Lesson Learn

Laboratory safety knowledge sharing | 2022. Feb.

I. Case Introduction

An undergraduate student researcher was working at the laboratory bench when the apparatus she was using exploded, sending glass fragments into her face and upper torso. The researcher was using a rotary evaporator (rotovap) to remove organic solvents from an azobenzene precipitate. She adjusted the bottom flask which then exploded sending glass towards her face, hitting her safety goggles and forehead. Lab personnel helped her to the safety shower and called 911. She was taken by ambulance to the hospital where she received stitches above her eyes and other treatment for her injuries. She was released from the hospital the same day.

On August 16, 2017 at around 8pm a first year graduate student was using a rotatory evaporator (rotovap) to work up a published reaction in the synthesis of fluorescent indicators. The 500mL round bottom flask had acetic anhydride, acetyl chloride, and other highly corrosive byproducts from the reaction when it fell into the 50oC water bath。

II. Introduction of Rotary evaporator

The rotary evaporator (English: rotary evaporator), is an experimental instrument commonly used in chemical laboratories, mainly for the continuous distillation of large quantities of volatile solvents under reduced pressure conditions. In particular, it is used for the concentration of extracts and the distillation of receiving liquids for

chromatographic separations, where the reaction products can be separated and purified.

III. Possible causes of accidents

Possible hazards include implosion due to the use of glassware containing defects, such as star cracks. Explosions may occur when unstable impurities are concentrated during the evaporation process, e.g. during the rotary evaporation of ether solutions containing peroxides。

The entangling action of the rotating parts of the equipment can pull the user, especially with loose clothing, hair or necklaces, resulting in broken glassware, burns and chemical exposure.

Operation with air-reactive materials under vacuum conditions, leaks may draw air into the equipment and violent reactions may occur.

When in use, too rapid evaporation may result in untimely condensation of the solvent, which in turn may enter the outside environment, a dangerous situation for flammable and explosive solvents. Evaporation should be carried out in a fume hood or in a well-ventilated area.

IV. General rules for usage of a rotary evaporator

1. When using, pump a small vacuum (about 0.03MPa) before turning on the rotation to prevent the distillation flask from slipping off; when stopping, stop the rotation first, hold the distillation flask by hand, pass the atmosphere, wait until the vacuum level drops to about 0.04MPa before stopping the vacuum pump to prevent the distillation flask from falling off and sucking backwards.
2. All interfaces, sealing surfaces, seals and joints must be coated with a layer of vacuum grease before installation.

3. The heating tank must be filled with water before powering up, no dry firing without water is allowed.
4. If the vacuum level is too low, check the gas tightness of the joints, vacuum tubes and glass vials.
5. If the viscosity of the sample is very high, slow down the rotation speed and rotate slowly by hand so that a new liquid surface can be formed to facilitate the evaporation of the solvent.
6. The temperature of the water bath should not exceed the boiling point of the solvent! For small quantities of common solvents, a water bath heater is not required.
7. A metal or Keck clip is used to secure the flask and the bump trap. The green one shown below fits 24/40 ground glass joints. Similar blue clips fit 19/22 joints and the yellow ones fit 14/20 joints.

8. The solvent should start collecting on the condenser and drip into the receiving flask. Some solvents (such as diethyl ether or dichloromethane) are so volatile that they will also evaporate from the receiving flask and be discharged down the drain. To prevent this, a cooling bath on the receiver or (on some models) use a dry-ice condenser can be used. In addition, an additional trap (with dry-ice or liquid nitrogen) can be placed between the vacuum source and the condenser unit. This is particularly important of a membrane pump is used as vacuum

source.

9. For flammable and explosive solvents, too rapid evaporation may result in untimely condensation of the solvent, which may then enter the outside environment. Evaporation should be carried out in a fume hood or in a well-ventilated area.

V. Daily maintenance

1. Distilled water should be used in the heating bath to minimize the scale build up in the bath, which coats the thermistor and heating coils. It is very difficult to remove and reduces the efficiency of the bath. In addition, regular tap water will promote the growth of spectacularly disgusting algae colonies, particularly during the summer months. The best protocol is a regular exchange of the water.
2. To remove algae gunk from the inside of a coiled water condenser, the condenser has to be removed from the rota-vap and the coil is soaked in a dilute nitric acid solution for a few hours. After carefully rinsing the insides, the rota-vap is reassembled. All standard safety precautions should be followed when working with nitric acid!

一定要牢记：拒绝侥幸，忌焦忌躁！

Nothing we do is worth getting hurt for !