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“Catalyzing and enabling the implementation of green chemistry and engineering throughout the global chemistry enterprise.”

Science
Advancing research & development for global challenges

Education
Advocating progress in education & communication of green chemistry principles

Industry
Accelerating industrial adoption of green chemistry

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Evan Thackaberry, Senior Scientist, Genentech
Peter Wueffling, Principal Scientist and Director, Merck

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“How to Create a Safer and More Sustainable Lab Through Green Chemistry”

David Finster
Professor of Chemistry and Chemical Hygiene Officer, Wittenberg University

Jeffrey Whitford
Director of Global Citizenship, Sigma-Aldrich

Joseph Fortunak
Professor of Chemistry, Howard University

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Co-produced with the ACS Green Chemistry Institute
How to Create a Safer and More Sustainable Lab Through Green Chemistry

Green Chemistry

Safe chemistry is motivated by...

... a desire to protect lab and industrial workers from the harmful effects of chemicals and chemical processes.

(The “OSHA” side of chemistry.)

Green (or sustainable) chemistry is motivated by...

... a desire to protect the environment as the “chemistry is done”.

(The “EPA” side of chemistry.)

Is there a connection between chemical safety and green chemistry?
Overview

Review of the 12 Principles of Green Chemistry

... and how they connect to in-lab (or in-plant) chemical safety – or not.

Inherently Safer Design and Green Chemistry

Green Chemistry as an integral step in lab safety

Audience Survey Question

How many of the 12 Principles of Green Chemistry, when followed in the lab, necessarily also make the lab a safer environment?

- 4
- 7
- 10
- 12
Well, some of this should be obvious... Green Principle #12:

#12. Inherently Safer Chemistry for Accident Prevention

Substances and the form of a substance used in a chemical process should be chosen to minimize the potential for chemical accidents, including releases, explosions, and fires.

The implied goal here is to protect the environment but, as this goal is addressed, the lab and manufacturing facility also become safer for employees.

“What Else?”

#1 Prevention

It is better to prevent waste than to treat or clean up waste after it has been created.

Wastes present hazards to individuals.

Less waste \(\rightarrow\) safer for employees.
"What Else?"

#2 Atom Economy

Synthetic methods should be designed to maximize the incorporation of all materials used in the process into the final product.

\[
\text{eg, Wittig Reaction}
\]

\[
\begin{align*}
\text{C}_6\text{H}_{12} \text{O} & \xrightarrow{\text{TlC}_2\text{H}_5} \text{C}_6\text{H}_{12} \\
\text{MW 98} & \text{MW 276}
\end{align*}
\]

\[
\begin{align*}
\text{C}_6\text{H}_{12} & \xrightarrow{\text{Cl}_2} \text{ClCH}_2\text{CH}_2\text{Cl} \\
\text{MW 96} & \text{MW 278}
\end{align*}
\]

% Atom Economy = (MM of atoms utilized / MM of all reactants) \times 100%
% Atom Economy = (96/374) \times 100% = 26%

vs.

\[
\text{CH}_2=\text{CH}_2 + \text{Cl}_2 \rightarrow \text{ClCH}_2\text{-CH}_2\text{Cl}
\]

% Atom Economy = 100%

Fewer (potentially) hazardous by-products \rightarrow safer for employees.

"Safe Chemistry is Green Chemistry"

#3 Less Hazardous Chemical Syntheses

Wherever practicable, synthetic methods should be designed to use and generate substances that possess little or no toxicity to human health and the environment.

Less toxic substances \rightarrow safer for employees.
“Safe Chemistry is Green Chemistry”

#4 Designing Safer Chemicals
Chemical products should be designed to effect their desired function while minimizing their toxicity.

Less toxic substances \(\rightarrow\) safer for employees.

“Safe Chemistry is Green Chemistry”

#5 Safer Solvents and Auxiliaries
The use of auxiliary substances (e.g., solvents, separation agents, etc.) should be made unnecessary wherever possible and innocuous when used.

