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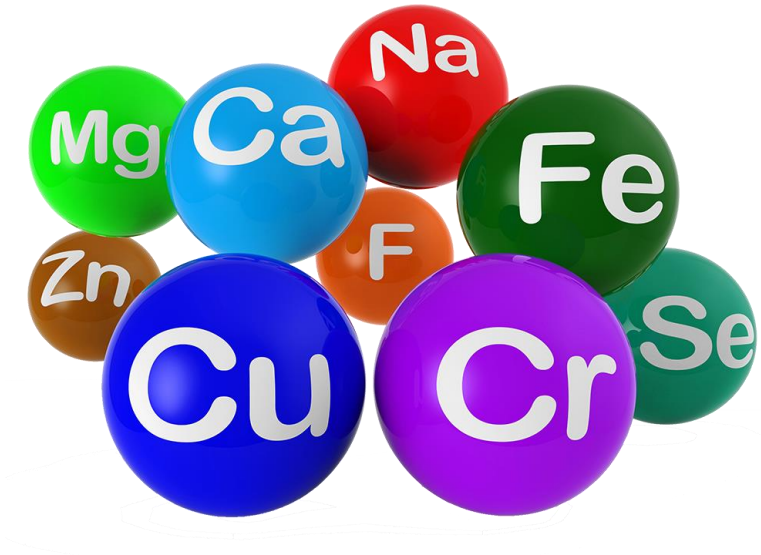

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Got a bit too
"swirl-happy"
#rookielabmistakes


Washed plastic
safety specs with
Acetone
#rookielabmistakes


BUMPED
SOLUTION ON
ROTOVAP
#rookielabmistakes

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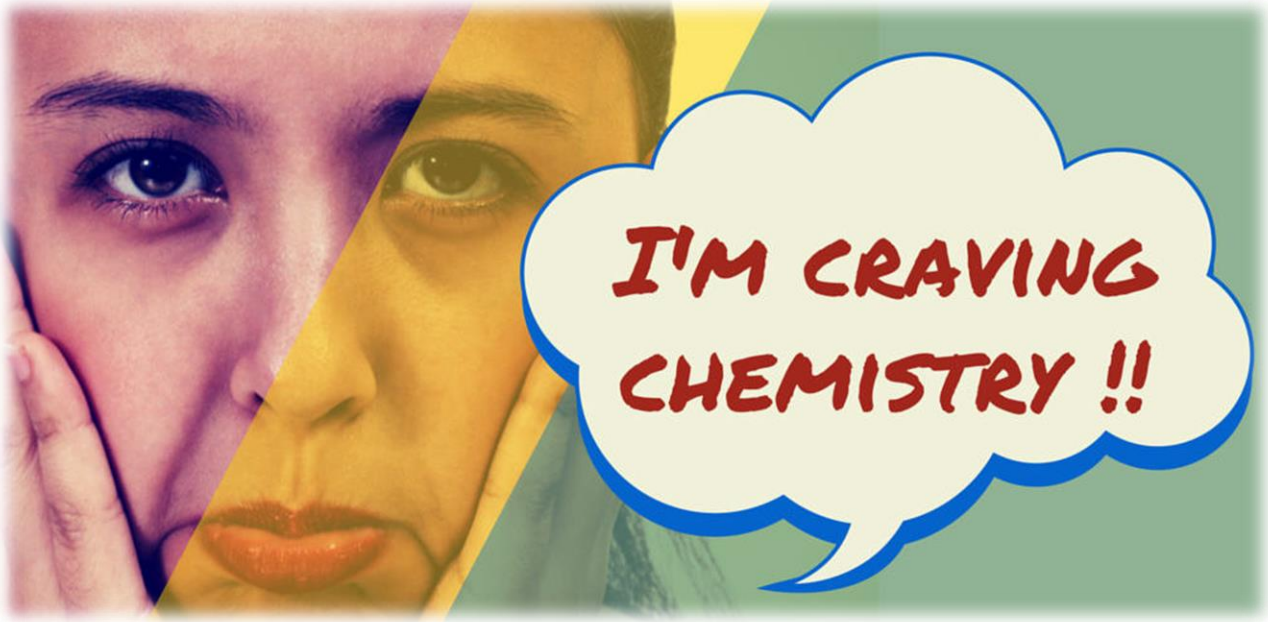

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Dr. Timothy Long, Professor, Department of Chemistry, Director of Macromolecules and Interfaces Institute, Virginia Tech University

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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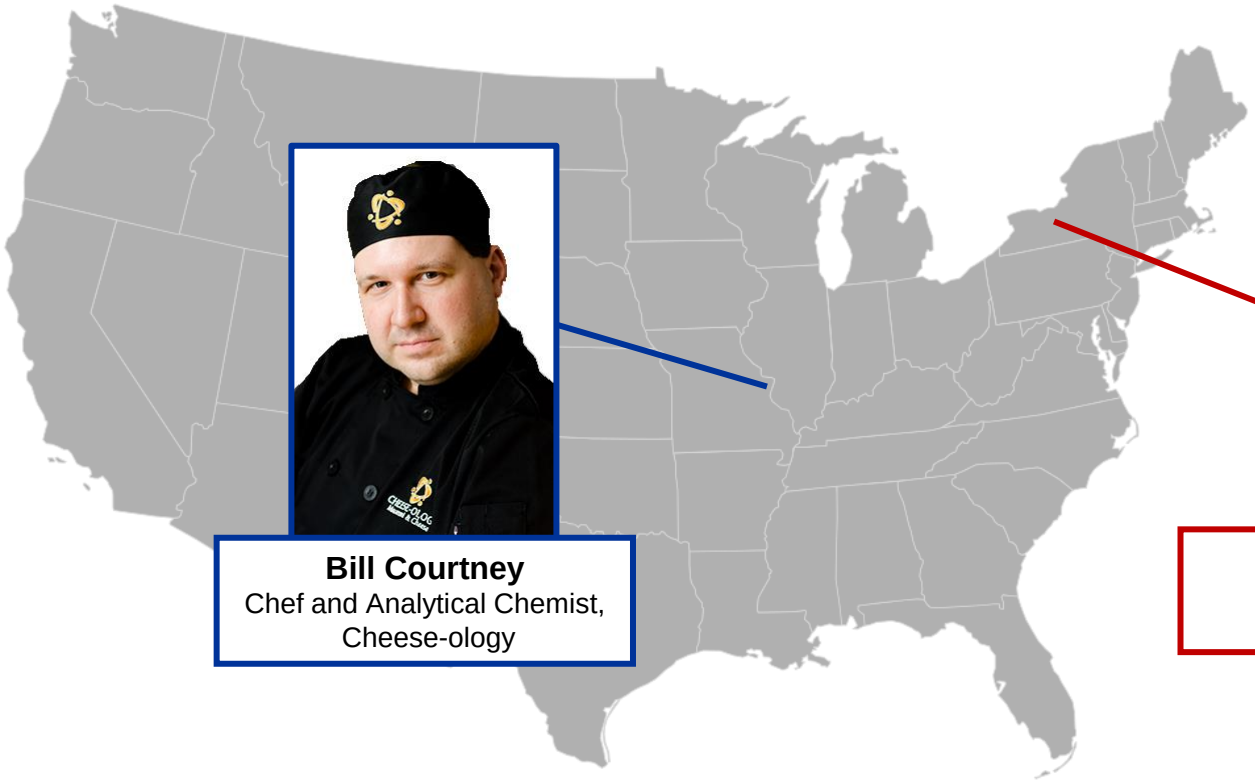
ACS Chemistry for Life®

