Highlights from the Flow Chemistry Literature 2012 (Part 1)

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In this section of the journal, the continuous flow chemistry literature of the preceding months is presented. Included are articles published in January and February 2012 and some articles from 2011 which received page numbers only in the beginning of 2012. Some key examples are highlighted in the form of graphical abstracts. The remaining publications in the field are then listed ordered by journal name, with review articles grouped at the end.

01/2012

Diisobutylaluminium Hydride Reductions Revitalized: A Fast, Robust and Selective Continuous Flow System for Aldehyde Synthesis

Damien Webb, Timothy F. Jamison*

Org. Lett. 2012, 14, 568–571 DOI: 10.1021/ol2031872

Continuous-flow DIBALH reduction of esters to aldehydes was demonstrated. Various parameters were optimized in the continuous-flow system – flow rate, temperature, residence time, and stoichiometry. The best results were obtained at higher flow rates due to better mixing and faster in-line quench using methanol. The reactions were complete within less than 60 s. Design of experiments (DOE) helped in finding the optimal conditions, favoring the formation of the desired aldehydes and diminishing the amounts of formed over-reduced products (alcohols). Reaction temperatures in the range of −20 to −40 °C gave promising results as compared to batch conditions, where the major products were the corresponding alcohols.

02/2012

A Solid-Supported Organocatalyst for Continuous-Flow Enantioselective Aldol Reactions

Carlec Ayats, Andrea H. Henseler, Miquel A. Pericas*

ChemSusChem 2012, 5, 320–325 DOI: 10.1002/cssc.201100570

Highly stereoselective aldol reactions between aromatic aldehydes and cyclic ketones under continuous-flow conditions using a solid-supported, proline-based organocatalyst are demonstrated. The organocatalyst has been prepared based on a “click”–chemistry cycloaddition. The catalyst could be reused more than seven times without loss of activity, retaining high diastereoo- and enantioselectivity during the runs. The reaction mixture was transferred to a flow process and nearly 5 g of product with 97% ee were obtained in a 30-h uninterrupted run.
A Ceramic Microreactor for the Synthesis of Water Soluble CdS and CdS/ZnS Nanocrystals with On-line Optical Characterisation

Sara Gomez-de Pedro, Mar Puyol, David Izquierdo, Inigo Salinas, J. M. de la Fuente, Julian Alonso-Chamarro*

Nanoscale 2012, 4, 1328–1335 DOI: 10.1039/c2nr11525e

The use of a computer-controlled microreactor for the generation of water-soluble CdS and CdS/ZnS nanocrystals with in situ monitoring of the synthetic progress is reported. The microfluidic device is made of ceramic tapes via low-temperature co-fired ceramics technology (LTCC). The use of an optical detection system for absorbance and fluorescence measurements allows efficient managing of reaction variables to obtain the desired results. Performing the reaction in an aqueous media can be considered as further advantage.

Visible-Light-Mediated Photochemistry: Accelerating Ru(bpy)$_3^{2+}$-Catalyzed Reactions in Continuous Flow

Farhan R. Bou-Hamdan, Peter H. Seeberger*

Chem. Sci. 2012, 3, 1612–1616 DOI: 10.1039/c2sc01016j

Several Ru(bpy)$_3^{2+}$-catalyzed reactions were performed under continuous-flow conditions using visible light. As a light source, two commercial 17-W white LED-lamps with emission lengths in the visible-light range (400–700 nm) were used without the need of additional cooling. Matching the high photon flux at 452 nm with the absorption wavelength of the Ru(II) photocatalyst promoted additionally the reaction progress. Short reaction times (≤30 min) and cleaner reaction profiles with good yields were obtained in comparison with batch samples.

