Section 5.7 **Title:** Heating and Cooling Chemical Mixtures

Revision Date: 11/01/19

Prepared By: Michael Roy **P.I.:** Prof. John F. Berry

Prior Approval: This procedure is NOT considered hazardous enough that prior approval is needed from the Principal Investigator.

Involves Use of Particularly Hazardous Substance (PHS)? No

___ Carcinogen ___ Reproductive Toxin ___ High Acute Toxicity

Does this procedure require medical surveillance? No

Does this require use of a fit-tested respirator? No

Brief Description of Procedure:

Overview of common heating and cooling methods used in the Berry labs.

Location: *List the locations (buildings/rooms) where this procedure may be performed. For use of a PHS indicate a more precise location within the room, if appropriate, as a designated area.*

Daniels Chemistry - All Berry group labs

Chemicals Involved:

Chemical	Physical or Health Hazard (e.g. carcinogen, corrosive)
Organic solvents (cooling)	Consult relevant SDSs for more details
Dry ice	Frostbite
Liquid nitrogen	Frostbite, asphyxiation

Other Hazards: *Include hazards, other than chemical, that may be present during operation of the procedure.*

Burns (heating) and frostbite (cooling).

Exposure Controls: *(Check all that apply)*

PPE: _X_ Safety Glasses ___ Face Shield ___Chemical Splash Goggles

___Chemical Apron _X_ Gloves (Nitrile) _X_ Lab Coat

___Respirator (type) ___Other:

Engineering Controls:

_X_ Fume Hood ___Biosafety Cabinet ___ Glove box

___ Vented gas cabinet ___Other:

Administrative Controls: *List any specific work practices needed to perform this procedure (e.g., cannot be performed alone, must notify other staff members before beginning, etc.).*

N/A

Task Hazard Control Table: *For procedures involving numerous steps, it may be convenient to indicate specific requirements for individual tasks in the table below:*

N/A

Waste Disposal: *Describe any chemical waste generated and the disposal method used.*

Dispose of the reagents involved as appropriate. Consult SDSs for more details.

Accidental Spills: *Describe the procedure for handling small chemical spills that may occur during this procedure. Note that for large spills it may be appropriate to call 911.*

Small spills may be cleaned with an absorbing material. The material should be placed in a fume hood to dry after the spill has been cleaned.

Decontamination Procedures (required for PHS use): *Describe the procedure for decontamination of personnel and equipment.*

N/A

Training: *Describe any training needed prior to performing this procedure. Include training performed in-lab and any required demonstrations of competency.*

No formal training or documentation is required. This procedure should be demonstrated by experienced lab members. New members should talk through their procedures with experienced lab members.

Principle Investigator Approval: I have reviewed this procedure and approved it for use. Note: Modifications to the procedure may require update to this form.

Name: John F. Berry Signature: ________________________ Date: ___________

Heating and Cooling Chemical Mixtures

Chemical procedures often require controlled temperatures above or below ambient. There are often multiple ways to go about heating or cooling chemical mixtures. Always consider the scale and temperature when deciding on a heating or cooling method. When working with heat sources or cooling agents, always be careful to avoid direct skin contact, as this can quickly result in burns or frostbite.

Heating Methods

General Notes:

- Never heat closed systems unless they are explicitly designed for high pressure. Consult with Prof. Berry before working with high pressure systems.
- Heated reactions, such as refluxes, often require cooling methods to prevent solvent from escaping the system. Always verify that your cooling method (chilled water, dry ice bath, etc.) is functioning before you apply heat.
- Most organic solvents are highly flammable. Do not have open containers of solvent present near heat sources.
- For gentle heating (up to about 80 °C), you may instead wish to use a water bath. Spilled water is much easier to clean than spilled oil. For extended heating, though, water baths will evaporate and may not be a suitable choice.

The most common heating method in the Berry labs is the use of oil baths. Both mineral oil and silicone oil are common heating bath liquids. Mineral oil has a maximum temperature of 130 °C, while silicone oil has a maximum temperature of 210 °C. Because of this, almost all oil baths in the Berry labs are silicone oil. When using a silicone oil bath:

