

# Implementation of Green Chemistry and Sustainable Lab Practices in the CU-Boulder Chemistry Department

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University of Colorado **Boulder**

# Learning Objectives

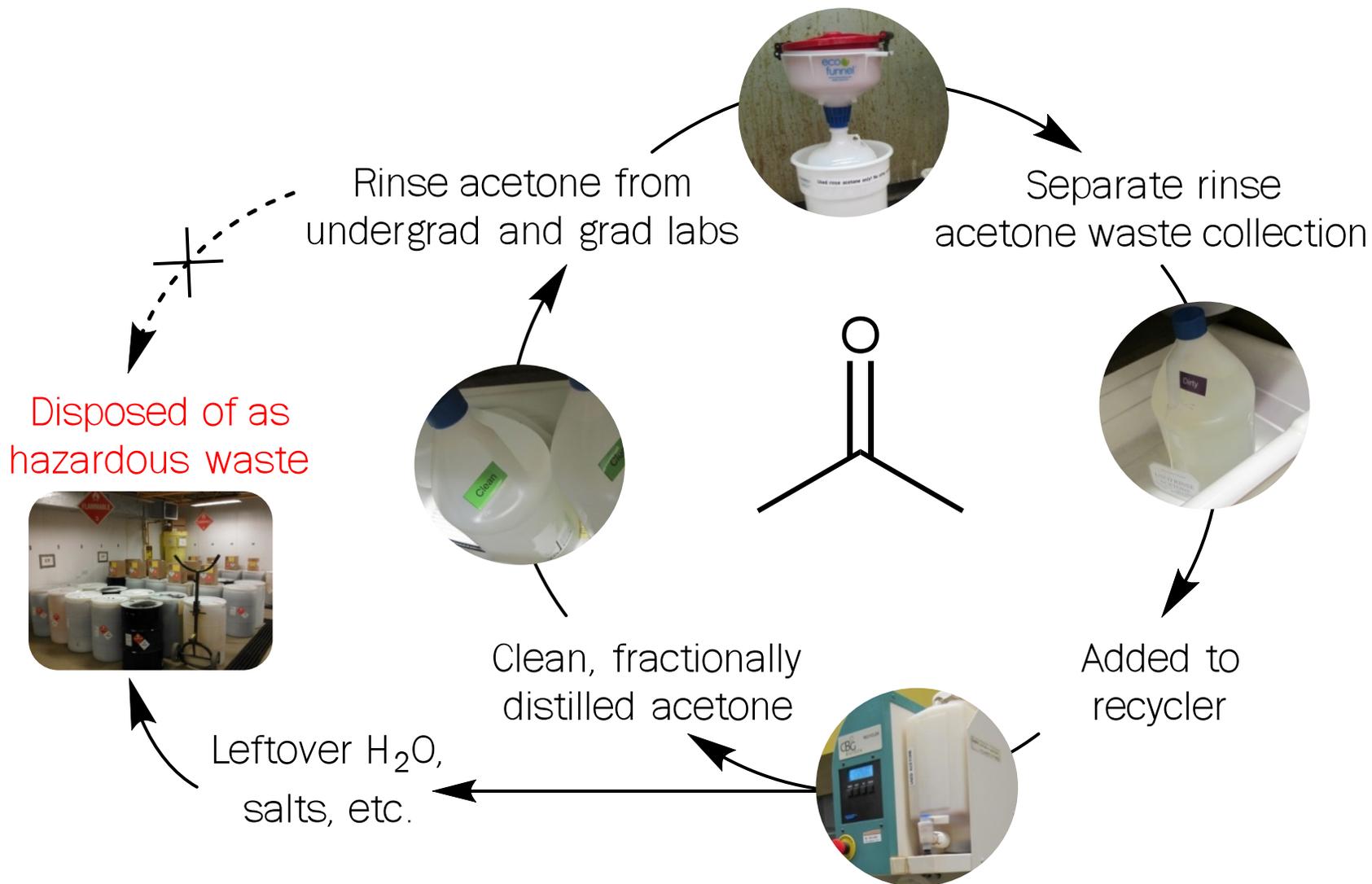
- Learn about green chemistry projects implemented at CU.
- Learn about the hazardous waste diversion and dollar savings resulting from those projects.
- Learn about impact those projects are having on raising awareness about green chemistry at CU.

