

Overview and walkthrough for TR-SAXS: Design, Fabrication, Testing, Running

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The purpose of this document is to provide an overview of the current state of fabrication and usage for time resolved SAXS chips at MacCHESS as of January 2018 (when Jesse H. finished his postdoc). It is organized into steps in the fabrication, followed by a section on running (mostly dealing with setting and the hardware and control software), with some additional appendices detailing other useful methods or failed fabrication attempts.

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1 Mixer design and simulation

The core mixer design is based on the design in (Kane et al., 2008), shown below. Original design was intended for a $\sim 50 \mu\text{m}$ beam in the vertical dimension, and to work with a wide range of flow rates.

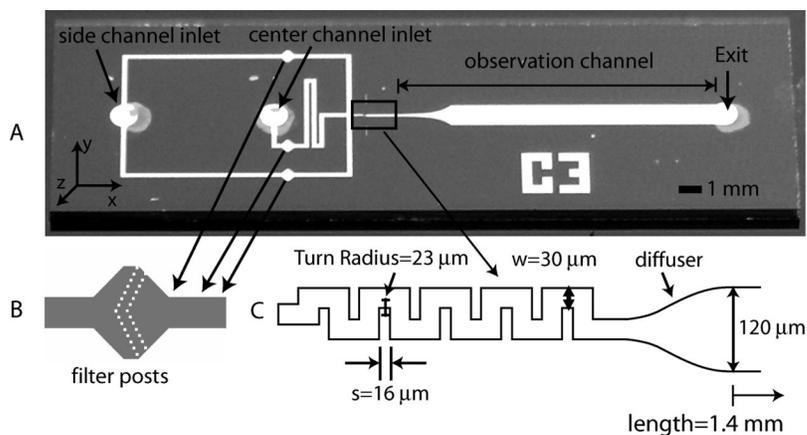


Figure 1 - Mixer as shown in Figure 1 of Kane et al.

Testing of specific mixer designs are done in COMSOL v5.1.

1.1 COMSOL

1.1.1 File locations and descriptions

All files are located on the CHESS auxiliary directory, available from the lnx201 CLASSE computing node. The base path is:

/nfs/chess/auxiliary/data1/macchess/biosaxs/jbh246/mixer_comsol

The most recent version of the mixer CAD is the *mixer3.mph* and *mixer3_2d_fixed.mph* files. The first file is the mixer in 3D, which takes a long time to simulate and can't handle a relatively large range of flowrates. The second file is the mixer in 2D, which runs much faster and gives results more or less equivalent to the 3D file. I generally use the 2D file for most of my simulations.

Additionally there are *mixer_crl.mph* and *mixer_crl_2d.mph* files that contain mixers with much smaller observation regions, such as we might use with a small, optimized CRL beam.

Output of COMSOL files are usually saved in separate folders, such as the *mixer_jan17_results* and *crl_results* folder.

Note: .mph files are comsol files.

1.1.2 Starting COMSOL for simulations

- 1) ssh into the lnx201 node with graphics forwarding enabled: *ssh -Y lnx201*
- 2) Access a free cluster node with a graphics card: *qrsh -q interactive.q -l cuda_free=1*
- 3) Run comsol: *comsol*

Note: The first time you use comsol, it is important to change the recovery file directory. This is described here:

<https://www.comsol.com/support/knowledgebase/1095/>

If you run a large simulation, you can fill up your home directory with the recovery file, and get an error. When I did this, CLASSE computing recommended creating a directory in /cdat/tem, described here:

<https://wiki.classe.cornell.edu/Computing/TemDisk>

1.1.3 Working with COMSOL files

Most of the parameters for the mixer are defined in the Global Definitions of the Parameters files. This lets you change diffusion coefficient, mixer channel widths, flow velocity, ratio of flow velocities in the buffer and sample inlets, etc.

