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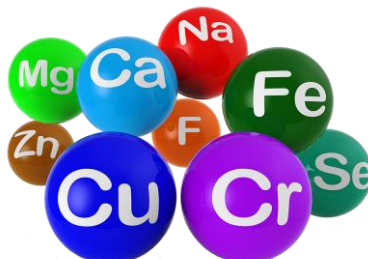
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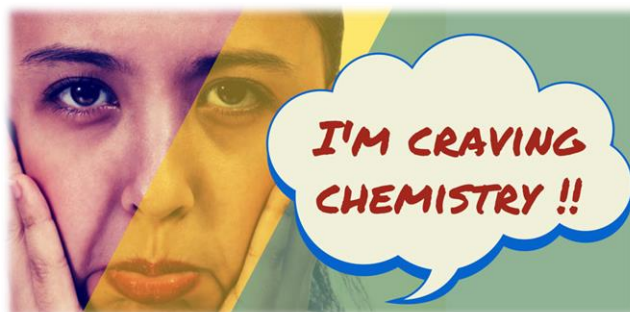
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Thursday, January 22, 2015

“3D Printing: From Molecules to Manufacturing”

Dr. Timothy Long, Professor, Department of Chemistry, Director of Macromolecules and Interfaces Institute, Virginia Tech University

Dr. Christopher Williams, Associate Professor, Department of Mechanical Engineering, Assistant Director of Macromolecular and Interfaces Institute, Virginia Tech University



Thursday, January 29, 2015

“2015 Drug Design & Delivery Symposium: Designing Better Drug Candidates”

Dr. Paul Leeson, Director, Paul Leeson Consulting, Ltd.

Dr. Rick Connell, Vice President, External Research Solutions, Pfizer

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“Rookie Lab Mistakes and Other Facts Not Found in Textbooks”



Bill Courtney
Chef and Analytical Chemist,
Cheese-ology

Dr. Alison Frontier
Professor of Chemistry,
University of Rochester

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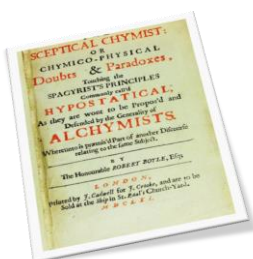
We all want to avoid A Nightmare Scenario...



...like these ones...

Photo Credit: <https://www.fema.gov/earthquake/fema-e-74-reducing-risks-nonstructural-earthquake-damage-14> ¹⁷

A Nightmare Reaction *To Begin With*



Theory suggests that the experiment should work. However, the only precedent you find is either:

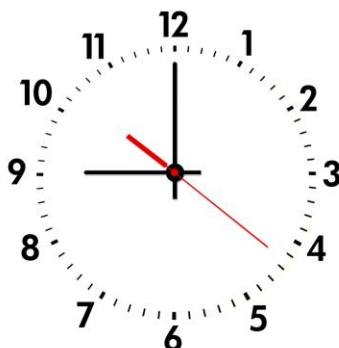
- a) in another language
- b) from 1927
- c) has no experimental details...**OR**

<http://chem.chem.rochester.edu/~nvd/pages/reaction.php?page=nightmare>

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A Nightmare Reaction

To Begin With



It's 9 PM. None of your glassware is clean. You can't decide what scale to run it on and you don't have any of the reagents.

<http://chem.chem.rochester.edu/~nvd/pages/reaction.php?page=nightmare>

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A Nightmare Reaction

Setting Up

You need to weigh four different reagents:

1. a hygroscopic solid that gets liquidy in the air,
2. 0.05 mg of catalyst,
3. a liquid that clogs syringes and must be distilled immediately before use, and
4. your precious reactant, which is heat and acid sensitive.

The reaction must be done at -30°C under argon using a complex glass apparatus, and requires three flasks for successive dropwise addition via cannula.

<http://chem.chem.rochester.edu/~nvd/pages/reaction.php?page=nightmare>

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A Nightmare Reaction

Monitoring the Reaction



- The reaction takes hours to complete: however, the product is unstable and decomposes slowly under the reaction conditions.
- Progress cannot be monitored by TLC.

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A Nightmare Reaction

Quench



The product mixture is highly reactive and requires dropwise addition of quenching reagent to prevent a volcano-like exothermic eruption.

