

Green Chemistry

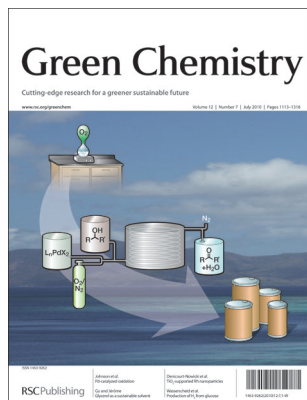
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IN THIS ISSUE

ISSN 1463-9262 CODEN GRCHFJ 12(7) 1113–1316 (2010)



Cover

See Johnson, Yates and Stahl *et al.*, pp. 1180–1186. A continuous-flow tube reactor developed by the Stahl group (Wisconsin) and Eli Lilly enables safe and scalable aerobic oxidation reactions to be implemented in pharmaceutical synthesis.

Image reproduced by permission of Shannon Stahl and Betsy True from *Green Chemistry*, 2010, **12**, 1180.



Inside cover

See Shchipunov *et al.*, pp. 1187–1195. Jellification of chitosan solutions through regulated self-organization with oppositely charged polysaccharides.

Image reproduced by permission of Yury Shchipunov from *Green Chemistry*, 2010, **12**, 1187.

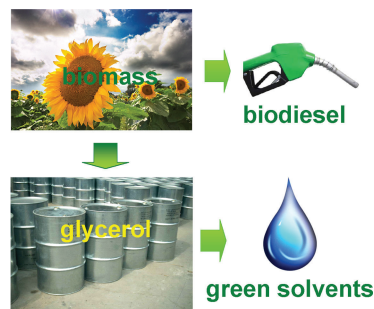
CRITICAL REVIEWS

1127

Glycerol as a sustainable solvent for green chemistry

Yanlong Gu* and François Jérôme*

This review summarizes the background, aspects and beneficial effects of using glycerol as a green solvent for organic syntheses, catalysis, biocatalysis and materials chemistry.

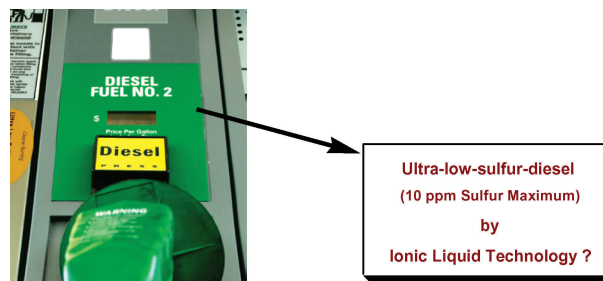


1139

Deep desulfurization of diesel fuel using ionic liquids: current status and future challenges

Prashant S. Kulkarni* and Carlos A. M. Afonso

Alternate strategies for deep desulfurization of diesel fuel are necessary to meet new legislation standards. The present review focuses on the state-of-the-art desulfurization technologies using ionic liquids with comments on future challenges and outlook.



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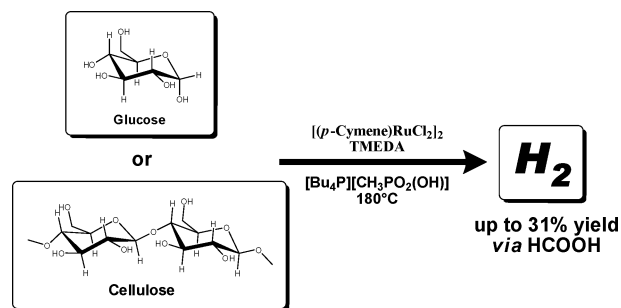
COMMUNICATIONS

1150

Catalytic production of hydrogen from glucose and other carbohydrates under exceptionally mild reaction conditions

N. Taccardi, D. Assenbaum, M. E. M. Berger, A. Bösmann, F. Enzenberger, R. Wölfel, S. Neuendorf, Volker Goeke, N. Schödel, H. -J. Maass, H. Kistenmacher and P. Wasserscheid*

Ionic liquids dissolve biogenic feedstock like cellulose, which can be used to produce hydrogen. The biomass thermally decomposes to formic acid that is catalytically transformed to hydrogen and CO₂.