Continuous Flow Production of Thermally Unstable Intermediates in a Microreactor with Inline IR-Analysis: Controlled Vilsmeier–Haack Formylation of Electron-Rich Arenes

Sbastiaan A. M. W. van den Broek, Jeroen R. Leliveld, Rene Becker, Marièlle M. E. Delvill, Pieter J. Nieuwland,* Kaspar Koch, Floris P. J. T. Rutjes*

Org. Process Res. Dev. 2012, 16, 934–938 DOI: 10.1021/op2003437

The Vilsmeier–Haack formylation is performed in a flow manner using a microreactor system equipped with inline IR-analysis of the reaction mixture. After optimization of reaction time, temperature and molar ratios, the reaction could be successfully used to formylate various arenes. Crucial for the synthesis is the use of IR-analysis as well as precise temperature control, since the formation of the formylating agent is an exothermal process and if not controlled could result in thermal runaways (batch). Finally, a scale-up run was successfully realized, allowing the isolation of nearly 6 g of formylated pyrrole.
A novel concept of a continuous-flow reactor employing a non-resonant microwave applicator as direct heating method is introduced. The instrument uses microwaves with frequency in the range of 2.4–2.5 GHz, allowing the reactor to work in a temperature regime up to 270 °C. The accurate temperature control is assured by multiple IR-sensors. Tubular reactors made out of different materials – SiC, ceramics, quartz, etc. can be used. Several standard reactions have been examined in this new microwave flow reactor – Claisen rearrangement, Diels–Alder cycloaddition, Fischer indole synthesis, and Suzuki–Miyaura and oxidative Heck C–C couplings. All of the shown examples proceeded well, resulting in full conversion and high isolated yields within few minutes reaction time.

Further Flow Chemistry Publications

“Transesterification of rapeseed oil under flow conditions catalyzed by basic solids: M–Al(La)–O (M=Sr, Ba), M–Mg–O (M=Y, La)”
O. V. Sherstyuk, A. S. Ivanova, M. Y. Lebedev, M. V. Bukhiyarova, L. G. Matvienko, A. A. Budneva, A. N. Simonov, V. A. Yakovlev
Application A: General 2011, 419, 73–83

“Meter-long and robust supramolecular strands encapsulated in hydrogel jackets”
D. Kiriya, M. Ikeda, H. Onoe, M. Takinoue, H. Komatsu, Y. Shimoyama, I. Hamachi, S. Takeuchi
Angewandte Chemie International Edition 2012, 51, 1553–1557

“Continuous-flow synthesis of the anti-malaria drug artemisinin”
F. Levesque, P. H. Seeberger
Angewandte Chemie International Edition 2012, 51, 1706–1709

“Towards quantitative conversion of microalgae oil to diesel-range alkanes with bifunctional catalysts”
B. Peng, Y. Yao, C. Zhao, J. A. Lercher
Angewandte Chemie International Edition 2012, 51, 2072–2075

“Triphase microfluidic-directed self-assembly: Anisotropic colloidal photonic crystal supraparticles and multicolor patterns made easy”
Z. Yu, C.-F. Wang, L. Ling, L. Chen, S. Chen
Angewandte Chemie International Edition 2012, 51, 2375–2378

“Continuous-flow catalytic asymmetric hydrogenations: Reaction optimization using FTIR inline analysis”
M. Rueping, T. Bootwicha, E. Sugiono
Beilstein Journal of Organic Chemistry 2012, 8, 300–307

“Microfluid technology: An economical and versatile approach for the synthesis of O-[(2-[18]F)fluoroethyl]-L-tyrosine ([18]FET)”
V. Bouvet, M. Wuest, P.-H. Tam, M. Wang, F. Wuest
Bioorganic & Medicinal Chemistry Letters 2012, 22, 2291–2295

“Modified zeolite ZSM-5 for the methanol to aromatics reaction”
M. Conte, J. A. Lopez-Sanchez, Q. He, D. J. Morgan, Y. Ryabenkova, J. K. Bartley, A. F. Carley, S. H. Taylor, C. J. Kiely, K. Khalid, G. J. Hutchings
Catalysis Science & Technology 2012, 2, 105–112