<http://chem.chem.rochester.edu/~nvd/pages/reaction.php?page=nightmare>

Photo Credit: <http://www.stevespanglerscience.com/lab/experiments/erupting-peroxide-volcano>

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A Nightmare Reaction

Workup

When an aqueous solvent is added to the diluted reaction mixture, an emulsion forms. All efforts to resolve the layers fail, your solution has swollen to gargantuan proportions, and you can't find or can't lift a separatory funnel large enough to hold it.

OR



<http://chem.chem.rochester.edu/~nvd/pages/reaction.php?page=nightmare>

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A Nightmare Reaction

Workup

Upon addition of aqueous bicarbonate, the organic layer becomes a graceful fountain, coating the inside of your fume hood.

OR



<http://chem.chem.rochester.edu/~nvd/pages/reaction.php?page=nightmare>

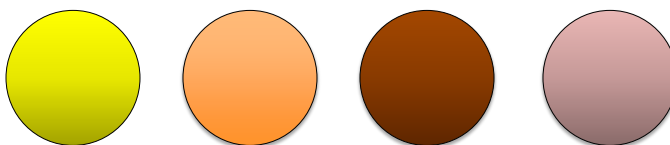
Photo Credit: http://www.prweb.com/releases/Lab_furniture_fume_hood/mistral_fume_hood/prweb11858713.htm

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A Nightmare Reaction

Workup

The aqueous solution you use to wash the organic layer turns yellow, orange, brown or pink- the first ten times you try it.



<http://chem.chem.rochester.edu/~nvd/pages/reaction.php?page=nightmare>

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A Nightmare Reaction

Purification

IMPURITIES

$$R_f = \frac{\text{distance of the spot on the TLC-plate}}{\text{distance of the solvent front}}$$



Boiling Point

Your compound is an oil. No purification method has been reported for its isolation. The crude reaction mixture has three or four minor impurities, all with similar boiling points and R_f values similar to your desired compound.

<http://chem.chem.rochester.edu/~nvd/pages/reaction.php?page=nightmare>

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Inherent challenges

+

The wizardry of expert chemists

=

Voodoo?

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Not Voodoo:

Demystifying Organic Laboratory Technique

In 2004: 11 Rookie Mistakes

In 2014: >250 Rookie Mistakes

- **About Not Voodoo:** [Development](#), [Wanted Items](#), [Charter Contributors](#), [Contributors](#)
- **Browse by Experience Level:**
 - [First-Time Independent Researchers](#)
 - [First-Year Ph.D. Students](#)
 - [Intermediate Graduate Students](#)
 - [Advanced Researchers](#)
- **The Tour of Collective Wisdom:** [Rookie Mistakes](#), [Toxic Reagents](#), [Pyrophoric and I](#)
[A Day in the Life](#), [May Require Mojo](#), [1, 2, 3... Ph.D.](#), [Q&A](#)
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In September 2014: Not Voodoo X:

<http://chem.chem.rochester.edu/~nvd/?page=home>

Not Voodoo X

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Magic Formulas	Tips and Tricks	Troubleshooting	How To	Rookie Mistakes	Chemists Weigh In	Chromatography	Reagents and Solvents	Workup Tricks	How to Run a Reaction
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Not Voodoo X @Not_Voodoo 27 Oct
 Rookie Mistakes Monday #RMM
 When we forget that PV=nRT:
chem.chem.rochester.edu/~nvd/pa

Not Voodoo X @Not_Voodoo 14 Oct
 Tricks for working up reactions in polar and water-miscible solvents:
chem.chem.rochester.edu/~nvd/pa

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Take a look at the [new and updated pages for Not Voodoo Redux](#)

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Audience Survey Question

Which of these Rookie Mistakes is the **MOST** common?

- Forgot to pre-weigh your round-bottom flask
- Burned hand on hot plate because it didn't look hot!
- Didn't check for cracks on clips for rotovap, or didn't use a clip. When vacuum is switched off, flask containing precious compound drops in bath.
- Didn't label a flask. One week later, have NO idea what is inside.
- Tried to drain sep funnel with stopper still in.