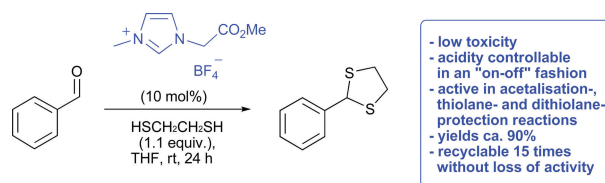


1157

Highly recyclable, imidazolium derived ionic liquids of low antimicrobial and antifungal toxicity: A new strategy for acid catalysis

Lauren Myles, Rohitkumar Gore, Marcel Špulák, Nicholas Gathergood* and Stephen J. Cannon*

Imidazolium derived catalysts have been developed which are aprotic and of low antimicrobial and antifungal toxicity, yet which can act as efficient Brønsted acidic catalysts in the presence of protic additives. These can be utilised at low loadings and can be recycled repeatedly.

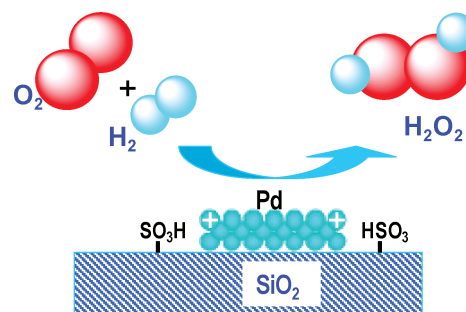


1163

Direct synthesis of hydrogen peroxide on palladium catalyst supported on sulfonic acid-functionalized silica

Gema Blanco-Brieva, M. Pilar de Frutos Escrig, Jose M. Campos-Martin and Jose L. G. Fierro*

Direct synthesis of H₂O₂ has been successfully achieved at high-concentration and high selectivity at realistic reaction temperatures (313 K) on Pd-loaded sulfonic acid-functionalized silica using non-acidic solutions.

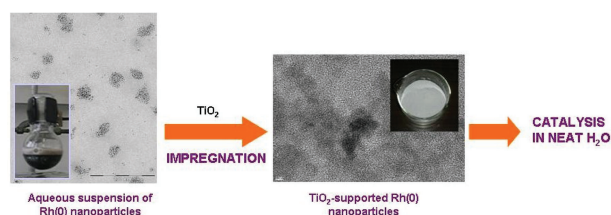


1167

TiO₂-supported Rh nanoparticles: From green catalyst preparation to application in arene hydrogenation in neat water

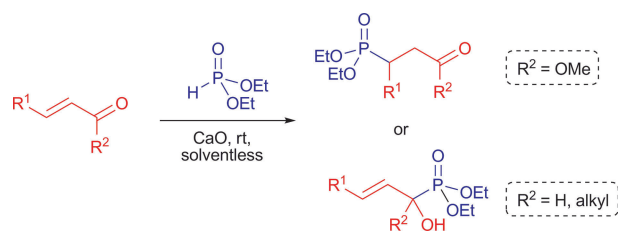
Claudie Hubert, Audrey Denicourt-Nowicki,* Patricia Beaunier and Alain Roucoux*

TiO₂-supported Rh(0) nanoparticles were synthesized by an easy method under mild conditions in neat water. The characterized materials with sizes between 3–4 nm proved to be highly active catalysts for arene hydrogenation in water with TOFs up to 33 333 h⁻¹.



COMMUNICATIONS

1171



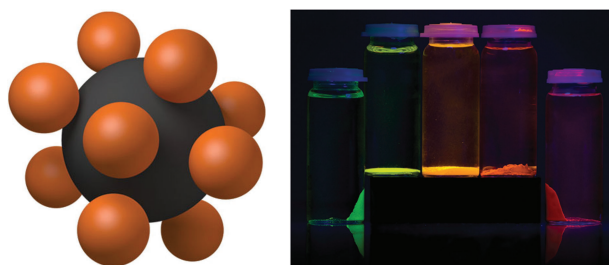
A green procedure for the regio- and chemoselective hydrophosphonylation of unsaturated systems using CaO under solventless conditions

Elisa Martínez-Castro, Óscar López, Inés Maya, José G. Fernández-Bolaños* and Marino Petrini*

Regioselectivity in the CaO-promoted nucleophilic addition of diethyl phosphite to α,β -unsaturated systems follows a definite trend depending on the nature of the substituent on carbonyl.