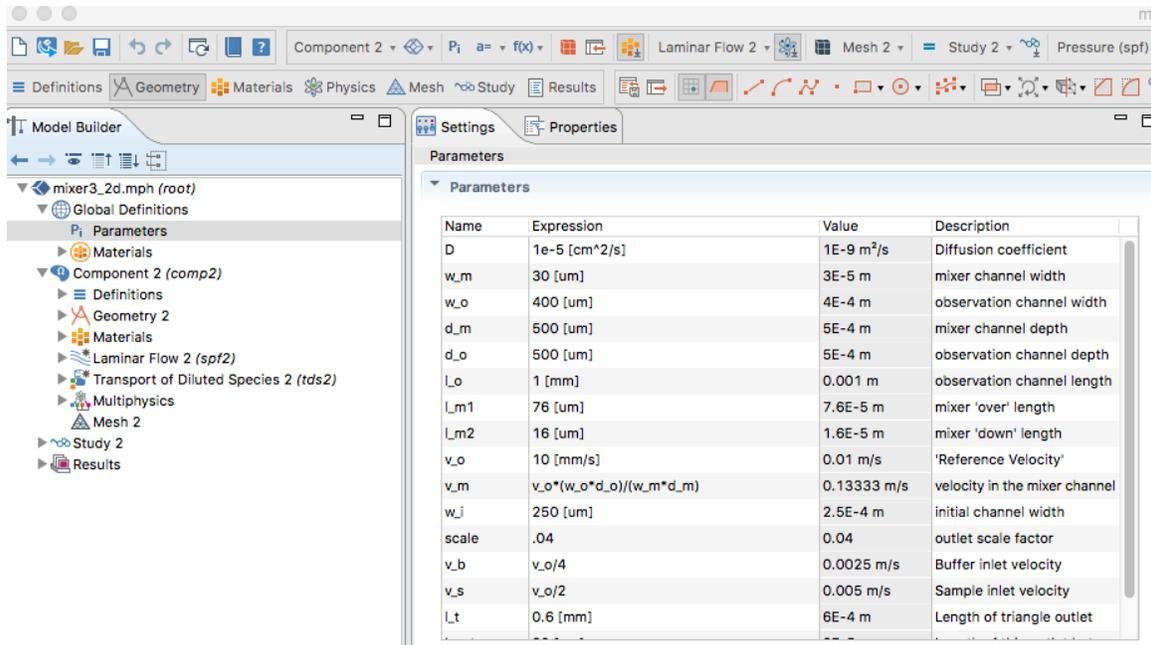


Figure 2 - Parameters available under the Global Definitions node.

Generally speaking, parameters ought to be well set in the physics part, though obviously should be modified as needed.

Generally speaking, COMSOL should be used for design testing, and for calibrating time points at a given flow rate. Calibrating time points will be described in the following section.

Note: For the 3D mixer, I had to create my own mesh to get it to run, even that is kind of rough. That meshing will probably have to be redone if you change the geometry.

1.1.4 Overview of mixer features

It is useful to have a working vocabulary for how I talk about the mixer. Here's a rough breakdown, using the COMSOL image below.

- 1 – Buffer/side inlets
- 2 – Sample/center inlet
- 3 – Mixing region
- 4 – Outlet region/taper
- 5 – Start of observation region/first observable point
- 6 – Observation region

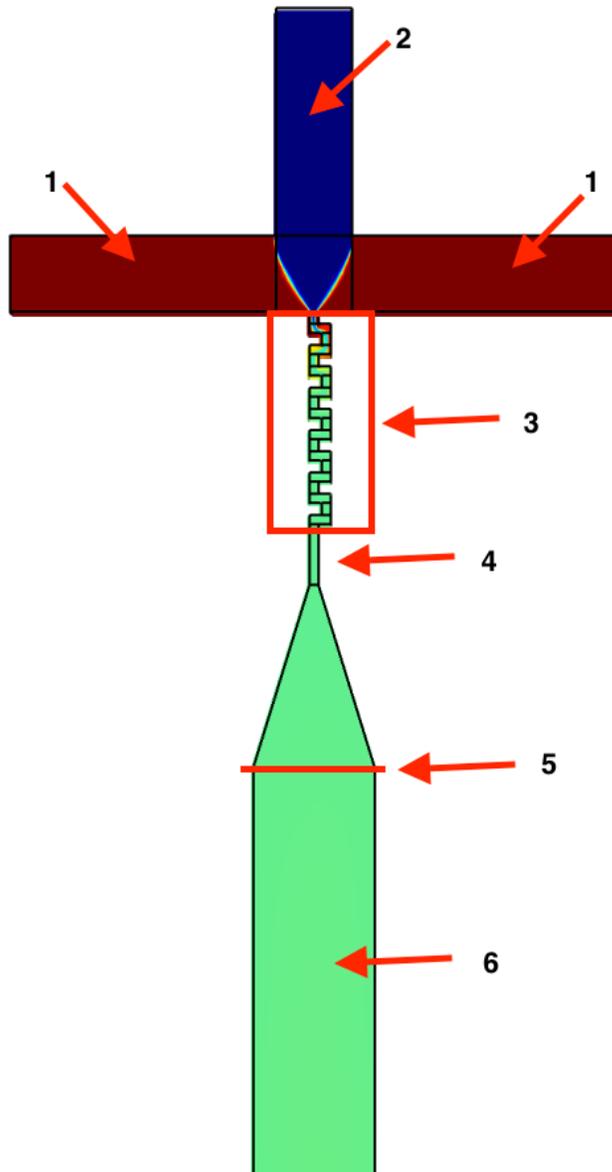


Figure 3 - Standard naming convention for regions of the mixer.

1.1.5 Calculating time points from COMSOL results

One of the biggest questions is how do you know what the time point (or distribution of time points) at a given position in the mixer is. This assumes you have COMSOL results from a simulation. The general approach I use is: have COMSOL calculate a set of streamlines for the device, and export the concentration and velocity along those streamlines. Then for each streamline, find where it first crosses a given concentration threshold (arbitrary, usually 0.4 out of a possible 0.5), and mark that as time 0. Calculate for each subsequent position along that line how long it takes to get there from time 0, and mark that as the time after mixing at that point. At each point in the mixer, average over every streamline to get the observed time since mixing, and get the spread in mixing times.

