

enabling and empowering students

strategies for maintaining a strong safety culture



Ian A. Tonks
University of Minnesota – Twin Cities

LSI Graduate Research Faculty Safety Award
August 22 2021



 @ianatonks
sites.google.com/umn.edu/tonksgroup

hazards in the Tonks Group
(early transition metal organometallic synthesis and catalysis)



strong oxidizers



chronic toxins



pyrophorics
(#1a concern)



high pressures
(#1b concern)



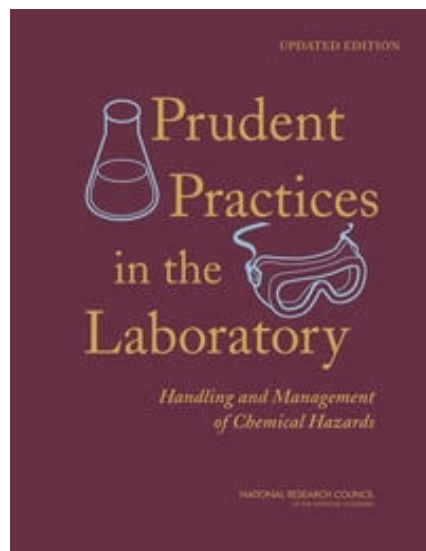
reactives and
explosives

what are the unique problems we face with graduate education and safety?

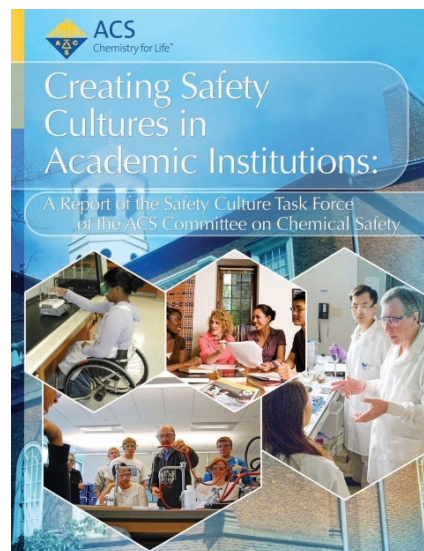
- we are always training new students
- expertise leaves with graduation
- awareness of safety (you don't know what you don't know!)
- miscalculations of risk (how bad could an explosion really be?)

how does this fit in to managing and advising my research group and departmental safety?

communication is key in safety culture



2011: *Prudent Practices*



2012: ACS



2014: National Academies



2016: APLU

Recent reports on safety and safety culture in academia consistently hit on three themes:

- *Setting core values and leading by example*
- *Empowering researchers to collaborate and practice safe science*
- *Implementing constructive and supportive infrastructure*



**communicating & building
better ways to communicate!**

National Research Council. *Prudent Practices in the Laboratory: Handling and Management of Chemical Hazards*; National Academies Press: Washington, D.C., 2011.

Creating Safety Cultures in Academic Institutions: A Report of the Safety Culture Task Force of the ACS Committee on Chemical Safety; 2012.

Safe Science: Promoting a Culture of Safety in Academic Chemical Research; 2014.

APLU Council on Research Task Force on Laboratory Safety. *A Guide to Implementing a Safety Culture in our Universities*; 2016.

Minnesota Safety Over ~8 Years:

- Joint Safety Team Creation
- Adoption of Standard Operating Procedures (SOPs)
- Safety Moments
- Safety Advertising (Stall Wall Moments)
- Faculty SOP Peer Reviews
- Anonymous Learning Experience Reports
- Safe Operating Cards