“Aqueous phase Fischer–Tropsch synthesis in a continuous flow reactor”
L. Liu, G. Sun, C. Wang, J. Yang, C. Xiao, H. Wang, D. Ma, Y. Kou
Catalysis Today 2012, 183, 136–142

“Continuous flow synthesis of conjugated polymers”
H. Seyler, D. J. Jones, A. B. Holmes, W. W. H. Wong
Chemical Communications 2012, 48, 1598–1600

“Continuous flow metal-free oxidation of picolines using air”
M. Hamano, Kevin D. Nagyz, K. F. Jensen
Chemical Communications 2012, 48, 2086–2088

“Maximising the efficiency of continuous photo-oxidation with singlet oxygen in supercritical CO2 by use of fluorous biphasic catalysis”
J. F. B. Hall, X. Han, M. Poliakoff, R. A. Bourne, M. W. George
Chemical Communications 2012, 48, 3073–3075

“Continuous flow photocatalytic oxidation of nitrogen oxides over anodized nanotubular titania films”
A. G. Kontos, A. Katsanaki, V. Likodimos, T. Maggos, D. Kim, C. Vasilakos, D. D. Dionysiou, P. Schmuki, P. Falaras
Chemical Engineering Journal 2012, 179, 151–157
“Heterogeneously catalyzed synthesis of performic acid in a microstructured reactor”
F. Ebrahimi, E. Kolehmainen, I. Turunen
Chemical Engineering Journal 2012, 179, 312–317

“Multiphase minireactor system for direct fluorination of ethylene carbonate”
P. Langa, M. Hill, I. Krossing, P. Woias
Chemical Engineering Journal 2012, 179, 330–337

“Direct carbonylation of nitrobenzene to phenylisocyanate using gas–liquid slug flow in microchannel”
Y. Takebayashi, K. Sue, S. Yoda, T. Furuya, K. Mae
Chemical Engineering Journal 2012, 180, 250–254

“Controllable synthesis of microscale titania fibers and tubes using colaminar microflows”
W. Lan, S. Li, J. Xu, G. Luo
Chemical Engineering Journal 2012, 181–182, 828–833

“Design of optimal multiphase reactors exemplified on the hydroformylation of long chain alkenes”
A. Peschel, B. Hentschel, H. Freund, K. Sundmacher
Chemical Engineering Journal 2012, 188, 126–141

“Monitoring on-chip Pictet–Spengler reactions by integrated analytical separation and label-free time-resolved fluorescence”
S. Ohla, R. Beyreiss, S. Fritzschke, P. Glaser, S. Nagl, K. Stockhausen, C. Schneider, D. Belder
Chemistry – A European Journal 2012, 18, 1240–1246

“Gas–liquid segmented flow microfluidics for screening Pd-catalyzed carbonylation reactions”
X. Gong, P. W. Miller, A. D. Gee, N. J. Long, A. J. de Mello, R. Vilar
Chemistry – A European Journal 2012, 18, 2768–2772

“Biodiesel process intensification in a very simple microchannel device”
E. Santacesaria, M. Di Serio, R. Tesser, R. Turco, M. Tortorelli, V. Russo
Chemical Engineering and Processing: Process Intensification 2012, 52, 47–54

“Hydrodeoxygenation of pyrolysis oil in a microreactor”
N. Joshi, A. Lawal
Chemical Engineering Science 2012, 74, 1–8

“Design and operation of a filter reactor for continuous production of a selected pharmaceutical intermediate”
K. M. Christensen, M. J. Pedersen, K. Dam-Johansen, T. L. Holm, T. Skovby, Skil
Chemical Engineering Science 2012, 71, 111–117

“Sequential immobilization of enzymes in microfluidic channels for cascade reactions”
S. Fornera, P. Kuhn, D. Lombardi, A. D. Schlüter, P. S. Dittrich, P. Walde
ChemPlusChem 2012, 77, 98–101

“Ritter reactions in flow”
L. Audiger, K. Watts, S. C. Elmore, R. I. Robinson, T. Wirth
ChemSusChem 2012, 5, 257–260