To do this, once you have results, go to (or add) a 2D plot node. Go to or add a Streamline. In the streamline, make the Streamline positioning tab make the positioning “On selected boundaries” and the number 1000. Set the selected boundary to be the top edge of the center channel (where ‘sample’ first enters the simulation). In the Quality section set the Resolution to “Coarse” and the Recover to “Off”. Add a Color Expression node and in this node set the color expression to the flow velocity (or the concentration, depending on the streamline). In the Export node, add a node for the streamlines by velocity and concentration. Export each as a spreadsheet. These are all set in the mixer files I use above.

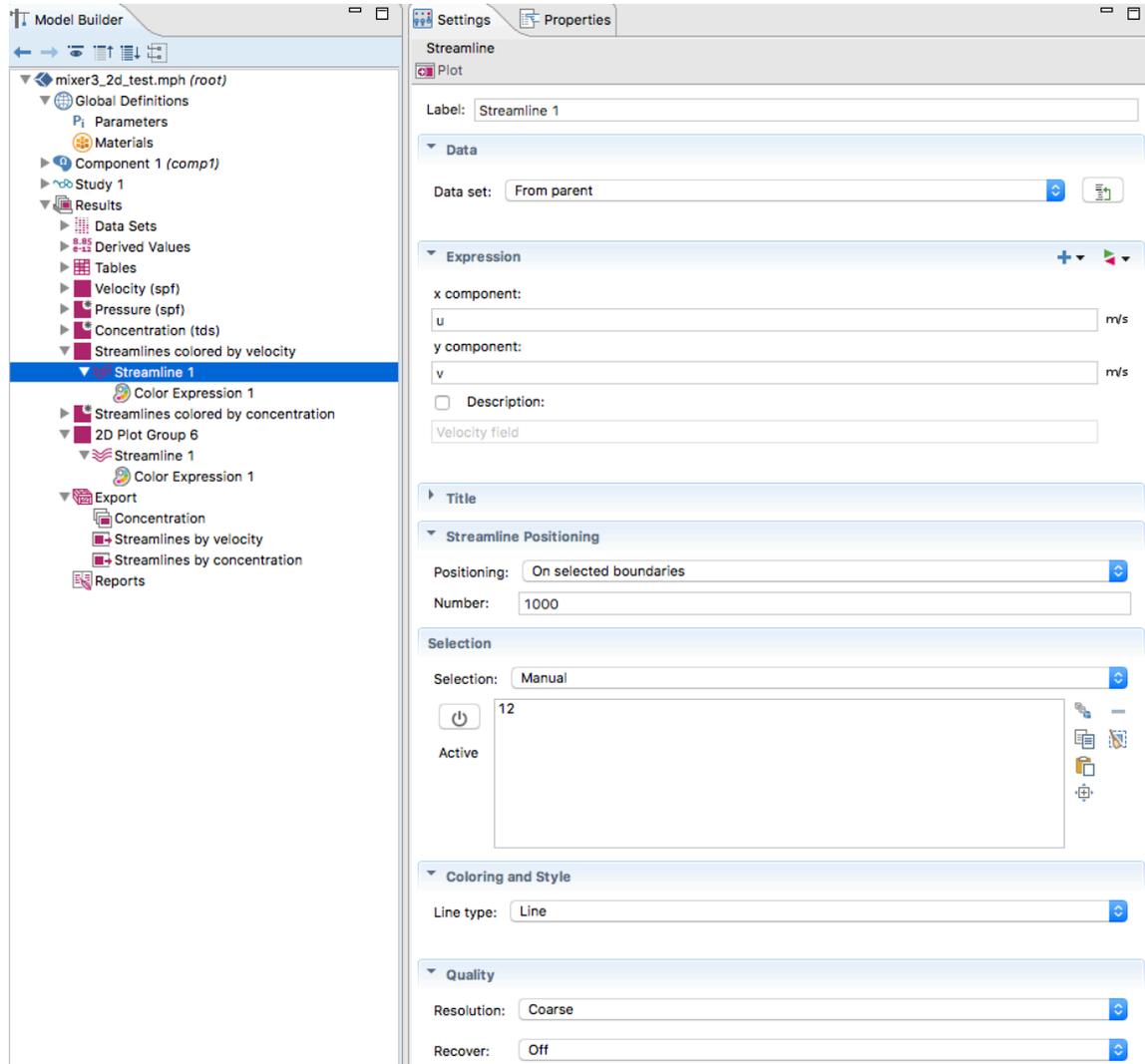


Figure 4 - Streamline settings tab for a 2D mixer.