Commentary

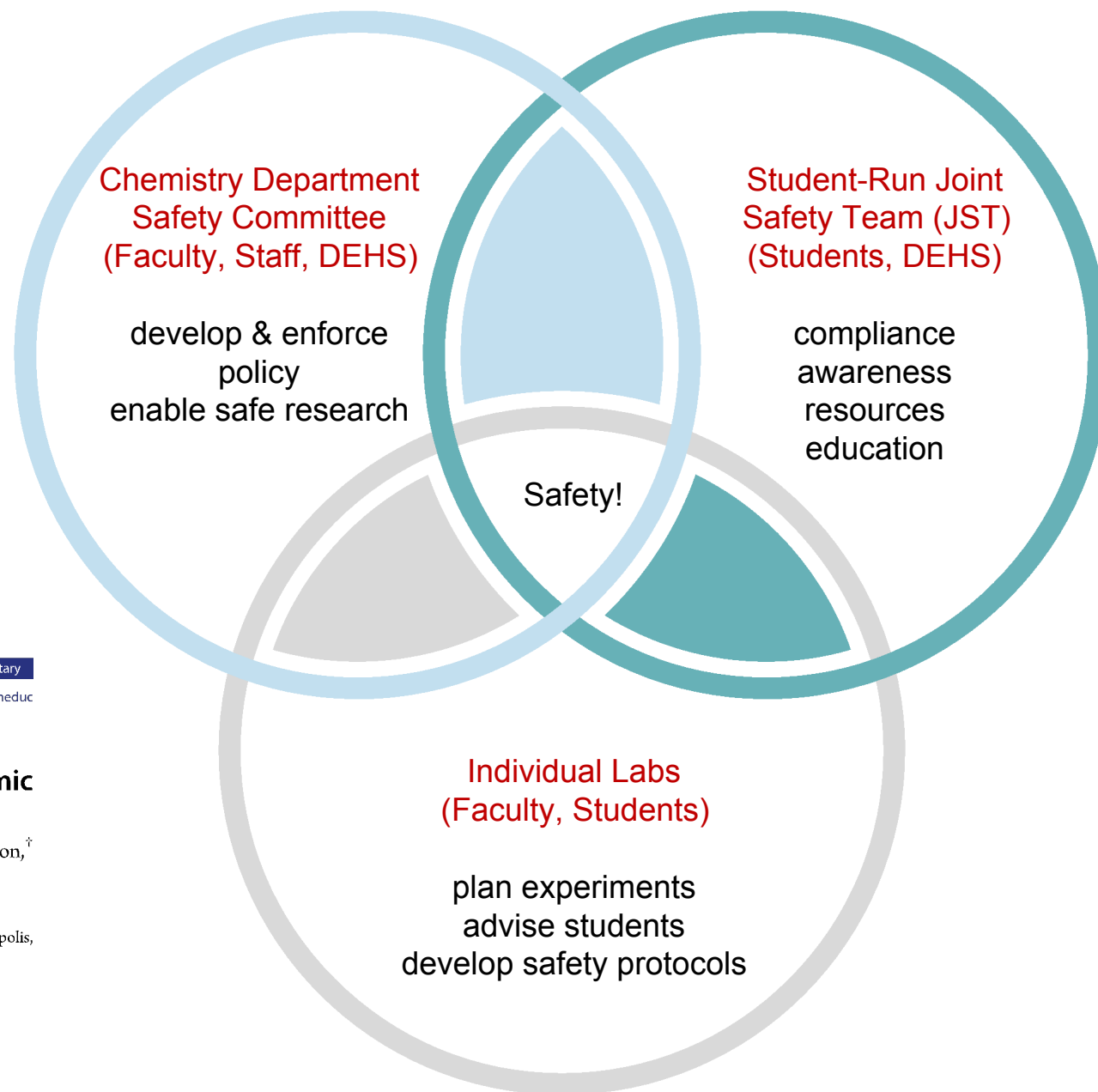
pubs.acs.org/jchemeduc

Student Involvement in Improving the Culture of Safety in Academic Laboratories

Kathryn A. McGarry,[†] Katie R. Hurley,[†] Kelly A. Volp,[†] Ian M. Hill,[‡] Brian A. Merritt,[‡] Katie L. Peterson,[†] P. Alex Rudd,[†] Nicholas C. Erickson,[‡] Lori A. Seiler,[§] Pankaj Gupta,[§] Frank S. Bates,[‡] and William B. Tolman^{*,†}

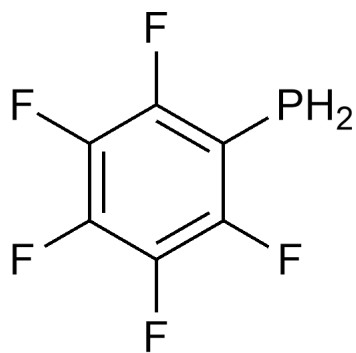
[†]Department of Chemistry and [‡]Department of Chemical Engineering and Materials Science, University of Minnesota, Minneapolis, Minnesota 55455, United States

[§]Dow Chemical Company, Midland, Michigan 48674, United States



outline: case studies in communication

Why is it important for us to communicate about “near-misses” and other potential dangers?



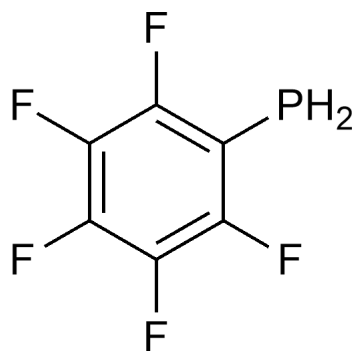
*don't make this using
the reported prep.*



Learning Experience Reports

outline: case studies in communication

Why is it important for us to communicate about “near-misses” and other potential dangers?



*don't make this using
the reported prep.*



Learning Experience Reports

Why is it important to have (and review) written standards and procedures?



*if you're gonna make this,
follow the directions.*

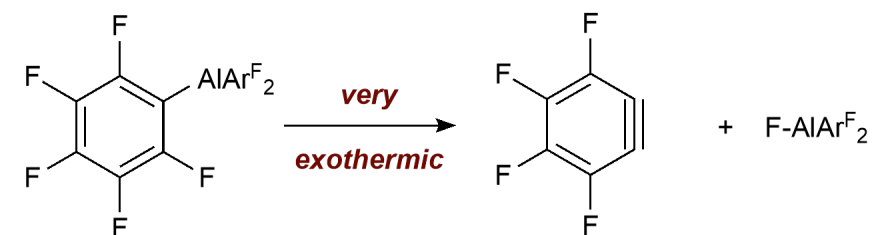
don't do it on process scale.



Standard Operating Procedures

fluorinated organics and Al or Mg: a bad combination

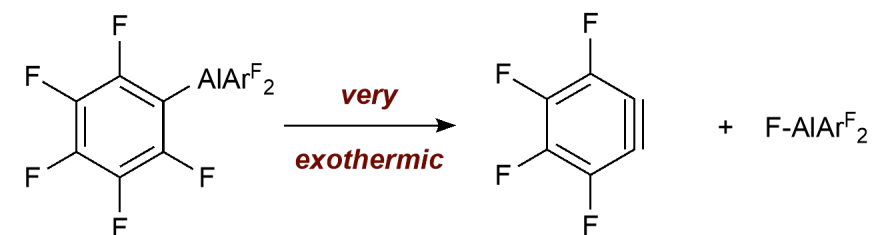
Preface: Al or Mg (and other hard, fluorophilic metals) will readily and exothermically β -F abstract from Ar-F



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Handling, Storage, and Precautions: extra caution should be exercised when handling this compound, especially the unsolvated form (due to its thermal and shock sensitivity)! An explosion was reported in an attempt to sublime the crude etherate prepared from the reaction of AlCl_3 and 3 equiv of $\text{C}_6\text{F}_5\text{MgBr}$ in ether or in an attempt to prepare the unsolvated $\text{Al}(\text{C}_6\text{F}_5)_3$ by heating a mixture of $\text{B}(\text{C}_6\text{F}_5)_3$ and AlEt_3 to 70°C .¹ Store the solid alane under inert (argon or nitrogen) atmosphere in a glove box and its solutions in a refrigerator inside the glove box.

