Industrial Organic Chemistry

Dec. 12th, 2017

Malek Ibrahim

Group meeting
• **Overview of the organic chemicals industry**

• **Bulk Chemicals**
  • Effect of raw materials on chemistry
  • Enabling technologies
  • Examples from industry

• **Fine & Specialty chemicals**
  • Comparison against bulk chemicals
  • Factors affecting GCMP
  • Future directions

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**Outline**

- Chemistry
  - catalyst
  - Kinetics
  - Energetics
  - Intermediates
  - Phases

- Economics
  - ROI
  - Sustainability
  - Optimization

- Engineering
  - Conditions
  - Equipment
  - Transport
  - Purification

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2
Overview

- 1850 plants & animals, coal
- 1920 acetylene (from coal)
- 1950 oil
- 1973 oil & gas

Bulk chemicals → Fine chemicals → Specialty chemicals

<table>
<thead>
<tr>
<th>volume</th>
<th>&gt; 10,000 t/y</th>
<th>&lt; 10,000 t/y</th>
<th>&lt; 1,000 t/y</th>
</tr>
</thead>
<tbody>
<tr>
<td># of products</td>
<td>100’s</td>
<td>1000’s</td>
<td>&gt; 10,000’s</td>
</tr>
<tr>
<td>price</td>
<td>5 $ /kg</td>
<td>5-20 $/kg</td>
<td>&gt; 20 $ kg</td>
</tr>
<tr>
<td>Value added</td>
<td>low</td>
<td>moderate</td>
<td>high</td>
</tr>
<tr>
<td>Key factor</td>
<td>cost</td>
<td>Function, cost</td>
<td>function</td>
</tr>
</tbody>
</table>
Examples of BC

- Primary BC: Ethylene, propylene, butadiene, benzene, p-xylene, methanol
- Secondary BC: ethylene oxide, ethylene chloride, ethylbenzene, acrylonitrile, terephthalic acid, MTBE, cumene
- Tertiary BC: vinyl chloride, polyester, styrene
Characteristics of BC industry

- Large scale, dedicated production lines
- High temperature and pressure for high throughput
- Continuous flow chemistry and purification is common
- Highly engineered processes
- Heterogeneous catalysis (exceptions hydroformylation and p-xylene oxidation)
- Large investment, industrial clusters
- R&D trend is towards using alternative feedstocks and greener production (2-5 %)

Big investments: BP, ExxonMobil, Shell, Chevron, SABIC, Sinopec
Process Licensors: Lurgi, Uhde, UOP, KBR, Lummus, Axens, Johnson Matthy
Why is this important for us?

> 1850 plants & animals, coal
> 1920 acetylene (from coal)
> 1950 oil
> 1973 oil & gas

• **Engineering solutions could be implemented in other sectors**
  (flow chemistry, heterogeneous catalysis…)

• **Well-studied reaction mechanisms**
  could be intercepted/modified for more complex chemistries

• **Awareness of the added value by each transformation**
  avoid developing “de-evaluating” chemistries

• **Help predict impurities in your feedstock and possible side reactions in your chemistry**
Opportunities for org. chemistry in renewables

• Engineering solutions could be implemented in other sectors (flow chemistry, heterogeneous catalysis…)

• Well-studied reaction mechanisms could be intercepted/modified for more complex chemistries

• Awareness of the value that each transformation adds avoid developing “de-evaluating” chemistries

• Help predict impurities in your feedstock and possible side reactions in your chemistry

• Awareness of what chemistries are needed to harness emerging feedstocks
Opportunities for org. chemistry in renewables

> 1850 plants & animals, coal
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> 1973 oil & gas
CO$_2$ and biomass

Bulk chemicals $\rightarrow$ Fine chemicals $\rightarrow$ Specialty chemicals

Examples:
- CO$_2$ capture (MOF, hydroxylamines, ionic liquids…)
- CO$_2$ reactions (carboxylation, RWGS…)
- C-O bond cleavage reactions