"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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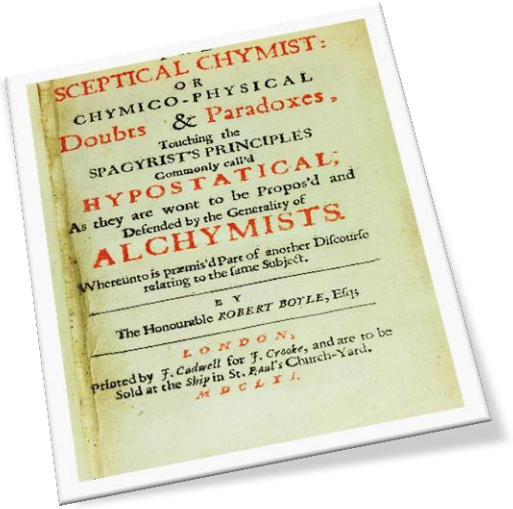
We all want to avoid
A Nightmare Scenario…

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

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

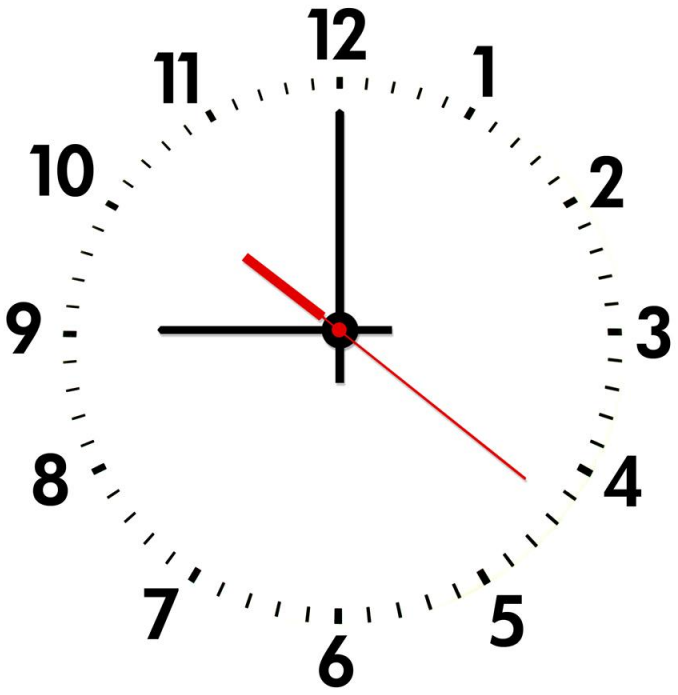
b) from 1927

c) has no experimental details

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

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

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

A Nightmare Reaction

Monitoring the Reaction

Progress cannot be monitored by TLC.

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

A Nightmare Reaction

Workup

The product mixture is *highly reactive* and requires *dropwise addition* of water to prevent a volcano-like exothermic eruption

OR

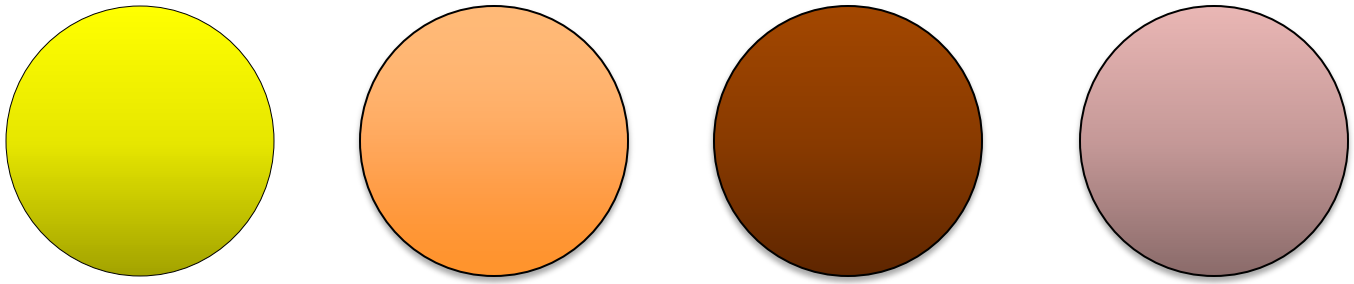
http://chem.chem.rochester.edu/~nvd/pages/reaction.php?page=nightmare

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

A Nightmare Reaction

Workup

The aqueous solution you use to wash the organic layer turns yellow, orange, brown or pink- *and ten washes later, it's still there!*



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

A Nightmare Reaction

Purification

I M P U R I T I E S

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

Your compound is an *oil*. No purification method has been reported for its isolation. The crude reaction mixture has your product and *three or four minor impurities*, all with the *same R_f value*.

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

Inherent challenges

+

The Wizardry of Expert Chemists

= *Voodoo?*

Photo Credit: http://trail.pugetsound.edu/2013/10/chemistry-magic-show-puget-sound-chemists-gear-up-for-a-night-of-enchantment/

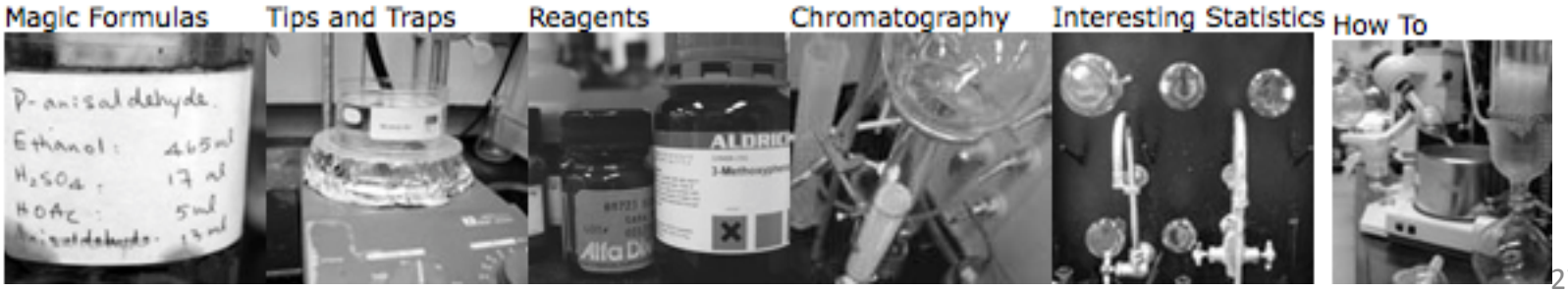
Not Voodoo:

Demystifying Organic Laboratory Technique

In 2004: 11 Rookie Mistakes

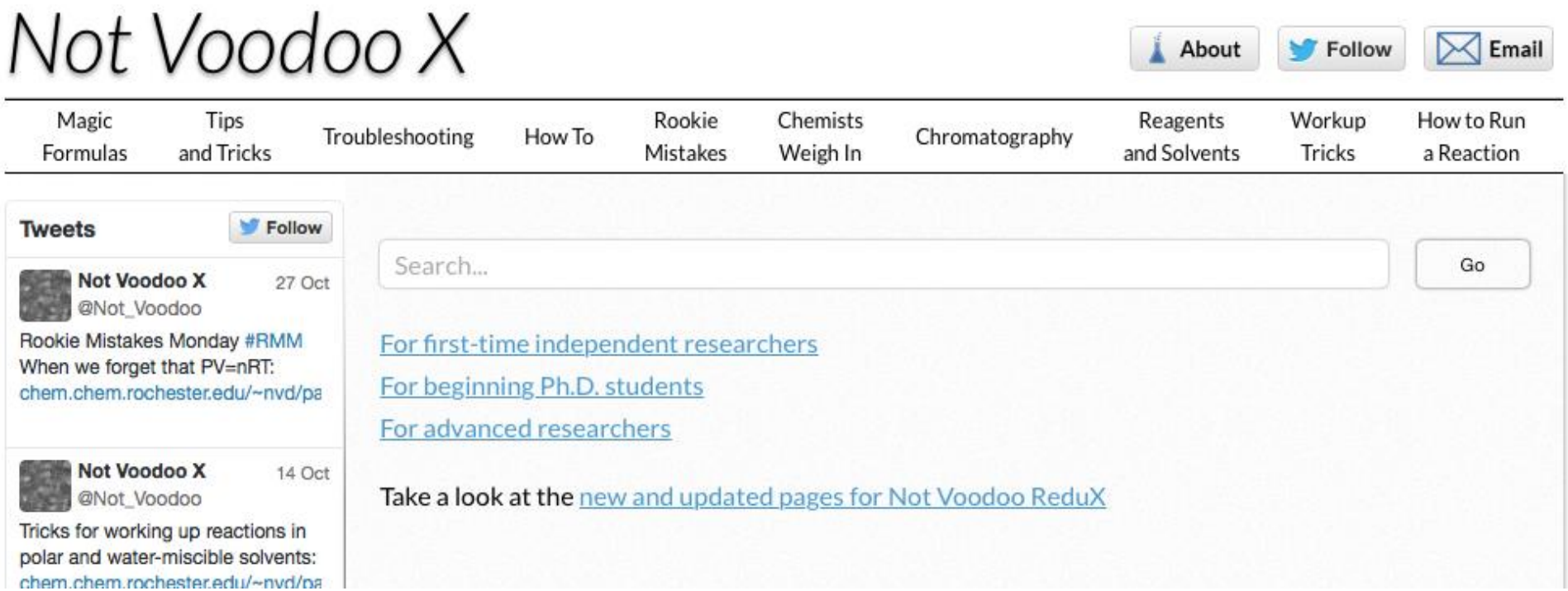
In 2014: >250 Rookie Mistakes

- **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
- **Search Not Voodoo:** Search



In September 2014: Not Voodoo X:

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



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.

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***

Most Common Rookie Mistakes

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

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.php

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.”

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

Most Common Rookie Mistakes

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

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

An Observation:

Everyone Forgets

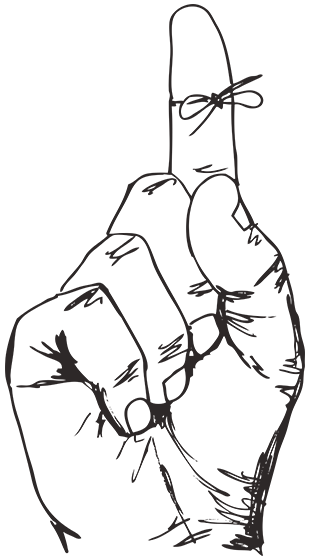
5: *Didn't label* a flask. One week later, have NO idea what is inside.

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*



Routines Help You Remember

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

Other things that should look wrong

- An occupied round-bottom flask without a label
- A sep funnel without an Erlenmeyer flask under it.
- An empty round-bottom flask under argon

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

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

Trends Emerging from the Mistakes

Not Voodoo X

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

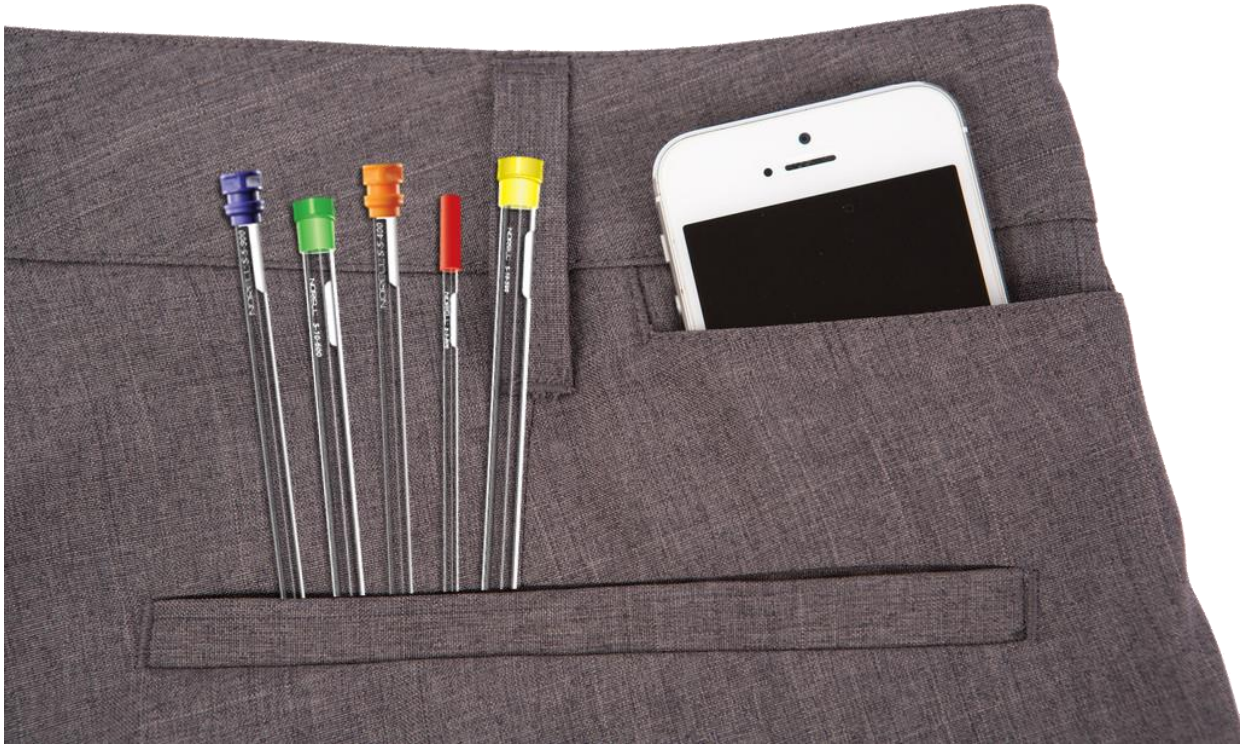
- About Rookie Mistakes
- Equipment
- Reagents
- Setup
- TLC
- Workup
- Column Chromatography
- Spectroscopy
- The Vacuum Apparatus
- Labels and Bookkeeping
- Ouch
- Under Pressure
- The Sound of Breaking Glass
- Fire in the Lab
- Just… Wow
- Top Ten
- Search

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.

The Rookie Mistakes did not exactly work out that way. What began as an innocent compilation of beginner bungles 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.

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 an unexpected source of solace after a frustrating day in the lab. That's partly because it's funny, but also because you don't feel quite so incompetent after reading about how other people accidently destroyed their experiments.

Ouch



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

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

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 dessicator shattering into a thousand pieces.”

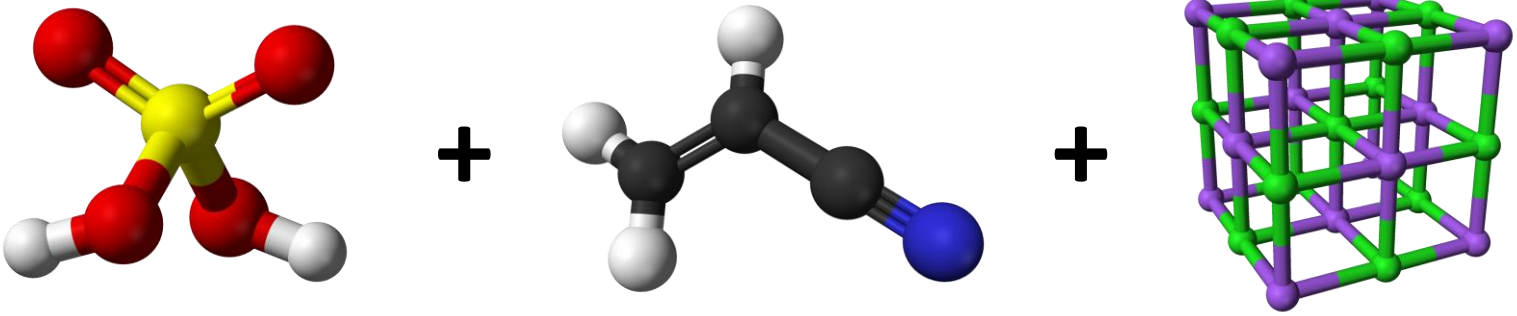
The Sound of Breaking Glass

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

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....."