30

According to Site Visitors (2004-2014)

- Forgot to pre-weigh your round-bottom flask **#2**
- Burned hand on hot plate because it didn't look hot! **#9**
- Didn't check for cracks on clips for rotovap, or didn't use a clip. When vacuum is switched off, flask containing precious compound drops in bath. **#6**
- Didn't label a flask. One week later, have NO idea what is inside. **#5**
- Tried to drain sep funnel with stopper still in. **#1**

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Most Common Rookie Mistakes



5: Didn't label a flask. One week later, have NO idea what is inside.

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Most Common Rookie Mistakes



4. Set up reaction under Ar, added in reagents and forgot to add stir bar.

Photo Credit: http://www.cellartek.com/products/lab_equipment_and_supplies/lab_ware/accessories.php³³



Most Common Rookie Mistakes



3. Poured a reaction mixture into a sep funnel without closing the tap. Recovered reaction mixture from the bottom of the fume hood.

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Most Common Rookie Mistakes



2. Forgot to pre-weigh your round-bottom flask

Photo Credit: http://us.mt.com/us/en/home/products/Laboratory_Weighing_Solutions/Accessories/ergoclips/11106746_ErgoClip_Round_Bottom_Flask.html ³⁵



Most Common Rookie Mistakes



1. Tried to drain sep funnel with stopper still in.

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Most Common Rookie Mistakes

5. Didn't label a flask. One week later, have NO idea what is inside.
4. Set up reaction under Ar, added in reagents and forgot to add stir bar.
3. Poured a reaction mixture into a sep funnel without closing the tap. Recovered reaction mixture from the bottom of the fume hood.
2. Forgot to pre-weigh your round-bottom flask
1. Tried to drain sep funnel with stopper still in

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Lessons Learned:

Everyone Forgets



- 5: *Didn't label* a flask. One week later, have NO idea what is inside.
4. Set up reaction under Ar, added in reagents and *forgot to add* stir bar.
3. Poured a reaction mixture into a sep funnel *without closing the tap*. Recovered reaction mixture from the bottom of the fume hood.
2. *Forgot to pre-weigh* your round-bottom flask
1. Tried to drain sep funnel *with stopper still in*

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Lessons Learned: *Routines Help You Remember*

When you come into the lab, you wear eye protection. You'd feel weird without it.

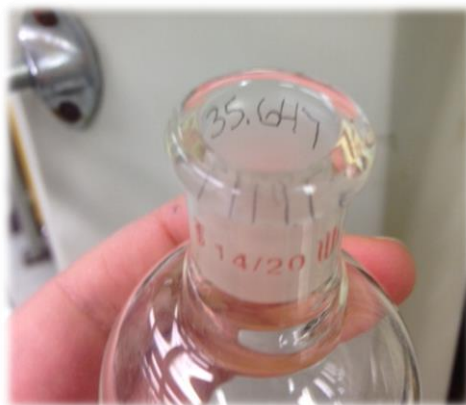
Here are some new routines to adopt:

- Always label your flasks. It's only a Sharpie away.
- Every time you put a sep funnel in a ring stand, put an Erlenmeyer flask under it.
- Every time you put a round-bottom flask under argon, make sure a stir bar is in it.

More Routines Here:

http://chem.chem.rochester.edu/~nvd/pages/collective_wisdom.php?page=always_and_never³⁹

2. Forgot to Pre-weigh Your Round-Bottom Flask



Tip: For all of your flasks, write the weight twice on the inside of the joint in pencil.

Lessons Learned: *More Advice for Rookies*

Always and Never

http://chem.chem.rochester.edu/~nvd/pages/collective_wisdom.php?page=always_and_never

Tips and Tricks to Improve Your Yield

http://chem.chem.rochester.edu/~nvd/pages/tips.php?page=improve_yield

Rules of Thumb

http://chem.chem.rochester.edu/~nvd/pages/tips.php?page=rules_of_thumb

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Trends Emerging from the Mistakes