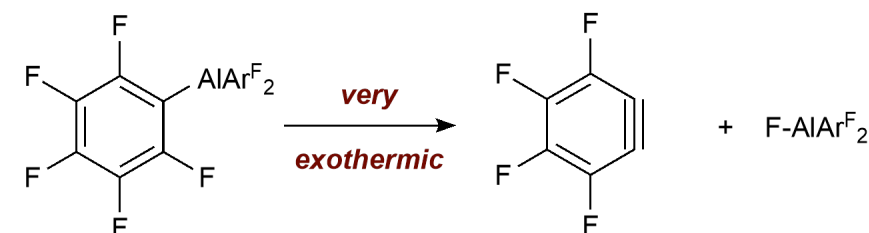


Tris(pentafluorophenyl)alane, *e-EROS*, 2012
<https://doi.org/10.1002/047084289X.rn01382>

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Boulder Scientific: Fatality in 2008

Accident Investigation Summary

Summary Nr: 201573466

Event: 05/16/2008

Employee Dies After Drying Oven Explodes

On May 16, 2008, Employee #1 was walking in a hall at a facility that used a vacuum drying oven to dry chemicals. The chemical in the oven was potassium tetrakis (pentafluorophenyl) borate which contained hexane and toluene. The oven was being used to remove the hexane and toluene. The chemicals ignited and caused an explosion that drove steel door off its hinges, turned the oven 45 degrees, and ruptured the condensate line. Additional hexane and toluene spilled into the area which caused a second explosion. The second explosion drove debris and material toward Employee #1 who was passing through the area in an exterior hall. Employee #1 suffered third-degree burns and unspecified internal injuries. He died due to internal bleeding.

US Department of Labor, OSHA Inspection: 311903751 - Boulder Scientific Company

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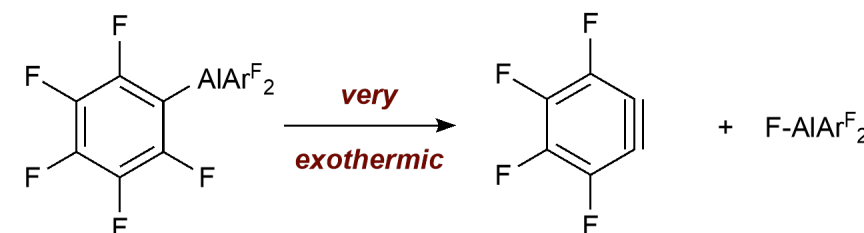
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On May 16, 2008, Employee #1 was walking in a hall at a facility that used a vacuum oven to dry **borate which contained hexane and toluene**. The oven was being used to remove the solvent from the borate. The oven was turned on, the temperature was increased to 45 degrees, and ruptured the condensate line. Additional borate debris and material toward Employee #1 who was passing through the area. Employee #1 was killed due to internal bleeding.

US Department of Labor, OSHA Inspection: 311903751 - Boulder Scientific Co



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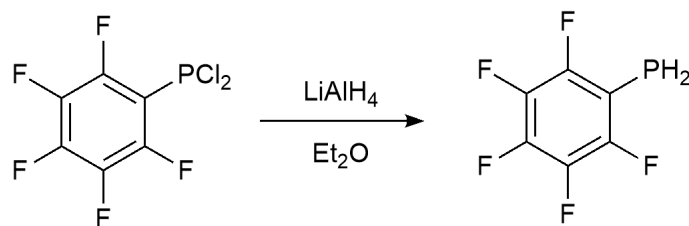
US Patent No. 5,473,036.

PROCESS FOR FORMING A BROMOMAGNESIUM TETRAKIS (FLUOROPHENYL) BORATE

A solution of 104 millimoles of pentafluorophenyl-magnesium bromide in 60 ml of ether was prepared using 2.89 g of magnesium and 25.9 grams of pentafluorophenyl bromide. Unreacted magnesium (0.32 g) was removed by filtration and the solution was diluted with 130 ml of dry butyl ether. This was then followed by the addition of 3.71 g of boron trifluoride etherate. The flask with the reaction mixture was then placed in an oil bath, and the bath temperature was increased to allow gradual ether removal by distillation. About 50 ml of solvent was removed. Any residual ether was then removed by vacuum distillation leaving about 40–50 ml of liquid. (According to the literature, complete removal of the solvent may result in an explosion). Fresh butyl ether in the amount of 120 ml was then added and the reaction mixture was stirred at room

$\text{C}_6\text{F}_5\text{PH}_2$: a missed opportunity to report hazards

Jan 2012, IAT: is this a good idea?



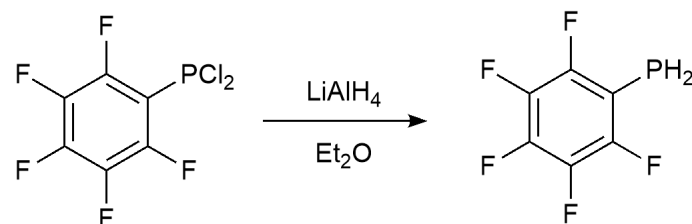
*Z. Naturforsch.***1966** 21b, 920

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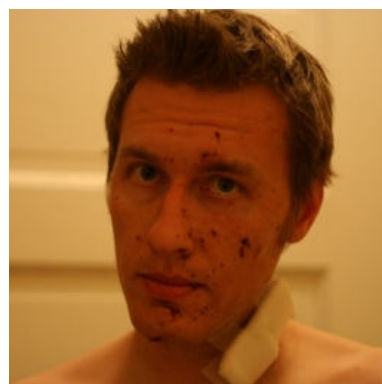
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Detonated prior to distillation:



Z. Naturforschg.1966 21b, 920



Safety Alert: Explosion During Prep Of $(\text{C}_6\text{F}_5)\text{PH}_2$

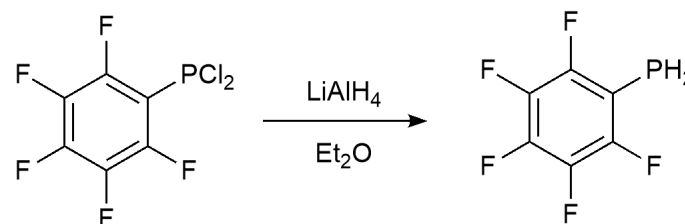
MAY 21, 2012 | APPEARED IN **VOLUME 90, ISSUE 21**

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MAY 21, 2012 | APPEARED IN VOLUME 90, ISSUE 21

Jan 30, 2018

Around 15 yrs ago a postdoc I was working with did the same reaction and his material also went off during the distillation. Luckily only a couple flasks were broken and nobody was hurt. We were both standing about 10 ft away. He said he wouldn't do that reaction again. :)

1 4 6

Ian Tonks @ianatonks · Jan 30, 2018

This is why people should share. I wouldn't have glass in my face if someone had noted that.