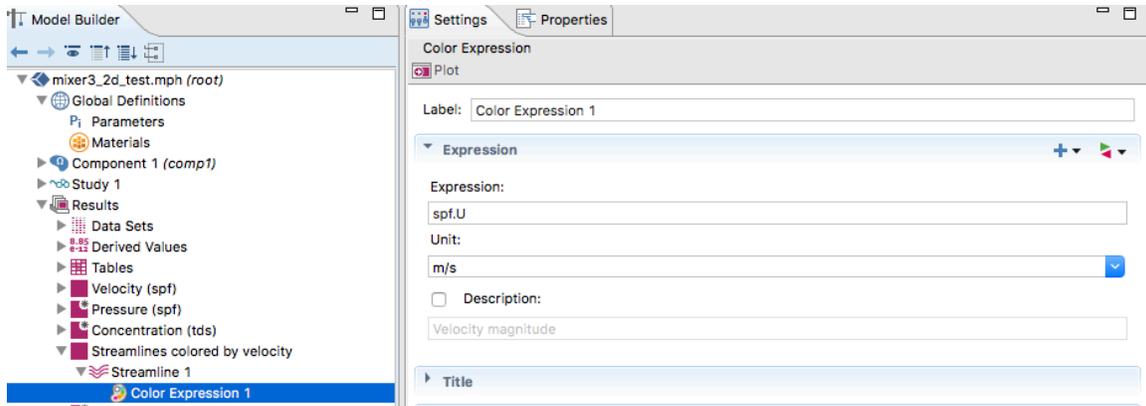


Figure 5 - Streamline color expression settings for a 2D mixer.

Appendix A includes a discussion of the appropriate number and quality of streamlines.

I have created a set of python scripts for processing the output of the streamlines into useable numbers. The core file is the mixer.py file in */nfs/chess/auxiliary/data1/macchess/biosaxs/jbh246/scripts*. Files that use these functions that make good examples are the *mixer_jan17.py*, *mixer_crl.py*, and *mixer_2d_results.py* (among others).

These functions load the streamlines from file, and then carry out the calculations as described above. A basic usage is:

```
v_data = 'filepath'
c_data = 'filepath2'
```

```
v_streamline = CStreamline(v_data)
c_streamline = CStreamline(c_data)
```

```
results = streamline_mixing_time_2d(v_streamline, c_streamline)
```

Note that there are separate functions for 2D (*streamline_mixing_time_2d*) and 3D (*streamline_mixing_time*)

The *streamline_mixing_time* functions take as input:

v_streamline (required) – A CStreamline (COMSOL streamline) object made from the velocity data

c_streamline (required) – A CStreamline object made from the concentration data

threshold (optional) – The concentration threshold at which mixing is said to have occurred. Defaults to 0.4

plane (optional) – The direction of ‘positive time’ through the mixer in COMSOL coordinates. Note that it defaults to ‘y’ (how all of my mixers are built), and that other coordinates are not supported (but should be easy to implement).

step (optional) – The step size of the interpolated results, defaults to 1e-5

conv (optional) – A unit conversion factor, defaults to 1

It returns as output:

eval_at – Position in the ‘plane’ coordinate (direction of positive mixing), in COMSOL coordinates

t_mix_mean – Mean mixing time at the positions in *eval_at*

t_mix_median – As *t_mix_mean* but median mixing time

std_mix – Standard deviation of the mixing time at the positions in *eval_at*

tmix_25p – 25th percential mixing time at the positions in *eval_at*

tmix_75p – 75th percentile mixing time at the positions in *eval_at*

times – The full array of times at each position in *eval_at*

These can then be used to plot the output. Note that the mixer coordinates are usually in [m], and that the mixer usually moves in the negative y direction. So to make a reasonable plot in [mm] and [ms] you might do:

```
plt.plot(eval_at*-1*1000, t_mix_median_p1*1000, 'b', label = '0.1 mm/s')
```

```
plt.legend(loc = 2)
```

```
plt.xlabel('Position along mixer in y direction (mm)')
```

```
plt.ylabel('Mixing time at position y (ms)')
```

```
plt.axvline(1.5, color = 'r',linestyle = '--')
```

```
plt.show()
```

This would create a plot like below (shown with 3 mixing times), which is mixing time in ms at a given flow rate as a function of position along the mixer in mm. The vertical dashed line shows where the first observable point in the mixer is, corresponding to the end of the triangular outlet in the mixing region. This you have to get from COMSOL (either by knowing how big you made each element in your geometry, or by reading it off the COMSOL image).