Not Voodoo X

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Magic Formulas	Tips and Tricks	Troubleshooting	How To	Rookie Mistakes	Chemists Weigh In	Chromatography	Reagents and Solvents	Workup Tricks	How to Run a Reaction
	About Rookie Mistakes			<p>This collection of pages began with a list of eleven mistakes in September 2004. The idea was that beginning experimentalists might learn from experienced chemists, chemists who have run hundreds of reactions, and made lots of mistakes.</p>					
	Equipment								
	Reagents								
	Setup								
	TLC								
	Workup								
	Column Chromatography								
	Spectroscopy								
	The Vacuum Apparatus			<p>The Rookie Mistakes did not exactly work out that way. What began as an innocent compilation of beginner bumbles evolved into an entertaining catalog of honest errors, freak accidents, relatively innocuous events that cascaded in horrific directions, and incidents worthy of a Darwin award. After a few years of this, reading the list involved scrolling through a vast catalog of catastrophe encompassing all the classes of chemical experimentation.</p>					
	Labels and Bookkeeping								
	Ouch								
	Under Pressure								
	The Sound of Breaking Glass			<p>Over time, I learned that "Rookie Mistakes" was appreciated for reasons far beyond its intended role as a guide to help people avoid common pitfalls in the lab. Incredibly, students told me that the list was <u>an unexpected source of solace after a frustrating day in the lab.</u> That's partly because it's funny, but also because you don't feel quite so incompetent after reading about how other people <u>accidentally destroyed their experiments.</u></p>					
	Fire in the Lab								
	Just... Wow								
	Top Ten								
	Search								

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Ouch



Sat down with NMR tubes in the back pocket of pants.

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Under Pressure *aka don't forget $PV=nRT$*



Attempted to dissolve something by putting the flask in a hot water bath ... without removing the stopper.

Photo Credit: <http://www.capitolscientific.com/Kimble-28008-50-KIMAX-50mL-Class-A-Volumetric-Flask-with-Yellow-PE-Snap-Cap-Calibrated-To-Conta>

44

Vacuum Apparatus

low pressure is as tricky as high pressure



Attached air line to vacuum desiccator without first breaking the vacuum seal. Lid burst off, went about 18" in the air and crashed down on top of the desiccator shattering into a thousand pieces.

45

The Sound of Breaking Glass



Dropped freshly washed glassware and tried to save it with my foot, but ended up kicking it across the lab.

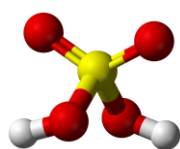
46

Just...wow!

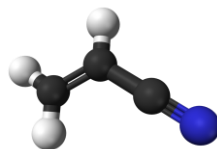
- Melted shoe to the ground with HCl.



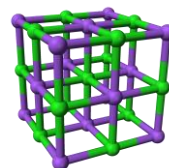
- Added concentrated **sulphuric acid** to a mixture of **acrylonitrile** and **sodium chloride**.....whoa! within seconds the entire reaction setup (flask, thermometer, condenser) got blown off and the overhead motor flew like a missile.....



+



+



47

Audience Survey Question

Which piece of equipment is most difficult to master?

- The Rotovap
- The Separatory Funnel
- The Glass Pipette
- The Glovebox
- Vacuum Apparatus

48

The Glass Pipette



While transferring small amount of product to an NMR tube, accidentally squeezed the bulb of the pasteur pipette, dumping sample onto the bench.

49

Vacuum Apparatus



The hose from the manifold vacuum port dropped into the oil bath and sucked the entire contents into the manifold.

Photo Credit: <http://www.cuhk.edu.hk/chem/en/resources/f6resourcebk-eimg12.html>

50

The Rotovap



Put product on rotovap at 40C and came back half an hour later to find that compound was very volatile and that flask is COMPLETELY empty.

Photo Credit: www.coleparmer.com

51

Lessons Learned:

How to Handle Volatile Compounds

- **Reduce the vacuum strength on your rotovap** as much as possible. If your compound is still ending up in the solvent trap, **try removing the solvent using conventional distillation**, using heat at atmospheric pressure.
- To run a reaction at elevated temperature with a volatile reagent, use a **Vigreux condenser** or consider **sealed-tube apparatus/** techniques.
- To purify a volatile product using column chromatography, choose your solvent system carefully. For example, you can substitute **pentane** for hexanes, and **avoid ethyl acetate**.
- **Store extra-volatile compounds at low temperature.**

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The Sep Funnel

“Accidentally dropped a **small** sep funnel **stopper** into a **large** sep funnel.”