2 5 26

UMN JST interview: <https://www.youtube.com/watch?v=3zqw8kKygm8>

C&EN interview: <https://cen.acs.org/safety/Podcast-Lessons-learned-lab-safety/97/i30>

Learning experience reports: an easy (anonymous!) way to share

LERs: A Joint Safety Team (JST) initiative inspired by Dow Chemical:
<http://www.jst.umn.edu/learning-experience-reports>

Learning Experience Reports

If you experienced or observed an incident, an accident, or an 'almost!' situation, please report it here:

[Learning Experience Report Form \(requires a University of Minnesota e-mail address\)](#)

Important:

If someone was injured (even minor cuts or exposures), please be sure a **[First Report of Injury form](#)** has been completed first! See our **[Incident Reporting](#)** page for more.

What is required:

The submission form is anonymous and only requires a brief description of the incident and the suggested measures that could be taken to solve the problem. Anyone with a UMN account can create a submission.

When to submit:

Please consider submitting an LER if you ever think there is an underlying condition or opportunity to learn. Even submitting a seemingly minor incident is important to develop the practice of sharing potential safety concerns.

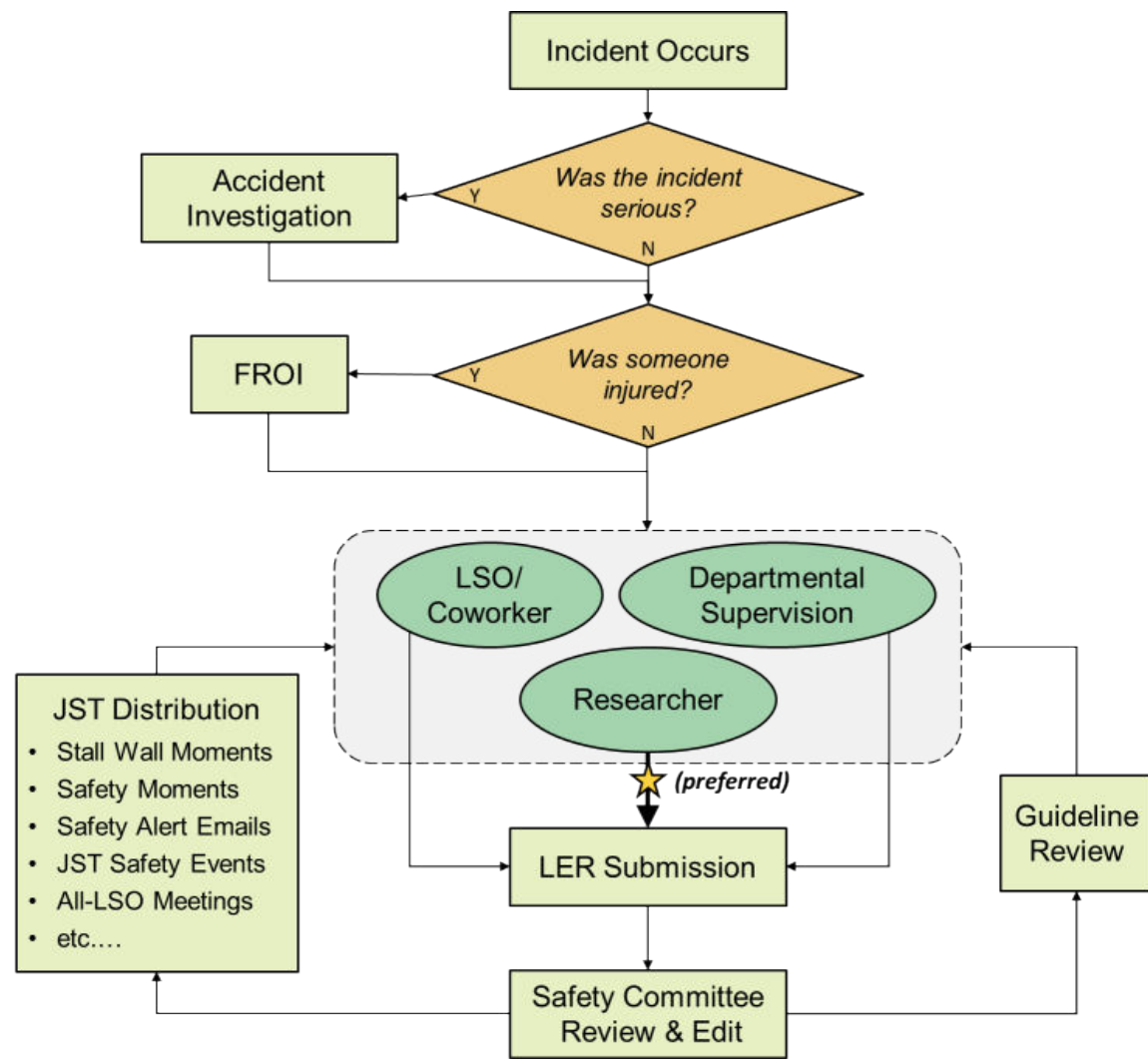
"I wish I had known."

...

"Why didn't anyone tell me?"

...