Yaghi et al. J. Am. Chem. Soc. 2017 139 (35), 12125
Chemistry of oil processing

**Challenge:** the fraction of oil that is rich in useful “and separable” chemicals is the same fraction that makes good gasoline...naphtha

Long term goal: energy comes from renewables, chemicals come from oil

For the time being, maximize the utility of extracted oil by converting the less useful fractions to more useful ones
Naphtha reforming

**Reactions:** dehydrogenation, isomerization, dehydrogenative aromatization

**Temperature:** 450-550°C

**Catalyst:** Pt/Al₂O₃ (chlorinated)
Naphtha reforming

**Reactions:** dehydrogenation, isomerization, dehydrogenative aromatization

**Temperature:** 450-550°C

**Catalyst:** Pt/Al$_2$O$_3$ (chlorinated)

**Challenges:**
- catalyst deactivation by heavy hydrocarbon deposition (coking)
- solution: continuous catalyst circulation
Naphtha reforming

**Reactions:** dehydrogenation, isomerization, dehydrogenative aromatization

Temperature: 450-550°C

**Catalyst:** Pt/Al₂O₃ (chlorinated)

**Challenges:**
- catalyst deactivation by loss of chlorine
- solution: continuous addition of chlorine source (PERC)
Naphtha hydrotreating

**Reactions:** dehydrogenation, isomerization, dehydrogenative aromatization

Temperature: 450-550 C

Catalyst: Pt/Al$_2$O$_3$ (*chlorinated*)

Challenges:

- catalyst deactivation by sulfur presence in the feed
- solution: naphtha pretreatment (hydro desulfurization, i.e. C-S bond scission with MoS$_2$/Al$_2$O$_3$ to form H$_2$S)

Naphtha hydrotreating

**Reactions:** dehydrogenation, isomerization, dehydrogenative aromatization

Temperature: 450-550°C

Catalyst: Pt/Al$_2$O$_3$ (chlorinated)

Challenges:

- catalyst deactivation by sulfur presence in the feed
- solution: naphtha pretreatment (hydro desulfurization...i.e C-S bond scission with MoS$_2$/Al$_2$O$_3$ to form H$_2$S)

**Catalytic cracking:** cleavage of C-C bonds in presence of solid acid catalysts (zeolite)

Convert less usable longer chains to shorter more useful alkanes and alkenes

Reaction time is very short "milliseconds" ... fluid catalyst riser

Formation of Carbocations on Acid Sites of Catalysts

- **Bronsted acid sites** – donate protons
- **Lewis acid sites** – accept electrons

**Olefin** $\text{C} = \text{C} \rightarrow C_2H_5^+$

$450^\circ \text{C}$

**Alkane** $\text{C} - \text{C} \rightarrow C_2H_5^+$

$450^\circ \text{C}$

**http://www.apc-network.com**
**Alkylation**:

*formation of longer chain alkanes from volatile, shorter alkane/ene*

- acid catalyzed by sulfuric or hydrofluoric acid in biphasic reaction
- efforts for finding solid-acid catalyst has been going on for decades, $\text{SbF}_5$ in acid washed silica, triflic acid on porous support…
- in 2015, first sold acid unit was commercialized by Albemarle “AlkyClean”
**Alkylation: formation of longer chain alkanes from volatile shorter alkane/ene**

Acid catalyzed by sulfuric or hydrofluoric acid in biphasic reaction

Efforts for developing solid-acid catalyst has been going on for decades

$\text{SbF}_5$ in acid washed silica, triflic acid on porous support…

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**STRATCO**

**Contactor™ Reactor**

A - Contactor Reactor Shell
B - Tube Bundle Assembly
C - Hydraulic Head Assembly
D - Motor, Turbine/Driver

http://www.dupont.com
Benzene to chemicals

- Ethyl benzene → styrene → polystyrenes
- Phenol
- Acetone
- Bisphenol A → solvents → epoxy resins → polycarbonate
- Cumene → acetone
- Cyclohexane
- Adipic acid
- Caprolactum → nylons
- Nitrobenzene → aniline
- Methylene diphenyl diisocyanate → polyurethanes
- Alkyl benzene → detergents
- Chlorobenzene
- Gas phase Friedel Craft: phosphoric acid has been replaced by zeolites
- Liquid phase oxidation with air in liquid phase to give phenol and acetone as a byproduct
- Decomposition of the peroxide in presence of acid
Benzene to chemicals

