Research Sample Causes Explosion in Refrigerator- April 2017

What happened?
A researcher worked up a reaction by using column chromatography with a 50:50 mixture of diethyl ether and hexanes as the eluent. The researcher was working on a small scale (millimolar, or 1-2g of sample) and concentrated approximately 300mL of the fractions containing the desired product via rotatory evaporation. The resulting sample was placed in a scintillation vial and stored inside the freezer portion of a commercial household refrigerator. The lab reported a 150% yield on this particular vial so some contamination was known to the lab.

Later that night around 7:45pm, a loud sound and smoke came from the refrigerator. The noise was heard from at least two labs over. There were no injuries and no one reported any symptoms of a chemical exposure. A member of the lab called University of Chicago Police Department, while others initiated an evacuation of neighboring labs, eventually to include the entire floor and the floor below. The lab was released back to the PI the following morning around 10:30am.

What was the cause?
While no definitive cause has been determined there was a contaminant in a research vial. With the use of diethyl ether, it is possible the contaminant was an explosive peroxide. There are documented examples of diethyl ether and other peroxide forming chemicals (PFC) exploding upon rotatory evaporation.
In addition to concentrating a PFC, the lab's practices around peroxides could have contributed to the incident. One main contributing factor is refilling smaller chemical containers from dated and unexpired bulk containers without using the entire volume of the chemical and rinsing the smaller container. Since peroxides form overtime after exposure to air the peroxide concentration in these smaller containers are likely to be higher from evaporation.
The lab also placed the research sample inside the freezer portion. Colder temperatures could have facilitated peroxide crystallization.

What were some of the things done well?
- Lab members responded to the incident well by evacuating immediately and communicating the situation to adjacent laboratories and to UCPD
- Personnel had UCPD phone number stored in cell phone (773.702.8181)
- Lab had detailed inventory of the refrigerator contents
- Lab labeled reaction samples well enough to be identified by personnel
- Lab remained on the scene to answer questions from UCPD and ORS
- The chemical storage inside the refrigerator was good and resulted in breaking of only three research sample vials.
- Lab had appropriate PPE for hazards present

Is your laboratory as well prepared for an incident as this one was?

What are some lessons learned from the incident?
Safe peroxide forming chemical management is important and detailed in the University's Chemical Hygiene Plan and guidance document of Peroxide Forming Chemicals. While the lab was managing their bulk peroxides according to University Policy some laboratory practices could have accelerated the peroxide formation. Additional steps can and should be implemented to prevent further incidents, including:

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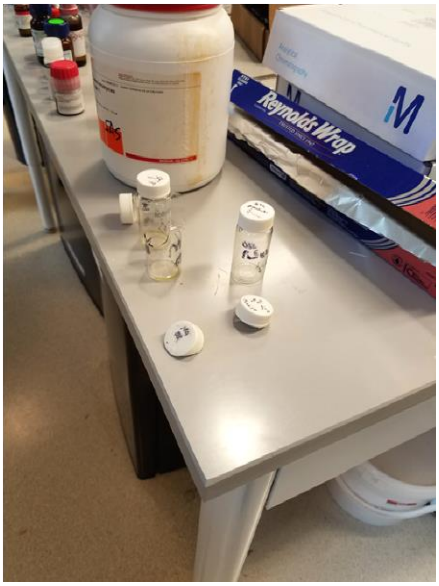
- Substitute other solvents for PFC (when possible by testing retention factors of other solvent combinations) when purifying with column chromatography
- Rotatory evaporation of PFC should be done behind a blast shield and away from other chemical hazards
- Test for peroxides before evaporating a sample containing a PFC to dryness
- Emptying, rinsing, and dating the smaller containers of PFC before refilling
- Consider using flasks with stoppers to store samples with PFCs instead of tightly sealed scintillation vials.

References and Resources

1. University of Chicago's Chemical Hygiene Plan template
https://researchsafety.uchicago.edu/sites/researchsafety.uchicago.edu/files/uploads/UChicago%20Chemical%20Hygiene%20Plan%20Editable.pdf
2. University of Chicago's Peroxide Forming Chemicals Guidance Document
https://researchsafety.uchicago.edu/sites/researchsafety.uchicago.edu/files/uploads/PFC.pdf
3. Kelly, R.J, Review of Safety Guidelines for Peroxidizable Organic Compounds, Chemical Health and Safety, 1996, 3 (5), 28-36.
4. Clark, D.E., Peroxides and Peroxide - Forming Compounds, *Chemical Health and Safety*, 2001, 8 (5), 12-21
5. National Research Council. 2011. Prudent Practices in the Laboratory: Handling and Management of Chemical Hazards, Updated Version. Washington, DC: The National Academies Press. doi:https://doi.org/10.17226/12654.



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