxoisindoline-4-carboxylic acids

Christopher P Gordon, Nolene Byrne and  
Adam McCluskey\*

Use of protic ionic liquids effects rapid and quantitative access to decorated oxoisindoles.

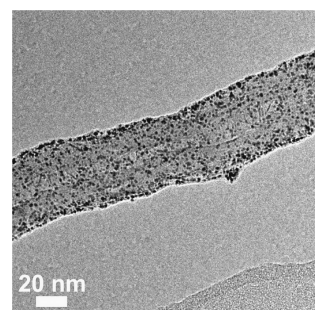


1007

### The solvent-free selective hydrogenation of nitrobenzene to aniline: an unexpected catalytic activity of ultrafine Pt nanoparticles deposited on carbon nanotubes

Zhenyu Sun, Yanfei Zhao, Yun Xie, Ranting Tao,  
Hongye Zhang, Changliang Huang and Zhimin Liu\*

Carbon nanotube supported Pt nanocatalysts have an unprecedented activity and selectivity for the hydrogenation of nitrobenzene to aniline in the absence of solvent under mild conditions.

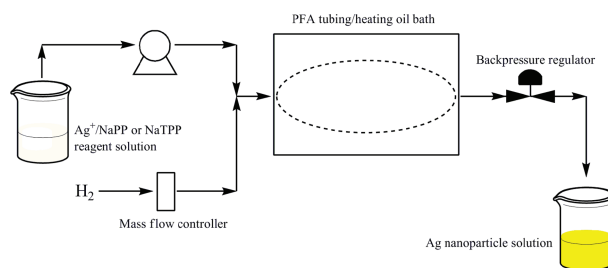


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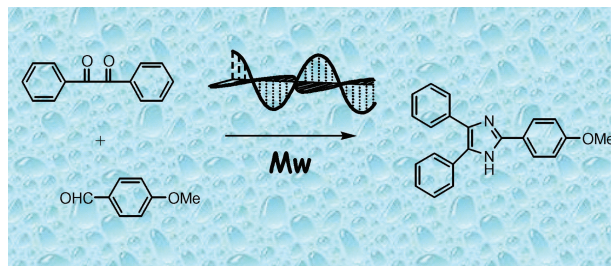
### Continuous flow synthesis of small silver nanoparticles involving hydrogen as the reducing agent

Karel J. Hartlieb, Martin Saunders, Roshan J. J. Jachuck and  
Colin L. Raston\*

Very small, 2–3 nm diameter, silver nanoparticles have been produced using a process intensification and green chemistry methodology by means of a narrow-channel reactor with hydrogen gas as a reductant in the presence of polyphosphates.



1018

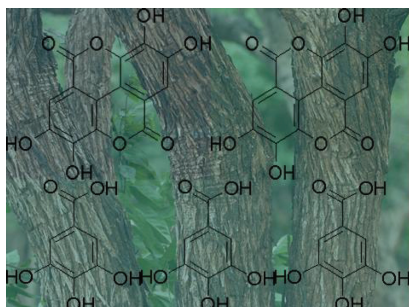


### Synthesis of 2,4,5-triarylimidazoles in aqueous solution, under microwave irradiation

Edouard Chauveau, Catherine Marestin,\* Frédéric Schiets and Régis Mercier

A series of 2,4,5-triarylimidazoles was synthesized from a new, highly efficient and green method. The reaction is performed in water, without the presence of any catalyst, and under microwave irradiation.

1023

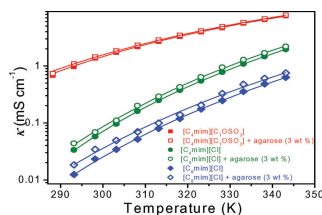
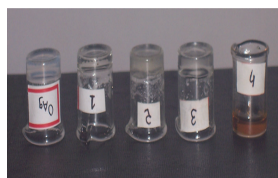


### Distillable ionic liquid extraction of tannins from plant materials

Shahana A. Chowdhury, R. Vijayaraghavan and D. R. MacFarlane\*

Distillable ionic liquids, such as DIMCARB, are used to extract valuable hydrolysable tannin materials such as ellagic acid in higher yields than conventional extraction methods.