# Solvent Recycling

- Fractional distillation unit purchased in June 2013 (CBG Biotech).
- Main target for solvent recycling is acetone, but other commonly used solvents may be addressed in the future

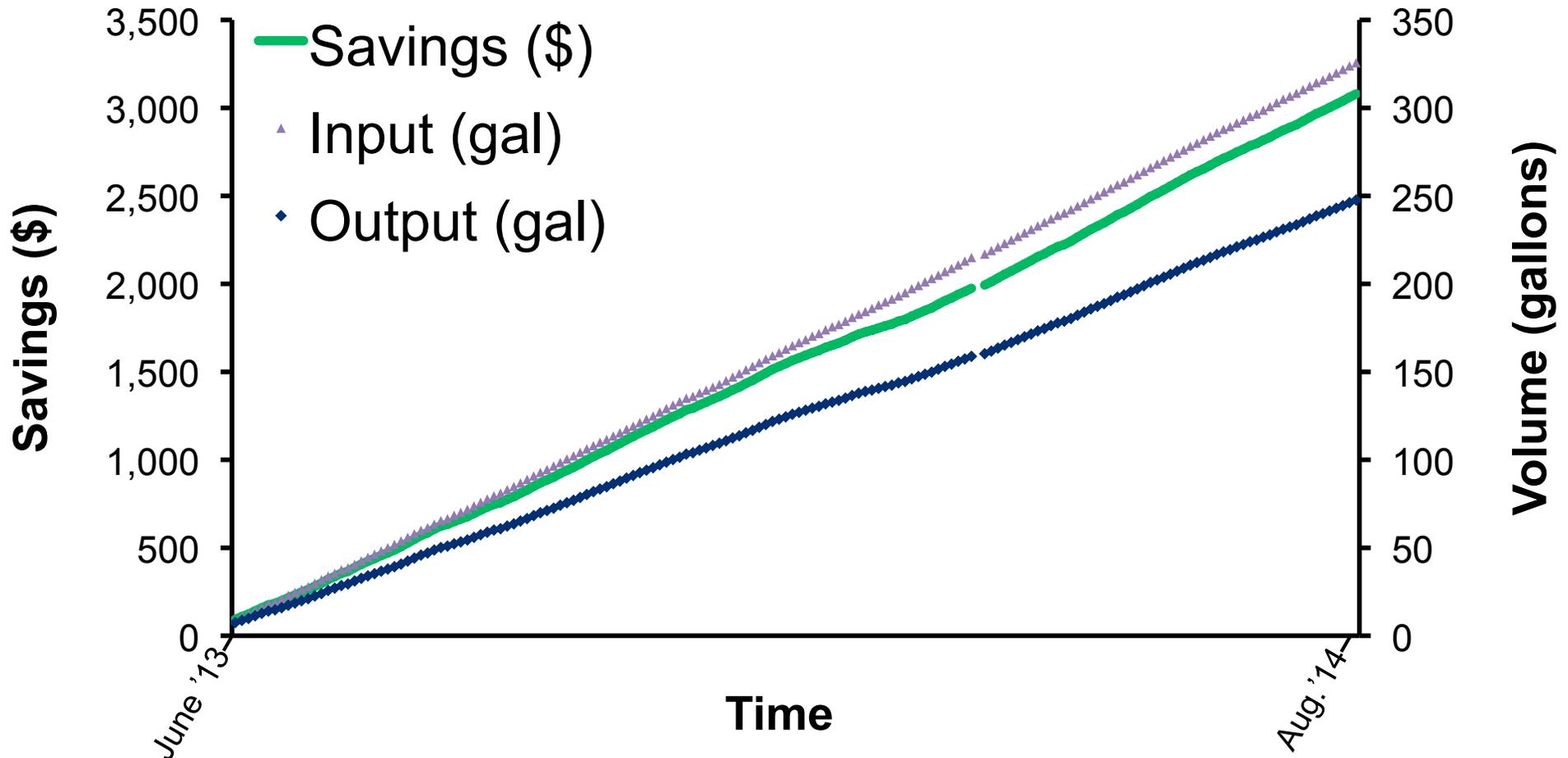


# Acetone Recycling: How it works



# Acetone Recycling: Savings

- Overall efficiency = 76.0%; overall savings = \$3,079.\*



\* Not including previous disposal costs



# Other Possible Solvents

- Common mixtures from column chromatography
  - Hexanes/ethyl acetate
    - **Testing in progress**
  - Dichloromethane/hexanes
  - Azeotrope formation could be problematic, i.e., resultant product could remain a mixture
- Other rinse solvent streams that are mixtures, i.e., acetone, methanol, and water



# Ethanol Reuse Program

- 120 gallons/year of used ethanol from cold traps was being disposed of as hazardous waste
- This volume is equivalent to a purchase price of \$1,560!
- Now this ethanol is being diluted and reused to wipe down biosafety cabinets in Biochemistry!



