A Greener Grignard Reaction

A case study prepared by Beyond Benign as part of the Green Chemistry in Higher Education program: A workshop for EPA Region 2 Colleges and Universities

A Greener Grignard Reaction

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A Greener Grignard Reaction

Summary:
A greener approach to a traditional Grignard Reaction has been implemented by Dr. Abby O'Connor at the College of New Jersey (TCNJ). The greener version of the laboratory exercise has been implemented in to Dr. O'Connor's Inorganic Chemistry laboratory course at TCNJ.

Reduction in waste and purchasing costs:

For every semester this reaction is implemented with 100 students, there is an estimated ***cost savings of $123.36*** in purchasing and waste disposal costs and a ***reduction in waste from 0.8 gallons to 0.4 gallons***. The greener version of the Grignard Reaction also ***decreases the use of ethers and eliminates the use of bromobenzene, benzophenone and magnesium***, all of which have human health and aquatic hazards.

Background:
This case study is a result of an EPA Region 2 Source Reduction grant[1] titled *Green Chemistry in Higher Education: A Workshop for Region 2 Colleges and Universities*. The Green Chemistry in Higher Education workshop was carried out at Siena College on July 18-21, 2013. 29 faculty members participated from 20 different institutions in New York and New Jersey. The workshop consisted of three main focus areas: green chemistry case studies for lecture and course work, green chemistry laboratory exercises, and toxicology and environmental impact.

During the workshop participants were able to test a variety of greener laboratory exercises for introductory and organic chemistry courses. One of the labs was an Aqueous Grignard Reaction.[2] Dr. Abby O'Connor from the College of New Jersey (TCNJ) tested the laboratory exercise during the workshop and made improvements on the lab for use in her own laboratory at TCNJ during the following academic years. This case study includes a comparison of a traditional Grignard Reaction versus the greener reaction. The following pages include purchasing and disposal costs, along with chemicals used and their associated hazards. The benefits of the Greener Grignard Reaction are outlined in this case study.

[1] **Disclaimer: Although the information in this document has been funded wholly or in part by the United States Environmental Protection Agency under assistance agreement X9-96296312 to Beyond Benign, it has not gone through the Agency's publications review process and, therefore, may not necessarily reflect the views of the Agency and no official endorsement should be inferred.**

[2] "Water-soluble catalysts: Aqueous analogue of the Grignard reaction." Warner, J.C., in *Greener Approaches to undergraduate chemistry experiments*, American Chemical Society, p. 23-24.

Additional Resources for Green Chemistry in General Chemistry and Beyond:

Greener Educational Materials (GEMs) Database (University of Oregon)

- Website: http://greenchem.uoregon.edu/gems.html
- Description: Searchable database with Green Chemistry educational materials uploaded by faculty members and educators world-wide
- Most curriculum is available for download (free-of-charge) or with primary literature information
- Google map of Green Chemistry educators

American Chemical Society's Green Chemistry Institute

- Website: www.acs.org/greenchemistry
- Description: Green Chemistry Resources for educators and students
- Experiments and Curriculum available for download
- List of ACS books on Green Chemistry

Green Chemistry Commitment

- Website: www.greenchemistrycommitment.org
- Description: A program of Beyond Benign to adopt Green Chemistry Learning Objectives in higher education.
- Case studies are available, university highlights, and curriculum resources

Beyond Benign

- Website: www.beyondbenign.org
- Description: Green Chemistry Curriculum available on-line (free-of-charge)
- Regional Outreach and Community Educational Events

GCEdNet - Green Chemistry Education Network

- Website: http://cmetim.ning.com/
- Description: A place where Green Chemistry educators share resources
- Blogs, discussions and chat rooms

University of Scranton Greening Across The Chemistry Curriculum

- Website: http://www.scranton.edu/faculty/cannm/green-chemistry/english/drefusmodules.shtml
- Description: Green Chemistry modules available for download
- Power point presentations, hand-outs available

Carnegie Mellon University Institute for Green Science

- Website: http://igs.chem.cmu.edu/
- Description: Green Chemistry modules available for download
- Power point presentations, hand-outs available

Traditional Experiment:

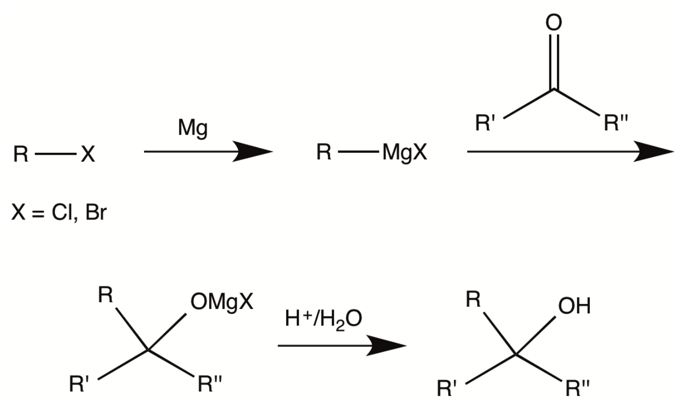
The Grignard reaction is a typical experiment that is performed in the undergraduate organic chemistry laboratory course to demonstrate carbon-carbon bond formation. The formation of carbon-carbon bonds is one of the most important reactions in organic chemistry.

The procedure involves placing magnesium powder into a dry reaction flask, adding the anhydrous diethyl ether. Bromobenzene in anhydrous diethyl ether is added to the reaction flask slowly (an exothermic reaction) and benzophenone dissolved in anhydrous ether is then added to the flask.

The conditions for this type of reaction typically require very anhydrous conditions due to the fact that the magnesium salt will react with any small amount of water in the solution. Therefore, the Grignard reaction is typically performed in anhydrous organic solvents such as tetrahydrofuran or diethyl ether.

Grignard Reaction
Traditional Experiment

Chemicals avoided per class of 100 students:
100 mL Bromobenzene
0.34 lb. Benzophenone
250 mL Hexanes
1.25 L anhydrous diethyl ether

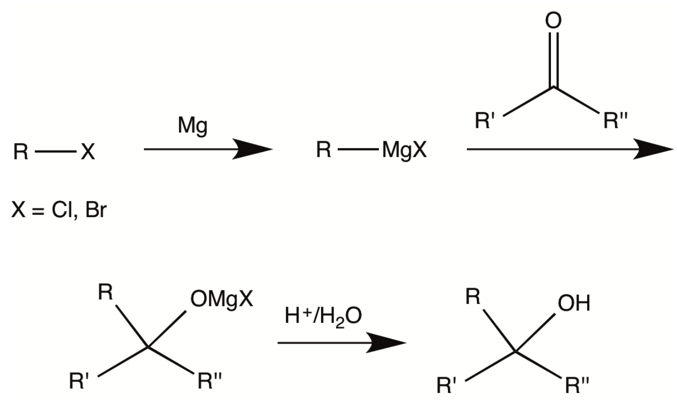


Classic Grignard Reaction

Chemicals used and hazards:
The chemicals that are typically used in this experiment are listed on the following page, along with a list of hazards. The amounts are estimated based on procedures that can be found on-line. Some procedures are smaller in scale and will use less of the chemicals listed, while others are larger scale and will use more. The procedure in this case study was chosen due to it being an average of typical procedures.[3]

[3] Moravian College, CH224, Organic Chemistry Laboratory II, Experiment #6, Grignard Reaction of Phenyl Magnesium Bromide with Benzophenone to form Triphenylmethanol [http://www.chem.moravian.edu/~rdlibby/_211-212Chem-PDF/Laboratory/Experiments/212-08Lab/Expt4-Alkene-Syn/exp6grignardprocedure.pdf, Accessed July 18, 2014]

Traditional Experiment, Continued:



Classic Grignard Reaction

Grignard Reaction
Traditional Experiment

Chemicals avoided per class of 100 students:
100 mL Bromobenzene
0.34 lb. Benzophenone
250 mL Hexanes
1.25 L anhydrous diethyl ether

Table 1. Chemicals used and hazards:

Chemical:	**Amount per group of 2 students:**	**Flammability:**[4]	**Human health toxicity:**[5]	**Aquatic toxicity:**[5]
Magnesium	0.45 g	*Pyrophoric solid: flammable on contact with water*		
Anhydrous Diethyl ether	25 mL	*Highly Flammable* *NFPA Code: 4* Flash Point: -45°C	*Low toxicity,* LD50 (oral, rat) 1,215 mg/kg; LD50 (dermal, rabbit) 14.2 g/kg	*Low toxicity,* LC50 (fish, 96 hr) 2,560 mg/l; EC50 (daphnia, 24 hr) 165 mg/l
Bromobenzene	2.0 mL	*Flammable* *NFPA Code*: 2 Flash Point: 51.0°C	*Low toxicity* LD50 (oral, rat) 2,383 mg/kg; LD50 (inh, mouse) 21,000 mg/m3	*High aquatic toxicity* LC50 (fish, 96 hr) 35.7 mg/l; EC50(daphnia, 24 hr) 1.6 mg/l
Benzophenone	3.12 g	n/a	*Moderate toxicity;* IARC Group 2B: Possibly carcinogenic to humans; LD50 (oral, rat) 10,000 mg/kg; LD50 (dermal, rabbit) 3,535 mg/kg	*High aquatic toxicity* LC50 (fish, 96 hr) 14.2 mg/l; EC50(daphnia, 24 hr) 0.28 mg/l
Ethanol	12 mL	*NFPA Code: 3* Flash Point: 9°C	*Low toxicity,* LD50 (oral, rat) 7,060 mg/kg	
3M HCl	10 mL		*Causes severe burns and eye damage*	
Sodium sulfate	5-10 g	n/a	*Low toxicity* LD50 (oral, mouse) - 5,989 mg/kg	*Moderate aquatic toxicity;* LC50 (fish, 96 hr) - 120 mg/l; LC50 (fish, 96 hr) - 4,380 mg/l
Hexanes	5 mL	*NFPA Code: 3* Flash Point: -26°C	*High chronic toxicity, Reproductive and Developmental hazard*	*High aquatic toxicity* LC50 (fish, 96 hr) 2.5 mg/l; EC50 (daphnia, 48 hr) 3,878 mg/l

[4] NFPA codes can be found here: http://en.wikipedia.org/wiki/NFPA_704#Red

[5] Human health and aquatic toxicity data was gathered from Globally Harmonized Safety Data Sheets, which can be obtained from Sigma-Aldrich [http://www.sigmaaldrich.com/united-states.html].

Traditional Experiment, Continued:

The purchasing and waste disposal costs associated with this procedure are estimated in the following table. Purchasing costs were estimated based on prices available from Sigma-Aldrich:[5]

Total amounts of chemicals used and disposed of per class of 100 students:

- ***0.33 gallons anhydrous diethyl ether***
- ***0.34 lbs benzophenone***
- ***0.03 gallons bromobenzene***
- ***0.07 gallons hexanes***
- ***0.81 gallons of liquid and 0.94 lbs solid waste***

Grignard Reaction
Traditional Experiment

Volume of waste and purchasing and waste disposal costs per class of 100 students:
0.81 gallons of liquid and 0.94 lbs solid waste
$326.15 in purchasing and disposal costs

Table 2. Purchasing and waste disposal costs:

Chemical:	Amount per 100 students:	Waste disposal cost[6]	Purchasing cost:[5]	Purchasing cost per 100 students:	Waste disposal cost per 100 students:	Total cost (per 100 students)
Magnesium	22.5 g (0.05 lb)	$1.35/lb	500g - $90.00	$4.05	$0.08	$4.13
Anhydrous Diethyl ether	1,250 mL (0.33 gal)	$11.27/gal	1 L - $129.00	$161.25	$3.72	$164.97
Bromobenzene density: 1.495 g/mL	100 mL (0.026 gal)	$11.27/gal	100 mL - $19.60	$19.60	$0.29	$19.89
Benzophenone	156 g (0.34 lb)	$1.35/lb	500 g - $35.20	$10.98	$0.46	$11.44
Ethanol, 200 proof	600 mL (0.16 gal)	$11.27/gal	1 L - $108.50	$65.10	$1.80	$66.90
3M HCl	500 mL (123.2 mL conc. HCl to make 500 mL 3M solution) (0.13 gal)	$11.27/gal	500 mL - $60.10 (conc. Solution)	$14.81	$1.47	$16.28
Sodium sulfate	250 g (0.55 lb)	$1.35/lb	500 g - $49.10	$24.55	$0.74	$25.29
Hexanes (density: 0.6548 g/mL)	250 mL (0.066 gal)	$11.27/gal	1 L - $66.00	$16.50	$0.74	$17.24
TOTAL	**0.7 gal and 0.94 lb. waste (0.81 gal total waste*)**			**$316.84**	**$9.30**	**$326.15**