"If only I had dealt with that problem or followed instructions earlier."



Vial Explosion by Warming LN2

Title

[Vial Explosion by Warming LN2](#)

Monday, June 3, 2019

Situation

The researcher was cryo-fracturing a sponge-like material (99% porosity) by first submerging the sample in liquid nitrogen, then tearing it apart using two pairs of tweezers. When finished the sample was transferred into a scintillation vial for storage. Because of the high porosity, an estimated amount of 1 cm³ LN2 was absorbed in the sponge-like material, and was also transferred to the vial. The researcher then made a mistake by capping the vial because the researcher wasn't paying too much attention while talking to a lab-mate. Soon after capping the vial, the vial exploded in their hand because of the volume expansion of the nitrogen gas. Fortunately, the researcher was all geared up (gloves, goggles and lab-coat) so there were just a couple of scratches on their hand. The coworkers in the lab quickly located gauze pads and bandages for the researcher, and the bleeding was stopped very soon.

Suggestions



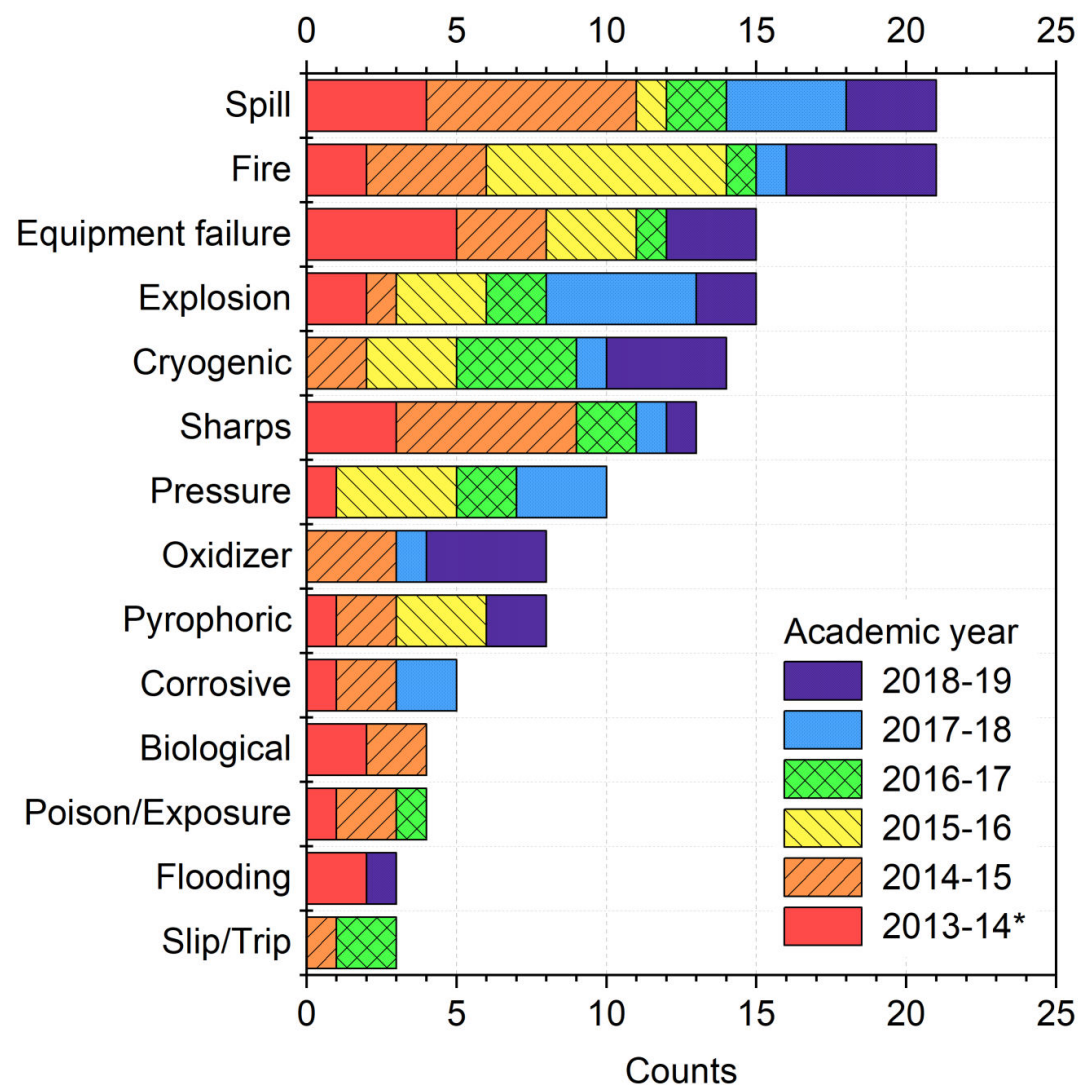
(1) Do not be in a rush to put any samples that were just submerged in liquid nitrogen in a sealed container wait a minimum of 24 hours for vapors to expand or LN2 liquid to drain out if still in the vapor phase. LN2 expands 600x in volume when warmed so very little amount of liquid nitrogen in a sealed environment could be devastating.

(2) Be on your toes when preparing samples using cryo-fracturing. Avoid any distractions during lab work and focus your attention completely on your work.

(3) Always fully gear up and never work alone in the lab. Add a blast shield and stay away while thawing. Or use a face shield with goggles and cut resistant gloves when handling materials that have not thawed. It is also recommended to perform cryo-fracturing in a fume hood. It won't just be minor scratches if the researcher was working without any protection on a weekend.

(4) A first report of injury should be completed for even minor injuries and exposures.

LERs help capture different data than formal EHS requirements



Ranked Hazard Type	LER (n = 85)	FROI (n = 72)
First	Spill, Fire (n = 21, 25%)	Sharp (n = 20, 28%)
Second	–	Slip/Trip (n = 14, 19%)
Third	Equipment failure, Explosion (n = 15, 18%)	Poison/Exposure (n = 12, 17%)

LER Data Provides...