1029

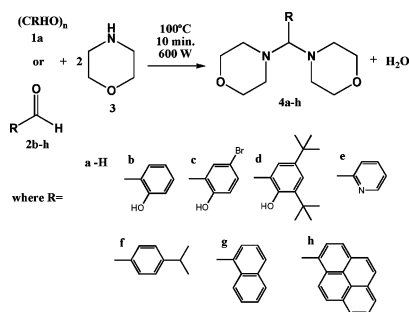


### Dissolution, regeneration and ion-gel formation of agarose in room-temperature ionic liquids

Tejwant Singh, Tushar J. Trivedi and Arvind Kumar\*

The solubility of agarose has been tested in ionic liquids, and agarose-based highly conducting soft ion-gels having small thermal hysteresis have been prepared and characterized.

1036



### Molecular design and QSAR study of low acute toxicity biocides with 4,4'-dimorpholyl-methane core obtained by microwave-assisted synthesis


Raúl Hernández-Altamirano, Violeta Y. Mena-Cervantes, Sandra Perez-Miranda, Francisco J. Fernández, Cesar Andres Flores-Sandoval, Victor Barba, Hiram I. Beltrán\* and Luis S. Zamudio-Rivera\*

Solventless microwave assisted synthesis led to eight 4,4'-dimorpholyl-methanes. They have shown modulated acute toxicity and high biocidal activity rationalized through structure-activity correlations.



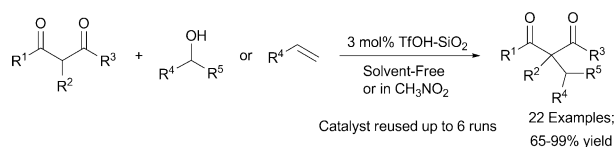
## PAPERS

1049

 **Triflic acid adsorbed on silica gel as an efficient and recyclable catalyst for the addition of  $\beta$ -dicarbonyl compounds to alcohols and alkenes**

Pei Nian Liu,\* Fei Xia, Qing Wei Wang, Yu Jie Ren\* and Jun Qin Chen

The readily available triflic acid supported on silica gel was applied as an efficient and recyclable catalyst for the heterogeneous addition of  $\beta$ -dicarbonyl compounds to alcohols and alkenes, which afforded moderate to excellent yields under solvent-free conditions or in nitromethane.

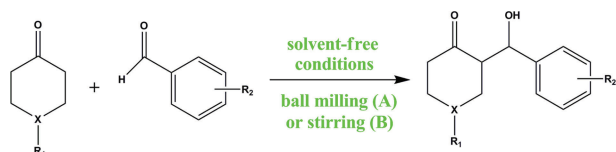


1056

**Application of molecular topology for the prediction of the reaction times and yields under solvent-free conditions**

Jorge Gálvez,\* María Gálvez-Llompart and Ramón García-Domenech

Molecular topology has been used to achieve mathematical models capable of predicting the yields and the reaction times of different reactions under solvent-free conditions. The results have implications for the efficacy of the methodology employed in helping experimentalists to achieve greener and more sustainable reactants and products.

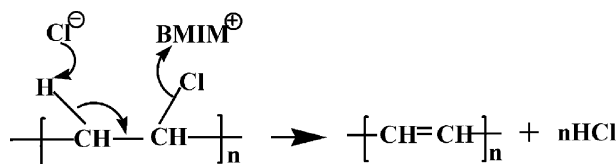


1062

**A highly efficient approach for dehydrochlorinating polyvinyl chloride: catalysis by 1-butyl-3-methylimidazolium chloride**

Tao Zhao, Qian Zhou,\* Xiao-Li He, Sha-Di Wei, Li Wang, Johannes M. N. van Kasteren and Yu-Zhong Wang\*

An easily accessible ionic liquid, 1-butyl-3-methylimidazolium chloride ([Bmim]Cl), is demonstrated to be a novel, green and efficient catalyst for PVC dehydrochlorination, which holds great promise for application in other dehalogenation processes.