Total purchasing and waste disposal costs per class of 100 students:

- ***$316.84 in purchasing costs***
- ***$9.30 in waste disposal costs***
- ***$326.15 total cost***

* The density of the waste is assumed to be 1 g/mL, therefore adding 0.11 gallons of waste to liquid waste total.
[5] Sigma-Aldrich [http://www.sigmaaldrich.com/united-states.html, Accessed July 18, 2014].
[6] Waste disposal costs are based on the EPA Cost Calculator Tool [http://www.epa.gov/p2/pubs/resources/measurement.html#calc, accessed December 2014].

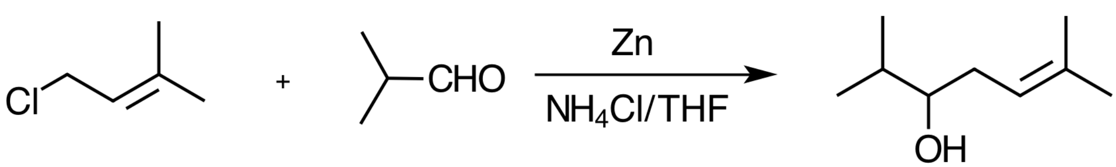
Greener Grignard Reaction
A Greener Approach

Volume of waste and purchasing and waste disposal costs per class of 100 students:
0.42 gallons of liquid and 0.56 lbs solid waste
$202.79 in purchasing and disposal costs

A Greener Approach:

Dr. Abby O'Connor at the College of New Jersey revised and implemented a greener approach to the traditional Grignard reaction. Dr. O'Connor was a participant at the summer green chemistry workshop at Siena College (summer of 2013) and she implemented the laboratory in her Fall 2013 Inorganic chemistry lab-based course and it is now standard in her Inorganic Chemistry courses. We present a summary of this new approach, along with a comparison of the hazards and costs associated with the greener approach versus the traditional approach.

Overview of the greener reaction:
A zinc-mediated process is used to perform a carbon-carbon bond formation reaction, which involved the use of zinc dust, magnesium sulfate, and a minimal amount of organic solvent in aqueous conditions.



Zinc Mediated coupling of 1-chloro-3-methyl-2-butene and isobutyraldehyde

It should be noted that the Greener reaction still uses anhydrous diethyl ether, but in smaller amounts. Because the zinc mediated process can be performed under aqueous conditions, reduced amounts of the ethers are necessary.

Table 3. Chemicals used, human health and aquatic toxicity data:

Chemical:	**Amount per group of 2 students:**	**Flammability:**[4]	**Human health toxicity:**[5]	**Aquatic toxicity:**[5]
Magnesium sulfate (sat. soln) (solubility 25.5 g/100 mL at 20 C)	25 mL of saturated solution (6.38 g/25mL)	n/a	*Low toxicity,* LD50 (oral, rat) >2,000 mg/kg; LD50 (inh, rabbit) >2,000 mg/l	*Low toxicity,* LC50 (fish, 96 hr) 2,820 mg/l; EC50 (daphnia, 48 hr) 344 mg/l
Zinc dust	1.8 mmol (0.12 g)	*Combustible in dust form*	*Low toxicity*	*High aquatic toxicity, H400: Acute aquatic toxicity,* LC50 (fish, 96 hr) 450 ug/l; LC50 (daphnia, 48 hr) 0.068 mg/l
Anhydrous Diethyl ether	6 mL	*Highly Flammable NFPA Code: 4 Flash Point: -45°C*	*Low toxicity,* LD50 (oral, rat) 1,215 mg/kg; LD50 (dermal, rabbit) 14.2 g/kg	*Low toxicity,* LC50 (fish, 96 hr) 2,560 mg/l; EC50 (daphnia, 24 hr) 165 mg/l
Tetrahydrofuran	0.2 mL	*Flammable NFPA Code: 3 Flash Point: -14°C*	*Low acute toxicity, Suspected human carcinogen,* LD50 (oral, rat) 2,050-2,850 mg/kg; LD50 (dermal, rat) 2,000 g/kg	*Low toxicity,* LC50 (fish, 96 hr) 2,160 mg/l; EC50 (daphnia, 24 hr) 382 mg/l
1-chloro-3-methyl-2-butene (density: 0.928 g/mL)	1.7 mmol (0.19 mL)	*Flammable NFPA Code: 3 Flash Point: 13°C*	*Respiratory hazard*	*High aquatic toxicity*
Magnesium sulfate	5 g	n/a	*Low toxicity,* LD50 (oral, rat) >2,000 mg/kg; LD50 (inh, rabbit) >2,000 mg/l	*Low toxicity,* LC50 (fish, 96 hr) 2,820 mg/l; EC50 (daphnia, 48 hr) 343.6 mg/l
Isobutyraldehyde (density 0.79 g/mL)	1.1 mmol (0.10 mL)	*Flammable NFPA Code: 2 Flash Point: -19°C*	*Low toxicity,* LD50 (oral, rat) 3,730mg/kg; LC50 (inh, rat) 23.9 mg/l, LD50 (dermal, rabbit) 5,630 mg/kg	*Moderate toxicity,* LC50 (fish, 96 hr) 23 mg/l; EC50 (daphnia, 48 hr) 277 mg/l