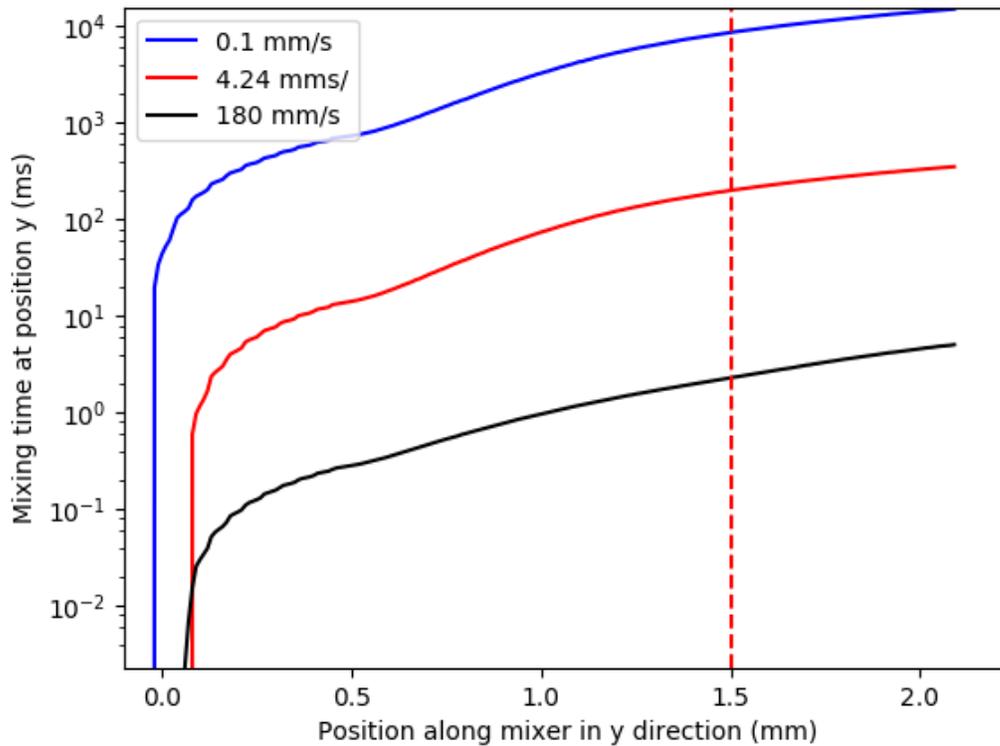


Figure 6 - Mixing time as a function of flow rate and position along the mixer.

This should in theory be done for every flow rate used. My plan, which I never finished, was to do this for a wide range of flow rates, then create a nice python program that loaded all that data, interpolated for each position and flow rate, and then could output the time point in the mixer at any given flow rate and position.

I generally find the median time and the 25/75 percentiles to be most sensible for mixing time and standard deviations. That's something someone else might want to do a careful investigation of.

1.2 Converting volume flow rates to linear flow rates, and quick estimation of time points

In addition to simulating the mixing, you want to be able to figure out how much sample you're using, and can also get a rough idea of what the time points are by knowing the minimum observable time (time at the first observation point), the total flow rate, and the observation channel size. This is what I attempted to do during the experiments in November 2017. Additionally, COMSOL uses mm/s as a velocity input, while our flow meters measure in $\mu\text{L}/\text{min}$, so conversion between those is necessary.

I have created an excel spreadsheet do this called *mixer_numbers* (sheet 2), which is in the folder associated with this summary. In essence it's pretty simple: you know the channel cross sectional dimensions (0.5 mm x 0.4 mm), so converting between an average linear velocity and the volume flow rate is simple.

Once you know the average linear velocity, you can calculate the delta t after the first observable mixing point based on how far down the channel you are simply by taking $dt = d_{\text{channel}}/v_{\text{flow}}$. Add this to the first observable mixing time (known from prior COMSOL calculation) and you can quickly estimate the time point measurable at any region in the mixer. This is probably good enough for on the beamline work, but you'd want to use the more accurate numbers from COMSOL for data analysis.

This approach also lets you go from flow velocity to volume flow rate, and thus calculate the fluid flow that you need to get certain time points, and how much sample consumption that would be.

Note: The sample consumption should account for the mixing ratio. So, for example, a linear flow rate of 50 mm/s is a volume flow rate of 10 uL/s, but at a 1:1 mixing ratio that's only 5 uL/s of sample!

2 Mixer fabrication

Mixer fabrication was done primarily at CNF. This section will focus on current working fabrication steps.

List of necessary CNF training for students getting started on this project. This is the minimum necessary to expose and develop SUEX layers on chips:

- 1) Take the required CLASSE safety tests
- 2) Fill out New User form, return to Kathy Springer
- 3) Take CNF New User training
- 4) General Photolithography training
- 5) SUEX/SU8 training + laminator (Beth Rhodes)
- 6) ABM contact aligner training (Ed Camacho)

Appendix B is devoted to describing failed attempts and Appendix C to speculative ideas on other methods of fabrication. The general outline of mixer fabrication is given here, followed by detailed sections on each step:

- 1) Laser cut out an acrylic baseplate in 0.5 or 0.7 mm acrylic
- 2) Apply kapton windows to the observation region of the acrylic baseplate
- 3) Laminate 500 um SUEX on the baseplate

- 4) Do standard contact aligner photolithography to expose the SUEX and pattern the mixer's fluidic channels.
- 5) Develop out the mixer fluidic channels.
- 6) Either, laser cut the piece into individual devices and apply a sealing top layer (which acts as an x-ray window), or apply a sealing top layer and then laser cut into individual devices.

2.1 Making a mask for photolithography and CAD for the versalaser

The first step in mixer fabrication is making a mask for the mixer photolithography and CAD for the versalaser baseplate. This wasn't included in the above steps because it only needs to be done when the mixer design is changed.

2.1.1 Mixer CAD

The mixer CAD has been done in LEdit, available in the CNF CAD room on any of the computers. The most recent version of the CAD is *mixer4*. Training on LEdit is available through CNF, and there shouldn't be anything particularly tricky about the CAD.

The most important thing to remember is that we are working with a negative photoresist, which means you need to expose the resist that you want to stay on the device. For us, this means >90% of the mask must be open. My approach is that I make the device pattern as I would for a positive resist, and then put a square around it and do a subtraction. This makes the appropriate negative resist pattern.