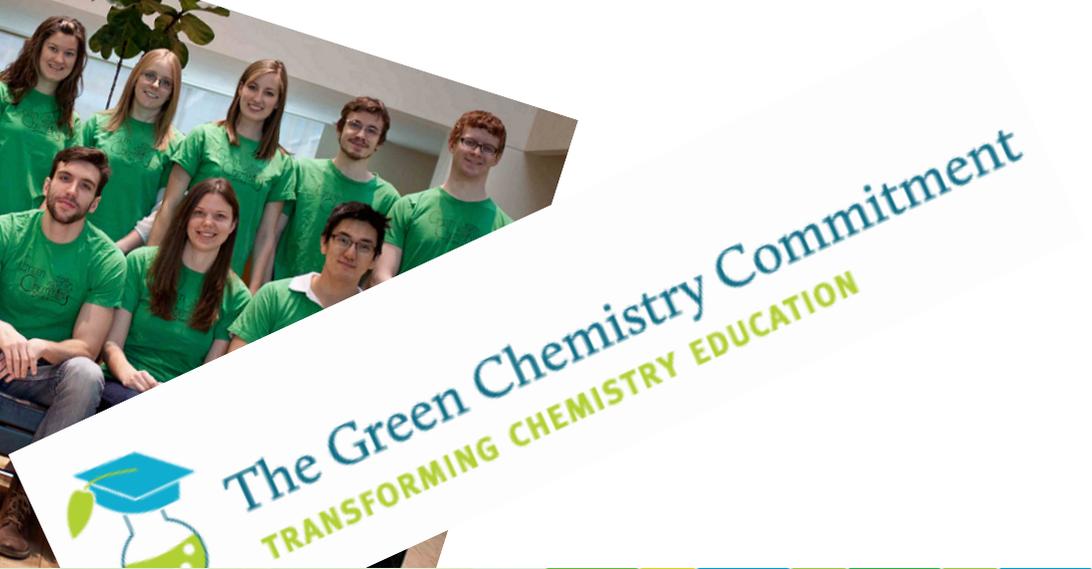
Ethanol Cold Trap



Biosafety Cabinet Sterilization



# Green Chemistry



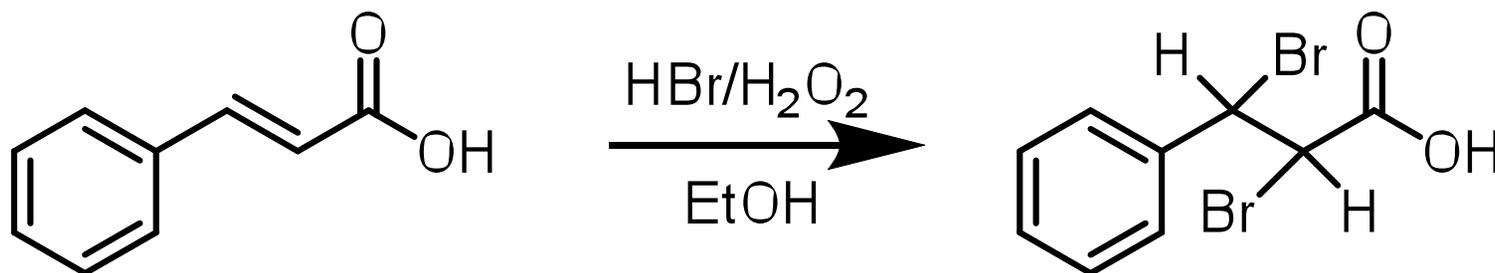
**Green Chemistry & Engineering:**  
Innovation for a Sustainable Future

# Green Chemistry in Teaching Labs

## *Endeavors*

- Scaling down of reactions
- Replacement of hazardous procedures
- Substitution for less hazardous reagents
- Undergraduate education about hazards, acetone recycling, etc.
- **A handful of reactions have been replaced and/or implemented**
- **Impacting up to 900 students/year**