Safer (or no!) solvent(s)/auxiliary agents \(\rightarrow\) safer for employees.
“Safe Chemistry is Green Chemistry”

#6 Design for Energy Efficiency

Energy requirements of chemical processes should be recognized for their environmental and economic impacts and should be minimized. If possible, synthetic methods should be conducted at ambient temperature and pressure.

Ambient T and P $\rightarrow$ safer for employees.

“Safe Chemistry is Green Chemistry”

#8 Reduce Derivatives

Unnecessary derivatization (use of blocking groups, protection/deprotection, temporary modification of physical/chemical processes) should be minimized or avoided if possible, because such steps require additional reagents and can generate waste.

Fewer reagents/intermediates $\rightarrow$ fewer hazards $\rightarrow$ safer for employees.
"Safe Chemistry is Green Chemistry"

#9 Catalysis

Catalytic reagents (as selective as possible) are superior to stoichiometric reagents.

![Energy diagram](image)

**Milder conditions ➔ safer for employees.**

"Safe Chemistry is Green Chemistry"

#11 Real-time analysis for Pollution Prevention

Analytical methodologies need to be further developed to allow for real-time, in-process monitoring and control prior to the formation of hazardous substances.

"You cannot control what you cannot measure."

![Cardiogram](image)

**Prevention of real-time, in-process hazards ➔ safer for employees.**
“Safe Chemistry is Green Chemistry”

Less Important Principles with Respect to Employee Safety:

#7 Use of Renewable Feedstocks

A raw material or feedstock should be renewable rather than depleting whenever technically and economically practicable.

#11 Design for Degradation

Chemical products should be designed so that at the end of their function they break down into innocuous degradation products and do not persist in the environment.

So: 10 of the 12 principles of Green Chemistry also protect people in the lab and in manufacturing facilities.

“Green Chemistry is Safe Chemistry”

Let’s reverse this: Is there any instance where making the lab safer for workers (“OSHA”) compromises a Green Chemistry Principle or otherwise harms the environment?

Lab/Plant Exhaust:

Using Chemical hoods

Plant Ventilation

A Sampling of Presidential Green Chemistry Awards

<table>
<thead>
<tr>
<th>Year</th>
<th>Award</th>
<th>Process</th>
<th>Safety Aspect</th>
</tr>
</thead>
<tbody>
<tr>
<td>2005</td>
<td>Synthetic Pathways</td>
<td>Efficient Synthesis of a Chemotherapy Anti-Nausea Drug</td>
<td>Eliminates operational hazards: sodium cyanide, dimethyl titanocene, &amp; gaseous ammonia</td>
</tr>
<tr>
<td>2006</td>
<td>Small Business</td>
<td>Safe Solvents &amp; Reclamation in Flexographic Printing</td>
<td>Elimination and reduction of hazardous solvents</td>
</tr>
<tr>
<td>2007</td>
<td>Reaction Conditions</td>
<td>Synthesis of H₂O₂ by Selective Nanocatalyst</td>
<td>Eliminates hazardous chemicals &amp; reaction conditions</td>
</tr>
<tr>
<td>2008</td>
<td>Academic</td>
<td>Preparing Boronic Esters</td>
<td>Eliminates solvent &amp; halides</td>
</tr>
<tr>
<td>2008</td>
<td>Small Business</td>
<td>Stabilized Alkali Metals</td>
<td>Reduces hazard by encapsulating alkali metals</td>
</tr>
<tr>
<td>2009</td>
<td>Synthetic Pathways</td>
<td>Solvent-Free Biocatalytic Process for Cosmetic and Personal Care Ingredients</td>
<td>Eliminates solvents, strong acid catalysts &amp; high temperatures</td>
</tr>
<tr>
<td>2009</td>
<td>Reaction Conditions</td>
<td>Protein Analysis without Hazardous Chemicals or High Temperatures</td>
<td>Eliminates sulfuric &amp; hydrochloric acid, sodium hydroxide, &amp; metal catalysts</td>
</tr>
<tr>
<td>2010</td>
<td>Reaction Conditions</td>
<td>Synthesis of Sitagliptin for Treatment of Diabetes</td>
<td>Eliminates high-pressure hydrogenation</td>
</tr>
<tr>
<td>2011</td>
<td>Synthetic Pathways</td>
<td>Production of Basic Chemicals from Renewable Feedstocks</td>
<td>Eliminates organic solvent, high pressure, &amp; high temperature</td>
</tr>
<tr>
<td>2012</td>
<td>Reaction Conditions</td>
<td>MAX HT® Bayer Sodalite Scale Inhibitor</td>
<td>Eliminates sulfuric acid &amp; hazardous mechanical tasks</td>
</tr>
<tr>
<td>2012</td>
<td>Synthetic Pathways</td>
<td>Biocatalytic Process to Manufacture Simvastatin</td>
<td>Eliminates hazardous reagents</td>
</tr>
</tbody>
</table>

