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

It's a busy summer for flow chemistry publications !

Contents

We appreciate all your feedback so please keep it coming.

Product Announcements

External control of Vapourtec system now possible

For all the users who have ever asked how they could integrate the Vapourtec system with other automation, there is now an API (application programming interface) which offers that capability.

The new interface permits an external program (either user written code or Labview, Matlab etc) to choose reactor setup, specify parameters of reactions, and run experiments.

It also gives access to all low level functions permitting novel processes (such as "stop flow", for example) to be researched.

And there is also the facility for remote access to the instrument (from PC, smartphone, tablet etc)

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Events

4th Beijing Conference and Exhibition on Instrumental Analysis

Vapourtec will be exhibiting at this event.
Come along and see the equipment up close.

Look for our distributor Tegent Scientific, Hall 12, booths 12013-12017, 12022-12026

More details

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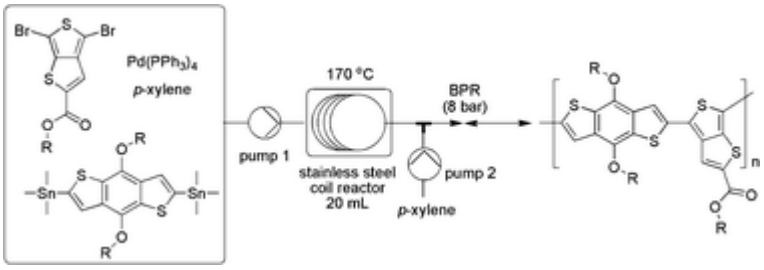
Publications

Continuous flow synthesis of conjugated polymers



Helga Seyler
David J. Jones
Andrew B. Holmes
Wallace W. H. Wong
Dept of Chemistry,
University of Melbourne, Australia

A selection of conjugated polymers, widely studied in organic electronics, was synthesised using continuous flow methodology. As a result of superior heat transfer and reagent control, excellent polymer molecular mass distributions were achieved in significantly reduced reaction times compared to conventional batch reactions.

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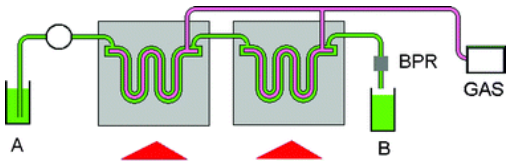
Continuous-flow, palladium-catalysed alkoxycarbonylation reactions using a prototype reactor in which it is possible to

load gas and heat simultaneously

Michael A. Mercadante
Nicholas E. Leadbeater
Department of Chemistry, University of Connecticut, USA



A prototype tube-in-tube reactor in which it is possible to load gas and heat simultaneously has been used in a continuous-flow approach to alkoxycarbonylation reactions of aryl iodides. In the stainless steel coil, liquid flows on the outside of a gas-permeable membrane. The coil can be heated and the temperature can be measured accurately via a probe touching the outer steel surface. A range of aryl iodides can be transformed to the corresponding esters in excellent conversion by reaction at 120 °C using 0.5 mol% palladium acetate as the catalyst with no additional ligand required. Small-scale optimization and substrate screening runs were followed by scale-up.

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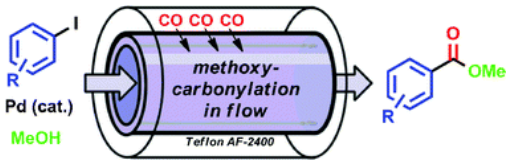
Teflon AF-2400 mediated gas/liquid contact in continuous flow methoxycarbonylations and in-line FTIR measurement of CO concentration

Peter Koos
Ulrike Gross
Anastasios Polyzos
Matthew O'Brien
Ian Baxendale
Steven V. Ley
Dept of Chemistry, University of Cambridge, UK



We report on the development of a continuous flow process for the palladium catalysed methoxycarbonylation of aryl, heteroaromatic and vinyl iodides and an aryl bromide using a Teflon AF-2400 based Tube-in-Tube reactor to mediate the selective permeation of carbon monoxide into solution at elevated pressures. The low volume of pressurised gas within the reactor (5.6 mL) offers the potential for an enhanced safety profile compared to batch processes. We also present preliminary results for the use of in situ FTIR to measure solution concentrations of carbon monoxide and demonstrate the use of a second reactor to effect the removal of carbon monoxide from the flow stream.

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Continuous flow photolysis of aryl azides: Preparation of 3H-azepinones

Farhan R. Bou-Hamdan
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Alexander G. O'Brien
Peter H. Seeberger

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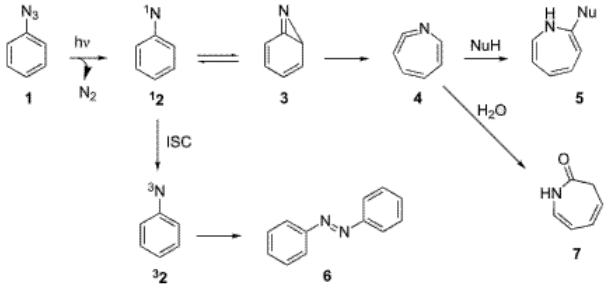

Photolysis of aryl azides to give nitrenes, and their subsequent rearrangement in the presence of water to give 3H-azepinones, is performed in continuous flow in a photoreactor constructed of fluorinated ethylene polymer (FEP) tubing. Fine tuning of the reaction conditions using the flow reactor allowed minimization of secondary photochemical reactions.