*Rookie Mistakes #1 and #3
involve sep funnels,
Also, we use sep funnels
for workup...*



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Even More Nightmares at Workup:

Combination of organic and aqueous solutions gives a ***gooey or insoluble precipitate, which floats between the two layers*** and obscures the border.

OR

When an aqueous solvent is added to the diluted reaction mixture, an ***emulsion*** forms.

OR

Addition of aqueous solution to your black organic reaction mixture leads to a ***uniform black mixture***.

54

Lesson Learned:

Workup Tips

Problem: Insoluble Goo

Tip: Keep washing with water until most of the goo is removed. Then use lots of drying agent, and with luck, the goo will be absorbed and you will be able to filter it away.

<http://chem.chem.rochester.edu/~nvd/pages/workup.php?page=workup>

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Lessons Learned:

How to Handle an Emulsion

Problem: An emulsion forms. A brine wash fails to resolve the layers.

Tips:

- Evaporating the reaction solvent *before* workup
- Wait.
- Add solid NaCl
- Dilute with copious organic solvent
- Filter the whole thing through Celite

<http://chem.chem.rochester.edu/~nvd/pages/workup.php?page=workup>

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Lessons Learned: *Uniform Black Mixture*

Problem: The solution in your sep funnel is opaque, and *you can't see the border between the organic and aqueous layers.*



Tip: Try adding ice, which will float on the water, between the layers.

<http://chem.chem.rochester.edu/~nvd/pages/workup.php?page=workup>

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The Glovebox



Tried to scratch an itch on my face... while my hands were inside the glovebox.

Photo Credit: <http://newartsci.case.edu/magazine/fall-2012/a-reason-to-stay>

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How about Magnetic Stir Bars?



Added stir bar to flask. Stir bar smashes a hole in bottom of the flask, contents of flask now all over fume hood.

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Searching the Rookie Mistakes...

Magic Formulas	Tips and Tricks	Troubleshooting	How To	Rookie Mistakes	Chemists Weigh In	Chromatography	Reagents and Solvents	Workup Tricks	How to Run a Reaction
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<ul style="list-style-type: none"> Reagents Setup TLC Workup Column Chromatography Spectroscopy The Vacuum Apparatus Labels and Bookkeeping Ouch Under Pressure The Sound of Breaking Glass Fire in the Lab Just... Wow Top Twenty-Five Search 	<p>Search by Keyword:</p> <p>stir bar <input type="button" value="Go"/></p> <p><i>May we suggest: acetone, forgot, oven, quench, stir bar, heat, acid, dropped, condenser, rotovap, glove box, bromine, water, TLC, sep funnel</i></p> <table border="1"> <thead> <tr> <th>Mistake</th> <th>Vote</th> <th># Rookies</th> </tr> </thead> <tbody> <tr> <td>Set up reaction under Ar, added in reagents and forgot to add stir bar.</td> <td><input type="button" value="👍"/></td> <td>240</td> </tr> <tr> <td>Used a little stir bar with a big flask.</td> <td><input type="button" value="👍"/></td> <td>165</td> </tr> <tr> <td>While cleaning beakers with stir bars inside, poured the stir bars down the drain.</td> <td><input type="button" value="👍"/></td> <td>159</td> </tr> <tr> <td>Poured stir bar into sep funnel, decided to shake it anyway and shattered the funnel.</td> <td><input type="button" value="👍"/></td> <td>39</td> </tr> <tr> <td>Poured beaker of solvent into organic waste container... forgot there was a stir bar inside. Found out plastic is too thick for magnet to retrieve stir bar.</td> <td><input type="button" value="👍"/></td> <td>32</td> </tr> <tr> <td>Added stir bar to flask. Stir bar smashes a hole in bottom of the flask, contents of flask now all over fume hood.</td> <td><input type="button" value="👍"/></td> <td>26</td> </tr> <tr> <td>Lost a magnetic stir bar in the solid waste container.</td> <td><input type="button" value="👍"/></td> <td>12</td> </tr> </tbody> </table>	Mistake	Vote	# Rookies	Set up reaction under Ar, added in reagents and forgot to add stir bar.	<input type="button" value="👍"/>	240	Used a little stir bar with a big flask.	<input type="button" value="👍"/>	165	While cleaning beakers with stir bars inside, poured the stir bars down the drain.	<input type="button" value="👍"/>	159	Poured stir bar into sep funnel, decided to shake it anyway and shattered the funnel.	<input type="button" value="👍"/>	39	Poured beaker of solvent into organic waste container... forgot there was a stir bar inside. Found out plastic is too thick for magnet to retrieve stir bar.	<input type="button" value="👍"/>	32	Added stir bar to flask. Stir bar smashes a hole in bottom of the flask, contents of flask now all over fume hood.	<input type="button" value="👍"/>	26	Lost a magnetic stir bar in the solid waste container.	<input type="button" value="👍"/>	12
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Lost a magnetic stir bar in the solid waste container.	<input type="button" value="👍"/>	12																							