# “A Greener Bromination of Stilbene”



*trans*-Cinnamic acid

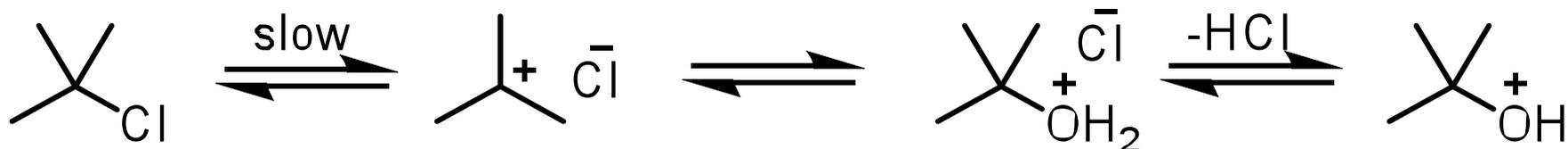
## Previously:

- Bromine solution, dichloromethane as solvent
- Large volumes of aq. sodium thiosulfate neutralized Br<sub>2</sub>

## Now:

- Br<sub>2</sub> generated in situ with H<sub>2</sub>O<sub>2</sub> and HBr, ethanol used as solvent, and no aq. sodium thiosulfate needed

# “Measuring solvent effects: kinetics of hydrolysis of tert-butyl chloride”



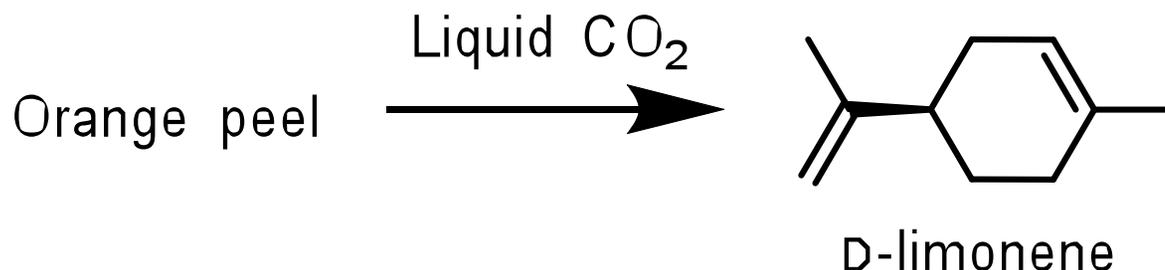
## Previously:

- Replaces an old kinetics lab, which generated less volume of waste, but students used more hazardous reagents (KOH pellets, volatile alkylhalides)

## Now:

- Milder reagents:
  - Uses recycled acetone and tap water as solvents
  - Prepared 0.04 M NaOH aqueous solution

# “CO<sub>2</sub> liquid extraction of limonene”



## Previously:

- Energy intensive steam distillation extraction procedure
- Required about 1 orange/student—high volume of discarded oranges

## Now:

- Liquid CO<sub>2</sub> is more efficient, new digital polarimeter allows lower detection of limonene → ~1 orange/5 students!

McKenzie, L. C. et al., *Green Chem.* **2004**, 6, 355–358.

# Green Chemistry in Research Labs

## *Endeavors*

- Solvent substitutions
- Findensers—water-free condensers
- Use of solvents from bio-based feedstocks as opposed to petroleum-based
- Green Chemistry Fund

# Solvent Substitutions

- A number of solvent selection guides are available

## Glaxo-Smith Kline

	Few issues (bp°C)	Some issues (bp°C)	Major issues
Chlorinated	...before using chlorinated solvents, have you considered TBME, isopropyl acetate, ethyl acetate, 2-Methyl THF or Dimethyl Carbonate?		
Greenest Option	Water (100°C)		
Alcohols	1-Butanol (118°C) 2-Butanol (100°C)	Ethanol/IMS (78°C) t-Butanol (82°C) Methanol (65°C)	1-Propanol (97°C) 2-Propanol (82°C) 2-Methoxyethanol **
Esters	t-Butyl acetate (95°C) Isopropyl acetate (89°C) Propyl acetate (102°C) Dimethyl Carbonate (91°C)	Ethyl acetate (77°C) Methyl acetate (57°C)	
Ketones		Methyl isobutyl ketone (117°C) Acetone (56°C) p-Xylene (138°C) Toluene ** (111°C)	Methyl ethyl ketone Benzene **
Aromatics		Isooctane (99°C) Cyclohexane (81°C) Heptane (98°C)	Petroleum spirit ** 2-Methylpentane Hexane
Hydrocarbons		t-Butyl methyl ether (55°C) 2-Methyl THF (78°C) Cyclopentyl methyl ether (106°C)	1,4-Dioxane ** 1,2-Dimethoxyethane ** Tetrahydrofuran Diethyl ether Diisopropyl ether **
Ethers			Dimethyl formamide ** N-Methyl pyrrolidone ** N-Methyl formamide ** Dimethyl acetamide ** Acetonitrile
Dipolar aprotics		Dimethyl sulfoxide (189°C)	