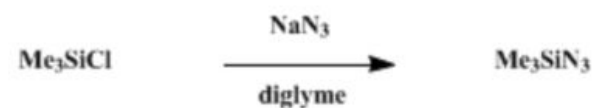
- Longitudinal data across labs on common hazards and mistakes
 - Waste carts: narrow carts led to more spills
 - Old Corning stirplates had a defect that led to uncontrolled heating in multiple labs (search-and-replace campaign)
- Help to ID training blind spots in labs
 - Inorganic labs were much more aware of cryogen safety than others
- DIFFERENT data than FROI
 - Only accidents that cause injury make it to FROI, LER captures a different set

explicit instruction and communication is key



*if you're gonna make this,
follow the directions.*

don't do it on process scale.



Org. Syn. **1970**, 50, 107

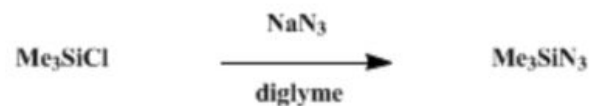
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July 2014: Devastating explosion in the UMN Chem Dept during a 200 g scale synthesis of TMSN_3 (student severely injured, has thankfully recovered):



Org. Syn. **1970**, 50, 107



This was from a materials group that used *A LOT* of TMSN_3 over the years.

Prep carried out on > 200 g scale 20+ times in the lab. What went wrong?

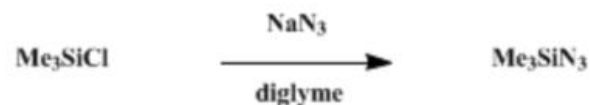
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Over the years, the procedure had evolved away from the *Org. Syn.* Prep in several dangerous ways, and students had stopped referring to the original sources. Failure to recognize the problems and dangers!

- Freshly distilled diglyme => PEG from a bottle
 - PEG is protic and often wet, could easily protonolyze to HN_3
- Mechanical stirring => magnetic stirring
 - Students were opening reactor to “break up” chunks of NaN_3 manually
- 200 g of a potential explosive is not suitable for engineering controls in a regular academic lab

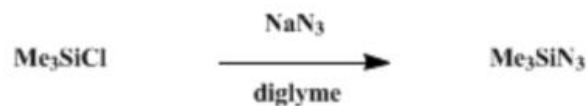
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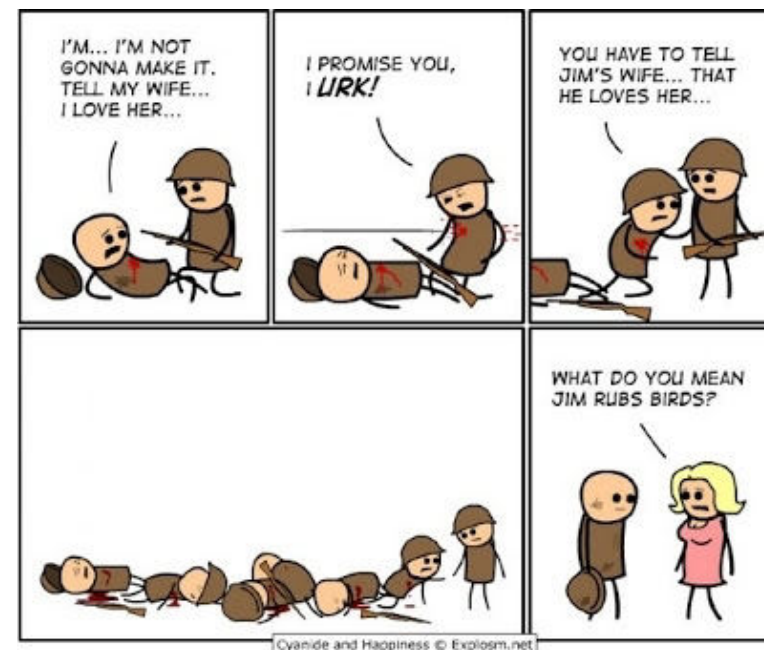
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Prep carried out on > 200 g scale 20+ times in the lab. What went wrong?

In essence, this was a game of telephone gone wrong. With no written procedures being followed, original safety protocols and logic were forgotten, changed, or ignored.

Formalized procedures and protocols are important for successful knowledge transfer over time!

Standard Operating Procedures (SOPs) are a great tool for this.



SOPs are everywhere now! Writing things down is a great way to break the game of telephone.

University of Minnesota – Twin Cities
Tonks Group Standard Operating Procedures

Degassing Solvents – Freeze, Pump, Thaw

Degassing solvents involves iteratively applying vacuum to a liquid in order to remove dissolved gases such as O_2 or N_2 from solution. In order to prevent the liquid from also boiling away, the liquid is cooled to a temperature such that it has a very low vapor pressure, even under vacuum.

A common misconception of FPT technique is that you must *freeze* your liquid. **This is untrue** – you just need to **cool your solution** such that the vapor pressure of the liquid is extremely low under vacuum. Unnecessary application of extreme cryogenics to fully freeze some liquids is dangerous and is a common source of explosion/implosion accidents while FPT'ing. Don't do this.

A. General Safety Guidelines & PPE:

- Working with Schlenk and vacuum lines presents implosion/explosion dangers.
- Working with cryogenics presents cold burn and liquid oxygen dangers.
- Working with solvent pots involves the use of potentially pyrophoric material.

Full PPE (goggles or facemask, gloves, and labcoat) must be worn at all times, and manipulations should be carried out in a working fume hood. Work with the safety sash in front of you at all times. Be aware of the location and use of fire safety equipment in the lab prior to using flammables.

B. Instructions:

1. The liquid to be degassed needs to be in a vessel capable of withstanding prolonged vacuum: a solvent bomb, Strauss flask, Schlenk flask fitted with a greased glass stopper, or a round-bottomed flask with a 180 degree adapter.
2. Turn on your vacuum line (see Schlenk line and High Vacuum Line SOPs). Make sure that the traps on your vacuum line (either Schlenk or High vacuum line) are filled with LN_2 to prevent solvent from entering your vacuum pump.
3. Attach your vessel to the vacuum line.
4. Use a chemical bath (usually dry ice/acetone) to cool your vessel.