“Organocatalyzed epoxidation of alkenes in continuous flow using a multi-jet oscillating disk reactor”
R. Spaccini, L. Liguori, C. Punta, H.-R. Bjørsvik
ChemSusChem 2012, 5, 261–265

“Highly efficient 1,4-addition of aldehydes to nitroolefins: Organocatalysis in continuous flow by solid-supported peptidic catalysts”
S. B. Otvös, I. M. Mandity, F. Fülöp
ChemSusChem 2012, 5, 266–269

“Room-temperature, acid-catalyzed [2+2] cycloadditions: Suppression of side reactions by using a flow microreactor system”
K. Kurahashi, Y. Takemoto, K. Takasu
ChemSusChem 2012, 5, 270–273

“The oxygen-mediated synthesis of 1,3-butadiynes in continuous flow: Using Teflon AF-2400 to effect gas/liquid contact”
T. P. Petersen, A. Polyzos, M. O’Brien, T. Ulven, I. R. Baxendale, S. V. Ley
ChemSusChem 2012, 5, 274–277

“Prilezhaev dihydroxylation of olefins in a continuous flow process”
B. A. M. W. van den Broek, R. Becker, F. Kössl, M. M. E. Delville, P. J. Nieuwland, K. Koch, F. P. J. T. Rutjes
ChemSusChem 2012, 5, 289–292

“Transfer of the epoxidation of soybean oil from batch to flow chemistry guided by cost and environmental issues”
D. Kralisch, I. Streckmann, D. Ott, U. Kürschik, E. Santacesaria, M. Di Serio, V. Russo, L. De Carlo, W. Linhart, E. Christian, B. Cortese, M. H. J. M. de Croom, V. Hessel
ChemSusChem 2012, 5, 300–311

“Sequential continuous flow processes for the oxidation of amines and azides by using HOF-MeCN”
C. B. McPake, C. B. Murray, G. Sandford
ChemSusChem 2012, 5, 312–319

“TEMPO-mediated electrooxidation of primary and secondary alcohols in a microfluidic electrolytic cell”
J. T. Hill-Cousins, J. Kuleshova, R. A. Green, P. R. Birkin, D. Fletcher, T. J. Underwood, S. G. Leach, R. C. D. Brown
ChemSusChem 2012, 5, 326–331
“Solid-supported gallium triflate: An efficient catalyst for the three-component ketonic Strecker reaction”
C. Wiles, Paul Watts
ChemSusChem 2012, 5, 332–338

“Practical synthesis of photochromic diarylethenes in integrated flow microreactor systems”
T. Asai, A. Takata, A. Nagaki, J.-i. Yoshida
ChemSusChem 2012, 5, 339–350

“Stereocontrolled synthesis of the cis-hydroxydecalin system: Towards biologically active 19-nor-clerodanes”
P. M. Mirzayans, R. H. Pouwer, C. M. Williams, P. V. Bernhardt
European Journal of Organic Chemistry 2012, 1633–1638

“β-Galactosidase-immobilised microreactor fabricated using a novel technique for enzyme immobilisation and its application for continuous synthesis of lactulose”
Y. S. Song, H. Y. Shin, J. Y. Lee, C. Park, S. W. Kim
Food Chemistry 2012, 133, 611–617

“E-factor minimized protocols for the polystyryl-BEMP catalyzed conjugate additions of various nucleophiles to α,β-unsaturated carbonyl compounds”
S. Bonollo, D. Lanari, J. M. Longo, L. Vaccaro
Green Chemistry 2012, 14, 164–169

“Catalysis in flow: Au-catalysed alkylation of amines by alcohols”
N. Zotova, F. J. Roberts, G. H. Kelsall, A. S. Jessiman, K. Hellgardt, K. K. (Mimi) Hii
Green Chemistry 2012, 14, 26–232

“In situ electrogeneration of o-benzoquinone and high yield reaction with benzenethiols in a microflow system”
T. Kashiwagi, F. Amemiya, T. Fuchigami, M. Atobe
Green Chemistry 2012, 14, 232–238