\*\* = EHS Regulatory Alerts: please consult the detailed solvent guide and the GSK Chemicals Legislation Guide for more information <http://solventguide.gsk.com/>

GSK SSG-MC-02 September 2010



## Pfizer

### Preferred

Water  
Acetone  
Ethanol  
2-Propanol  
1-Propanol  
Ethyl Acetate  
Isopropyl acetate  
Methanol  
MEK  
1-Butanol  
t-Butanol

### Usable

Cyclohexane  
Heptane  
Toluene  
Methylcyclohexane  
TBME  
Isooctane  
Acetonitrile  
2-MeTHF  
THF  
Xylenes  
DMSO  
Acetic Acid  
Ethylene Glycol

### Undesirable

Pentane  
Hexane(s)  
Di-isopropyl ether  
Diethyl ether  
Dichloromethane  
Dichloroethane  
Chloroform  
NMP  
DMF  
Pyridine  
DMAc  
Dioxane  
Dimethoxyethane  
Benzene  
Carbon tetrachloride

*Green Chem.* **2011**, *13*, 854–862.

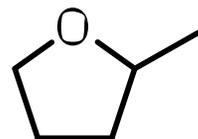
*Green Chem.* **2008**, *10*, 31–36.



# Solvent Substitutions

- Solvent substitutions are also available for chromatography, which uses high volumes of solvents, often chlorinated solvents and hexanes or ethyl acetate and hexanes.
- References:
  - *Green Chem.* **2012**, *14*, 3016–3019.
  - *Green Chem.* **2012**, *14*, 3020–3025.
- Also, alternative types of chromatography have been tested that use less solvent:
  - *Synthesis* **2001**, *25*, 2431–2434; *Syn. Lett.* **2014**, *25*, 0058–0063.

# Bio-based solvents



2-Methyltetrahydrofuran (2-MeTHF)

## *Advantages*

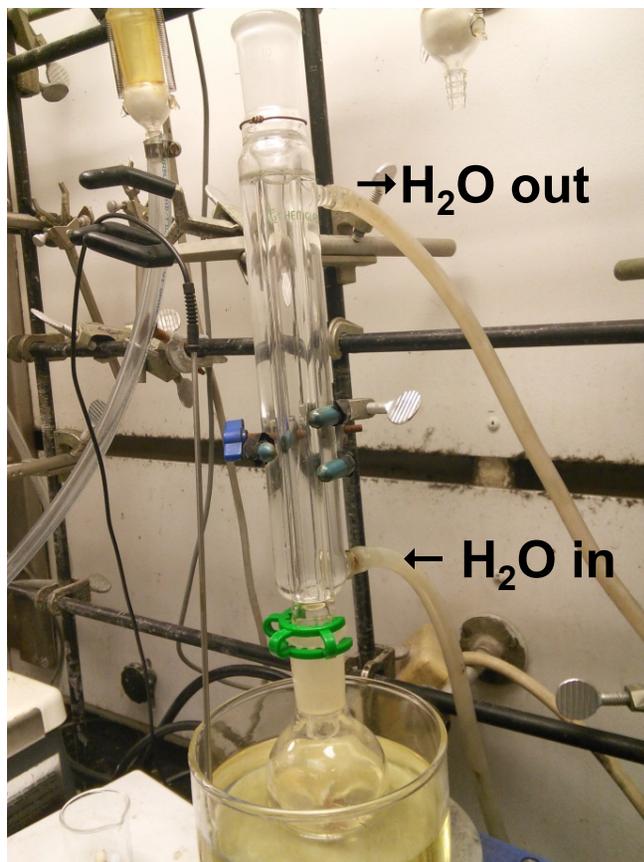
- Substitute for petrol-based tetrahydrofuran (THF)
- Immiscible with water
- Reactions may proceed better