PAPERS

1175

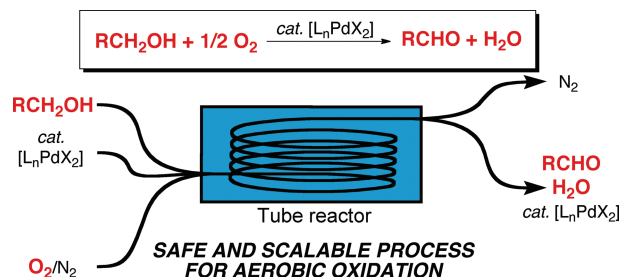


Nanosized luminescent superparamagnetic hybrids

Cameron W. Evans, Colin L. Raston and K. Swaminathan Iyer*

We demonstrate an aqueous self-assembly route to fabricate nanohybrids combining cadmium telluride quantum dots and magnetite nanoparticles. The resulting nanocomposite displays both photoluminescence and superparamagnetic behaviour.

1180

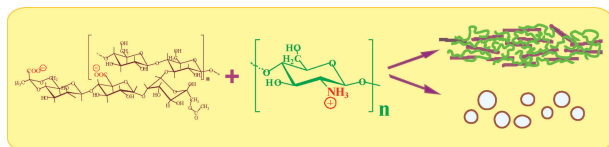


Development of safe and scalable continuous-flow methods for palladium-catalyzed aerobic oxidation reactions

Xuan Ye, Martin D. Johnson,* Tianning Diao, Matthew H. Yates* and Shannon S. Stahl*

A continuous flow reactor has been developed for the application of Pd-catalyzed aerobic oxidation reactions in process-scale pharmaceutical synthesis.

1187



Hydrogels formed through regulated self-organization of gradually charging chitosan in solution of xanthan

Yury Shchipunov,* Sergei Sarin, Il Kim and Chang-Sik Ha

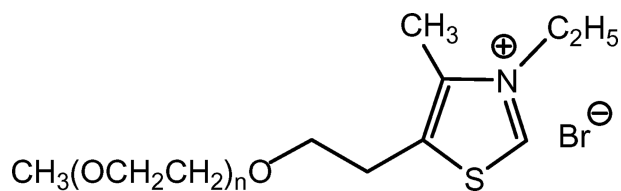
Monolithic hydrogels or capsules are formed through the electrostatic interactions of anionic polysaccharide with chitosan that are gradually charged in the course of a progressive pH decrease by chemical acidulant.

1196

Polyether-substituted thiazolium ionic liquid catalysts – a thermoregulated phase-separable catalysis system for the Stetter reaction

Fengli Yu, Ruili Zhang, Congxia Xie* and Shitao Yu

A novel thermoregulated phase-separable catalysis (TPSC) system of a polyether-substituted thiazolium ionic liquid catalyst has been developed for resolving the problems associated with separation and reuse of Stetter catalysts.

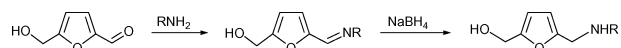


1201

Production of bio-based HMF derivatives by reductive amination

Ana Cukalovic and Christian V. Stevens*

A simple procedure for the conversion of HMF to (5-alkyl- and 5-arylamino-methyl-furan-2-yl)methanol has been developed. Reactions were conducted without the use of a catalyst and under very mild conditions.