It should be noted that some of the above chemicals still pose some human health and environmental hazards. Green chemistry is a process and improvements can always be made. Therefore, the above might be a better alternative for current traditional processes today, but improvements can be made tomorrow.

[4] NFPA codes can be found here: http://en.wikipedia.org/wiki/NFPA_704#Red

[5] Human health and aquatic toxicity data was gathered from Globally Harmonized Safety Data Sheets, which can be obtained from Sigma-Aldrich [http://www.sigmaaldrich.com/united-states.html].

Greener Grignard Reaction
A Greener Approach

Volume of waste and purchasing and waste disposal costs per class of 100 students:
0.42 gallons of liquid and 0.55 lbs solid waste
$202.79 in purchasing and disposal costs

A Greener Approach, Continued:

The purchasing and waste disposal costs associated with this procedure are estimated in the following table. Purchasing costs were estimated based on prices available from Sigma-Aldrich:[5]

Total amounts of chemicals used and disposed of per class of 100 students:

- ***Reduction in use of anhydrous diethyl ether (0.08 gal)***
- ***Tetrahydrofuran (0.003 gal)***
- ***Aqueous solvent (0.33 gal)***
- ***Zinc catalyst (0.01 lb)***
- ***0.42 gallons liquid and 0.56 lbs solid waste***

Table 4. Purchasing and waste disposal costs:

Chemical:	**Amount per 100 students:**	**Waste disposal cost[6]**	**Purchasing cost:[5]**	**Purchasing cost per 100 students:**	**Waste disposal cost per 100 students:**	**Total cost (per 100 students)**
Magnesium sulfate (sat soln) (water solubility: 25.5 g/100 mL at 20 C)	1,250 mL (319 g/1,250 mL) (0.33 gal)	$11.27/gal	500 g - $93.70	$59.78	$3.72	$63.50
Zinc dust	6 g (0.013 lb)	$1.35/lb	100 g - $42.30	$2.54	$0.02	$2.56
Anhydrous Diethyl ether	300 mL (0.079 gal)	$11.27/gal	1 L - $129.00	$38.70	$0.89	$39.59
Tetrahydrofuran	10 mL (0.0026 gal)	$11.27/gal	100 mL - $47.30	$4.73	$0.03	$4.76
1-chloro-3-methyl-2-butene (density: 0.928 g/mL)	9.5 mL (0.00225 gal)	$11.27/gal	50 g - $252.50	$44.52	$0.03	$44.55
Magnesium sulfate	250 g (0.55 lb)	$1.35/lb	500 g - $93.70	$46.85	$0.74	$47.59
Isobutyraldehyde (density 0.79 g/mL)	4.05 mL (0.0012 gal)	$11.27/gal	1 L - $46.30	$0.19	$0.23	$0.01
TOTAL	**0.42 gal waste and 0.55 lbs solid waste**			**$197.35**	**$5.44**	**$202.79**

Total purchasing and waste disposal costs per class of 100 students:

- ***$197.35 in purchasing costs***
- ***$5.44 in waste disposal costs****
- ***$202.79 total cost***