- Ethyl benzene → Styrene → Polystyrenes
- Cumene
  - Phenol → Epoxy resins
  - Acetone → Polycarbonate
  - Bisphenol A → Solvents
- Benzene
  - Cyclohexane → Adipic acid → Nylons
  - Caprolactum
- Nitrobenzene → Aniline → Methylene diphenyl diisocyanate → Polyurethanes
- Alkyl benzene → Detergents
- Chlorobenzene

Chemical reaction:

\[
\begin{align*}
\text{HO-CH}_2\text{CH}_3 + \text{CO} + \text{HO-CH}_2\text{CH}_3 \\
&\xrightarrow{\text{H}^+} \text{H}_2\text{O} \\
\text{HO-C}_6\text{H}_4\text{OH} \rightarrow \text{HO-C}_6\text{H}_4\text{CH}_3\text{CH}_3\text{CH}_3\text{OH}
\end{align*}
\]
Aromatics to chemicals

Less applications compared to benzene and xylenes

Trans-alkylation (disproportionation): gas phase conversion of toluene to benzene and xylenes

Zeolite catalysis
Aromatics to chemicals

Where do these aromatics come from?

Homogeneous catalyst Co, Mn bromide + HBr
Acetic acid solvent
Highly corrosive, slow reaction...expensive alloy, large volume

Solution: titanium cladding

Where do these aromatics come from?
Aromatics to chemicals

Xylenes separation
Simulated Moving Bed SMB, using zeolite
https://www.youtube.com/watch?v=miUgGJO8ptc
Xylenes separation by SMB

Oil to Olefins

Naphtha again...”cracking” this time
- high temperature fire heater reactors
- besides Naphtha, LPG and ethane are often used too
Ethylene to ethylene oxide

- Unique ability of silver to molecularly adsorb O₂
- Adsorbed atomic chlorine favors this ads. mode versus the dissociative mode
- Maximum achievable selectivity is 80%
- High silver loading (up to 15%) on alumina or other oxide supports

https://www.industry.usa.siemens.com
Benzene to chemicals

- propylene
  - isopropyl alcohol
  - acrylonitrile
  - polypropylene
  - propylene oxide
    - polyol
    - propylene glycol
    - glycol ethers
    - acrylic acid
    - acrylic polymers
    - allyl chloride
    - epichlorohydrin
    - epoxy resins
Benzene to chemicals

SOHIO Ammoxidation process:

\[ \text{Propylene} + \text{NH}_3 + \frac{3}{2} \text{O}_2 \rightarrow \text{HCN} + 3 \text{H}_2 \text{O} \]


- HCN and MeCN are the two main byproducts
Opportunities for org. chemistry in renewables?

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> 1920 acetylene (from coal)
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CO₂ and biomass

Explosion at Oppau 1921
http://www.reimerei.net/Oppauammoniak_(FRB).htm

A number of accidents lead most countries to prohibit work with high P acetylene, BASF limit was 1.5 bar

Reppe’s goal: use acetylene produced from CaC₂ as a primary building block

Reppe wants to go higher than 25 bar

Reppe focuses on developing equipment for safe operation to allow for studying the chemistry:
  - tube reactors instead of vessels
  - stainless steel (to avoid explosive salts formation with copper…)
  - delivery in a plurality of smaller parallel tubes
  - dilute with nitrogen

Walter Reppe (1892-1969)
Photo: www.euchems.org
PhD in Chemistry 1921

http://www.reimerei.net/Oppauammoniak_(FRB).htm
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BASF butanediol unit today with the reactors built under Walter Reppe in 1943
Photo: www.basf.de