"Inherently Safer Design and Green Chemistry Principles"

**Inherently Safer Design**

- **Substitute**: replacing one material with another that is less hazardous
- **Minimize**: reducing the amount of hazardous material in the process
- **Moderate**: using less hazardous process conditions such as lower pressures or temperatures
- **Simplify**: designing processes to be less complicated, and therefore less prone to failure.

**Green Chemistry Principles**

- # 2 Atom Economy
- # 4 Designing Safer Chemicals
- # 5 Safer Solvents and Auxiliaries
- # 6 Design for Energy Efficiency
- # 8 Reduce Derivatives
- # 9 Catalysis
- # 12 Inherently Safer Chemistry for Accident Prevention
**Audience Survey Question**

ANSWER THE QUESTION ON BLUE SCREEN IN ONE MOMENT

**Which strategy is most effective with regard to protecting chemists in labs?**

- Personal Protective Equipment (PPE)
- Lab Safety Training
- Engineering Controls (e.g., chemical hoods)
- Elimination of Lab Hazards

“Green Chemistry is Safe Chemistry”

Another way to see how Green Chemistry fits into laboratory safety from an OSHA perspective...

Adapted from: [https://www.osha.gov/dsg/safer_chemicals/why_transition.html](https://www.osha.gov/dsg/safer_chemicals/why_transition.html)


Contributed by Shelly Bradley, Campus Chemical Compliance Director, Hendrix College; Dr. David C. Finster, Professor of Chemistry, Wittenberg University, and Dr. Tom Goodwin, Elbert L. Fausett Professor of Chemistry, Hendrix College
Simple Techniques to Make Everyday Lab Work Greener

### Solvent Selection

1. **Use dry ice/isopropanol for cooling baths**
   - Makes assembly the same temperature as dry ice baths (73°C to −70°C), but the lower volatility of isopropanol minimizes vapor emissions and inhalation, and makes the bath last longer.

2. **Use heptane instead of hexanes**
   - Heptane has almost identical chemical properties to hexane, but is significantly less toxic due to the odd number of carbon atoms, which affect its metabolic product in the body.

3. **Use 2-MeTHF instead of THF**

4. **Substitute DCM in column chromatography**
   - One of the largest contributors to contaminated solvent waste is chromatography. While selecting a new solvent system may seem challenging, J. P. Tognotti, et al. (Green Chem. 2012, 14, 3012-3022) have already done the work for you.

### Waste Reduction

5. **Recycle wash solvents**
   - Wash solvents are ideal for reusing because they are typically too insoluble. Simply wash your glassware as usual, collecting the liquid in a separate container. When full, transfer to the fume hood and drip into a cleanup flask.

6. **Recycle solvents isolated from distillation/rotovaping**
   - If you are going to remove the solvent from the distillate, why not reuse it? When you are done your purification, go to a quick check of the purity of the solvent. Then, pour it in another reaction or as a wash solvent. This is ideal for small solvent systems.

