

# 828/DEA Batching Manual

- McCoy Group -

-NMT-

-2022 Update-

## Introduction

Getting accurate, reliable data on epoxy starts with creating well-made epoxy samples. Epoxy has two primary components: a resin, and a hardener. Other additives may be included in order to optimize certain material characteristics. This manual details the batching instructions to make an 828 resin, DEA hardener epoxy mixture.

## Safety Information

- Make sure that one or more qualified lab workers are available to help you batch before you begin.
- Personal protection equipment is required. Please consult the “Materials” section for specifics.
- Long pants and closed-toed shoes should be worn. Long hair should be worn up. Gloves should be changed in the event of an epoxy spill.
- Uncured epoxy resins will cause skin sensitivities to develop if contact is experienced on a regular basis. After the epoxy mixture has cured, the material is non-reactive and safe to be handled without gloves or safety glasses.
- Particular care must be taken with additives such as DBA, which is both corrosive and flammable. Carefully read all additives’ MSDS sheets before batching.
- **In the case of epoxy (EPON 828) or hardener (DEA) exposure to skin:** Rinse thoroughly with soap and water. Monitor site of exposure for adverse reactions, and seek medical attention if necessary.
- **In the case of epoxy, 828, or DEA exposure to eyes:** Remove contacts if wearing. Go to the nearest eye-wash station and rinse eyes for fifteen minutes. Monitor eyes for adverse reactions, and seek medical attention if necessary.

## Procedure

This manual contains the following sections:

- A. Before Starting
- B. Preparation
- C. Mixing Chemicals
- D. Degassing
- E. Pouring and Cure
- F. Cleanup
- G. Recovering Cylinders

## Materials

- Personal Protection Equipment: Safety glasses, a lab coat, and nitrile/latex gloves. All of these can be found in 130C.



*Personal protective equipment*



*10mL syringe*

- Chemicals: Diethanolamine(DEA), EPON 828 Epoxy-Resin, additives if necessary
- Bunsen Burner and lighter
- Syringe rack
- Top loading balance
- Three (3) Popsicle sticks
- 2 Pipettes
- Acetone
- Eight (8) 10mL syringes
- Waste disposal container
- Labeling Tape
- Batching Sheet

**Batching:****Before Starting:**

1. Allow for an hour beforehand to heat the 828 Epon Epoxy-Resin (EPON 828) and Diethanolamine (DEA) in an oven set to 70°C. This reduces viscosity and makes mixing easier. As the chemicals and prepared mixture come to room temperature the consistency will become more viscous.

*Note:* If batching at temperatures above 80°C, do not pre-heat the DEA. Volatile additives (Phenol, Octanol, etc.) should not be heated above 80°C and should not be pre-heated for long periods of time.

2. Fill out a batching sheet as shown in sample below. Accurate weights should be recorded and included with reported property testing on the resulting material. Procedures (e.g., preheat resin...) should be checked off as accomplished. Modifications to procedure should be noted.

**828/DEA Batching Sheet**

*828 DBA + Octanol*

Batch No.	Date	<i>9/22</i>
Materials Used		Mass (g)
Use mix ratio of 100:12 (828:DEA) by weight		
Resin: Epon 828	<i>50 g 828</i>	<i>13.35 g Octanol</i>
Hardener: Diethanolamine		<i>7.37 g DBA</i>
<b>Procedure</b>		
<input checked="" type="checkbox"/>	Pre-heat resin and hardener at 70°C for at least one hour prior to use. **Prepare the DSC pans and cylinder molds that will be used for this batch.	
<input checked="" type="checkbox"/>	Weigh out the appropriate masses of resin and hardener.	
<input checked="" type="checkbox"/>	Mix hardener and resin thoroughly until the mixture becomes white.	
<input checked="" type="checkbox"/>	Place the mixture in the oven at 70°C for 5 minutes.	
<input checked="" type="checkbox"/>	After 5 minutes check to see if the epoxy mixture has cleared	
<input checked="" type="checkbox"/>	— If not, place back into the oven and check in 30 second intervals - removing and stirring until the mixture becomes clear.	
<input checked="" type="checkbox"/>	Make samples	
<b>DSC Pans</b>		<b>Compression Cylinders</b>
**prior to batching, pans must be weighed out and sample envelopes labeled		Degas batch at 1-3 torr for a maximum of 3 minutes.
Place 1 drop of mixed epoxy in the prepared pan with a pipette, ensuring it doesn't exceed the pan top.		Carefully pour epoxy into the cylinders, angling them such that the epoxy can slide smoothly from the cup to the cylinders.
Crimp the lid onto the pan.		Place samples into the oven to cure at 70°C for 24 hours.
Test the samples immediately or store them in the freezer for testing at a later date.		
<b>Samples Made:</b>		
		<i>8 cylinders</i>
Clean up work space and ensure all materials used (gloves, stir sticks, etc.) are also cured at 70°C for 24 hours.		
<b>Notes:</b> <i>Very clear after a few minutes in oven</i> <i>Very easy to pour</i>		