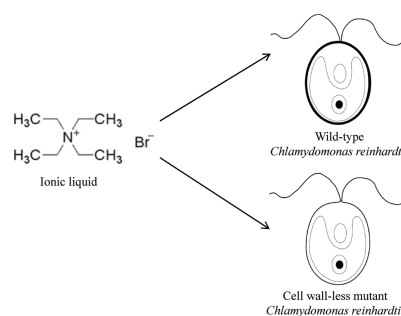


1066

**The role of the cell wall in the toxicity of ionic liquids to the alga *Chlamydomonas reinhardtii***

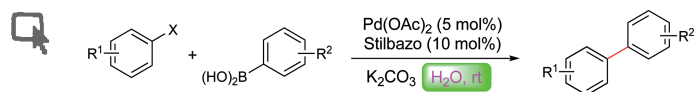
David W. Sena,\* Konrad J. Kulacki, Dominic T. Chaloner and Gary A. Lambert

We investigated the toxicity of five ionic liquids to wild and cell wall-less mutant strains of the freshwater phytoplankton *Chlamydomonas reinhardtii* to elucidate the role of the cell wall in mitigating ionic liquid toxicity.



## PAPERS

1072

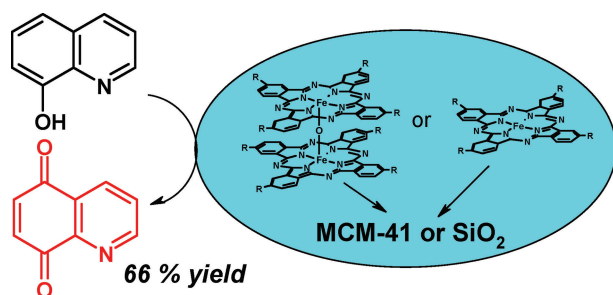


### Room-temperature highly efficient Suzuki–Miyaura reactions in water in the presence of Stilbazolone

Yi-Yuan Peng,\* Jinbiao Liu, Xiaoli Lei and Zenlan Yin

Stilbazolone was found to be a promoter for the synthesis of biaryl compounds by a ligand-free Suzuki–Miyaura reaction in water at room temperature.

1076

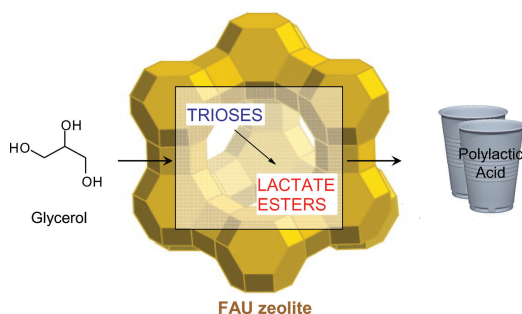


### Clean catalytic oxidation of 8-hydroxyquinoline to quinoline-5,8-dione with <sup>t</sup>BuOOH in the presence of covalently bound FePcS–SiO<sub>2</sub> catalysts

Olga V. Zalomaeva, Alexander B. Sorokin\* and Oxana A. Kholdeeva

The heterogeneous oxidation of 8-hydroxyquinoline afforded the corresponding quinone, a structural fragment of drugs, in 66% yield.

1083

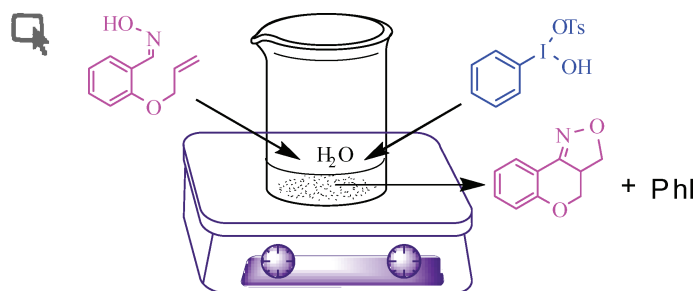


### Zeolite-catalysed conversion of C<sub>3</sub> sugars to alkyl lactates

Paolo P. Pescarmona, Kris P. F. Janssen, Chloë Delaet, Christophe Stroobants, Kristof Houthoofd, An Philippaerts, Chantal De Jonghe, Johan S. Paul, Pierre A. Jacobs\* and Bert F. Sels\*

*En route* to lactates from glycerol: USY zeolite efficiently catalyses the conversion of C<sub>3</sub> sugars into alkyl lactates.

1090




### 'On-water' synthesis of chromeno-isoxazoles mediated by [hydroxy(tosyloxy)iodo]benzene (HTIB)

Mustafa J. Raihan, Veerababurao Kavala, Chun-Wei Kuo, B. Rama Raju and Ching-Fa Yao\*

An efficient and handy method for the synthesis of chromeno-isoxazole/isoxazolines under 'on-water' conditions is described, together with a thorough mechanistic study.

