Flow Synthesis Online - Winter 2012

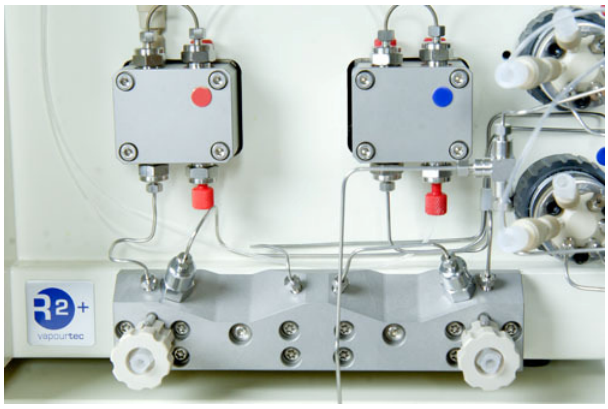
As the year draws to a close, here is the final newsletter of the year.

There are some interesting new application notes, including
- more solvent free chemistry, offering both higher throughput and a much greener approach
- some high pressure flow chemistry, using liquefied gaseous reagents at elevated temperatures
- a new application showcasing the Vapourtec *acid resistant* gas/liquid membrane reactor.

And there are more interesting new publications
- Flow synthesis of organic electronic materials
- Preparation of arene chromium tricarbonyl complexes in flow
- Light-Initiated preparation of functionalized polystyrene monoliths

Have a peaceful holiday, and see you in the New Year

Product Developments



High Pressure Pump Module

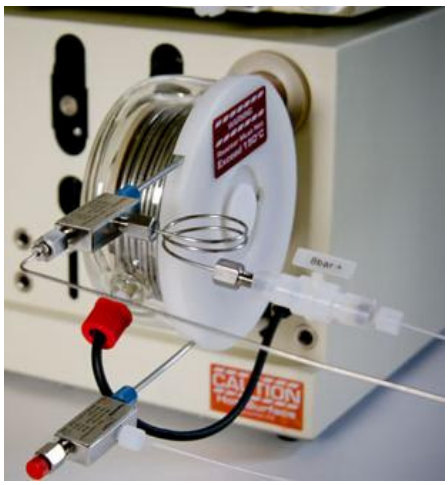
Earlier in the year Vapourtec announced the availability of the high pressure pump module (capable of pumping reagents at up to 200 bar).

In this issue we show the system in use, in a new application note in which dimethyl amine is reacted at 235ºC. Using a reaction pressure of 140bar it is possible to treat this reagent (gaseous at room temperature) as a liquid throughout. Scroll down to see the application note.

Click here for more details of the high pressure pump module

Acid Resistant Gas/Liquid reactor

The Vapourtec tube-in-tube reactor has been popular since its launch, as it slots straight into existing Vapourtec reactor manifolds and permits heating at the same time gas is added.



It is compatible with all R-Series systems and with the new E-Series system

Now, a **new** variant is available, which can tolerate a wide range of acids.

Scroll down to see a new application note which uses this variant for an oxidation with oxygen that is catalysed with HCl.

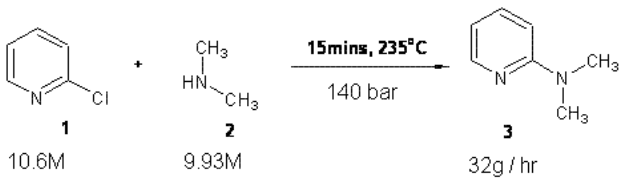
Click here for more details about the gas/liquid reactor

Applications

The following new application notes have recently been made available on the Vapourtec website.

High Pressure and Temperature Process Scale SNAr Reaction under Solvent Free (neat, 10.6M) Conditions using Liquefied Dimethylamine

This application note illustrates the use of the Vapourtec R-Series system to run reactions under solvent free (neat) conditions and its ability to



- pump liquefied gases
  thereby achieving precise control over reaction stoichiometry.
- react the liquefied gas at high temperatures and pressures.

This expands on work carried out in Application note 29

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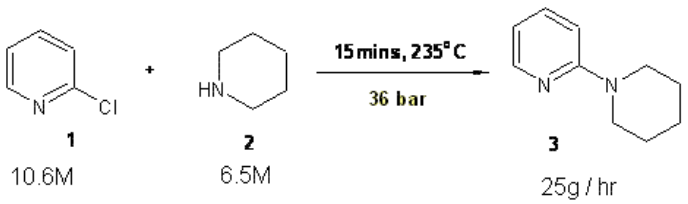
Process Scale Uncatalysed Amination of 2-Chloropyridine (SNAr) under Solvent Free (neat, 10.6M) Conditions.