The current mask is designed to produce a 3x3 square of mixers. You can buy SUEX in 98 mm square sheets, and each mixer is 30 mm x 30 mm, so this tiles nicely into the SUEX without wasting it, and leaving a little edge around each. Typically, you produce all the mixers in a batch, then use the laser cutter to cut out the individual units.

Additionally, remember that your pattern should be centered at (0,0).

Currently, the inlet channels are 0.25 mm wide and taper to 0.03 mm where they meet, the mixer channels are 0.03 mm wide, and the observation channel is 0.4 mm wide.

2.1.2 CorelDraw CAD

CorelDraw is available on the computers in the CNF CAD room, and on the VersaLaser computer. The CAD for all pieces associated with the mixers is currently located in the *All_User_Patterns/jbh246/tr_saxs* folder on the desktop of the VersaLaser machine.

The base for the acrylic can be cut out of any thickness, I tend to use 0.5 or 0.7 mm thick, and have noticed little difference between the two. You'll see in the CAD that it has four layers each in a different color. The black small holes are the inlets, outlets,

and x-ray window for the mixer. The red squares are to cut out individual mixing units. The blue outline is just a guide for the size of a SUEX sheet. The green outline is the cutout for the baseplate.

Typically, fabrication is done on an entire baseplate simultaneously, allowing production of 9 mixer units at a time. That means you want to cut just the black and green layers, not the red (you never want to cut the blue). Alternatively, if you want to work with individual baseplate pieces, you can cut the red layer as well.

The three inlets at the top are spaced so that the inlet channels in the LEdit CAD can be centered in them. The same is true of the outlet at the bottom. The inlets are currently 2 mm diameter, and the observation window is currently 10 mm long and 0.8 mm wide. The frame of an individual mixer piece is 30 mm square.

2.1.3 Make a mask

Masks are made using a 5" quartz mask (available from the CNF store, at the same time also get an orange mask holder). I currently use the Heidelberg DWL2000. Because most of the area has to be exposed, it takes ~1.75-2 hours to write the mask. It takes an additional 30 minutes or so to do the development and processing after the mask is written.

2.2 Laser cut a baseplate

The first step in making the mixers is to laser cut the acrylic baseplates for the mixer. As with the mask, it is typical to cut a 3x3 mixer baseplate, to make a batch of 9 mixers at once. The appropriate CAD layers to cut are described in section 2.1.2. This laser cutting is done on the VersaLaser. I typically use the red lens, and typical settings are:

0.5 mm:

17% power

10% speed

1000 PPI

0.6 mm Z

0.7 mm:

24% power

10% speed

1000 PPI

0.8 mm Z

Note that power settings should be checked regularly.

I have also used 0.8 mm UV transparent acrylic with settings:

27% power

10% speed

1000 PPI
0.9 mm Z

2.3 Apply kapton windows to baseplate

The observation window in the baseplate now has to have the window material applied. The typical way I do this is to apply 7.5 um thick Kapton (polyimide) sheet across the window region, though other materials could potentially be used. The kapton used is obtained from the CHESS staff cabinet, and is the standard chemplex films. Some 100 mm square sheets of the same thickness kapton are available in the storage box (box 202, with Soren and Richard's names on it) in the cleanroom if you want to try to bond the whole baseplate at once.

Typical window application is done by:

- 1) Cut small pieces of kapton to just larger than the observation window area
- 2) Apply as thin a layer as possible of 5 minute epoxy around an observation window
- 3) Carefully adhere the kapton to the baseplate.

Do this for all the windows on the baseplate.

Tips:

- Use a small pipette tip to apply several drops of epoxy on each side of the observation window cutout.
- Use the edge of the pipette tip to scrape the epoxy around the window, removing as much excess as possible (pipette tip should be essentially flat, and scrape along the acrylic)
- If you get a string of epoxy on the acrylic where it shouldn't be, gentle swabbing with a 50% IPA 50% water solution shortly after it sets will easily remove it.
- Be careful not to allow epoxy to run into the PMMA window as this can block and interfere with x-rays.
- Do not press down on the Kapton to flatten it out against the PMMA. With time, the epoxy will spread and the Kapton will lay flat. Adding pressure can push epoxy into the PMMA window.

Note: If any smoothing of the baseplate is going to be done, it should be done before the window is applied. For example, wet sanding to remove burrs from laser cutting (see section 5). In this case, you may want to use an alternative method of window bonding such as chloroform bonding by wetting the surface of the chip with chloroform, and then placing it onto the kapton. As long as the kapton is very flat, this should minimize surface ridges.