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Audience Survey Question

Which reagent is most difficult to work with?

- Triphenylphosphine
- Thiols
- DMSO
- Dicyclohexylcarbodiimide (DCC)
- Tributyltin hydride

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Rookie Mistake #8



Upset coworkers (and/or self) by handling sulfur compounds or other noxious volatiles outside of the fume hood.

Photo Credit: <http://www.masabainc.com/industries/sulfur>

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Lessons Learned: *Working with Thiols*

A **Thiol** is an organosulfur compound that contains a carbon-bonded sulfhydryl ($-C-SH$ or $R-SH$) group (where R represents an alkane, alkene, or other carbon-containing group of atoms).

- BLEACH!
- Zip-Lock Bags!
- Latex gloves!



Photo Credit: <http://www.jerrysartarama.com/discount-art-supplies/general-equipment/safety-gear.htm>⁶³

Workup Tricks

Magic Formulas	Tips and Tricks	Troubleshooting	How To	Rookie Mistakes	Chemists Weigh In	Chromatography	Reagents and Solvents	Workup Tricks	How to Run a Reaction				
	<ul style="list-style-type: none"> About Workup Tricks How to Manage an Emulsion Drying Methods Aluminum Hydride Reductions DCC Coupling mCPBA Oxidation Chromium Oxidations Removing Tin Byproducts Removing Copper Salts Removing Triphenylphosphine Removing Titanium Byproducts Removing Bromine or Iodine Removing Amines Removing Alcohols Reactions in Benzene Reactions in THF/Dioxane Reactions in Acetonitrile Reactions in DMF or DMSO Rookie Mistakes 							<h3>Workup for Removing Tin Byproducts</h3> <p>Six different workup methods for removal of tin byproducts from a reaction mixture are listed below:</p> <ol style="list-style-type: none"> Byproducts of tin based reactions such as Bu_3SnBr can be removed by treatment with $AlMe_3$ to create the nonpolar Bu_3SnMe or $NaOH$ to create the polar Bu_3SnOH. See: P. Renaud, E. Lacote, L. Quaranta Tetrahedron Lett. 1998, 39, 2123. (partially taken from the Merlic Group at UCLA, The Organic Companion) Filter through a mixture of KF/Celite <ol style="list-style-type: none"> Dilute rxn with suitable organic solvent Add aqueous phase (water, sat. NH_4Cl, etc.) Remove aq layer and wash organic phase 2-3 times with 1M KF aq solution. Shake in sep funnel for up to 1 minute for each wash. Solid Bu_3SnF precip may form at organic/aqueous interface. If this is a problem, filter through celite. Wash aq 1 X brine, dry organic phase and remove solvent In many Stille reactions, the Bu_3SnX junk can be almost entirely removed by filtering through silica made up with ~2-5% triethylamine in the eluent (or immediately purifying by standard column chromatography). Run the flash column using the same solvent. This method is quicker than stirring with aq, KF, and more fun than grinding a big bowl of toxic KF with silica or celite for a good dispersion. (contributed by Graham Cumming) 					

Audience Survey Question

Which reagents most commonly cause fires in labs?