*Batching sheet for 828-DBA with Octanol*

Preparation:

I. Preparing syringes to hold epoxy samples.

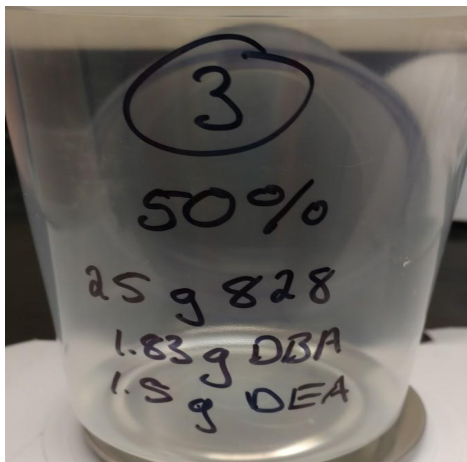
1. Unwrap 10mL syringes.
2. Unplug all syringes. Save only one plunger- the rest may be disposed of.
3. Set up the syringe rack next to the workspace.

4. Turn on gas and light Bunsen burner.
5. Carefully melt the needle adapter of the syringe (the tip where liquid would be pushed out) by holding it over the flame. Let gravity help you seal the opening by rotating the syringe on its side. Take caution during this step: you should not ignite the plastic, just heat it enough to liquify it.



**Note:** Keep the open flame away from the chemicals used for batching.

6. Place the freshly sealed syringe tip-down in the rack to cool.
  7. Repeat steps 5 and 6 until all syringes have been sealed.
  8. Turn off the Bunsen Burner and put it away.
  9. Allow each syringe to cool for several minutes.
  10. Using the plunger you saved earlier, attempt to push air through the cooled syringes. If properly sealed, the plunger should resist the motion, and should slide backwards once released. If the syringe does not pass this test, repeat steps 5 and 6.
- II. Set up mixing workspace.
11. Prepare workspace inside the fume hood by laying out paper towels around the balance and plugging the balance into the wall outlet. Check the bubble level to make sure the balance is level, and adjust as necessary. The rack holding the sealed syringes should be nearby and readily available.
  12. Label a plastic container with the proportions of your batch (as in photo).  
**Note:** All materials that come in contact with the epoxy/hardener mixture must be cured in the oven for 24 hours before going into the trash (after this period, the resin is fully cured and is no longer hazardous).



*Plastic epoxy mixing container, labeled with batch proportions.*



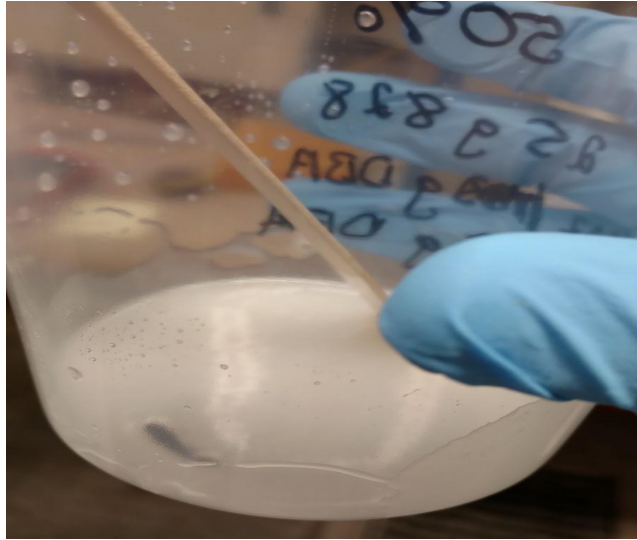
*Tared Scale*

### Mixing Chemicals:

1. Begin by positioning your plastic container on the balance in the fume hood. Tare (with the plastic cup on the balance) by pressing the “zero” button.
2. Take the 828 out of the oven and carefully pour it into the plastic container. As you get closer to your desired figure, use the designated 828 pipette to add the resin at a more controlled rate. Try your best to get within 0.1g of the target mass. Put the 828 in the chemical cabinet when you are done with it. (Record exact mass, e.g., "50.1g")
3. Tare the scale again, and add the appropriate amount of DEA using its designated pipette. Again, try to get within 0.1g of the target mass and record exact mass added. Put the DEA back in the oven when you are done using it.

**Note:** Some curatives, such as dibutylamine (DBA) are more hazardous than DEA and should only be handled under the supervision of an experienced student. Please check with supervisor before using any chemical in the lab.