Greener Grignard Reaction
Summary

Waste avoided:
Reduction in 0.4 gallons liquid and 0.38 lbs solid waste
Reduction in use of diethyl ether from 0.33 gal to 0.08 gal
Cost comparison:
Reduction in purchasing and disposal costs of $123.36

Traditional Experiment Summary:

Total amounts of chemicals used and disposed of per class of 100 students:
- ***0.33 gallons anhydrous diethyl ether***
- ***0.34 lbs benzophenone***
- ***0.03 gallons bromobenzene***
- ***0.07 gallons hexanes***
- ***1.25 L (0.33 gal) anhydrous diethyl ether***
- ***0.81 gallons of liquid and 0.55 lbs solid waste***

Total purchasing and waste disposal costs per class of 100 students:
- ***$316.84 in purchasing costs***
- ***$9.30 in waste disposal costs***
- ***$326.15 total cost***

A Greener Approach Summary:

Total amounts of chemicals used and disposed of per class of 100 students:
- ***Reduction in use of anhydrous diethyl ether (0.08 gal)***
- ***Tetrahydrofuran (0.003 gal)***
- ***Aqueous solvent (0.33 gal)***
- ***Zinc catalyst (0.01 lb)***
- ***0.42 gallons liquid and 0.55 lbs solid waste***

Total purchasing and waste disposal costs per class of 100 students:
- ***$197.35 in purchasing costs***
- ***$5.44 in waste disposal costs****
- ***$202.79 total cost***

Conclusions:

In summary, by implementing a greener version of the classic Grignard type reaction, there are many benefits. Students are exposed to cutting edge reactions that are being explored that move beyond the typical boundaries of classical reactions. The greener version of the Grignard reaction described herein can be performed under aqueous conditions, whereas the classic Grignard experiment requires very anhydrous conditions and has a very high fail rate for students, making the reaction even more wasteful.

As with all green chemistry experiments, there are still some hazards associated with the reagents and solvents. Therefore, students should be challenged to inquire about greener versions of the current experiments they are performing and explore even better options for reducing hazards and costs associated with the experiments.

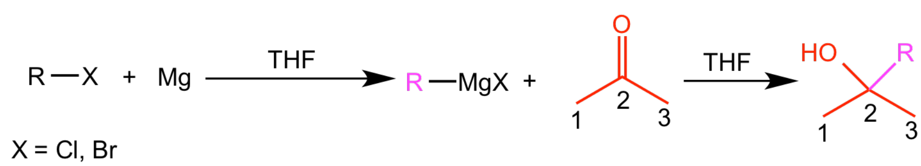
APPENDIX A: A greener approach to the Grignard Reaction

O'Connor
CHE 332, Fall 2013
Labs

Adapted from:
J. Org. Chem. **1989**, *54*, 3087-3091.
Greener Approaches to undergraduate chemistry experiments "Water-soluble catalysts: Aqueous analogue of the Grignard reaction."

Introduction:
The formation of carbon-carbon bonds is one of the most important reactions in organic chemistry. There are a number of ways to make a carbon-carbon bond, but the most ubiquitous reaction is the Grignard reaction. Many C-C bonds are formed using organometallic precursors. An organometallic complex is a species that contains a metal and organic fragment. An example of a Grignard reagent would be methyl magnesium bromide. Due to the presence of the metal, the carbon-metal bond is polarized; the carbon acquires a negative charge and can function as a nucleophile. These species are particularly reactive with electrophilic species, such as carbonyls. An example is shown below.

Scheme 1. Standard Grignard Reaction.

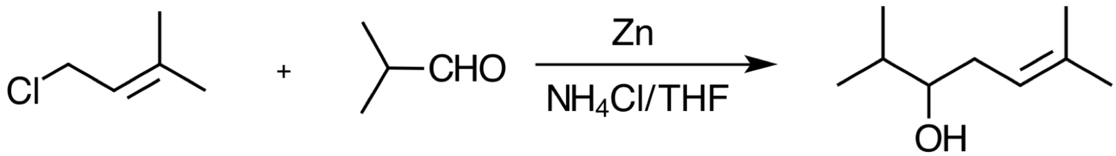


Although the Grignard reaction is critically important to organic chemists, there are lots of issues with this reaction. The chemistry is limited to substrates that do not possess acidic moieties. These reactions are also often conducted at low temperatures. Rigorously anhydrous and degassed organic solvents such as tetrahydrofuran and diethyl ether must be used in order to prevent degradation of the organomagnesium reagent. As discussed in class, ether solvents are employed to aid in stabilization of the reactive organometallic species. Additionally, there are concerns over the cost of the metal and disposal of waste. Overall not a very green process!

