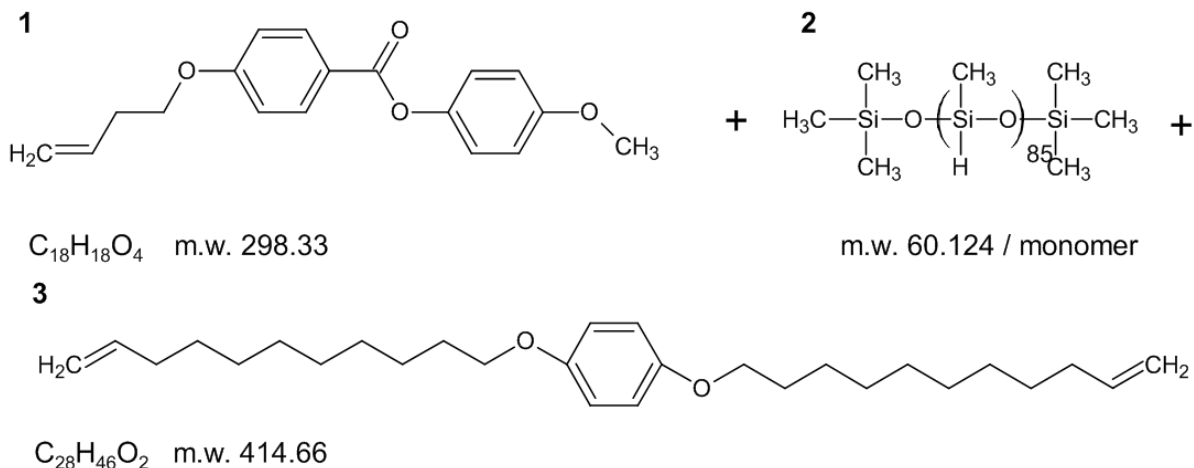


Side chain LCE

Finkelmann Group

NLCMF material preparation procedure



Materials

Chemicals	CAS #
1) Nematic mesogen	
2) Homopolymer	
3) Crosslinker	
4) Pt catalyst (COD)	12080-32-8
5) Toluene	108-88-3

✂ The platinum catalyst is prepared as a 1% w/w solution in methylene chloride

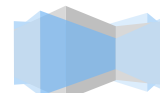
Spreadsheet:

Enter the crosslinker density you would like for you sample in the equivalents column of the crosslinker row. The required amounts of material will then be calculated automatically

Compound	m.w.	mass (mg)	mmol	equivalents	actual
mesoge (1)	298.33	258.99889	0.868162	0.87	
polymer (2)	60.127	60	0.997888	1	
crosslinker (3)	414.66	26.89597	0.064863	0.065	
Pt catalyst 1%		15.384615	uL		
Toluene			1 mL		

✂ *nota bene* Each cross linker molecule has 2 reactive vinyl groups that must be accounted for when calculating the amount of required cross-linker.

Actual amounts:



Enter the actual measured amounts in the **actual (mg)** column of the spreadsheet to calculate the true crosslinker density of the material.

Compound	m.w.	target mass	mmol	equivalents	actual (mg)	corrected eqv
1	298.33		0.86813	0.87	258.99	0.870
2	60.127		0.99789	1	60	1.000
3	414.66		0.06485	0.065	26.89	0.065

Pt catalyst 1% 15.38461538 uL

Toluene 1 mL

Please enter the mass in the above spreadsheet for the target mass

Synthesis Procedure

- Pre-heat the spin casting apparatus to 65°C before measuring out the chemicals. The spin casting head should be lined with a piece of Teflon support film.
- All materials (crosslinker, mesogen, siloxane polymer, and solvent) are measured using an analytical balance. Try to measure precisely as possible. Do not be concerned if a few mg extra of the crosslinker or mesogen are added. However, the spreadsheet calculations will no longer be accurate. Enter the weighed mass amounts into the spreadsheet to obtain the correct crosslink density
- Materials **(1)**, **(2)**, and **(3)** (see spreadsheet above) are measured on an analytical balance and transferred to a glass vial in the following order:
 - The polymer **(2)** (liquid) is transferred using a pipettor. The amount of polymer transferred is determined using the balance, not the pipettor.
 - The crosslinker **(3)** is transferred next, using a spatula, followed by the mesogen **(1)**. If too much mesogen is added, it may be very carefully removed using a spatula. This is not the case for the crosslinker, as it comes in direct contact with the siloxane polymer. If too much of a crosslinker is added (> 2% mol), use the spreadsheet to recalculate the amount of polymer required and carefully add additional polymer as needed
- The materials are dissolved in thiophene free toluene. The materials spontaneously dissolve in ~60 minutes. Alternatively, gently vortex or heat to speed up the process. Be sure no undissolved materials are left on the side of the vial. Allow the vial to return to room temperature before adding the catalyst.
- Add the platinum catalyst solution using a pipettor or Hamilton syringe and gently swirl for a few seconds. The catalyst may be added directly to the reaction solution, or added to the solution after it is placed in the spin casting head.
- Collect the solution in a syringe (disposable or glass). Place a 0.2 um syringe tip filter on the syringe and pass the reaction solution through the filter and into the spin casting apparatus. The material may be prepared without using the filter
- Tightly seal the spin casting head so that no liquid may escape.
- Centrifuge apparatus @ 65°C, for sixty to seventy minutes. After the spinning is complete, cool the centrifuge using liquid nitrogen or dry ice. Please note that you are cooling the elastomers film inside the spin casting head to slightly below room temperature, so while the spin casting head may be very cold, it is the temperature of the LCE film that is of concern. If the LCE is