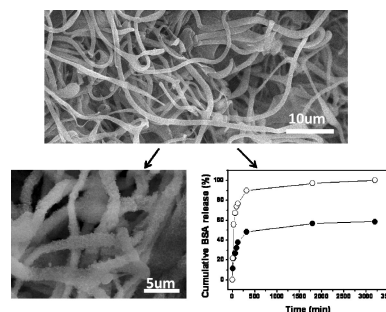


1207

Green synthesis of chitosan-based nanofibers and their applications

Lei Qian and Haifei Zhang*

Chitosan-based nanofibers are produced by a freeze-drying method and then used for the preparation of hybrid nanofibers, the adsorption of Cu^{2+} from aqueous solutions, and the controlled release of Rhodamine B and bovine serum albumin at 50 wt % loading (●) and 100 wt % loading (○).

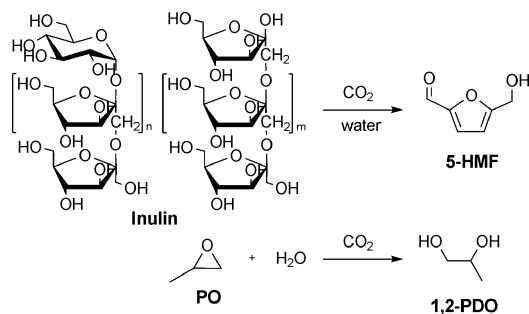


1215

Effect of CO_2 on conversion of inulin to 5-hydroxymethylfurfural and propylene oxide to 1,2-propanediol in water

Suxiang Wu, Honglei Fan, Ye Xie, Yan Cheng, Qian Wang, Zhaofu Zhang and Buxing Han*

CO_2 can enhance the conversion of inulin to 5-hydroxymethylfurfural (5-HMF) and of propylene oxide (PO) to 1,2-propanediol (1,2-PDO) in water.



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.



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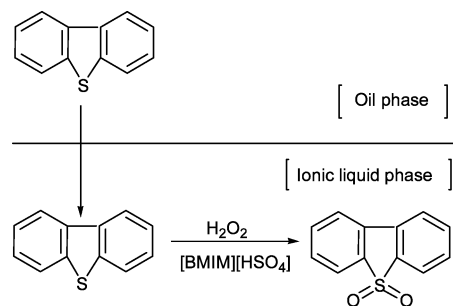
PAPERS

1220

Extraction and oxidative desulfurization of diesel fuel catalyzed by a Brønsted acidic ionic liquid at room temperature

Hongshuai Gao, Chen Guo,* Jianmin Xing, Junmei Zhao and Huizhou Liu*

The Brønsted acidic IL [BMIM][HSO₄] can be used as extractant and catalyst for desulfurization of diesel at room temperature. This process is simple, mild, and may be a complementary technology for the HDS process.

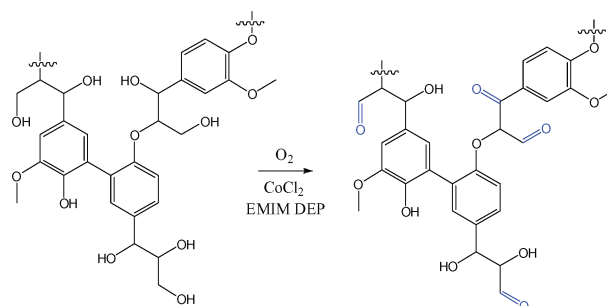


1225

Transition metal catalyzed oxidation of Alcell lignin, soda lignin, and lignin model compounds in ionic liquids

Joseph Zakzeski, Anna L. Jongerius and Bert M. Weckhuysen*

Lignin was dissolved in 1-ethyl-3-methylimidazolium diethylphosphate and oxidized using cobalt catalysts and molecular oxygen under mild conditions. Catalytic activity was elucidated using ATR-IR spectroscopy and by examining the reactivity of lignin model compounds.