For these reasons, other means to make C-C bonds have been explored. One example includes the use of palladium catalysts to stitch together carbon containing groups. This chemistry is heavily used by the pharmaceutical industry and is aligned with the principles of green chemistry because a catalyst is used rather than a stoichiometric reagent. However, this process must be conducted under anhydrous conditions using an expensive metal catalyst and in the presence of large amounts of organic solvents. Another avenue, which will be explored here, is the use of a zinc-mediated process. Some advantages for this methodology include the ability to conduct the reaction under "wet" conditions and in the presence of a minimal amount of organic solvent. Additionally, substrates that possess acidity moieties such as alcohols can be used in this reaction. We can see that this new reaction adheres to the principles of green chemistry! A downside of the reaction is that it is limited to the use of allylic substrates as the nucleophile and the reaction is stochiometric in zinc.

Although the mechanistic details of this reaction are not well understood it is proposed that the zinc metal reacts with the allyl halide to make a reactive zinc-alkyl that is similar to an alkyl lithium or Grignard reagent. Zinc can insert into a C-X bond just like magnesium can insert into a C-X bond. Radical pathways have not been ruled out in the mechanism. The presence of the metal polarizes the bond making the alkyl fragment possess a partial negative charge and act as a nucleophile.

Scheme 2. Zinc Mediated coupling of 1-chloro-3-methyl-2-butene and isobutyraldehyde



Chemicals:

Report safety information for substances that you will use in the lab
Isobutyraldehyde
Magnesium sulfate
1-chloro-3-methyl-2-butene
Zinc powder
Ammonium chloride
Tetrahydrofuran (THF)
Glass wool
Diethyl ether

Procedure:

Week 1 (scale - ~ 100-200 mg of Zinc)
1. Prepare a saturated ammonium chloride (NH_4Cl) solution (~25 mL of solution) (look up how much ammonium chloride is needed to make a saturated solution)
2. Add 1.8 mmol of zinc dust and 10 mL of the saturated ammonium chloride solution to a 50 mL Erlenmeyer flask containing a stir bar
3. Add 1.1 mmol of isobutryaldehyde to the flask followed by 0.2 mL THF
4. Stir solution for 1 minute
5. Add 1.7 mmol of 1-chloro-3-methyl-2-butene and stir for 45 minutes
An immediate reaction should take place with loss of Zn
6. After 45 min add 2 mL of diethyl ether and filter sample through a plug of glass wool (glass wool in a pipet, to remove the excess Zn and salts). Collect liquid into a small flask.
7. Rinse filter with another 2 mL of diethyl ether
8. Transfer the filtrate into a separatory funnel and separate the organic and aqueous layers. Note more ether may need to be added.
9. Wash the aqueous phase with 2 mL of diethyl ether
10. Dry organic layer of $MgSO_4$
11. Filter sample, collecting liquid into a tared vial. Blow off ether with air-line
12. Save product for week 2.

Week 2:
1. Obtain mass of product. Calculate % yield and % atom economy for the reaction.
2. Take IR of product and NMR of product.
To prepare NMR sample – put the tip of a glass pipet into the vial containing the product, place a ***small*** amount of product into an NMR tube and dissolve in 0.50 mL of $CDCl_3$.

Waste:

The aqueous layer can be flushed down the drain with copious amounts of water.
Place the drying agent and precipitate into the solid waste container in the hood.
The liquid in the NMR tubes should be disposed of in the halogenated waste.

Analysis:

Evaluate NMR and IR spectra. Clearly, identify and assign regions on the spectra. For example, circle the alcohol peak and assign the different signals in your NMR spectrum. These values will be clearly reported in a table in your lab report. Be sure to discuss in your report how you know that your reaction worked or did not work based on your data. Follow all guidelines listed in the syllabus.

Post lab Questions:

1. Running the reaction in water is greener than performing the reaction in an organic solvent. Can you think of any new problems that water might pose environmentally?

2. Draw the structure of the product that would be obtained if benzaldehyde were used as the carbonyl-containing compound in this reaction.

3. Can you suggest other ways to make this experiment greener?

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