This application note illustrates the use of the Vapourtec R-Series system to run reactions under solvent free (neat) conditions.
It expands the work previously carried out in Application note 29, mentioned in the previous newsletter.



The challenge when running this reaction neat is the salt formation as it can quickly form a solid in the system. This is overcome in this study.

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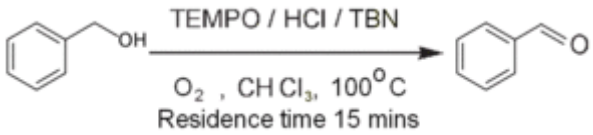
Oxidation Using a Gas-Liquid Membrane Reactor

This study looks at a flow implementation of the TEMPO/HCl/$NaNO_2$ catalytic system for alcohol oxidation with oxygen.



By using the new acid resistant Vapourtec gas/liquid reactor, it is possible to convert a batch process taking 10 hrs into a flow process with 15 minute residence time, that gives 98% conversion and 100% selectivity.

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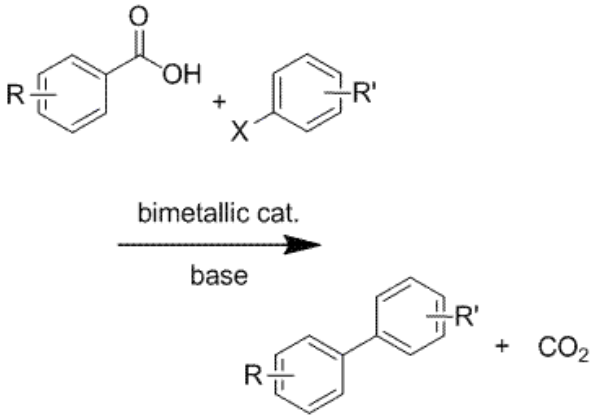
Decarboxylative Cross-Couplings with a Soluble Catalyst System

This study outlines a novel approach to decarboxylative cross-coupling reactions.



The combination of the Vapourtec R-Series system and a new completely soluble catalyst system allows the successful decarboxylative cross-coupling of a range of electron-deficient and heterocyclic aromatic carboxylic acids in moderate to good yields. It is noteworthy to mention that the reaction can be performed from the carboxylic acid rather than the preformed potassium carboxylate (see batch processes) without detecting considerable amounts of protodecarboxylation as byproduct.

Furthermore, no special precautions were necessary as to the purification of starting materials or solvents or the exclusion of moisture and air.

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First time ?

If this is the first newsletter you've received, bear in mind you can find all previous issues in the online archive.

Newsletter Archive

Events

LEGOMEDIC Launch Symposium - 21st January 2013, Belgium

LEGOMEDIC is a regionally funded consortium based on the partnership between various Belgian companies and academic groups, with expertise in synthetic chemistry, engineering, micromechanics and automation of industrial processes.

The LEGOMEDIC project concerns "Development and optimization of microreactors for the continuous flow manufacturing and purification of organic fine chemicals and biomolecules."

The project startup meeting will feature a selection of speakers and vendor displays.

Click here to see details of the meeting

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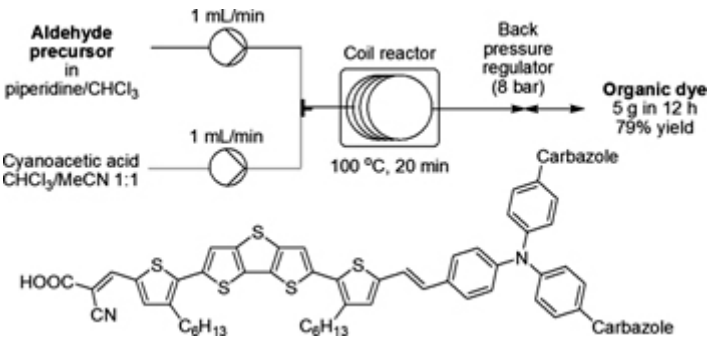
Publications

Continuous Flow Synthesis of Organic Electronic Materials :

Case Studies in Methodology Translation and Scale-up

Helga Seyler[1]
Stefan Haid[2]
Tae-Hyuk Kwon[1,3]
David J. Jones[1]
Peter Bäuerle[2]
Andrew B. Holmes[1]
Wallace W. H. Wong[1] *



[1] Bio21 Institute, University of Melbourne, Australia.
[2] Institute of Organic Chemistry II and Advanced Materials, University of Ulm, Germany.
[3] Current address: Interdisciplinary School of Green Energy, Ulsan National Institute of Science and Technology, Republic of Korea.