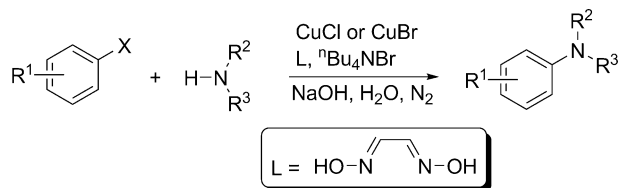
## PAPERS

1097

 **Efficient copper-catalyzed N-arylations of nitrogen-containing heterocycles and aliphatic amines in water**

Xufeng Li, Daoshan Yang, Yuyang Jiang and Hua Fu\*

A simple and efficient copper-catalyzed method has been developed for N-arylations of nitrogen-containing heterocycles and aliphatic amines in water. The protocol uses (1*E*,2*E*)-oxalaldehyde dioxime (OADO) as the ligand, and water as the solvent, and shows good tolerance towards various functional groups.

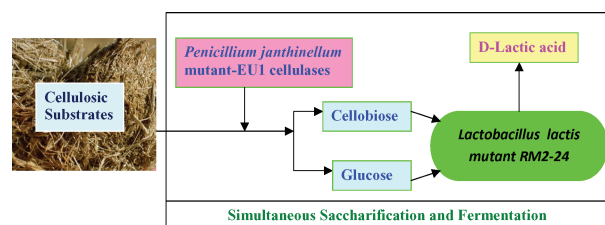


1106

 **D-(-)-Lactic acid production from cellobiose and cellulose by *Lactobacillus lactis* mutant RM2-24**


Mamta Singhvi, Dipti Joshi, Mukund Adsul, Anjani Varma and Digambar Gokhale\*

Simultaneous saccharification and fermentation (SSF) of cellulosic substrates to D-lactic acid using EU1 cellulases and *Lactobacillus lactis* mutant RM2-24. The SSF was carried out in screw-cap flasks at 42 °C with shaking at 150 rpm.



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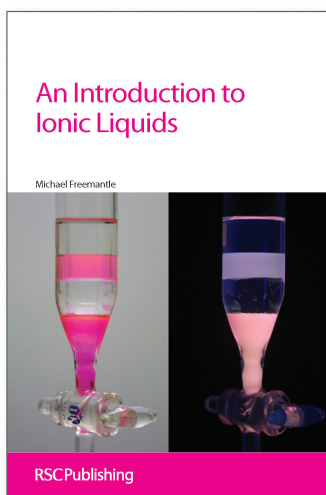
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\* Indicates the author for correspondence: see article for details.