7. **Use a closed-loop cooling system for condensers**
   - Closed-loop cooling systems eliminate wastewater and accidental laboratory flooding. Use a commercially available closed-loop water recirculator, an aluminum condenser, or for high-boiling liquids simply use air.

8. **Use Dry Column Vacuum Chromatography to purify large samples**
   - This is a relatively new technique that can dramatically reduce the amount of solvents used. For larger purifications, it’s better than flash chromatography and the columns can be easily recycled. For more information see D. J. Pelchen and E. S. Reed, Anal. Chem. 2003, 75, 2453–2458.

### Energy Reduction

9. **Close your fume hood**
   - A variable volume fume hood will save 60% more energy-efficient when the sash is closed.

10. **Turn off/unplug stuff when you are done with it**
    - It just makes sense.

---

**Conclusion(s):**

"**Green Chemistry is Safe Chemistry**"

and

"**Safe Chemistry is Green Chemistry**"
Our Purpose

SIGMA-ALDRICH

Maximizing  Minimizing
What are the biggest opportunity areas for Green Chemistry?

- More engagement among scientists.
- Better products to implement Green Chemistry.
- More information about “greeness” and what makes a product “greener.”
- Spurring innovation – new ideas we have not conceived yet.

**Greener Chemistry: Roadmap**

**12 PRINCIPLES OF GREEN CHEMISTRY**

1. Waste Prevention
2. Atom Economy
3. Less Hazardous Chemical Syntheses
4. Designing Safer Chemicals
5. Safer Solvents and Auxiliaries
6. Design for Energy Efficiency
7. Use of Renewable Feedstocks
8. Reduce Derivatives
9. Catalysis
10. Design for Degradation
11. Real-time analysis for Pollution Prevention
12. Inherently Safer Chemistry for Accident Prevention

**SIGMA-ALDRICH'S HOLISTIC APPROACH**

- Reengineered Products/Product Scoring
- Product Portfolio Expansion
- Marketing
- Packaging/Labeling
- Shipping
- Use
- Education

Greener Alternatives Platform

3 CATEGORIES

- Re-Engineered
- 12 Principles Aligned
- Enabling Technologies
  - Enzyme & MatSci Products for Alternative Energy and Energy Storage

The Sigma-Aldrich Approach to Green Chemistry

Leverage Global Capability to Enable Greener Chemistry

9 Green Chemistry Centers
40 Scientists
30+ Products Re-Engineered
Greener Alternatives: Re-engineered Products

<table>
<thead>
<tr>
<th>PRINCIPLE</th>
<th>% IMPROVEMENTS</th>
<th>REMARKS</th>
</tr>
</thead>
<tbody>
<tr>
<td>#1 Waste Prevention</td>
<td>55</td>
<td>Eliminated organic solvent usage and reduced 50% overall waste generation</td>
</tr>
<tr>
<td>#2 Atom Economy</td>
<td>52</td>
<td>Increased yield and reduced amount of raw materials used</td>
</tr>
<tr>
<td>#3 Less Hazardous Synthesis</td>
<td>96</td>
<td>Replaced organic solvents with water-based solutions and removed toxic filtering agents</td>
</tr>
<tr>
<td>#5 Safer Solvents</td>
<td>100</td>
<td>Eliminated all organic solvent usage</td>
</tr>
<tr>
<td>#6 Energy Efficiency</td>
<td>100</td>
<td>New process eliminated need for elevated temperature and pressure</td>
</tr>
<tr>
<td>#7 Renewable Feedstock</td>
<td>71</td>
<td>More efficient use of sweet potatoes while reducing auxiliary chemicals</td>
</tr>
<tr>
<td>#10 Design for Degradation</td>
<td>No change</td>
<td>No increased environmental impact with new procedure</td>
</tr>
<tr>
<td>#12 Accident Prevention</td>
<td>54</td>
<td>Eliminated flammability and reactivity dangers</td>
</tr>
</tbody>
</table>