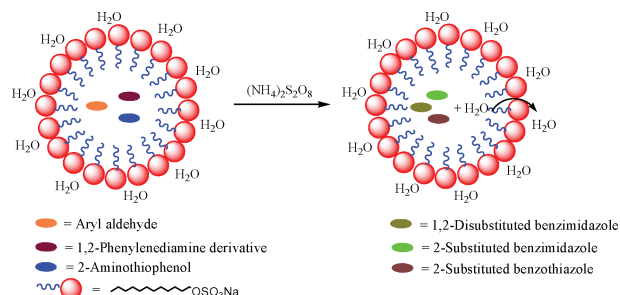


1237

Synthesis of 1,2-disubstituted benzimidazoles, 2-substituted benzimidazoles and 2-substituted benzothiazoles in SDS micelles

Kiumars Bahrami,* Mohammad M. Khodaei* and Akbar Nejati

A practical and convenient synthetic method has been developed for the facile synthesis of 1,2-disubstituted benzimidazoles, 2-substituted benzimidazoles and 2-substituted benzothiazoles.

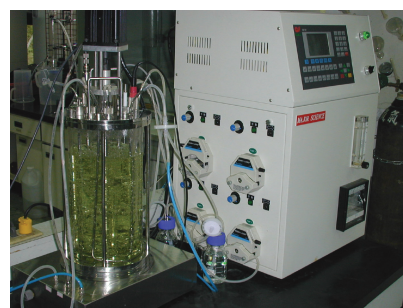


1242

Tandem production of levan and ethanol by microbial fermentation

Ing-Lung Shih,* Li-Dar Chen, Tsaor-Chin Wang, Jane-Yii Wu and Kuo-Shen Liaw

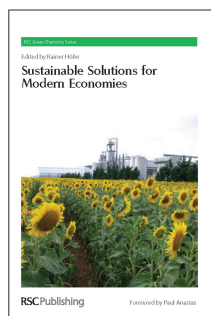
This process is green and eco-friendly in that sucrose substrate was fully utilized, two invaluable biomaterials (levan and ethanol) were produced and the amount of organic solvent for levan recovery was dramatically reduced.



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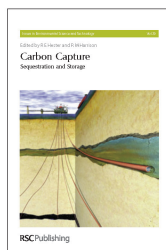
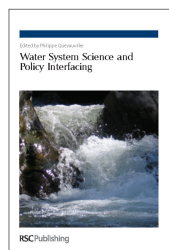
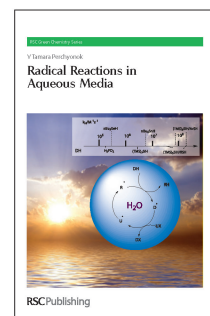
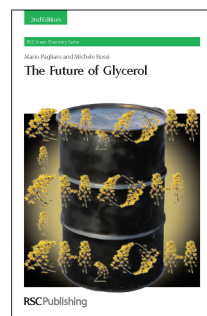
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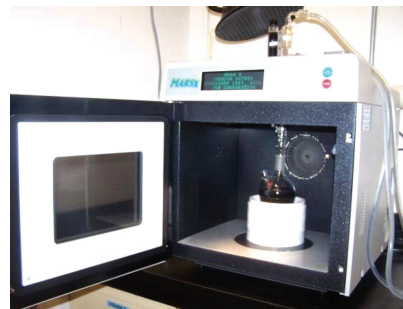
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PAPERS

1248

Green synthesis of tunable $\text{Cu}(\text{In}_{1-x}\text{Ga}_x)\text{Se}_2$ nanoparticles using non-organic solvents

Layla Al Juhaiman, Ludmila Scoles, David Kingston, Bussaraporn Patarachao, Dashan Wang and Farid Bensebaa*

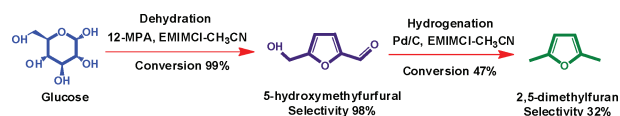
Microwave synthesis setup used for aqueous-based synthesis of $\text{Cu}(\text{In}_x\text{Ga}_{1-x})\text{Se}_2$ nanoparticles.

