

Welcome to the July 2011 issue of the Flow Synthesis Online newsletter.

There's a lot of interesting new process work highlighted in this issue.

Contents

- Product announcements
 - convenient inert gas blanketing of reagents
 - new polymer coated stainless steel tube reactors
- Other announcements
- A selection of interesting publications

We appreciate all your feedback so please keep it coming.
Have a great summer !

You have received this email because you have in the past expressed an interest in Vapourtec Flow Chemistry products. If you'd prefer not to receive this newsletter any more, use the unsubscribe link at the end of the email.

Product Announcements

Inert Gas Manifold for Blanketing Air/Moisture sensitive reagents

There are many situations in which reagents need to be isolated from air or moisture. For short periods balloons may suffice but for prolonged use a low pressure supply of dry inert gas is required.

Vapourtec's new manifold takes care of all the details, regulation of pressure and distribution to up to 8 containers.



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Chemically Inert Coated Stainless Steel Reactors

Certain pressure and/or temperature conditions can exceed the limits of PFA or PTFE reactor tubing, yet the alternative (stainless steel) is not resistant to strong acids. Even when special surface treatments or proprietary grades of material are used, corrosive attack is only slowed, not prevented. And in some cases metallic contact of any kind interferes with the reaction outcome.

But now a new polymer coated stainless steel coil is available, which offers complete chemical resistance and zero metallic contact with reagents, yet offers strength and thermal conductivity not achievable with PTFE or PFA tubing.



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Announcements

Vapourtec are pleased to announce the appointment of AST Science Corporation as exclusive Vapourtec distributor in Taiwan.

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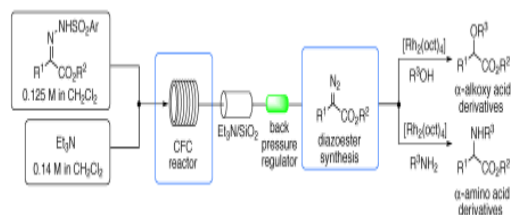
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Publications

Rapid Access to α -Alkoxy and α -Amino Acid Derivatives through Safe Continuous-Flow Generation of Diazoesters

Hannah E. Bartrum¹,
David C. Blakemore²,
Christopher J. Moody¹,
Christopher J. Hayes¹



¹School of Chemistry, Univ. of Nottingham, UK

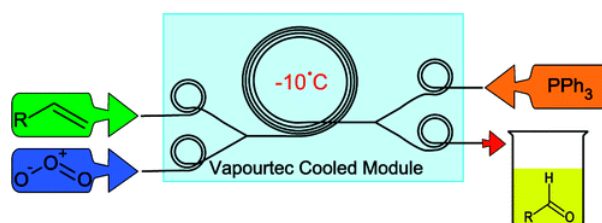
²Pfizer Global Research and Development, Sandwich, UK.

A highly efficient continuous-flow process has been developed for the synthesis of diazoesters from arylsulfonylhydrazones by means of in-flow Bamford–Stevens reactions. Furthermore, a range of α -alkoxy and α -amino acid derivatives have been prepared in excellent yield through rhodium(II)-mediated OH and NH insertions, without the need to isolate or handle the potentially hazardous diazo species

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Ozonolysis in Flow Using Capillary Reactors

M. D. Roydhouse¹,
A. Ghaini²,
A. Constantinou²,
A. Cantu-Perez²,
W. B. Motherwell¹,
A. Gavriilidis²



¹ Department of Chemistry, University College London, UK

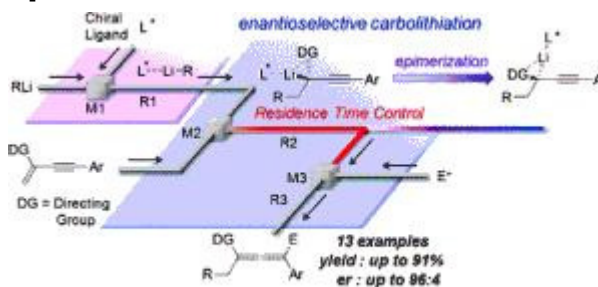
² Department of Chemical Engineering, University College London, UK

Reactions of *n*-decene with ozone and subsequent quenching of the formed ozonides were carried out under flow conditions using the standard Vapourtec flow system equipped with a cooled flow cell. The reactions were performed continuously and in the annular flow regime within the circular cross-section channels. The cooling system provided a safe and efficient control of the highly exothermic reaction system. The configuration of the system allowed the production of chemically significant amounts (1.8 g h⁻¹ at 1.3 ozone equivalents), with minimal amounts of ozonides present at any time.

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Asymmetric Carbolithiation of Conjugated Enynes: A Flow Microreactor Enables the Use of Configurationally Unstable Intermediates Before They Epimerize

Yutaka Tomida, Aiichiro Nagaki,
Jun-ichi Yoshida
Dept of Synthetic Organic
Chemistry, Kyoto University, Japan



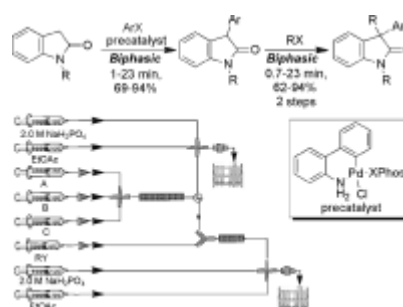
It was found that a flow microreactor system enables the generation of a configurationally unstable chiral organolithium intermediate and allows for its use in a reaction with an electrophile before it epimerizes. Based on this method, the enantioselective carbolithiation of conjugated enynes followed by the reaction with electrophiles was accomplished to obtain enantioenriched chiral allenes.