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## AUTHOR INDEX

- Adsul, Mukund, 1106  
 Agbossou-Niedercorn, Francine, 961  
 Alonso, David Martin, 992  
 Barba, Victor, 1036  
 Beckers, Jurriaan, 939  
 Beltrán, Hiram I., 1036  
 Bond, Jesse Q., 992  
 Braga, Antonio Luiz, 957  
 Byrne, Nolene, 1000  
 Chaloner, Dominic T., 1066  
 Chan, Albert S. C., 949  
 Chauveau, Edouard, 1018  
 Chen, Jun Qin, 1049  
 Chowdhury, Shahana A., 1023  
 De Jonghe, Chantal, 1083  
 Delaet, Chloë, 1083  
 Deprez, Benoit, 961  
 Deprez-Poulain, Rebecca, 961  
 Dumesic, James A., 992  
 Feng, Li-Chun, 949  
 Feng, Wei, 953  
 Fernández, Francisco J., 1036  
 Flores-Sandoval, Cesar Andres, 1036  
 Fu, Hua, 1097  
 Gálvez, Jorge, 1056  
 Gálvez-Llompart, María, 1056  
 García-Domenech, Ramón, 1056  
 Godoi, Marcelo, 957  
 Gokhale, Digambar, 1106  
 Gordon, Christopher P, 1000  
 Gul, Kashif, 957  
 Hartlieb, Karel J., 1012  
 Haumann, Marco, 979  
 He, Xiao-Li, 1062  
 Hernández-Altamirano, Raúl, 1036  
 Herrero, Santiago, 965  
 Houthoofd, Kristof, 1083  
 Huang, Changliang, 1007  
 Jachuck, Roshan J. J., 1012  
 Jacobs, Pierre A., 1083  
 Janssen, Kris P. F., 1083  
 Jiang, Yuyang, 1097  
 Jida, Mouhamad, 961  
 Jiménez-Aparicio, Reyes, 965  
 Joshi, Dipti, 1106  
 Kavala, Veerababurao, 1090  
 Kee, Choon Wee, 953  
 Kholdeeva, Oxana A., 1076  
 Kohler, Florian, 979  
 Kuhlmann, Esther, 979  
 Kulacki, Konrad J., 1066  
 Kumar, Arvind, 1029  
 Kuo, Chun-Wei, 1090  
 Laconde, Guillaume, 961  
 Lam, Kim-Lung, 949  
 Lamberti, Gary A., 1066  
 Lei, Xiaoli, 1072  
 Leow, Dasheng, 953  
 Li, Xufeng, 1097  
 Liu, Hongjun, 953  
 Liu, Jinbiao, 1072  
 Liu, Pei Nian, 1049  
 Liu, Zhimin, 1007  
 Lu, Xingmei, 968  
 MacFarlane, D. R., 1023  
 Malaquin, Sandra, 961  
 Marestin, Catherine, 1018  
 McCluskey, Adam, 1000  
 Mena-Cervantes, Violeta Y., 1036  
 Mercier, Régis, 1018  
 Narayanaperumal, Senthil, 957  
 Ondruschka, Bernd, 985  
 Palkovits, Regina, 972  
 Pan, Yuanhang, 953  
 Paul, Johan S., 1083  
 Peng, Yi-Yuan, 1072  
 Perez-Miranda, Sandra, 1036  
 Perles, Josefina, 965  
 Pescarmona, Paolo P., 1083  
 Philippaerts, An, 1083  
 Priego, José L., 965  
 Procelewska, Joanna, 972  
 Qin, Ying, 968  
 Raihan, Mustafa J., 1090  
 Raju, B. Rama, 1090  
 Raston, Colin L., 1012  
 Ren, Yu Jie, 1049  
 Rinaldi, Roberto, 972  
 Rodrigues, Oscar Endrigo Dorneles, 957  
 Rogers, Robin D., 968  
 Roth, Daniel, 979  
 Rothenberg, Gadi, 939  
 Roussel, Pascal, 961  
 Ruppert, Agnieszka, 972  
 Saunders, Martin, 1012  
 Schiets, Frédéric, 1018  
 Sels, Bert F., 1083  
 Sena, David W., 1066  
 Serrano-Ruiz, Juan Carlos, 992  
 Singh, Devender, 957  
 Singh, Tejwant, 1029  
 Singhvi, Mamta, 1106  
 Sorokin, Alexander B., 1076  
 Stolle, Achim, 985  
 Stroobants, Christophe, 1083  
 Sun, Ning, 968  
 Sun, Ya-Wei, 949  
 Sun, Zhenyu, 1007  
 Tajvidi, Kameh, 972  
 Tan, Choon-Hong, 953  
 Tang, Wei-Jun, 949  
 Tao, Ranting, 1007  
 Thorwirth, Rico, 985  
 Trivedi, Tushar J., 1029  
 Urbanos, Francisco A., 965  
 van Kasteren, Johannes M. N., 1062  
 Varma, Anjani, 1106  
 Vijayaraghavan, R., 1023  
 Wang, Li, 1062  
 Wang, Qing Wei, 1049  
 Wang, Yu-Zhong, 1062  
 Wasserscheid, Peter, 979  
 Wei, Sha-Di, 1062  
 Xia, Fei, 1049  
 Xie, Yun, 1007  
 Xu, Li-Jin, 949  
 Yang, Daoshan, 1097  
 Yao, Ching-Fa, 1090  
 Yin, Zenlan, 1072  
 Zalomaeva, Olga V., 1076  
 Zamudio-Rivera, Luis S., 1036  
 Zhang, Hongye, 1007  
 Zhao, Tao, 1062  
 Zhao, Yanfei, 1007  
 Zhao, Yujun, 953  
 Zhou, Qian, 1062  
 Zhou, Zhongyuan, 949



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