1253

A two-step approach for the catalytic conversion of glucose to 2,5-dimethylfuran in ionic liquids

Mandan Chidambaram and Alexis T. Bell*

12-Molybdophosphoric acid (12-MPA) enabled quantitative conversion of glucose into 5-hydroxymethylfurfural (HMF) in an ionic liquid–acetonitrile mixture and 2,5-dimethylfuran, a promising fuel additive, could be produced by hydrogenation of HMF in the same solvent system using carbon-supported Pd.

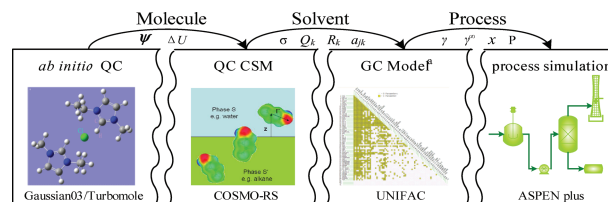


1263

Multi-scale simulation of the 1,3-butadiene extraction separation process with an ionic liquid additive

Xiao Tian, Xiangping Zhang,* Lu Wei, Shaojuan Zeng, Lei Huang and Suojiang Zhang*

A multi-scale simulation method is proposed to enable screening of ionic liquids as entrainers in extractive distillation, and its feasibility was validated by the 1,3-butadiene production process with acetonitrile.

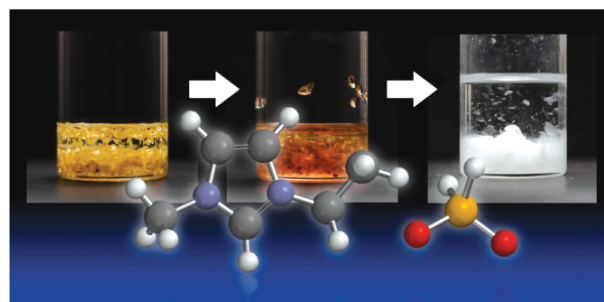


1274

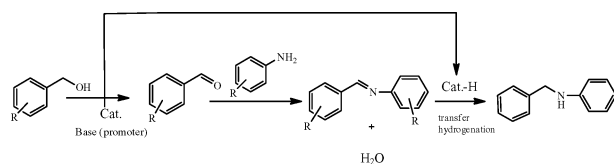
Extraction of polysaccharides from bran with phosphonate or phosphinate-derived ionic liquids under short mixing time and low temperature

Mitsuru Abe, Yukinobu Fukaya and Hiroyuki Ohno

The extraction of polysaccharides, mainly cellulose, from bran was carried out under mild conditions and a short treatment time with phosphonate or phosphinate-derived ionic liquids.



1281

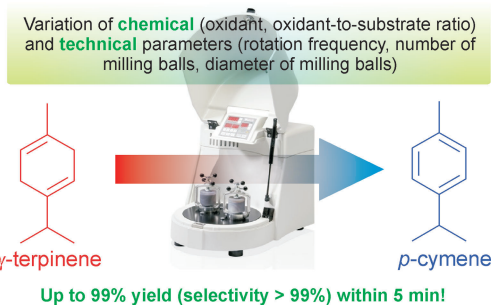


Highly active and selective supported iron oxide nanoparticles in microwave-assisted *N*-alkylations of amines with alcohols

Camino Gonzalez-Arellano, Kenta Yoshida, Rafael Luque* and Pratibha L. Gai

Ironing your chemistry: the efficient microwave-assisted *N*-alkylation of amines with benzyl alcohols can be performed using supported iron oxide nanoparticles on mesoporous materials *via* a hydrogen autotransfer process.

1288

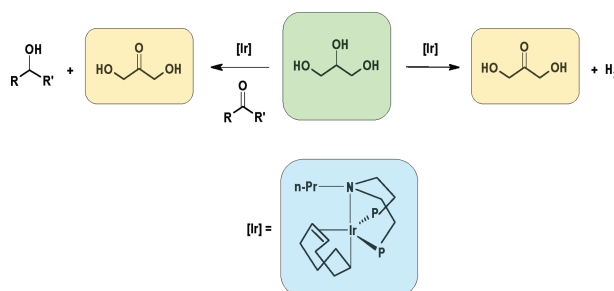


Solvent-free dehydrogenation of γ -terpinene in a ball mill: investigation of reaction parameters

Tony Szuppa, Achim Stolle,* Bernd Ondruschka and Wieland Hopfe

A solvent-free dehydrogenation reaction in a ball mill is used to assess chemical and technical variables for this reaction model. Experiments revealed that KMnO_4 can be replaced by less hazardous oxidants (NaIO_4 , Oxone[®]), retaining excellent selectivity.