- Lithium aluminum hydride
- Sodium hydride
- Palladium on carbon
- Sodium metal
- Ether

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Visitor Experience 2004-2014

ot Voodoo X

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Magical Formulas	Tips and Tricks	Troubleshooting	How To	Rookie Mistakes	Chemists Weigh In	Chromatography	Reagents and Solvents	Workup Tricks	How a Reagent Works
About Chemists Weigh In	Rookie Mistakes								
	Pyrophoric Reagents						Fires	Explosions	
Should I Buy it or Make it Myself?	1. Sodium metal						289	84	
Can I use it right out of the bottle?	2. Lithium aluminum hydride (LAH, LiAlH ₄)						283	63	
May Require Mojo	3. Palladium on carbon (Pd/C)						301	31	
Desert Island Oxidants	4. tert-Butyllithium (t-BuLi)						240	4	
Desert Island Protecting Groups	5. Sodium hydride (NaH)						221	16	
A Day in the Life	6. Diethyl ether (Et ₂ O)						136	20	
1, 2, 3... Ph.D.	7. n-Butyllithium						131	6	
Proverbs	8. Potassium Metal						101	25	
Quotations And Advice	9. Sodium Hydride (NaH; dry, oil free)						101	3	
Always and Never	10. Raney nickel (dry)						96	4	
First Time Through Leaving the Lab									
Kende's 20 Points									
More Desert Island Resources									

http://chem.chem.rochester.edu/~nvd/pages/collective_wisdom.php?page=pyrophoric_reagents⁶⁶

20 of the top 25 pyrophoric reagents on the list **(80%)** are:



- ***Alkali metals*** and other elemental metals, metal alloys
- ***Hydrides*** (NaH, KH, R₂AlH)
- ***Metal Alkyls*** (RLi, R₂Zn, R₃Al, R₃B)

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Rookie Mistakes: **FIRE**



+



Had to sneeze whilst handling sodium - covered my mouth - gloves on fire!!

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Rookie Mistakes: **FIRE**



Didn't believe post-doc when he said "**KH is much more reactive than NaH**"...sink fire.

Photo Credits: <http://periodictable.com>

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Rookie Mistakes: **FIRE**



Add **Pd/C** catalyst to an ongoing **hydrogenation** without removing the H_2 first: fire!!

Photo Credits: <http://periodictable.com>

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Beware: Pyrophoric and Explosive Reagents

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About Follow Email Search...

Magic Formulas Tips and Tricks Troubleshooting How To Rookie Mistakes **Chemists Weigh In** Chromatography Reagents and Solvents Workup Tricks How to Run a Reaction

About Chemists Weigh In
Rookie Mistakes
Pyrophoric Reagents
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Leaving the Lab
Kende's 20 Points
More Desert Island Resources (Coming Soon)

Many lab fires and explosions are caused by the same few common reagents. The list below is provided to emphasize the dangers associated with working with these reagents. **However, even if your reagent is not on this list, it could still cause a fire or explosion.**

NEW: [UCSD Instructional Videos - Working with Pyrophoric Reagents](#)

Check out this [pyrophorics bulletin](#) and this [pyrophorics video](#)

To keep accurate data, please come back and record each fire/explosion you witness.

	Fires	Explosions	Vote
1. Sodium metal	289	84	<input type="checkbox"/>
2. Lithium aluminum hydride (LAH, LiAlH ₄)	283	63	<input type="checkbox"/>
3. Palladium on carbon (Pd/C)	301	31	<input type="checkbox"/>
4. tert-Butyllithium (t-BuLi)	240	4	<input type="checkbox"/>
5. Sodium hydride (NaH)	221	16	<input type="checkbox"/>

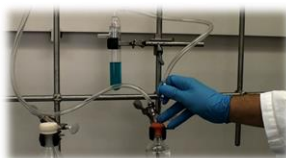
http://chem.chem.rochester.edu/~nvd/pages/collective_wisdom.php?page=pyrophoric_reagents⁷¹

How to Work with Pyrophoric Reagents

Check out these videos by Dr. Haim Weisman from UCSD



1. Getting Ready



2. Transferring Pyrophoric Liquids




3. Working with Reactive Metals

http://chem-courses.ucsd.edu/CoursePages/Uglabs/143A_Weizman/EHS/EHS.html⁷²

How to Add Your Rookie Experiences

TLC		
Mistake	Vote	# Rookies
spot on the TLC plate.	<input checked="" type="checkbox"/>	165
nd walked away to to something else... remembered the TLC	<input checked="" type="checkbox"/>	86
ting plate. Didn't realize until visualization stage.	<input checked="" type="checkbox"/>	80
vo solvents that were immiscible.	<input checked="" type="checkbox"/>	22
ia TLC with an ink pen	<input checked="" type="checkbox"/>	18
apillary tube through thin rubber septum to obtain TLC sample.	<input checked="" type="checkbox"/>	5
iger...trip to the emergency room!		