There are many options here, depending on your solvent. Keep in mind that you do **not** need to freeze your solvent, just significantly cool it. In the Tonks group, **using LN_2 to cool your vessel is banned**. Cooling a closed system with LN_2 is a recipe for condensing liquid O_2 , and that risk is not worth saving a couple mL of solvent. With a few exceptions for very low-boiling solvents, cooling to $-78^\circ C$ will result in only minimal solvent loss upon exposure to vacuum.

5. After your vessel has equilibrated with the cold bath, open it to vacuum.

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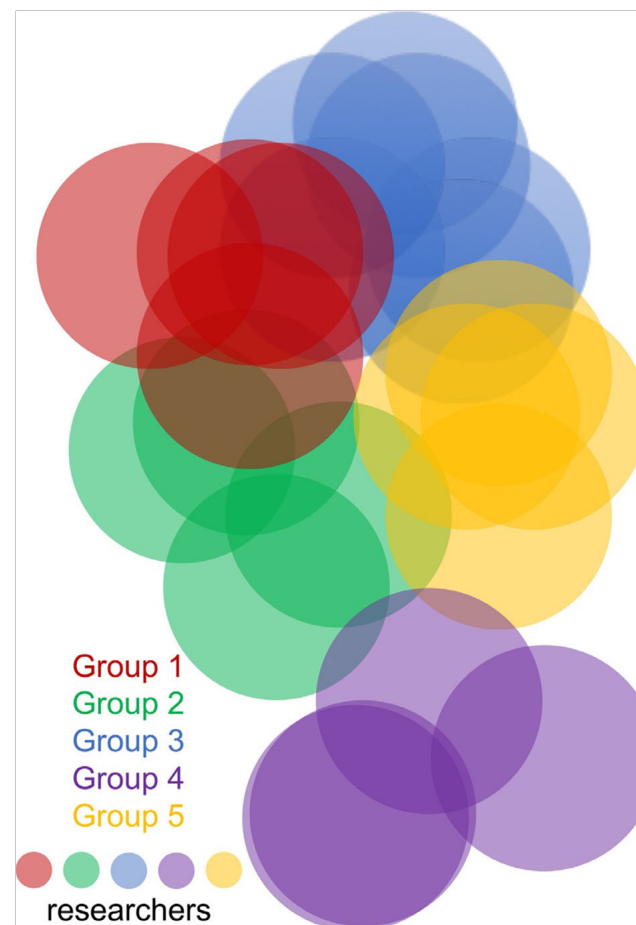
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BUT!

How do you know that your way is the **BEST** way?



We often have different, potentially non-overlapping areas of expertise.

How do you know that your way is the *BEST* way?

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1



pubs.acs.org/OPRD

Article

A Versatile Method of Ambient-Temperature Solvent Removal

Igor V. Kolesnichenko,* Galina Z. Goloverda,* and Vladimir L. Kolesnichenko*

Cite This: *Org. Process Res. Dev.* 2020, 24, 25–31

Read Online



Figure 2. Distillation apparatus with a cylindrical condenser/receiver. The receiver shown is filled with a frozen solvent.

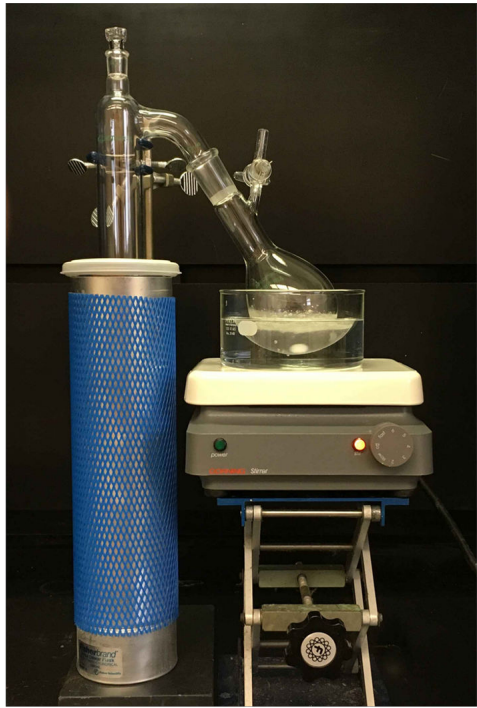


Figure 4. Vacuum distillation with a liquid nitrogen-chilled receiver.

How do you know that your way is the ***BEST*** way?

University of Minnesota – Twin Cities
Tonks Group Standard Operating Procedures

Degassing Solvents – Freeze, Pump, Thaw

Degassing solvents involves iteratively applying vacuum to a liquid in order to remove dissolved gases such as O₂ or N₂ from solution. In order to prevent the liquid from also boiling away, the liquid is cooled to a temperature such that it has a very low vapor pressure, even under vacuum.

A common misconception of FPT technique is that you must *freeze* your liquid. **This is untrue**— you just need to **cool your solution** such that the vapor pressure of the liquid is extremely low under vacuum. Unnecessary application of extreme cryogenics to fully freeze some liquids is dangerous and is a common source of explosion/implosion accidents while FPT'ing. Don't do this.

A. General Safety Guidelines & PPE:

- Working with Schlenk and vacuum lines presents implosion/explosion dangers.
- Working with cryogenics presents cold burn and liquid oxygen dangers.
- Working with solvent pots involves the use of potentially pyrophoric material.

Full PPE (goggles or facemask, gloves, and labcoat) must be worn at all times, and manipulations should be carried out in a working fume hood. Work with the safety sash in front of you at all times. Be aware of the location and use of fire safety equipment in the lab prior to using flammables.