Reppe’s efforts lead to a number of industrial processes

Walter Reppe (1892-1969)
Photo: www.euchems.org
PhD in Chemistry 1921

\[
\begin{align*}
\text{HC≡CH} + \text{ROH} & \xrightarrow{\text{cat}} \text{HOR} \\
\text{HC≡CH} + \text{HCN} & \xrightarrow{\text{cat}} \text{HCN} \\
\text{HC≡CH} + \text{HCl} & \xrightarrow{\text{cat}} \text{HCl} \\
\text{HC≡CH} + \text{RCHO} & \xrightarrow{\text{cat}} \text{RC(OH)R} \\
\text{HC≡OH} + \text{RCHO} & \xrightarrow{\text{cat}} \text{HO-C≡C-OH}
\end{align*}
\]
Coal and NG to chemicals

Diagram of the Lurgi Dry-Ash Gasifier
How to make Aspirin in the era of petrochemicals?
Opportunities for org. chemistry in renewables?

➢ Small molecules: active ingredients in pharma and agro applications, pigments, flavors & fragrance, sealants & coatings, organometallics…

➢ Big molecules: peptides, proteins… mwt > 700

• Batch STR reactors, multipurpose production lines
• Homogeneous and bio catalysis
• In-house production or outsourced (CM)
• Besides petrochemicals, extraction from plants, animals and bioproduction is common

• R&D activities are designing the synthetic routes and to a lesser extent scale-up and optimization (5 to 10%)

Small to large size companies: Lonza, Evonik-Degussa, Bayer, Millipore Sigma, Monsanto
Opportunities for org. chemistry in renewables?

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| Bulk chemicals | Fine chemicals | Specialty chemicals |

Chemical(s) to serve a certain function: lubrication, adhesive, surfactant, cosmetics…

When in mixture called formulation.
Batch reactions

R&D efforts is to deliver better function rather than a certain chemical >10%
BASF, AkzoNobel, Lubrizol, Albemarle, Cabot
Two arms of pharmaceutical industry:

1. medicinal chemistry (high throughput screen, SAR)
2. process chemistry (economic, safe, reproducible, scalable and green)

Routes serving these two arms can be different due to different goals

Need for GCMP is driven by: regulations and the increasing production cost (20% in 10 years):
• Innovative engineering solutions: flow chemistry, biocatalysis, high pressure…
• Standardized measurable evaluation criteria for production routes:

1. Materials cost: reactants, solvent, catalyst, additives, eluents, extractants….are they sustainable?
2. Atom economy

\[ AE = \frac{\text{MW(product)} \times 100%}{\sum \text{MW(raw materials)}} \]

Route 1: 24%
Route 2: 75%

Standardized measurable evaluation criteria for production routes:

1. Materials cost: reactants, solvent, catalyst, additives, eluents, extractants…are they sustainable?
2. Atom economy > 70%
3. Yield, based on limiting reactant. Convergent routes are more favored than sequential.

Besides loss of materials, the formed side products can be costly in terms of reactor cleaning and waste disposal, e.g. genotoxic or explosive impurities.

4. Volume Time Output VTO

\[
VTO = \frac{\text{nominal volume of all reactors} [\text{m}^3] \times \text{time per batch} [\text{h}]}{\text{output per step} [\text{kg}]}
\]

4. **Volume Time Output VTO:** < 1 m³ h/kg

\[
VTO = \frac{\text{nominal volume of all reactors} \times \text{time per batch}}{\text{output per step}}
\]

*Reactor volume 100 m³
Op. cost 20 M USD/year
24h/day, 330 day/year
Capacity utilization 60%
Cost: 42 USD/m³. h*

\[R= H, \ \text{VTO} = 8.2 \ m^3 \ h/kg\]
\[R= \text{Boc}, \ \text{VTO} = 0.1 \ m^3 \ h/kg\]

5. **E-factor or PMI**: 25-100

\[
E = \frac{\sum \text{mass of materials in [kg]} - \text{mass of product out [kg]}}{\text{mass of product out [kg]}}
= \frac{\text{mass of waste [kg]}}{\text{mass of product out [kg]}}
\] (5)