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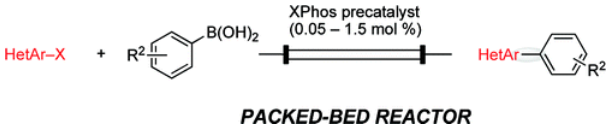
Suzuki-Miyaura Cross-Coupling of Heteroaryl Halides and Arylboronic Acids in Continuous Flow

Timothy Noel
Andrew J. Musacchio
Department of Chemistry, MIT , USA



General continuous-flow conditions for the Suzuki-Miyaura cross-coupling of heteroaryl halides and (hetero)arylboronic acids have been developed. A wide range of heterobiaryl products is obtained in excellent yields (20 examples) employing low catalyst loadings (0.05-1.5 mol % Pd).

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Safe, Convenient ortho-Claisen Thermal Rearrangement Using a Flow Reactor

Juan A. Rincon[1]
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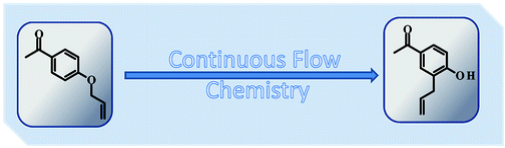

The [3,3] Claisen rearrangement is a well-known reaction that has been very useful for the synthesis of o-allyl phenols. The thermally induced rearrangement

could present safety and operational issues at large batch scale. Herein, we report a process that utilized a tube reactor to make 80 g of an early phase intermediate in a short time while mitigating the potential chemistry hazards. Thus, both project material demands and flow chemistry proof of concept were achieved.

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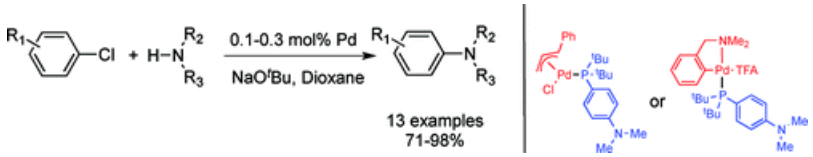
Highly Active Well-Defined Palladium Precatalysts for the Efficient Amination of Aryl Chlorides



Anthony Chartoire
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The efficient preparation of [Pd(Amphos)(cinnamyl)Cl)] and [Pd(Amphos)(TFA)(k²-N,C-C$_6$H$_4$-CH$_2$NMe$_2$)] (Amphos = 4-(di-tert-butylphosphino)-N,N-dimethylaniline and TFA = trifluoroacetate), two new well-defined palladium precatalysts, is reported. These complexes prove highly active in the Buchwald–Hartwig amination reaction, allowing the coupling of a wide range of (hetero)aryl chlorides, including unactivated, neutral, and sterically hindered substrates, with a wide range of amines, including primary and secondary amines. Finally, the catalytic systems have proven efficient at low catalyst loadings ranging from 0.1 to 0.3 mol %.

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Controllable Preparation of Poly (butyl acrylate) by Suspension Polymerization in Co-axial Capillary Microreactor

Zhendong Liu
Yangcheng Lu
Bodong Yang
Guangsheng Luo

In this work, a new controllable and continuous free radical polymerization process was developed and characterized in a co-axial capillary microreactor. In this process, the monomer solution was first dispersed into mono-dispersed droplets followed by thermal-initiated polymerization in following capillary immersed in oil bath. Experimental results in the microreactor showed that the molecular weight distribution was mainly determined by the size of the droplet, while the molecular weight of the polymer could be adjusted by changing the reaction temperature and AIBN concentration. This type of microreactor can potentially be applied to research involving the mechanisms of highly exothermic free radical

polymerization processes and can also be used as an efficient tool for their controllable preparation.

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Microreactor System for High-Pressure Continuous Flow Homogeneous Catalysis Measurements

Jaroslav Keybl
Klavs F. Jensen

Department of Chemical Engineering, MIT, USA

A high-pressure gas/liquid system is presented for determining homogeneous catalyst kinetic parameters. Microreactors enable segmented flow with very predictable gas/liquid contacting, reducing the effects of mass transfer as well as facilitating isothermal operation. The system is capable of studying homogeneous catalysis at high temperature and pressure (<350 °C and <100 bar) under continuous flow. By varying the pressure, temperature, and concentrations of both gas and liquid species, it is possible to determine kinetic parameters. Both inline and offline analyses are performed using attenuated total reflectance Fourier transform infrared (ATR-FTIR) spectroscopy and gas chromatography (GC). The hydroformylation of 1-octene is studied to demonstrate the utility of this microreactor system as a laboratory tool for kinetic measurements. The system is capable of providing both parameter estimation and mechanistic insight. A discussion is also included that explores the types of chemical systems that can be studied practically in microreactors.

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Announcements

New flow journal launched

The Flow Chemistry Society launched their **Journal of Flow Chemistry** at the start of the month at the ACS Fall Expo.

Every article in the first issue of the journal is available for free download.

Click here to see the journal online

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

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