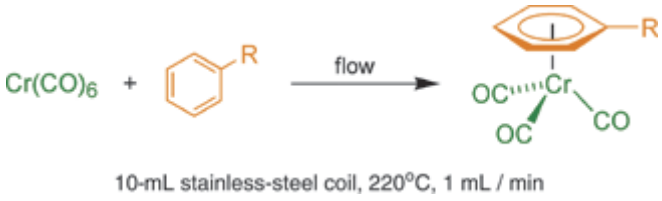
The continuous flow synthesis of functional thiophene derivatives was examined. Methodology for the lithiation of thiophene building blocks was developed using a commercial bench-top flow reactor. In addition, the advantages of flow processing were demonstrated in the synthesis of a high performance organic dye in gram scale.

Click here to go straight to the publication

Preparation of Arene Chromium Tricarbonyl Complexes Using Continuous-Flow Processing: (n6-C6H5CH3)Cr(CO)3 as an Example

Christopher (Xiang) Lee[1]
Elizabeth A. Pedrick[1]
Nicholas E. Leadbeater[1,2]

[1]Department of Chemistry, University of Connecticut, USA
[2]Department of Community Medicine and Health Care, University of Connecticut Health Center, USA



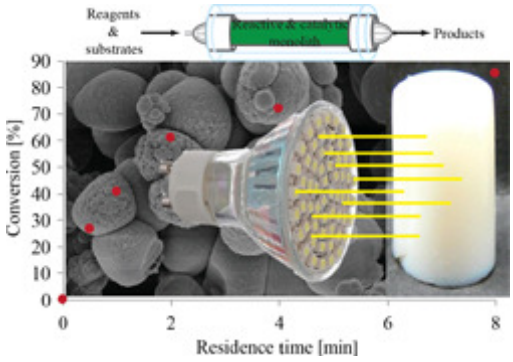
A continuous-flow approach to the direct synthesis of arene chromium tricarbonyl complexes is presented. By working in flow mode, it is possible to avoid some of the problems of batch synthesis, especially sublimation of the Cr(CO)6 starting material and the competitive decomposition of the product during the lengthy reaction times. Heating at 220 °C and operating with a residence time of 10 min through the heated zone allows for the synthesis of (n6-C6H5CH3)Cr(CO)3 as an example, along with a selection of other (arene)Cr(CO)3 complexes.

Click here to go straight to the publication

Visible Light-Initiated Preparation of Functionalized Polystyrene Monoliths for Flow Chemistry

Farhan R. Bou-Hamdan [1]
Kathleen Krüger [1]
Klaus Tauer [1],*.
Tyler McQuade [1],[3], *
Peter H. Seeberger [1],[2]

[1] Max Planck Institute of Colloids and Interfaces Potsdam, Germany.
[2] Institute of Chemistry and Biochemistry, Freie Universität Berlin , Germany.
[3] Department of Chemistry and Biochemistry, Florida State University, USA.



Styrenic monoliths are produced using a novel visible light-initiated method. Monoliths with varying pore sizes are produced using 1-dodecanol and 1-dodecanol/THF mixtures and it was demonstrated that the more volatile i-PrOH can replace 1-dodecanol while still providing the same porogenic properties. In addition, the visible light-initiation protocol enables the facile incorporation of monomers that are incompatible with thermal or UV-initiated monolith formation methods. In particular, a reactive N-hydroxysuccinimidyl (NHS)-ester can be incorporated into the monolith and then subsequently used as an attachment point for a catalyst. Lastly, we demonstrate that the functionalized monolith supports acylation reactions well and that the loading of the catalyst impacts the reaction rate.

Click here to go straight to the publication

Technical articles are in PDF form. Publications may require a subscription to access.

See you in the New Year

Vapourtec Ltd
Park Farm Business Centre
Fornham St Genevieve
Bury St Edmunds, England IP28 6TS
UK

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