6. **Quality Service Level**: how reproducible a reaction is from batch to batch: QSL: >98%

7. **Process Excellence Index**: how reproducible a process is in terms of yield and cycle time: PEI > 98

# Process chemistry

## 8. Ecoscale

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Question</th>
<th>Criterion</th>
<th>Points</th>
<th>RCM Route 1</th>
<th>RCM Route 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Yield [%]</td>
<td>What is the yield?</td>
<td>&gt; 95%</td>
<td>10</td>
<td>5</td>
<td>8</td>
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<tr>
<td></td>
<td></td>
<td>80-95%</td>
<td>7</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>60-80%</td>
<td>3</td>
<td></td>
<td></td>
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<tr>
<td>2 Quality [A or Wt%] of Product by GC, HPLC etc.</td>
<td>What is the quality?</td>
<td>&gt; 98%</td>
<td>10</td>
<td>7</td>
<td>10</td>
</tr>
<tr>
<td></td>
<td></td>
<td>95-98%</td>
<td>7</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>&lt; 95%</td>
<td>3</td>
<td></td>
<td></td>
</tr>
<tr>
<td>3 Workup &amp; Purification</td>
<td>Filtration before final crystallization possible?</td>
<td>Yes</td>
<td>10</td>
<td>NA</td>
<td>NA</td>
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<tr>
<td></td>
<td>Easy separation of suspension?</td>
<td>No</td>
<td>0-9</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Easy drying in tumble or paddle dryer possible?</td>
<td>Yes</td>
<td>10</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>No</td>
<td>0-9</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4 Equipment</td>
<td>Multipurpose reactors suitable?</td>
<td>Yes</td>
<td>10</td>
<td>0</td>
<td>10</td>
</tr>
<tr>
<td></td>
<td></td>
<td>No</td>
<td>0-9</td>
<td></td>
<td></td>
</tr>
<tr>
<td>5 Reaction Time [h]</td>
<td>What is the reaction time?</td>
<td>&lt; 3 h</td>
<td>10</td>
<td>3</td>
<td>10</td>
</tr>
<tr>
<td></td>
<td></td>
<td>3-6 h</td>
<td>7</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>&gt;10 h</td>
<td>3</td>
<td></td>
<td></td>
</tr>
<tr>
<td>6 Reaction Temperature [°C]</td>
<td>What is the reaction temperature?</td>
<td>Room T</td>
<td>10</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>&lt; 90°C</td>
<td>8</td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>90-150°C</td>
<td>5</td>
<td>8</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td></td>
<td>&gt; 150°C</td>
<td>3</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>&lt; -10°C</td>
<td>3</td>
<td></td>
<td></td>
</tr>
<tr>
<td>7 Raw Materials</td>
<td>Is chlorinated solvent used?</td>
<td>No</td>
<td>10</td>
<td>0</td>
<td>10</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Yes</td>
<td>0-9</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Price for solvents &lt; $7/kg?</td>
<td>Yes</td>
<td>10</td>
<td>10</td>
<td>10</td>
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<tr>
<td></td>
<td></td>
<td>No</td>
<td>0-9</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>All components are commodities?</td>
<td>Yes</td>
<td>10</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td></td>
<td></td>
<td>No</td>
<td>0-9</td>
<td></td>
<td></td>
</tr>
<tr>
<td>8 EHS</td>
<td>Reaction highly exothermic?</td>
<td>No</td>
<td>10</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td></td>
<td>Hazardous or toxic material needed?</td>
<td>No</td>
<td>10</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Yes</td>
<td>0-9</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Highly flammable or explosive material needed?</td>
<td>No</td>
<td>10</td>
<td>10</td>
<td>10</td>
</tr>
</tbody>
</table>

| TOTAL                            | 73                                            | 103             |
Process chemistry

Proposed weight of different factors by BI
Complete overview!