“Continuous stream processing: A prototype magnetic field induced flow mixer”
P. Koos, D. L. Browne, S. V. Ley
Green Processing and Synthesis 2012, 1, 11–18

“Liquid–liquid flow in a capillary microreactor: Hydrodynamic flow patterns and extraction performance”
J. Jovanovic, E. V. Rebrov, T. A. (Xander) Nijhuis, M. T. Kreutzer, V. Hessel, J. C. Schouten
Industrial Engineering Chemistry Research 2012, 51, 1015–1026

“Continuous production of CuZnSnS4 nanocrystals in a flow reactor”
A. Shavel, D. Cadavid, M. Ibanez, A. Carrete, A. Cabot
Journal of the American Chemical Society 2012, 134, 1438–1441

“Microfluidic study of fast gas–liquid reactions”
W. Li, K. Liu, R. Simms, J. Greener, D. Jagadeesan, S. Pinto, A. Günther, E. Kumacheva
Journal of the American Chemical Society 2012, 134, 3127–3132

“Alumina-grafted macro-/mesoporous silica monoliths as continuous flow microreactors for the Diels–Alder reaction”
A. Sachse, V. Hulea, A. Finiels, B. Coq, F. Fajula, A. Galameau
Journal of Catalysis 2012, 287, 62–67

“Dynamic control of gold nanoparticle morphology in a microchannel flow reactor by glucose reduction in aqueous sodium hydroxide solution”
T. Ishizaka, A. Ishigaki, H. Kawanami, A. Suzuki, T. M. Suzuki
Journal of Colloid and Interface Science 2012, 367, 135–138

“Low cost and non-surfactant synthesis of fluorinated alumina modified with magnesium for condensation of aniline to diphenylamine”
C. Zheng, X. Sun
Journal of Fluorine Chemistry 2012, 135, 373–378

“Simple enzyme immobilization inside glass tubes for enzymatic cascade reactions”
S. Fornera, T. Bauer, A. D. Schlüter, P. Walde
Journal of Materials Chemistry 2012, 22, 502–511

“Preparation of well-dispersed PdAu bimetallic nanoparticles on reduced graphene oxide sheets with excellent electrochemical activity for ethanol oxidation in alkaline media”
Z. Huang, H. Zhou, C. Li, F. Zeng, C. Fua, Y. Kuang
Journal of Materials Chemistry 2012, 22, 1781–1785

“Direct synthesis of dextran-coated superparamagnetic iron oxide nanoparticles in a capillary-based droplet reactor”
K. Kumar, A. M. Nightingale, S. H. Krishnadasan, N. Kamaly, M. Wylenzinska-Arridge, K. Zeissler, W. R. Branford, E. Ware, A. J. deMello, J. C. deMello
Journal of Materials Chemistry 2012, 22, 4704–4708

“Flash flow pyrolysis: Mimicking flash vacuum pyrolysis in a high-temperature/high-pressure liquid-phase microreactor environment”
D. Cantillo, H. Sheibani, C. O. Kappe
Journal of Organic Chemistry 2012, 77, 2463–2473

“Efficient and continuous monoacylation with superior selectivity of symmetrical diamines in microreactors”
R. A. Maurya, P. H. Hoang, D.-P. Kim
Lab on a Chip 2012, 12, 65–68
“Coated gas bubbles for the continuous synthesis of hollow inorganic particles”
J. Wan, H. A. Stone
*Langmuir* **2012**, *28*, 37–41

“Gas sensing with nano-indium oxides (In$_2$O$_3$) prepared via continuous hydrothermal flow synthesis”
S. Elouali, L. G. Bloor, R. Binions, I. P. Parkin, C. J. Carmalt, J. A. Darr
*Langmuir* **2012**, *28*, 1879–1885

“Continuous steroid biotransformations in microchannel reactors”
M. P. C. Marques, P. Fernandes, J. M. S. Cabral, P. Znidarsic-Plazl, I. Plazl
*New Biotechnology* **2012**, *29*, 227–234