4. If appropriate, tare the scale again and add the appropriate amount of any additional curative (such as DBA) using the designated pipette for this chemical. For small amounts, use a more sensitive scale.
5. Set a timer for five minutes. Carefully stir the epoxy mixture with a popsicle stick for the duration of this period. Keep the container under the fume hood at all times while stirring, and point the opening away from you to minimize exposure to fumes. The mixture should turn from clear to opaque after five minutes of stirring and fumes will subside as the small molecules are bound to the epoxy network.
6. Place the mixture in the 70°C oven for five minutes. Take out and stir, checking to see if the mixture has reverted from opaque to clear; if not, place in the oven for another five minutes. Repeat this process until mixture is clear.



*This mixture, still slightly opaque, could use more time in the oven.*

### Degassing:

1. Place the mixture in the pressure chamber, and close the chamber door securely. Turn on the pump using the switch located on the lower right side of the pressure chamber.
2. Next, turn on pressure gauge using the switch labelled below.



*Vacuum Pump Switch*



*Vacuum Controls*

3. Turn the release knob to the right as far as possible. If the valve is not sealed, air will enter the chamber and you will hear a whistling sound.
4. Switch on the vacuum. Begin to turn the vacuum knob to the left. This will start reducing the pressure.
5. Maintain a pressure of about 10-20 Torr. Keep an eye out for overboiling in your mixture: miniscule, quickly rising bubbles are quite normal, but larger bubbles are a bad sign. To curb boiling, increase the pressure by slightly twisting out the release knob.



*Pressure gage on top of vacuum chamber*

6. If after five minutes the mixture seems adequately degassed (that is, if the bubbling has significantly slowed), you may equalize the pressure in the chamber. Simultaneously twist *out* the release knob (to open the valve) and twist *in* the vacuum knob (to stop vacuuming), and wait until the chamber reaches equilibrium to open the vacuum door.

### Pouring and Cure:

1. Place your degassed mixture along with your sealed syringes + syringe rack under the fume hood.
2. Bend the lip of the mixture container into a spout shape by slightly pressing the two sides together.
3. Hold the opening of the syringe to the makeshift spout so that the sides of the two containers are near parallel. The quality of this configuration is what will determine how much air is incorporated back into the mixture during the pouring process, so line this up carefully.

**Note:** The goal of degassing is to remove air from the samples and decrease inconsistencies. Thus, it is of utmost importance that while pouring the degassed epoxy into our molds, no air is re-incorporated into the mixture.

4. Slowly pour the mixture down the side of the syringe. Fill the syringe up to at most a centimeter from the top. Overfilling the syringe can make the epoxy removal process difficult, while underfilling can lead to wasted epoxy when it comes time to cut samples for testing.
5. Carefully place the filled syringe back onto the rack.
6. Repeat steps 3-5 until you run out of epoxy. Seal and cool extra syringes if needed; no epoxy should go to waste.
7. Place the rack of filled syringes inside the oven. Cure for the desired time – the typical time is 24 hours.



**Note:** The cure time is usually in the magnitude of several hours, but should nonetheless be handled with precision; as such, make sure that either you or another qualified lab assistant will be there to take out the epoxy cylinders exactly when specified.

### Cleanup

1. Any trash that has come in contact with any of the chemicals should be stored in the mixing container and cured in the 70°C oven for twenty-four hours. It is only after this curing period that the trash can be disposed of.



*Trash in the curing oven*



*Acetone and paper towels*

2. Wipe your work surfaces with acetone and paper towels. Dispose of used towels in zip-lock bag for Hazardous Waste Disposal
3. Put your lab coat and safety glasses away.
4. Wash your hands for at least a minute to ensure no chemicals linger on your skin.

### Recovering Cylinders:

1. Suit up with proper PPE. Safety glasses, thick heat gloves (located in the drawers of 130C), and closed-toe shoes are a must for this step. Wearing a lab coat is recommended.
2. Lay out all necessary tools for this process- hammer, pliers, ziploc baggies, and sharpie. The first two can be found in the 130C toolbox, and the last two on the counter in 130B or 130C.
3. Using the heat gloves, take the rack of cured epoxy cylinders out of the oven.

4. While still wearing heat gloves (which will protect you from both the heat of the epoxy and the potential impact of the hammer) begin hammering at the sealed tip of a syringe with firm but controlled taps. The objective is not to crush the syringe tip, but rather to knock the epoxy cylinder down the length of the syringe. Aim to get the cylinder down at least a centimeter before proceeding with the pliers.

**Note:** Hammering the syringes can send sharp plastic debris into the air. Be sure to wear your safety glasses to prevent injury. Hammer on a surface not easily damaged (e.g., steel table or wooden block)

5. Using the pliers, pinch the newly empty tip of the syringe in order to push the epoxy cylinder further out. Continue with this pinching action down the length of the syringe until a good portion of the epoxy protrudes from the syringe, at which point you can simply use your hands to completely pull the cylinder out.
6. Repeat steps 4 and 5 until all epoxy cylinders have been extracted.
7. Store the cylinders in baggies labeled with the material, the appropriate batching number, and (if necessary) the cure schedule.
8. Throw away the syringe husks and sweep up the plastic debris from the hammering step. Put your tools and PPE away.