Audience Survey Question

Which piece of equipment is most difficult to master?

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

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.”

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/f6resoursebk-eimg12.html

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

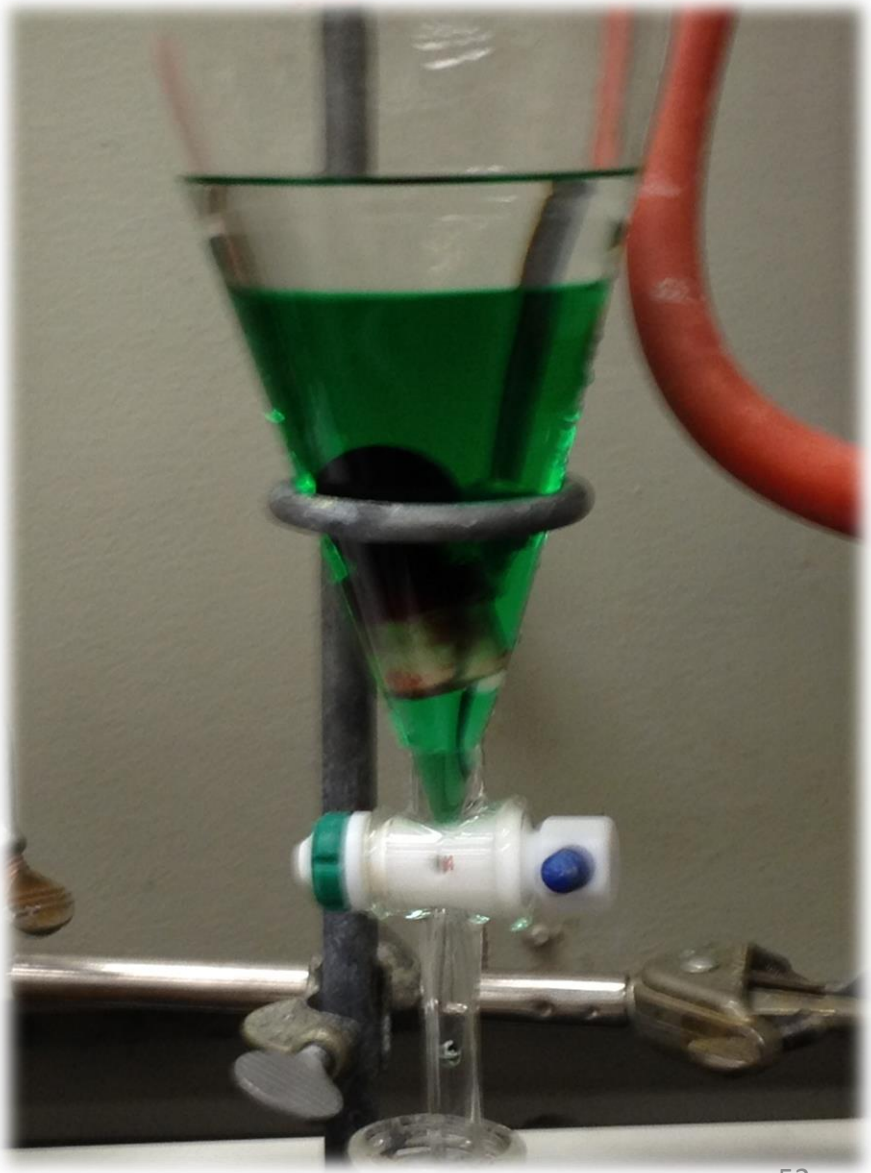
Lessons Learned:

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 ***Vigreaux 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***.

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…

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***.

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

Lessons Learned:

How to Handle an Emulsion

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

Tips:

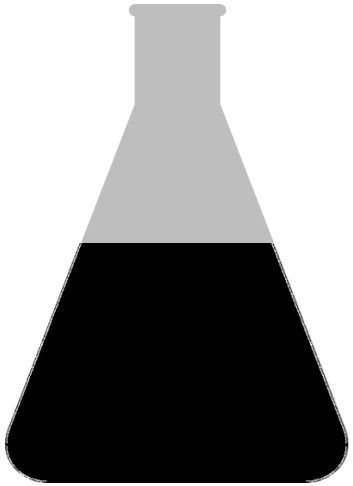
- Evaporating the reaction solvent *before* workup
- Wait.
- Add solid NaCl
- Dilute with copious organic solvent
- Filter the whole thing through Celite

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

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

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

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."

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

- 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**

Search by Keyword:

stir bar | Go

May we suggest: acetone, forgot, oven, quench, stir bar, heat, acid, dropped, condenser, rotovap, glove box, bromine, water, TLC, sep funnel

Mistake	Vote	# Rookies
Set up reaction under Ar, added in reagents and forgot to add stir bar.		240
Used a little stir bar with a big flask.		165
While cleaning beakers with stir bars inside, poured the stir bars down the drain.		159
Poured stir bar into sep funnel, decided to shake it anyway and shattered the funnel.		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.		32
Added stir bar to flask. Stir bar smashes a hole in bottom of the flask, contents of flask now all over fume hood.		26
Lost a magnetic stir bar in the solid waste container.		12

Audience Survey Question

Which reagent is most difficult to work with?

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

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

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

Workup Tricks

Workup for Removing Tin Byproducts

Six different workup methods for removal of tin byproducts from a reaction mixture are listed below:

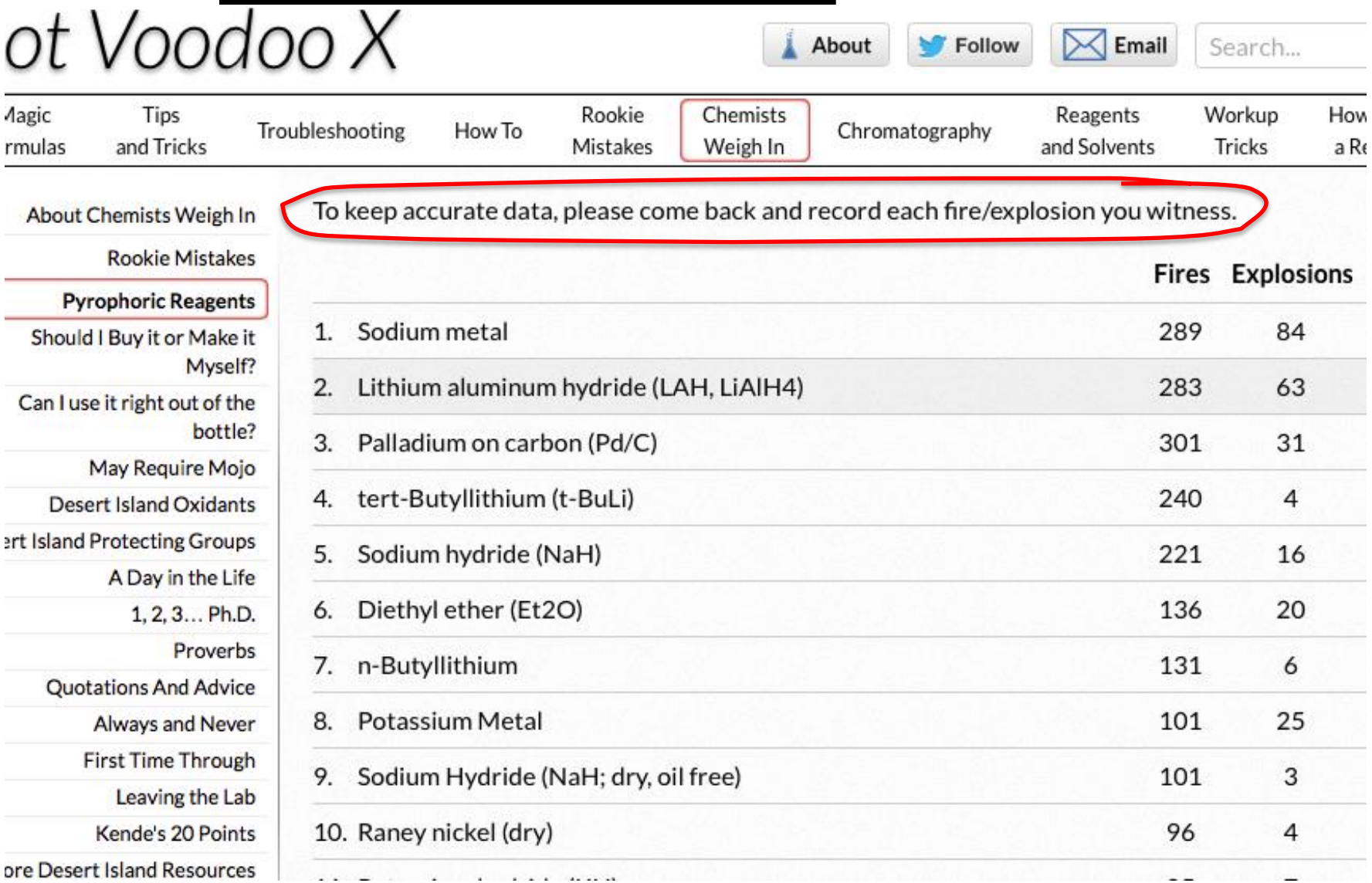
1. Byproducts of tin based reactions such as Bu_3SnBr can be removed by treatment with $AlMe_3$ to create the nonpolar Bu_3SnMe or NaOH to create the polar Bu_3SnOH. See: P. Renaud, E. Lacote, L. Quaranta Tetrahedron Lett. 1998, 39, 2123. (partially taken from the Merlic Group at UCLA, The Organic Companion)
2. Filter through a mixture of KF/Celite
3. a. Dilute rxn with suitable organic solvent
   b. Add aqueous phase (water, sat. NH_4Cl, etc.)
   c. 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.
   d. Wash aq 1 X brine, dry organic phase and remove solvent
4. 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

Visitor Experience 2004-2014



ot Voodoo X

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About Chemists Weigh In
Rookie Mistakes
Pyrophoric Reagents
Should I Buy it or Make it Myself?
Can I use it right out of the bottle?
May Require Mojo
Desert Island Oxidants
ert Island Protecting Groups
A Day in the Life
1, 2, 3… Ph.D.
Proverbs
Quotations And Advice
Always and Never
First Time Through
Leaving the Lab
Kende's 20 Points
ore Desert Island Resources

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

		Fires	Explosions
1.	Sodium metal	289	84
2.	Lithium aluminum hydride (LAH, LiAlH4)	283	63
3.	Palladium on carbon (Pd/C)	301	31
4.	tert-Butyllithium (t-BuLi)	240	4
5.	Sodium hydride (NaH)	221	16
6.	Diethyl ether (Et2O)	136	20
7.	n-Butyllithium	131	6
8.	Potassium Metal	101	25
9.	Sodium Hydride (NaH; dry, oil free)	101	3
10.	Raney nickel (dry)	96	4

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

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

- ***Alkali metals*** and other elemental metals, metal alloys
- ***Hydrides*** (NaH, KH, R_2AlH)
- ***Metal Alkyls*** (RLi, R_2Zn, R_3Al, R_3B)

Rookie Mistakes: FIRE

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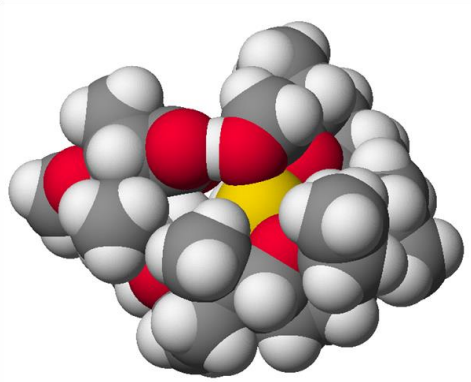
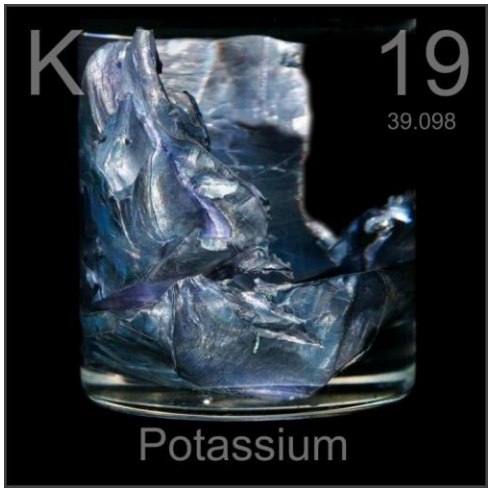
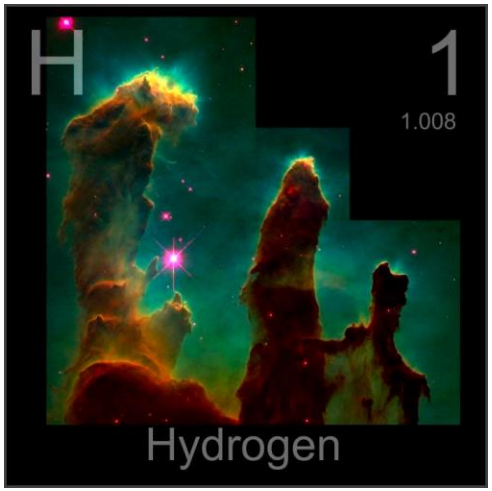


“Had to sneeze whilst handling **sodium** - covered my mouth - gloves on fire!!”

Rookie Mistakes: FIRE

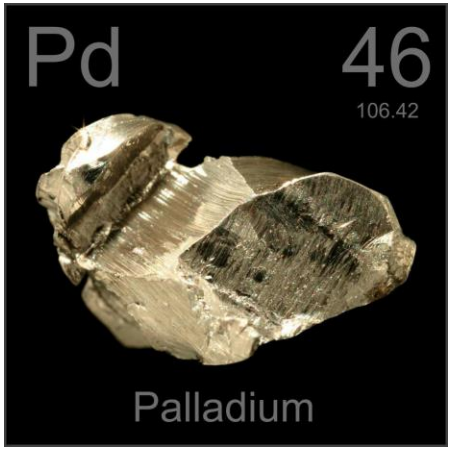




"Didn't believe post-doc when he said **"KH is much more reactive than NaH"...**sink fire."