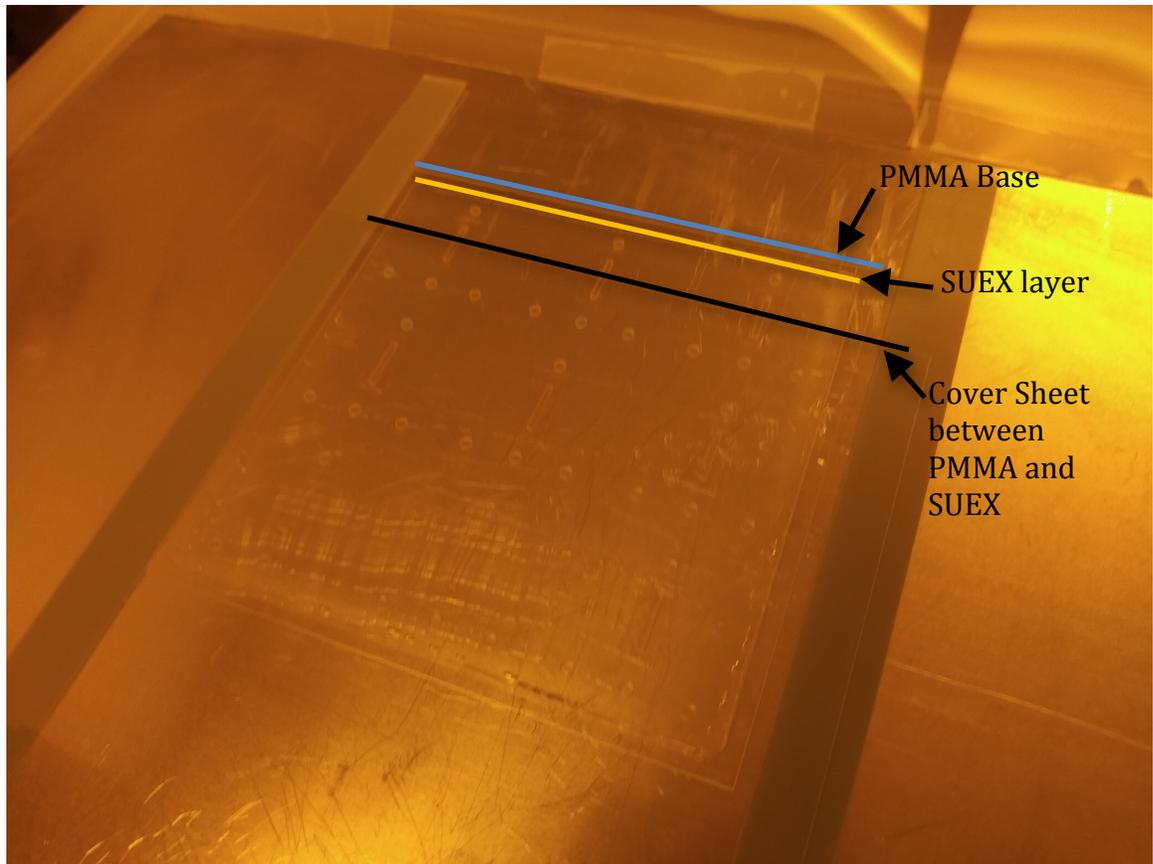
2.4 Laminate SUEX

Once the baseplate has windows applied, SUEX can be laminated on top. I typically use 500 um SUEX in 98 x 98 mm sheets. This is available from DJDev Corp (<https://djmicrolaminates.com/>). Most recently their US distributor was Integrated Micro Materials, Richard should have the most recent quote.

Lamination is done on the SUEX laminator in the Class 2 resist room at CNF. The settings used are: Speed 1, Temperature 48 C indicated 75 C actual, using 1 shim if the baseplate is 0.5 mm and two shims if the baseplate is >0.5 mm. The hazy PET sheet is left on during lamination. A post lamination bake is done for 15 minutes at 65 C. Removing the haze PET sheet after lamination is recommended as it can cause defects in the SUEX during the bake. Unexposed SUEX assemblies (either full 3x3 sheets or individual pieces) can be stored in the marked orange mask carrier.

Tips:

- Laminating an entire 3x3 sheet of PMMA baseplates before photolithography and subsequent laser cutting is recommended. The SUEX sheet is slightly smaller than the PMMA baseplates but if centered carefully, will cover all 9 chips upon laser cutting.
- A cover sheet between the PMMA and SUEX layers helps to eliminate any air bubbles and defects during lamination. The cover sheet must be pulled out before the PMMA and SUEX goes into the laminator (see below figure).
- Place the laminated sample on a pre-heated silicon wafer during the bake. Post bake, let the sample cool for 1-2 minutes before removing it from the silicon wafer. This prevents still warm SUEX from sticking to the wafer.



Note: 500 um SUEX is nominally 500 um, but often a bit thinner, more like 480 um.

2.5 Photolithography

After lamination, the SUEX is brought into the contact aligner room. Exposure is done on the ABM, with the filter in. A total exposure dose of 3600 mJ should be delivered.

Place the baseplate+SUEX assembly on the sample chuck baseplate side down. Align the mask with the inlet holes, outlet holes, and observation window using first your eye and then the alignment microscopes. It is possible to get the two alignment scopes on the two outside devices on a 3x3 device mask (so columns 1 and 3 of devices), which allows good alignment. Note that aligning the bottom right chip and the top left chip (two opposing corners) will ensure proper alignment of the entire assembly.

In order to avoid excessive heating, the exposure sequence should: 45 s exposure, 45 s rest, repeated as necessary. Total exposure time (not counting rests) should be around 420 s, but it will depend on the exact intensity of the UV lamp, always check this before exposure.