Add Your Experience



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How to Add Your Rookie Experiences

Voodoo X

About Follow Email Search...

Tips Troubleshooting How To **Rookie** Chemists Chromatography Reagents Workup How to Run

Add Experiences Add a New Mistake

Check the box if you've made the mistake

Used DMSO to dissolve the only 5 mg sample available for an NMR and then couldn't remove DMSO afterward.

Left reaction to reflux, came back 4 hours later to find I had forgotten to switch the hotplate on.

Drew up solvent from Sure-Seal bottle into large plastic syringe. Pulled furiously on plunger to increase flow-rate while holding solvent bottle to chest. Plunger popped out - solvent in the face!

Reaction in THF with potassium (molten) violently foams out of the reflux condenser resulting in river of fire.

Set up a complicated system for the reaction, then discovered that the lab did not have enough of the required solvent.

Enter Enter & Get More Close

Silica plom in my face...and lungs. 74

How to Add Your Rookie Experiences

Tips Troubleshooting How To Rookie Chemists Chromatography Reagents Workup How to Run

Add Experiences Add a New Mistake

Add a Mistake

Mistake Id be avoided by making a Rookie Mistakes list!

Preview: Assumed all mistakes could be avoided by making a Rookie Mistakes list!

Show Formatting Tips

Save Close

Under Pressure Separated Impurity.

Sound of Breaking Glass Poured silica gel before placing the cotton plug in the column, watched the silica gel flow right out. 64

Fire in the Lab

Just... Wow

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Acknowledgements

- Harry Stern
- Hiatt Zhao
- Colin Kinz-Thompson
- Chris Bauer
- Website visitors since 2004
- NSF (Division of Chemistry)

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“Rookie Lab Mistakes and Other Facts Not Found in Textbooks”



Bill Courtney
Chef and Analytical Chemist,
Cheese-ology



Dr. Alison Frontier
Professor of Chemistry,
University of Rochester

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Dr. Christopher Williams, Associate Professor, Department of Mechanical Engineering, Assistant Director of Macromolecular and Interfaces Institute, Virginia Tech University



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Dr. Paul Leeson, Director, Paul Leeson Consulting, Ltd.

Dr. Rick Connell, Vice President, External Research Solutions, Pfizer

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“Rookie Lab Mistakes and Other Facts Not Found in Textbooks”



Bill Courtney
Chef and Analytical Chemist,
Cheese-ology

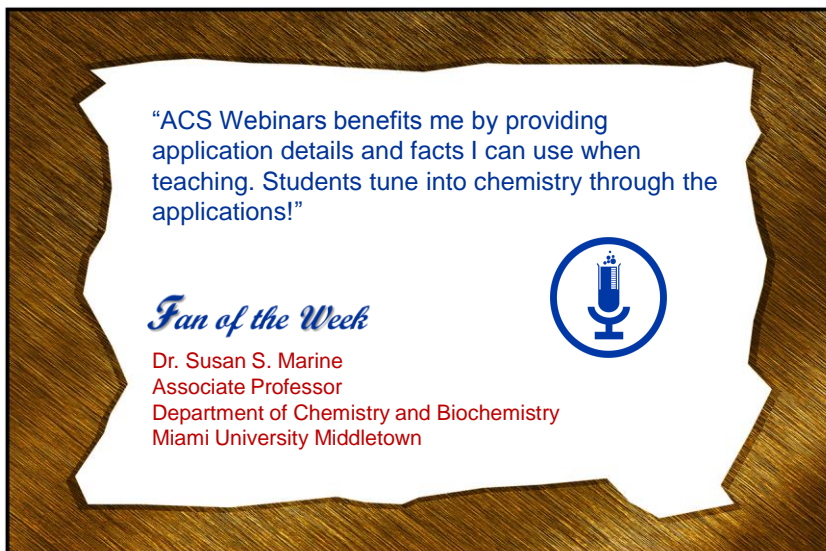
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