TOTAL 95%

AGGREGATE SCORE

 Previous Process Reengineered Process

SCORING MATRIX

100

SIGMA-ALDRICH
1-Aminobenzotriazole
Old Process

**STEP 1**
\[
\begin{align*}
\text{2-Nitramine} + \text{HCl} & \rightarrow \text{2-Nitro-aminobenzotriazole} \\
\text{2-Nitro-} & \text{aminobenzotriazole} + \text{HCl} \rightarrow \text{2-Nitro-aminobenzotriazole}
\end{align*}
\]

**STEP 2**
\[
\begin{align*}
\text{2-Nitro-aminobenzotriazole} & + \text{HCl} \rightarrow \text{2-Nitro-} \\
\text{aminobenzotriazole} & + \text{HCl} \rightarrow \text{2-Nitro-aminobenzotriazole}
\end{align*}
\]

**STEP 3**
\[
\begin{align*}
\text{2-Armct} & + \text{AlCl}_3 \rightarrow \text{2-Armct}
\end{align*}
\]

**STEP 4**
\[
\begin{align*}
\text{2-Armct} & + \text{AlCl}_3 \rightarrow \text{2-Armct}
\end{align*}
\]

**STEP 5**
\[
\begin{align*}
\text{2-Armct} & + \text{AlCl}_3 \rightarrow \text{2-Armct}
\end{align*}
\]

Greener Alternatives:
Re-engineered Products

### 1-AMINOBENZOTRIAZOLE

<table>
<thead>
<tr>
<th>PRINCIPLE</th>
<th>% IMPROVEMENTS</th>
<th>REMARKS</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Waste Prevention</td>
<td>75</td>
<td>Reduced 40% of organic solvent usage</td>
</tr>
<tr>
<td>2. Atom Economy</td>
<td>72</td>
<td>Increased the yield and reduced amount of raw materials used</td>
</tr>
<tr>
<td>3. Less Hazardous Synthesis</td>
<td>72</td>
<td>Eliminated the hazardous hydrogenation procedure</td>
</tr>
<tr>
<td>4. Safer Solvents</td>
<td>72</td>
<td>Used lesser volume of solvents</td>
</tr>
<tr>
<td>5. Energy Efficiency</td>
<td>77</td>
<td>Eliminated the heating in the procedure</td>
</tr>
<tr>
<td>6. Renewable Feedstock</td>
<td>72</td>
<td>Used renewables and reduced auxiliary chemicals</td>
</tr>
<tr>
<td>7. Accident Prevention</td>
<td>96</td>
<td>Eliminated the PLC use which in turn eliminated the potential for accident</td>
</tr>
<tr>
<td><strong>TOTAL</strong></td>
<td><strong>77%</strong></td>
<td></td>
</tr>
</tbody>
</table>

**AGGREGATE SCORE**

<table>
<thead>
<tr>
<th>Previous Process</th>
<th>Reengineered Process</th>
</tr>
</thead>
<tbody>
<tr>
<td>94</td>
<td>22</td>
</tr>
</tbody>
</table>

**SCORING MATRIX**

<table>
<thead>
<tr>
<th>10</th>
<th>9</th>
<th>8</th>
<th>7</th>
<th>6</th>
<th>5</th>
<th>4</th>
<th>3</th>
<th>2</th>
<th>1</th>
</tr>
</thead>
</table>

1 = most desirable
Greener Alternatives: Aligned

Reduce the generation of Waste and CO₂ emissions with Cyclopentyl methyl ether (CPME)

**CPME PROCESS**

- **REACTION**
  - Product: CPME + THF → THF + CPME
  - Reaction: CPME + THF → THF + CPME
- **EXTRACTION**
  - Product: THF + THF + CPME
  - Recovery: CPME + THF + THF
- **CONCENTRATION**
  - Product: THF + THF + CPME
  - Recovery: CPME + THF + THF
- **PRODUCT:**
  - Recovery rate: THF + THF + CPME