Photo Credits: http://periodictable.com

Rookie Mistakes: FIRE



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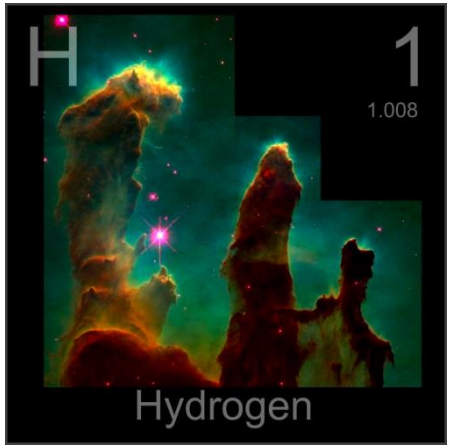

"Added **Pd/C** catalyst to an ongoing **hydrogenation** without removing the H_2 first: fire!!"

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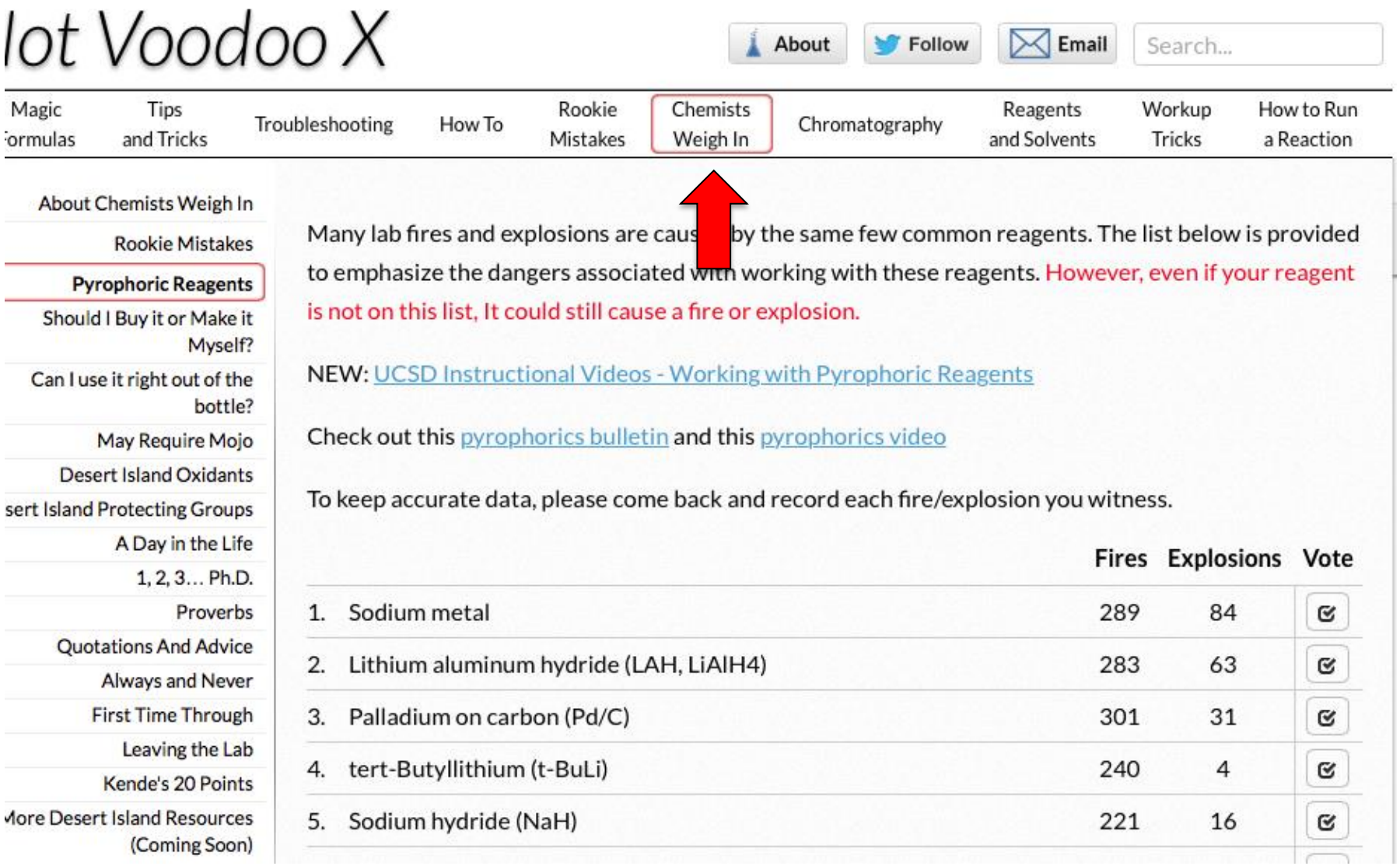
Beware: Pyrophoric and Explosive Reagents



lot Voodoo X

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- **Pyrophoric Reagents**
- Should I Buy it or Make it Myself?
- Can I use it right out of the bottle?
- May Require Mojo
- Desert Island Oxidants
- sert Island Protecting Groups
- A Day in the Life
- 1, 2, 3… Ph.D.
- Proverbs
- Quotations And Advice
- Always and Never
- First Time Through
- Leaving the Lab
- Kende's 20 Points
- More Desert Island Resources (Coming Soon)

Many lab fires and explosions are caus 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	
2.	Lithium aluminum hydride (LAH, LiAlH4)	283	63	
3.	Palladium on carbon (Pd/C)	301	31	
4.	tert-Butyllithium (t-BuLi)	240	4	
5.	Sodium hydride (NaH)	221	16	