B. Instructions:

1. The liquid to be degassed needs to be in a vessel capable of withstanding prolonged vacuum: a solvent bomb, Strauss flask, Schlenk flask fitted with a greased glass stopper, or a round-bottomed flask with a 180 degree adapter.
2. Turn on your vacuum line (see Schlenk line and High Vacuum Line SOPs). Make sure that the traps on your vacuum line (either Schlenk or High vacuum line) are filled with LN₂ to prevent solvent from entering your vacuum pump.
3. Attach your vessel to the vacuum line.
4. Use a chemical bath (usually dry ice/acetone) to cool your vessel.

There are many options here, depending on your solvent. Keep in mind that you do **not** need to freeze your solvent, just significantly cool it. In the Tonks group, **using LN₂ to cool your vessel is banned**. Cooling a closed system with LN₂ is a recipe for condensing liquid O₂, and that risk is not worth saving a couple mL of solvent. With a few exceptions for very low-boiling solvents, cooling to -78 °C will result in only minimal solvent loss upon exposure to vacuum.

5. After your vessel has equilibrated with the cold bath, open it to vacuum.



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Article

A Versatile Method of Ambient-Temperature Solvent Removal

Igor V. Kolesnichenko,* Galina Z. Goloverda,* and Vladimir L. Kolesnichenko*

Cite This: *Org. Process Res. Dev.* 2020, 24, 25–31

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* ORIGINAL ARTICLE This notice is a correction

Addition to "A Versatile Method of Ambient-Temperature Solvent Removal"

The following section should be added to the original paper:

Safety Notes

ARTICLE SECTIONS

Jump To

The procedure described in this article should be performed only by an experienced researcher/experimenter who is properly trained in vacuum techniques and handling cryogenics. The experimenter must be aware of a potential cryogenic burn hazard associated with dry ice and liquid nitrogen and must recognize that liquid oxygen can accumulate in a system or apparatus that is being chilled with liquid nitrogen *if it is vented to the atmosphere* (this includes but is not

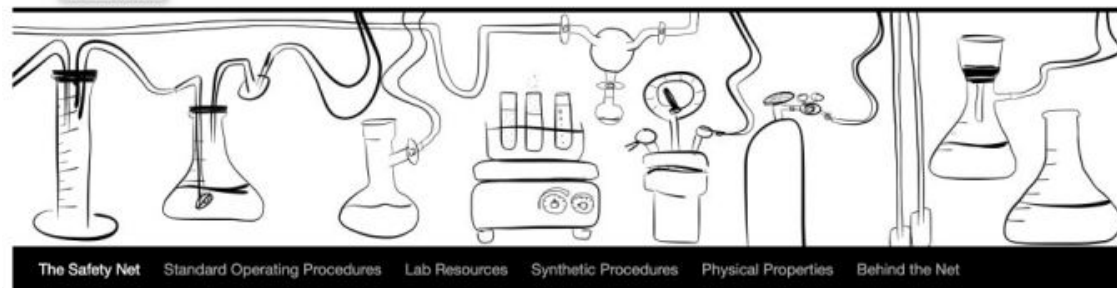
How do you know that your way is the **BEST** way?

Inorganic Safety Net: an *open* collection of SOPs

The Safety Net

The Safety Net

Safety knows no season.



Alex Miller, UNC



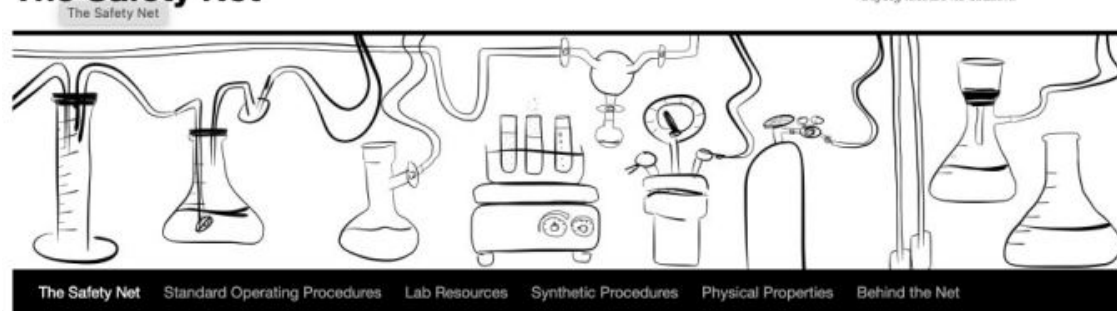
(see also:
NotVoodoo,
ILPI/Safety
Emporium)

If we share our methods and talk about how/why we do them, everyone will be safer!

How do you know that your way is the **BEST** way?

Inorganic Safety Net: an *open* collection of SOPs

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Alex Miller, UNC



(see also:
NotVoodoo,
ILPI/Safety
Emporium)

UMN Faculty SOP Peer Review

- annual, rotating group partners
- free-form, but safety committee prompts a few “areas of interest”

MEMORANDUM

SUBJECT: SOP Peer Review Meeting – Tonks and Tolman Groups

ATTENDEES: Dr. William Tolman, Dr. Ian Tonks, Jimmy Chiu, Debanjan Dhar, Courtney Elwell

DATE: Tuesday, May 19th, 2015

TIME: 10:00 AM

The general topics below were discussed in the SOP peer review meeting:

- ☐ Emergency contacts
- ☐ Lab notebook page format
- ☐ Sample labeling
- ☐ Standard Operating Card (SOC) SOP's
- ☐ Inclusion of lab specific syntheses, in particular hazardous reactions
- ☐ Chemical Inventory (www.quartzy.com)
- ☐ Group member checkout checklist (storage and disposal of synthesized chemicals)
- ☐ Chemical waste manifestation
- ☐ Scale up procedures
- ☐ Vacuum transfer of solvents (LN₂ vs. dry ice)

If we share our methods and talk about how/why we do them, everyone will be safer!

thank you!



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Dr. Victor Young Jr (XRD)

special thanks to all the students of the UMN Joint Safety Team & ACS DCHAS!