**THF PROCESS**

- **REACTION**
  - Product: THF + THF + CPME
  - Reaction: THF + THF + CPME
- **EXTRACTION**
  - Product: THF + THF + CPME
  - Recovery: THF + THF + CPME
- **CONCENTRATION**
  - Product: THF + THF + CPME
  - Recovery: THF + THF + CPME
- **PRODUCT:**
  - Recovery rate: THF + THF + CPME

<table>
<thead>
<tr>
<th>Recovery rate</th>
<th>Waste</th>
<th>CO₂*</th>
</tr>
</thead>
<tbody>
<tr>
<td>THF</td>
<td>93%</td>
<td>14.8 MT</td>
</tr>
<tr>
<td>CPME</td>
<td>99%</td>
<td>0.2 MT</td>
</tr>
<tr>
<td>Natural Capital Savings</td>
<td>11.5 MT Reduction</td>
<td>1.8 MT</td>
</tr>
</tbody>
</table>

*The figures on CO₂ emissions were calculated from incineration of waste streams.

What’s Coming Next

**ALTERNATIVE SOLVENTS:**

- Rebased NMP/DMF Replacement
- Rebased Acetic Acid
- Rebased Iobutanol
- Rebased Paraxylene

---

Greener Alternatives: Aligned

**REACTIONS IN WATER – JUST ADD YOUR SUBSTRATE**

Introducing a new single-use kit for the facile set-up of commonly used cross-couplings and other reactions done in water using the Lipshutz surfactant, TPFS-750-M.

**Kit Components**

- Reaction vial with stir bar
- Premixed additives and precatalysts
- TPFS-750-M in water
- Syringe and septum cap
- Fume hood magnet with standard instructions

**Products Offered**

- Suzuki-Miyaura Coupling
- Buchwald-Hartwig Amination
- Sonogashira’s Coupling
- Heck Coupling
- Stille Coupling
- Negishi Coupling
- Miyaura Borylation
- Olefin Metathesis
Transparency at Sigma-Aldrich

Introducing a new level of transparency regarding the why we label a product as a greener alternative.

1. Product category
2. DCOZN score (if applicable)
3. Up to 3 main drivers of “greener”
4. Link to 3rd party published paper on why it’s considered “greener”
5. Link to other products for easy navigability

You can find these products and more information at www.sigmaaldrich.com/greener
Customer Engagement

YOU DON’T KNOW WHAT ALL YOU USE UNTIL YOU DO.

Sigma-Aldrich Sustainability Opportunity Dashboards

HIGHLIGHTING:
• Potential Solvent Switches
• Packaging Improvements
• Polyethylene Coke Return Program
• Carbon Footprint Reduction Opportunities

We're starting the conversation on Green Chemistry in labs around the world.

---

Audience Survey Question
ANSWER THE QUESTION ON BLUE SCREEN IN ONE MOMENT

What do you think the biggest challenge holding back Green Chemistry implementation?

• My science works - why change it?
• It’s too expensive.
• Not enough experience with Green Chemistry.
• What are you talking about...we are green!
Green Chemistry Education Advocacy

WHY CHANGE WHAT WORKS?

- RESOURCE CONSTRAINT
- ENVIRONMENTAL CHALLENGES
- CHANGING CUSTOMER TASTES AND BUYING HABITS

GREEN CHEM EDUCATION PARTNERSHIP

SIGMA-ALDRICH® + beyondbenign

PILOT PHASE
Summer 2015 - May 2016

PHASE 2 EXPANSION
Summer 2016 - December 2016

You can find these products and more information at www.sigmaaldrich.com/greener
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Professor of Chemistry and Chemical Hygiene Officer, Wittenberg University

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Meredith Jolie, Attorney, Immigration Solutions Group, PLLC

Thursday, September 24, 2015
“Delivery Options to Support Dose Escalation in Preclinical Toxicology and Pharmacodynamic Activity Studies”
Evan Thackaberry, Senior Scientist, Genentech
Peter Wuelfing, Principal Scientist and Director, Merck

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