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

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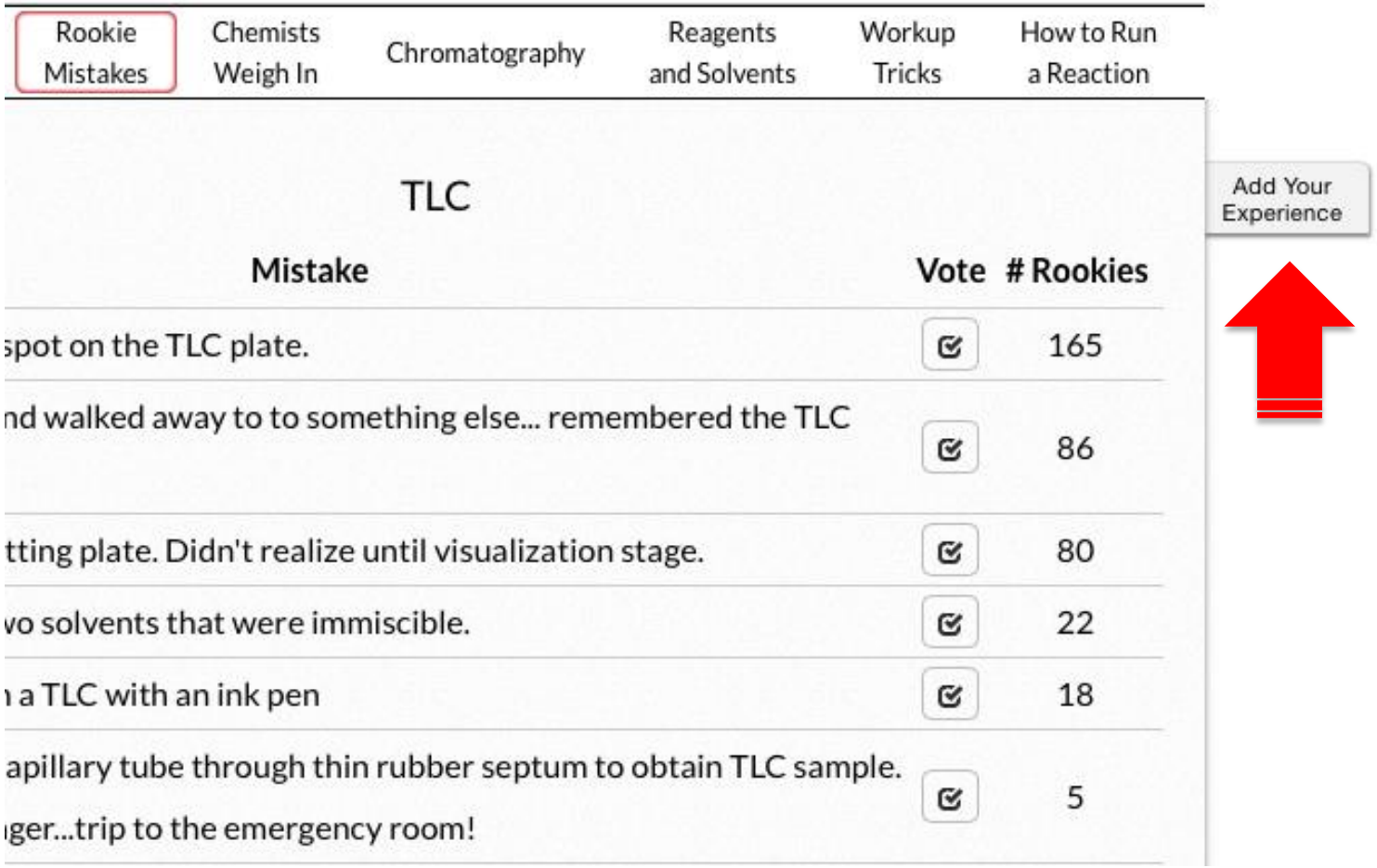
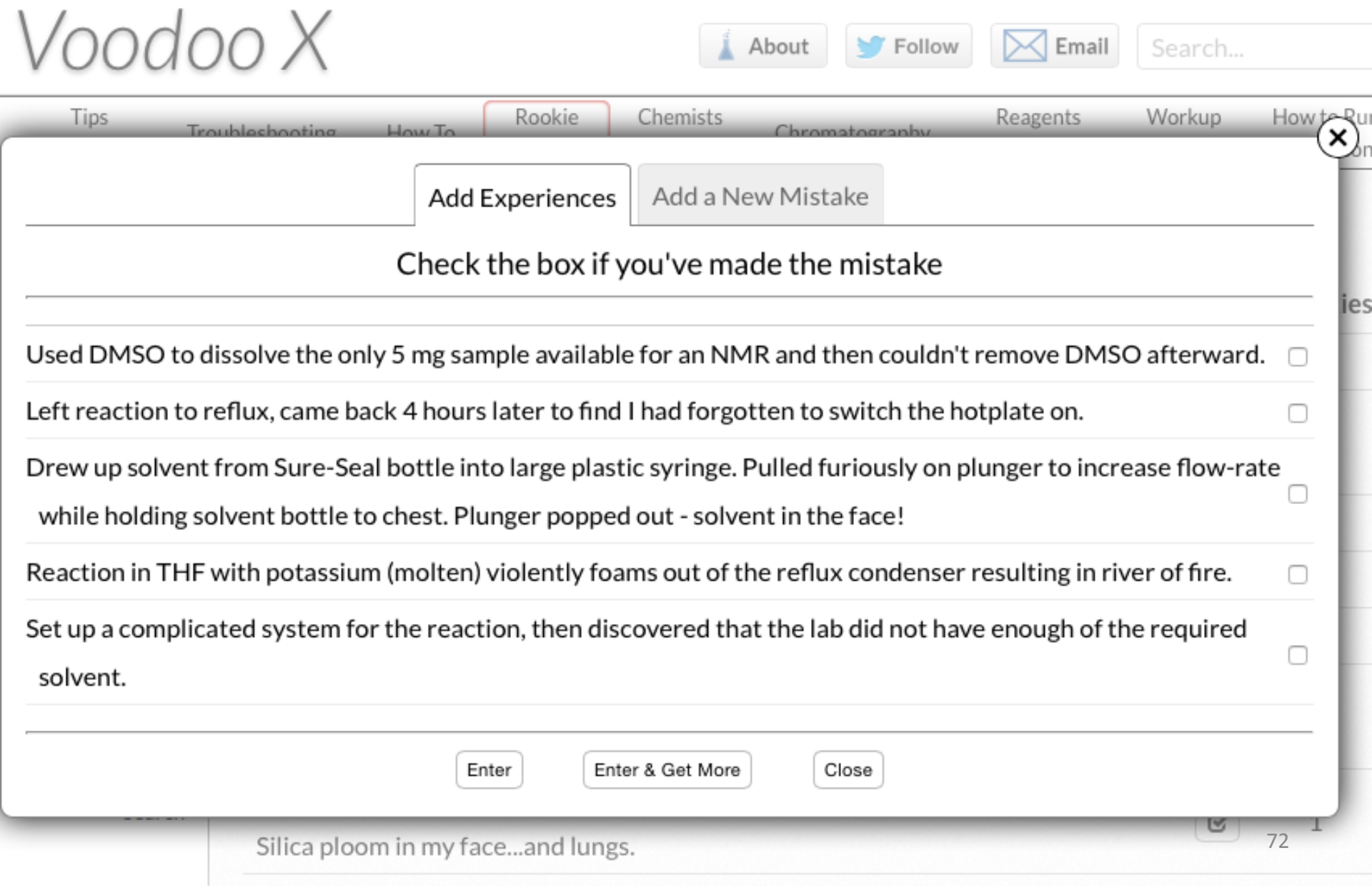
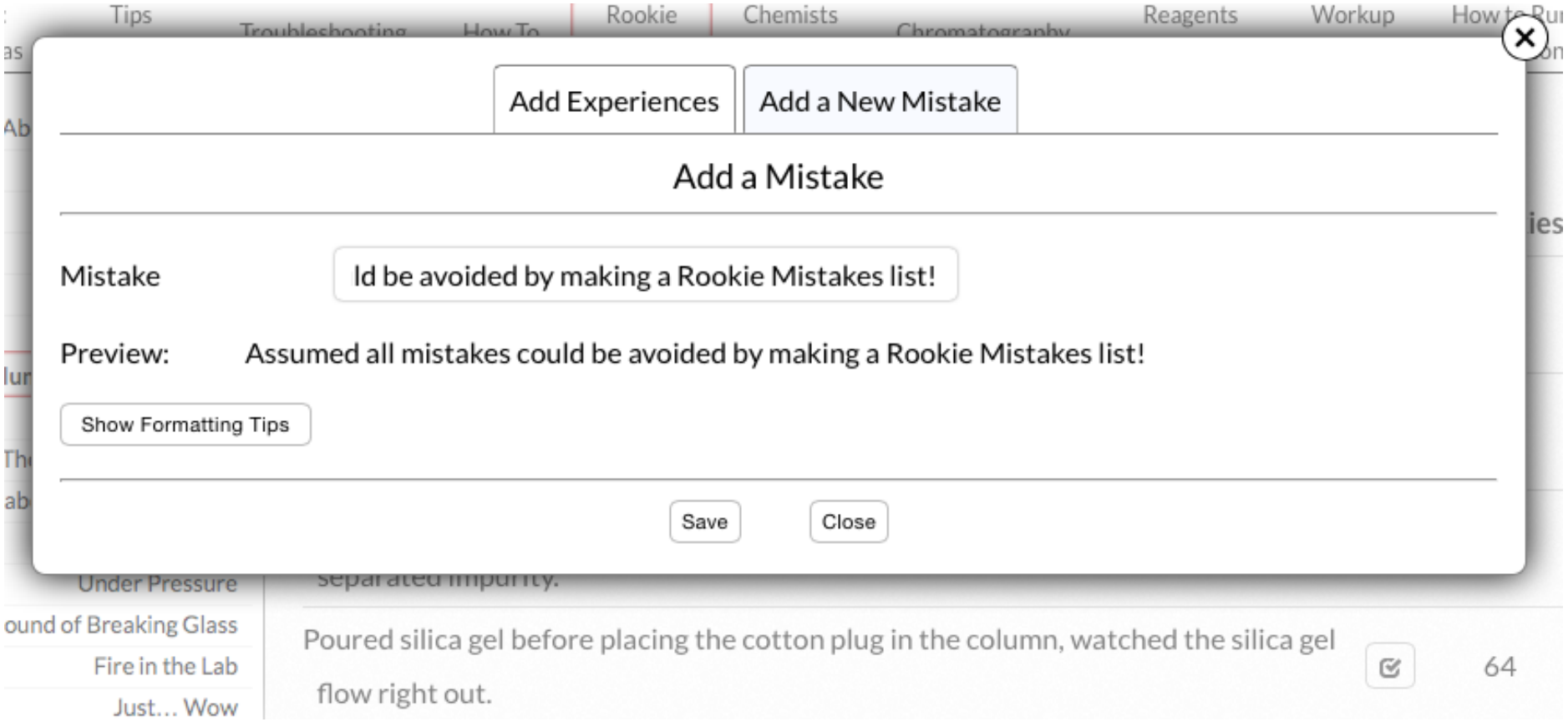