“Application of a Burkholderia cepacia lipase immobilized silica monolith microbioreactor to continuous flow kinetic resolution for transesterification of (R,S)-1-phenylethanol”
K. Kawakami, M. Ueno, T. Takei, Y. Oda, R. Takahashi
*Process Biochemistry* **2012**, *47*, 147–150

“Lipase-catalyzed regioselective acylation of sugar in microreactors”
L.-H. Du, X.-P. Luo
*RSC Advances* **2012**, *2*, 2663–2665

“Scale-up of flow-assisted synthesis of C$_2$-symmetric chiral PyBox ligands”
C. Battilocchio, M. Baumann, I. R. Baxendale, M. Biava, M. O. Kitching, S. V. Ley, R. E. Martin, S. A. Ohnmacht, N. D. C. Tappina
*Synthesis* **2012**, *44*, 635–647

“Highly efficient and convenient Strecker reaction of carbonyl compounds and amines with TMSCN catalyzed by MCM-41 anchored sulfonic acid as a recoverable catalyst”
M. G. Dekamin, Z. Mokhtari
*Tetrahedron* **2012**, *68*, 922–930

“Highly efficient thermal cyclization reactions of alkylidene esters in continuous flow to give aromatic/heteroaromatic derivatives”
L. Lengyl, T. Z. Nagy, G. Sipos, R. Jones, G. Dorman, L. Úrge, F. Darvas
*Tetrahedron Letters* **2012**, *53*, 738–743

“High-temperature continuous flow synthesis of 1,3,4-oxadiazoles via N-acylation of 5-substituted tetrazoles”
B. Reichart, C. O. Kappe
*Tetrahedron Letters* **2012**, *53*, 952–955

“Evaluation of a flow-photochemistry platform for the synthesis of compact modules”
M. Nettekoven, B. Püllmann, R. E. Martin, D. Wechsler
*Tetrahedron Letters* **2012**, *53*, 1363–1366

“Flow microreactor synthesis of tricyclic sulfonamides via N-tosylaziridinylolithiums”
E. Takizawa, A. Nagaki, J.-i. Yoshida
*Tetrahedron Letters* **2012**, *53*, 1397–1400

“Ultrasound-promoted intramolecular direct arylation in a capillary flow microreactor”
L. Zhang, M. Geng, P. Teng, D. Zhao, X. Lu, J.-Xin Li
*Ultrasonics Sonochemistry* **2012**, *19*, 250–256

Reviews

“Recent changes in patenting behavior in microprocess technology and its possible use for gas–liquid reactions and the oxidation of glucose”
I. Dencic, V. Hessel, M. H. J. M. de Croon, J. Meuldijk, C. W. J. van der Doelen, K. Koch
*ChemSusChem* **2012**, *5*, 232–245

“Application of metal-based reagents and catalysts in microstructured flow devices”
T. Chinnusamy, S. Yudha S, M. Hager, P. Kreitmeier, O. Reiser
*ChemSusChem* **2012**, *5*, 247–255

“Continuous reactions in supercritical carbon dioxide: Problems, solutions and possible ways forward”
X. Han, M. Poliaikoff
*Chemical Society Reviews* **2012**, *41*, 1428–1436

“Continuous flow reactors: A perspective”
C. Wiles, P. Watts
*Green Chemistry* **2012**, *14*, 38–54

“Biodiesel synthesis in microreactors”
T. Xie, L. Zhang, N. Xu
*Green Processing and Synthesis* **2012**, *1*, 61–70

“Process intensification in green synthesis”
V. Kumar, K. D. P. Nigam
*Green Processing and Synthesis* **2012**, *1*, 79–107

“A review on numerical studies of slug flow hydrodynamics and heat transfer in microtubes and microchannels”
V. Talimi, Y. S. Muzychka, S. Kocabiyik
*International Journal of Multiphase Flow* **2012**, *39*, 88–104