1295

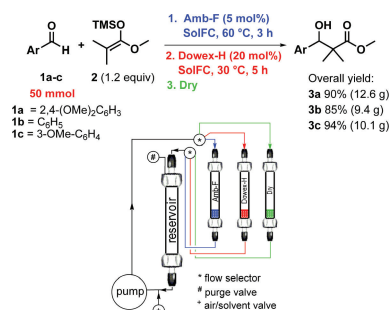


Dehydrogenation of glycerol to dihydroxyacetone catalyzed by iridium complexes with P–N ligands

Corrado Crotti, Jan Kašpar and Erica Farnetti*

Glycerol transfer dehydrogenation to dihydroxyacetone is catalyzed by $[\text{Ir}(\sigma,\eta^2\text{-C}_8\text{H}_{13})(\text{PNP})]$ ($\text{PNP} = \text{Pr}^n\text{-N}(\text{CH}_2\text{CH}_2\text{PPh}_2)_2$) without a basic cocatalyst; the occurrence of acceptor-less glycerol dehydrogenation is also demonstrated.

1301



An E-factor minimized protocol for the preparation of methyl β -hydroxy esters

Francesco Fringuelli, Daniela Lanari, Ferdinando Pizzo and Luigi Vaccaro*

An automated protocol for the Mukaiyama aldol reaction on a cyclic continuous-flow reactor allowed minimization of the waste production.

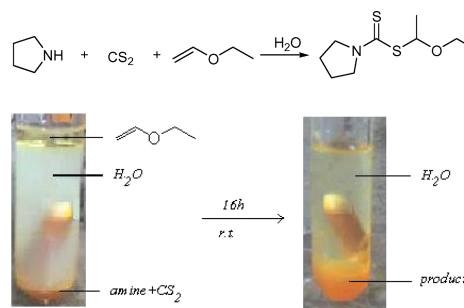
PAPERS

1306

Synthesis of dithiocarbamate by Markovnikov addition reaction in aqueous medium

Azim Ziyaei Halimehjani, Katayoun Marjani and Akram Ashouri

Highly efficient, one-pot and three component reactions of amines and carbon disulfide with alkyl vinyl ethers *via* Markovnikov addition reaction were carried out in water under a mild and green procedure with excellent yields and complete regioselectivity.

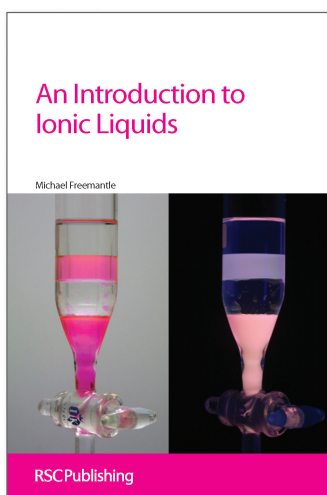


1311

Ruthenium-catalyzed estragole isomerization: high *trans*-selective formation of anethole

Beatriz Lastra-Barreira and Pascale Crochet*

Rapid and fully-selective access to *trans*-anethole is described.



An Introduction to Ionic Liquids

Michael Freemantle

This is the first single-author book on ionic liquids and the first introductory book on the topic. *An Introduction to Ionic Liquids* is written in a clear, concise and consistent way and provides a useful introduction to ionic liquids for those readers who are not familiar with the topic. It is also wide ranging, embracing every aspect of the chemistry and applications of ionic liquids. The book draws extensively on the primary scientific literature to provide numerous examples of research on ionic liquids. These examples will enable the reader to become familiar with the key developments in ionic liquids chemistry over recent years.

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