How to Add Your Rookie Experiences



How to Add Your Rookie Experiences



Acknowledgements

- Harry Stern
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- Chris Bauer
- Website visitors since 2004
- NSF (Division of Chemistry)

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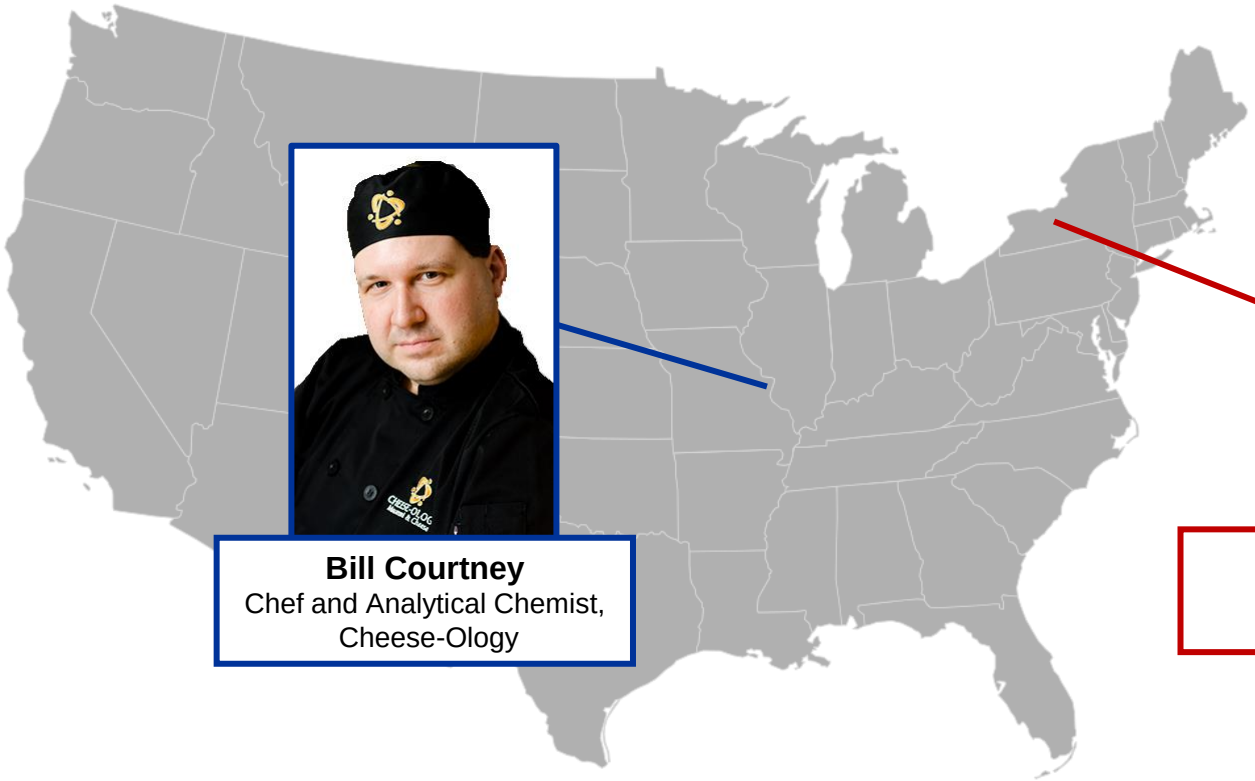

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Chef and Analytical Chemist,
Cheese-Ology

Dr. Alison Frontier
Professor of Chemistry,
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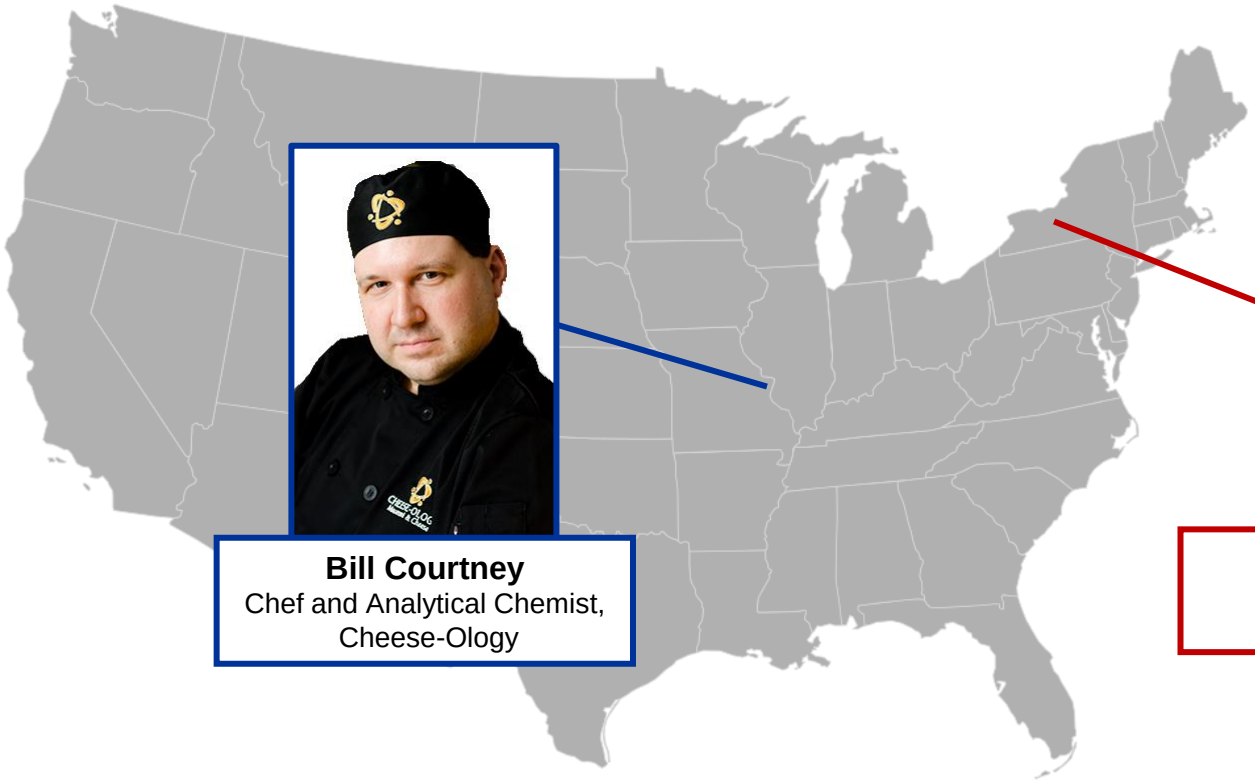

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