Quenching of Solvent Still Results in a Fire May 2017

What happened?
A researcher was quenching sodium metal from a 1L heptanes distillation apparatus when it ignited sending flames across an aisle and onto a lab bench. No one was in the lab at the time, however after hearing an explosion two students checked to see what had occurred before seeking assistance from a more experienced lab member. The more experienced lab member was able to extinguish the fire quickly while another lab member notified University of Chicago Police (UCPD-**123**). City of Chicago Fire Department was subsequently notified by UCPD and confirmed the fire was extinguished before releasing the lab back to the Principal Investigator. There were no injuries and very little damage to the lab, however similar incidents have happened at other institutions[1] and have resulted in serious injuries and damage.

What was the cause?
The lab member indicated that he thought the metal was completely quenched when he added approximately 100mL of isopropanol to the solvent distillation head to slowly drip into the still pot. The researcher normally uses a side arm to add the isopropanol to the still pot, however this time he wanted to add through the distillation head like an additional funnel so that he could leave the lab and do some analytical work. While adding the isopropanol, he inadvertently shut off the nitrogen flow, producing a closed system. The researcher walked away and was not in the lab when there was a sudden release of the built up pressure and ignited a fire. Paper towels across from the fume hood (sash was left half opened) caught fire along with some combustibles inside the hood.

As sodium is quenched with alcohols and water it produces sodium hydroxide, hydrogen gas, and heat. Researchers will normally start the quenching process with isopropanol before switching to ethanol, methanol, and water. The presence or absence of bubbles (hydrogen gas) is used to determine if the sodium is sufficently quenched. However a coating can form on the sodium that falsely gives the researcher the impression that the sodium has been quenched. When the coating dissolves the fresh sodium rapidly reacts with any alcohol or water present. In a closed system this results in the pressurization of hydrogen gas and heat. The addition of flammable liquids and production of flammable gas and heat also makes this a highly hazardous operation.

What were some of the things done well?
- The solvent still was not run to dryness
- The research was experienced in quenching procedure, had access to standard operating procedures developed by other chemistry groups at other universities and was aware of the hazards
- Lab members responded to the incident well by quickly extinguishing the fire and reporting to UCPD by calling **123**.
- Lab members are given a detailed orientation[2] before they begin work that includes location of safety equipment and general laboratory rules.
- Lab remained on the scene to answer questions from UCPD and Chicago Fire Department
- Lab has and uses a solvent purification column for their commonly used solvents
- Lab had appropriate PPE (Flame resistant lab coat, goggles, and nitrile gloves) for hazards present
- The fume hood had been cleared of flammable solvents that could have spread the fire

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What are some lessons learned from the incident?

Additional steps can and should be implemented to prevent further incidents, including:

- Highly hazardous operations should never be left unattended and the work being performed should be clearly communicated[3] to others in the lab.
- Emergency equipment should always be available and accessible.
- Good housekeeping should be employed to minimize the amount of combustibles and to prevent the spread of a fire.
- When working with Schlenk lines and glassware with multiple stopcocks it is important to:
    - have a method for determining when they are opened/ closed;
    - have a method and/or equipment to monitor pressure and temperature;
    - mentally visualize the process to prevent a closed pressurized system;
    - consider "what if" scenarios and plan appropriate emergency responses.
- Standard operating procedures[4] and lab specific training should include consideration of reacting incompatible chemicals, such as sodium and alcohol/ water.
- A checklist for procedures done regularly should be developed.
- Special attention, including consultation with other lab members and/or the Principal Investigator, is important when adjusting a procedure.
- Fume hood sashes should be closed when not in use.
- A glass coated stir bar can be added to the still pot for the quenching procedure
- Use of the *flowerpot technique*[5] when quenching sodium or potassium metal should be considered.

References and Resources

1. UC Irvine Incident Investigation http://www.ehs.ucsb.edu/files/docs/ls/UCI_fire.pdf
2. Research Personnel Orientation Checklist https://researchsafety.uchicago.edu/sites/researchsafety.uchicago.edu/files/uploads/Research%20Personnel%20Orientation%20Checklist.docx
3. Overnight or unattended reaction sheet https://researchsafety.uchicago.edu/sites/researchsafety.uchicago.edu/files/uploads/Overnight%20Reaction%20Form.docx
4. University of Chicago Standard Operating Procedure template https://researchsafety.uchicago.edu/page/standard-operating-procedure-templates
5. "Facile and Environmentally Friendly Disposal of Sodium and Potassium with Water" Roesky, H. W. **Inorg. Chem.** 2001, 40, 6855-6856.