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formulations, recipes...
Process chemistry

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• Standardized measurable evaluation criteria for production routes:
RESEARCH AND INDUSTRIAL ORGANIC CHEMISTRY

By Professor JAMES F. NORRIS
MASSACHUSETTS INSTITUTE OF TECHNOLOGY

The research was made by the division to bring about cooperative research in certain industries. The response by executives was not, at first, encouraging. The president of a large petroleum company told me it was not the business of the chemist to meddle in the affairs of the industry. His company had millions of dollars invested in plants. It was satisfied with the results. He did not want anybody to do anything to make these plants obsolete.

A high executive in another industry told me he did not believe it good business to spend money on research to develop new products or methods. He had found it better to let some one else bear the expense of such work, most of which proved of no value. If anything worth while came out of it he could afford to buy it. What a change has come about in seven years. The two industries to which I have just referred are now spending millions of dollars in research.

In recent years the methods and personnel of the industrial research laboratories have changed rapidly. The empirical approach to the solution of a problem has largely disappeared and has been replaced by coordinated investigation carried out with the use of the scientific methods and with a knowledge of the latest findings and instruments of fundamental science. The industrial laboratories keep informed as to the new knowledge resulting from research in the universities. Requests are received from such laboratories for reprints of papers which apparently are only of theoretical interest.

The change in the industrial laboratories has come about as the result of a change in personnel. I was consulted several years ago in regard to the appointment of a director of a proposed research laboratory for a chemical industry, which was conducted largely by rule-of-thumb methods. The directors insisted on the appointment of a well-informed organic chemist, preferably a university professor, who had no experience in the industry which he was expected to develop. In this case there was good judgment in the point of view.

Opportunities are rapidly decreasing for the advancement in his profession of the chemist whose scholastic experience is limited to a four years' course leading to the degree of bachelor of science. To get ahead he must put chemistry behind him and become a manager or executive. The men to-day who are developing the industry are some of the best of those chemical equilibrium and ionization, catalysis, x-rays, and different types of energy with varying intensity factors—these and other important facts and generalizations have broadened the methods of research in organic chemistry, and have been applied in the solution of industrial problems.

For the research chemist who enters the industrial field a knowledge is important of the principles of chemical engineering which follow from the application of those laws of physics and physical chemistry which have to do with the physical relationships between molecules.

Those who direct the education of students of organic chemistry should see to it that the preparation for future work includes training in the principles of physical chemistry and their use. It was not so long ago that one of the leading universities of the country had no requirement in physical chemistry for men awarded the doctorate in organic chemistry. Happily, conditions are improving.

The growth of the use of research in the industries based on chemistry is indicated by the rapid increase in the number of men awarded the degree of doctor of philosophy in American universities. A compilation made by the National Research Council shows that much larger number of these degrees is in the field of chemistry. Many of the young men awarded this degree have entered industrial research laboratories, where they are happily at work on problems of great interest, are supplied with every facility.
Enabling Technologies

Laboratory Experiment

Student-Fabricated Microfluidic Devices as Flow Reactors for Organic and Inorganic Synthesis

Z. Vivian Feng*, Kate R. Edelman, and Benjamin P. Swanson
Chemistry Department, Augsburg College, Minneapolis, Minnesota 55454, United States

DOI: 10.1021/ed5005307
Publication Date (Web): January 16, 2015
Copyright © 2015 The American Chemical Society and Division of Chemical Education, Inc.

*E-mail: feng@augsburg.edu.

Figure 7. Synthesis of azo dyes: (a) 2-hydroxy-5-(2-phenyl diazenyl)-benzoic acid, and (b) 4-(2-phenyl diazenyl)phenol in PDMS flow reactors.
The Hitchhiker’s Guide to Flow Chemistry

Matthew B. Plutschack§†, Bartholomäus Pieber§†, Kerry Gilmore*†, and Peter H. Seeberger*†‡,
† Department of Biomolecular Systems, Max-Planck Institute of Colloids and Interfaces, Am Mühlenberg 1, 14476 Potsdam, Germany
‡ Institute of Chemistry and Biochemistry, Department of Biology, Chemistry and Pharmacy, Freie Universität Berlin, Arnimallee 22, 14195 Berlin, Germany

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Resources

bulk and commodity chemicals

Fine chemicals