## *Cons*

- Much more expensive
  - Not enough demand
- 

# Water-free Condensers

- Save water, eliminate flooding



**Normal condenser**



**Water-free "Findenser"**

# FY '14 - '15 Green Chemistry Fund

- \$5,000 fund to cover cost of materials for lab members to try their ideas
- Committee of grad students, EH&S, & CU Green Labs to review requests
- Successes will be publically shared with campus
- Grow the green chemistry culture on campus and get labs involved in coming up with ideas
- Ideas that committee could also promote:
  - Replace EtBr with less toxic products for DNA viewing
  - Safer & greener alternatives to Piranha acid baths
  - Mercury free efforts



# Other Recycling

## *Current recycling*

- Carboy, plastic film, pipette tip box, Styrofoam

## *In progress*

- Metal, select brown glass bottles



**Carboys**



**Metal Reagent Cans & Solvent Drums**



**Brown Glass**



# Questions?

- Contact info

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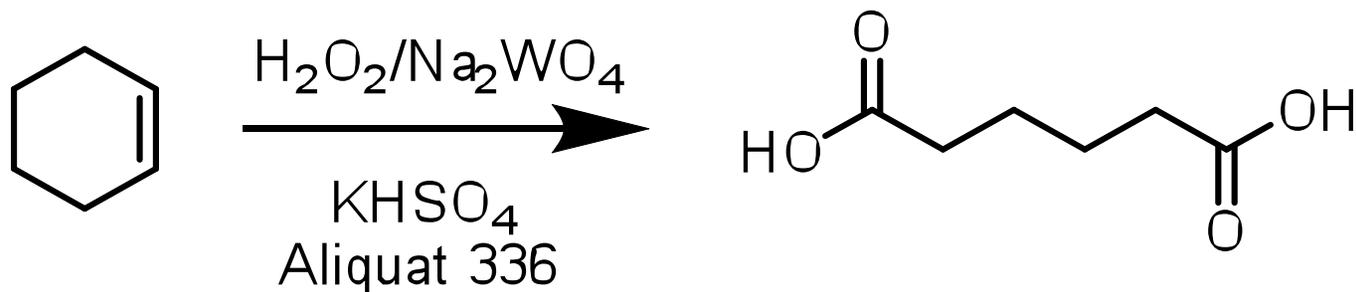
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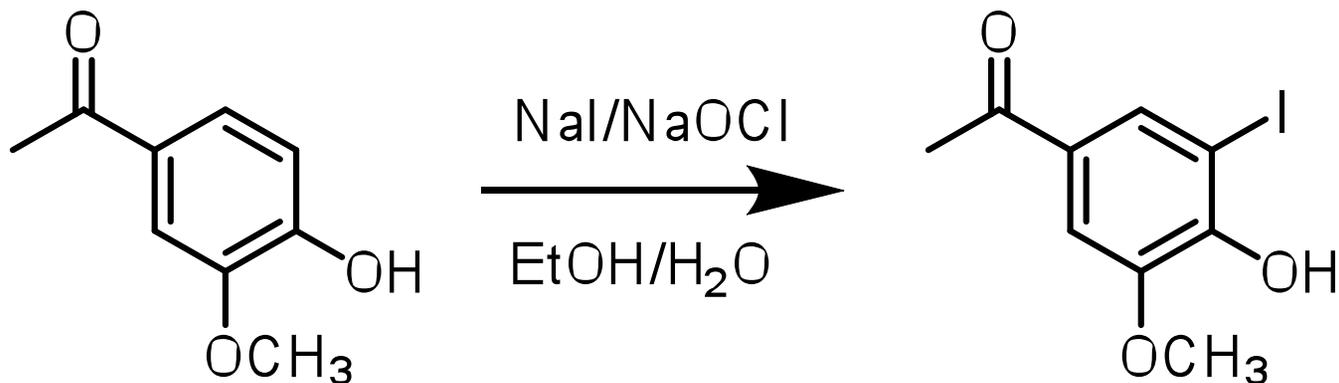
# “Synthesis and recryst. of adipic acid”



## “Synthesis and recryst. of adipic acid”

- Not a direct replacement for any particular lab at CU
- Affects: Majors OChem I students (~ 40 people/yr).

# “Electrophilic Aromatic Iodination”



- Not a direct replacement for any particular lab at CU
- Affects: Majors OChem II students (30-40 people/yr)
- Very successful



# “Palladium-catalyzed alkyne coupling”

- Uses product from electrophilic aromatic iodination
- Will be introduced in spring 2015
- Affects: Majors OChem II students (30-40 people/yr)