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Continuous-Flow Synthesis of 3,3-Disubstituted Oxindoles by a Palladium-Catalyzed α -Arylation/Alkylation Sequence

Dr. Pengfei Li, Prof. Stephen L. Buchwald
Dept of Chemistry, MIT, USA

Pd-catalyzed α -arylation of oxindoles in continuous flow involves a biphasic system, a precatalyst, and a packed-bed microreactor. Furthermore, this reaction was integrated into a two-step continuous-flow sequence for rapid, modular, and efficient syntheses of 3,3-disubstituted oxindoles.

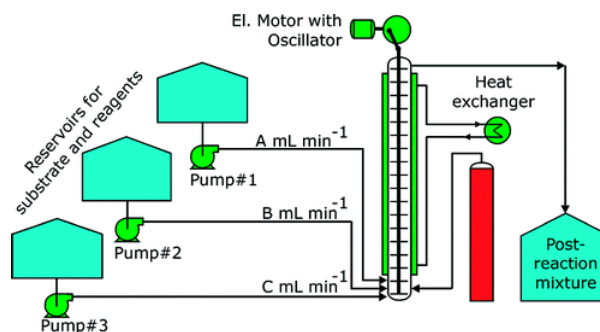


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Multijet Oscillating Disc Millireactor: A Novel Approach for Continuous Flow Organic Synthesis

Lucia Liguori¹,
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¹*Fluens Synthesis, Norway*
²*Department of Chemistry, University of Bergen, Norway*



This report discloses proof of concept and experimental results from a project involving design, development, and investigation of a novel approach for flow chemistry and the realization of equipment operating according to this new approach. This device is named multijet oscillating disk (MJOD) reactor and is dedicated to continuous flow organic synthesis in milliscale. Characteristics such as the importance of the multijet disk unit, with or without oscillating, and possible limitations, such as back-mixing, have been explored, and the flow system is benchmarked with other technologies. Several well-known reactions and syntheses usefully both in the chemical industry as well as in the research laboratory have been conducted using the new system, which have been benchmarked with batch- and microreactor protocols. In particular the Haloform reaction, the Nef reaction, nucleophilic aromatic substitution, the Paal-Knorr pyrrole synthesis, sodium borohydride reduction, O-allylation, the Suzuki cross-coupling reaction, the Hofmann rearrangement and N-acylation were performed during the study of the MJOD reactor performance. Our investigations revealed that the MJOD millireactor system can produce various organic compounds at a high rate concomitant with an excellent selectivity. A Hofmann rearrangement was conducted, a reaction that involves handling of a slurry of the substrate. This reaction was successfully conducted, achieving a quantitative conversion into the target molecule.

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High temperature epoxidation of soybean oil in flow –

speeding up elemental reactions wanted and unwanted

Bruno Cortese
M. De Croon
Volker Hessel

*Dept of Micro Flow Chemistry and Process Technology
Technical University of Eindhoven, Netherlands*



The soybean oil epoxidation reaction is investigated theoretically through kinetic modeling of temperature effects enabled through flow processing under superheated conditions. Different from previous studies on such processing, here a complex reaction network superimposed by multiphase transport is considered; with one elemental step – the hydrogen peroxide decomposition – which can defeat the much boosted product formation. For such a delicate reaction network, the accessibility of accurate and reliable kinetics is absolutely essential – especially when exploring this completely new temperature range. Initially, an overview of the actual kinetic models is given, this gives rise to implications for the study developed here considering high temperature flow processing, heat removal efficiency, hotspot formation, and the effect of different hydrogen peroxide decomposition kinetics. Subsequently an optimized process involving the use of microreactors at different temperatures is proposed for the process management of the reaction heat and to yield a commercial grade product under notably intensified conditions. The results are then benchmarked with quantitative, challenging process improvement criteria set by an industrial partner.

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Electrochemical Microreactor Design for Alkoxylation Reactions

Jiri Kristal , Roman Kodym , K Bouzek , Vladimir Jiricny , and J Hanika
Institute of Chemical Process Fundamentals of the ASCR, Prague, Czech republic

Two types of the bipolar electrochemical microreactor (BEMR) were investigated for methoxylation of 4-methylanisole: A block type BEMR with a fixed number of parallel electrodes and a filter-press type BEMR with a variable number of parallel electrodes. Based on a numerical parametric study, the current utilization factor η was presented for the two microreactors as a function of the dimensionless current I^* for different system parameters. Conversion and selectivity of the alkoxylation reaction was studied experimentally and the performance of both BEMR was compared. Even though the optimization of the reactor design is still needed particularly with respect to liquid distribution into the individual parallel microchannels, heat management and gas removal, the presented results indicated that the bipolar arrangement of electrochemical microreactors certainly offers a great potential for the intensification of alkoxylation reactions.

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