- Ensure that the oil bath is the appropriate size for your flask. Larger or smaller baths can be prepared as needed.
- Check that the oil is clean. Mineral and silicone oil are both clear and colorless. If the oil is cloudy or colored, clean or replace the oil. Silicone oil can often be cleaned completely by gravity filtration through filter paper.
- Place the oil bath on a hot plate. Make sure the oil bath has a paper clip or other stirring device. Stirring oil baths helps maintain a constant temperature throughout the bath.
- Lower the temperature probe of the hot plate into the oil bath. The end of the probe should be well-covered in oil and away from the sides and bottom of the bath.
- When setting up a reflux, sublimation, or other technique that requires cooling water, test connections before heating the oil bath. Having water spill into hot oil can cause the hot oil to splatter.
- Begin stirring the oil bath.
- Depending on your chemistry, you may want to pre-heat your oil bath. Otherwise, you can lower your flask into the bath, keeping it away from the sides and bottom of the bath, and begin heating.
- Take care when transferring chemicals over oil baths, especially if they are hot.
- Most heating plates in the Berry lab are either older blue Ika C-Mag HS7 or newer black Ika C-Mag HS7 Control hotplates. The older model automatically shuts off if the temperature probe does not heat (i.e. the probe is not in the oil bath). The newer models

need to have this setting turned on. Consult the Ika manual for instructions on turning on Error 5.
- The newer Ika hotplates can also have a timer set to turn off the heat after a certain amount of time. Consult the Ika manual if you wish to use this feature.

When your chemistry requires temperatures above 200 °C, the best option is to use a sand bath in a heating mantle. Heating mantles are controlled by variable transformers, often called Variacs. Never plug a heating mantle directly into a standard outlet, as it will overheat. When using a heating mantle:

- Select a heating mantle that is the appropriate size for your flask. If the flask does not fit perfectly, use a larger heating mantle filled with sand has a heat transfer agent. Sand is available near the large oven in 6319.
- Plan your reaction. Sand baths can take a long time to reach high temperatures (around 90 minutes for a large heating mantle to reach 200 °C). You may wish to begin heating the sand bath before setting up the rest of your reaction.
- Set up your entire heating apparatus before plugging in the heating mantle. Place a high-temperature thermometer or the temperature probe from a hot plate in the sand bath to monitor the temperature.
- When you are ready to begin heating, verify that the Variac is off and then plug in the heating mantle.
- If you know what Variac setting is appropriate for your chemistry, set the Variac to that setting and turn it on. If you do not know what the appropriate setting is, set the Variac low (~10 %) and turn it on. As the heating mantle equilibrates, slowly increase the setting on the Variac until the desired temperature is reached.
- Never leave heating mantles unattended until you know that the desired temperature has been reached and is stable. It is very easy to accidentally overheat heating mantles.
- When you are done heating, switch off the Variac and unplug the heating mantle. Allow the bath to cool completely before moving it.

Cooling Methods

General Notes:

- Chemical refrigerators (~5 °C) are located in rooms 6319 and 6325. A chemical freezer (–30 °C) is located in room 6319. These are suitable for storage of temperature-sensitive reagents and low-temperature crystallizations. All chemicals kept in the refrigerators and freezer must be sealed and clearly and completely labeled.

The easiest temperature to attain is 0 °C. Ice is available near the 5th floor research storeroom. Ice baths, consisting of mostly ice with enough water to facilitate heat transfer, are a simple and effective way to cool chemical mixtures. Adding rock salt, available in the 5th floor research storeroom, can lower the temperature by several degrees. Monitor this temperature with a thermometer or the temperature probe from a hot plate.

Dry ice baths can achieve a variety of temperatures between down to –78 °C. Mixtures of ethylene glycol are convenient for temperatures between –20 and -78 °C. When preparing a dry ice bath, fill an appropriately sized dewar with the desired volume of liquid. Then, in a fume hood, slowly add dry ice to cool the liquid. The dry ice will initially sublime rapidly, so don't add too much. Once the bubbling subsides, additional dry ice should be added until the desired temperature, monitored with a low temperature thermometer, is reached. For baths warmer than –78 °C, dry ice needs to be periodically added to keep the temperature correct. For –78 °C baths using isopropanol or acetone, excess dry ice can be added to keep the bath cold for several hours.

Liquid nitrogen slurries can be used for temperatures between below –78 °C. As with dry ice baths, the appropriate liquid is added to a dewar and liquid nitrogen is slowly added until the liquid just begins to freeze.

Large lists of cooling bath methods are available online. The following lists are from the Wikipedia page "Cooling Bath" (retrieved April 10, 2019):

Ethylene glycol / Ethanol / Dry ice

% Glycol in EtOH	Temp (°C)
0	–78
10	–76
20	–72
30	–66
40	–60
50	–52
60	–41
70	–32
80	–28
90	–21
100	–17

Other common cooling bath mixtures

Cooling Agent	Organic Solvent	Temp (°C)
Dry ice	Acetonitrile	–41
LN_2	n-Butanol	–89
LN_2	Acetone	–94
LN_2	Ethanol	–116