- much above room temperature, the toluene may evaporate from the film too quickly, effecting LC alignment. If the LCE is ice cold, it can be difficult to work with during the handling process.
9. Open the spin casting head, the LCE film should now be formed. If the material is still a liquid inside the spin casting head, add a few μL of catalyst solution, and spin for an additional 30 to 40 minutes.
 10. Remove the elastomer strip from the spin casting apparatus using tweezers, grasping the Teflon support film behind the LCE film. The LCE will stay on the Teflon support film if you handle it gently. Lay on LCE | Teflon support film on a large piece of Teflon film, LCE side up. Secure both ends of the LCE | Teflon film with tape. The LCE is now swollen with toluene and is isotropic.
 11. During the spin casting process, formation of chemical crosslinks is initiated, but not completed. At this stage, the LCE is a swollen polymer network that is partially crosslinked. After the LCE film is removed from the spin casting head, it is hung and weights are attached, uni-axially stretching the LC-polymer network while the final crosslinks are formed, establishing an aligned network.
 12. You now have approximately 20 to 25 minutes to hang the LCE film. First cut the LCE into 2 or 3 pieces of equal length. As the toluene evaporates, stress builds inside the LCE, and it will twist and curl on the Teflon support film, and may crack. Sectioning the film helps to relieve this stress.
 13. Attach a small piece of Tefzel tape, approximately $1\text{cm} \times 1\text{cm}$, to the center of a metal thin metal, wood, or rod, diameter $\sim 4\text{mm}$. Carefully attach the tape to one end of the LCE film, and gently pull up the LCE from the support film. Place the rod in a support so the LCE may hang freely. It will also stretch as weights are added to the systems, so be sure to leave room below the LCE to allow for an increase in length. Ideally, the distance between the bottom of the LCE and the bench surface should be at least 3X the length of the LCE film. Place a large piece of Teflon film underneath the area where the LCE are hung in case the films fall. Hang all pieces of LCE before proceeding to the next step.
 14. After all LCE strips are hung, the need to be watched as may fall off from the tape. In the initial 30 minutes after the LCE are hung, as the toluene evaporates, the LCE tends to lift from the tape, and may fall.
 15. If it appears the LCE is about to fall, lift the LCE by its support rod and gently lay the LCE film on the large Teflon sheet below the LCE. Place a new piece of tape on the rod, and re-hang the LCE. In most instances, the LCE will only need to be re-hung one time.
 16. Next, cut a 3cm strip of Tefzel tape, and attach a paperclip to the bottom third of the tape. The paperclip will serve as a weight to stretch the LCE and should be parallel to the LCE film. The mass of the paperclip we use is approximately 0.3 grams.
 17. Attach the tape | paper clip to the bottom of the LCE so approximately the bottom 5mm of the film is covered. The LCE will begin to stretch but should not break. At this point, the tape | paperclip may slide off the LCE. In this case, get use a fresh piece of tape, and attach the paperclip a second time.
 18. After approximately 30 minutes after all the paper clips are attached to the LCE, it is now time to place a second piece of tape on the opposite side of the LCE from the initial piece of tape at the top and bottom of the LCE.
 19. Approximately every 30 to 40 minutes, hang another paper clips until a total mass of approximately 2 to 2.5 grams is suspended from the LCE. It is better to use many light



paperclips than one heavy clip. After each paperclip is attached to the LCE, carefully inspect the LCE for cracks along the edge of the film. If cracks appear, remove the paperclip.

20. With time, the LCE will turn cloudy and then clear again. This process may take several hours. Let hang with weight for 18 to 24 hours.
21. Remove $\frac{2}{3}$ to $\frac{3}{4}$ of the paperclips, and place the LCE in an oven around 50°C for 36 to 48 hours to complete all chemical reactions.
22. Remove the LCE film from the oven. Take off all paperclips, but the tape may be left on the LCE film. Store the LCE on Teflon film. The films are now complete and ready to be washed. [Click here for information on washing the LCE films.](#)