After exposure, take the assembly back to the Class 2 room for a post exposure bake. Put the assembly on a carrier sheet e.g. a silicon wafer, and then on a room temperature hot plate (one of the ones you can reserve). Ramp the hotplate temperature to 65 C at 5-8 C per minute (depending on how patient you are). The SU-8 hotplates set to a ramp rate of 8 achieve this result. Do a 5 minute bake at 65 C, then ramp the temperature to 95 C at the same ramp rate. Bake for 20 minutes at 95 C, then turn off the hotplate and let it ramp to room temperature over the course of an hour.



Figure 7 - Post exposure back with acrylic/suex on a carrier sheet.

Tips:

- Make sure that the substrate vacuum holes on the ABM sample chuck aren't aligned with any of the cutouts in the acrylic.
- Due to the non-uniformity of the assembly, the sample vacuum may not hold the assembly to the chuck tightly. To account for this, move/rotate the stage very slowly during the alignment as to not displace the assembly.
- You will have to move the SU-8 further down from the mask to align it after leveling than the ABM manual says. Probably two full turns, if not more. Movement should feel free, not sticky, so adjust accordingly.

- Even with careful resting between exposures, the SUEX can sometimes stick to the mask. To minimize this, lower the SUEX away from the mask very slowly.
- Don't forget to clean the mask after use: wipe it down with Acetone and IPA, then blow dry.

Note: make sure to remove the hazy PET sheet before exposure

2.6 Development

SUEX devices should be developed SUEX side down in EBR-10A. I like to use the kind of stand you would normally use for a stirbar, upside down (see picture below) to minimize contact of the SUEX with anything else (maximize fully free/exposed area to solvent). If you have pieces, you should use a piece basket, and make sure the pieces are oriented so the SUEX is facing down (as much as possible, pieces end up on a tilt).

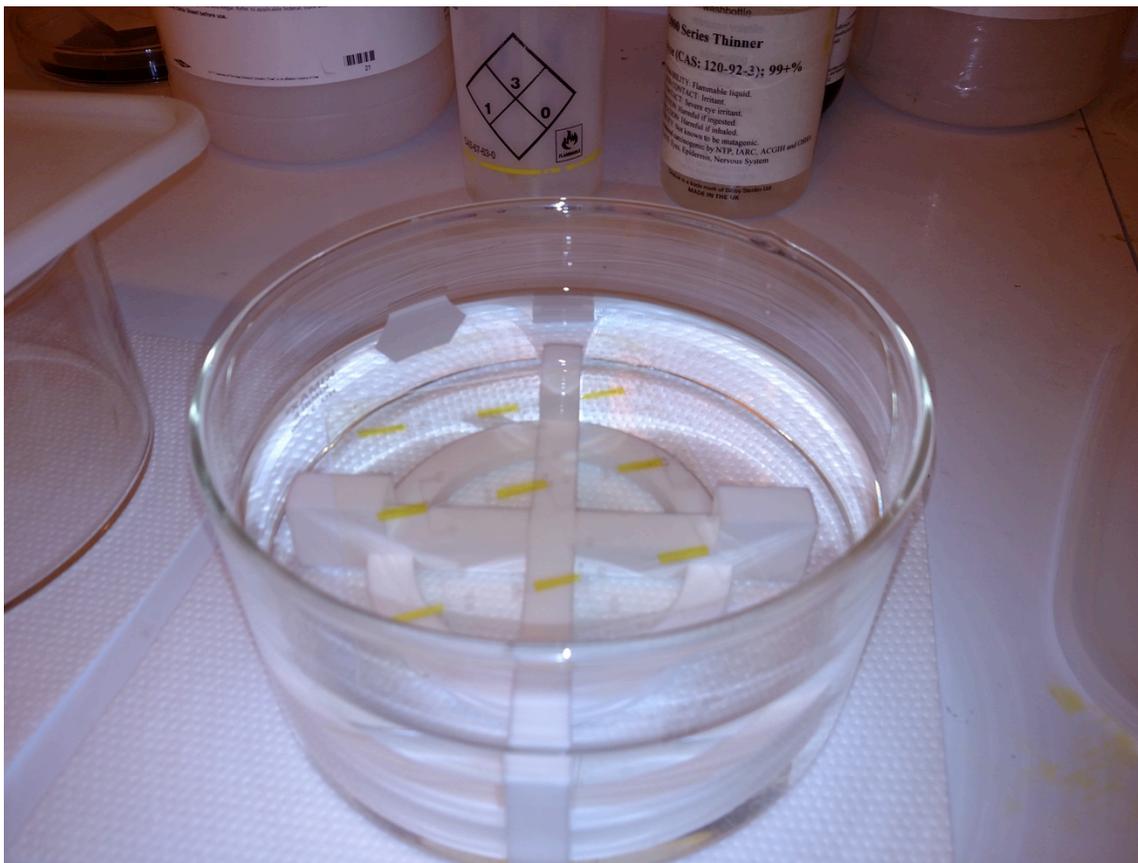


Figure 8 - Developing the SUEX in the hood. The acrylic is in EBR-10A on a stand. The yellow Kapton windows are easily visible.

I found for about a year that development times of 14-18 h were necessary. However, in November 2017 CNF got a new batch of EBR-10A, and I have now found

that development times are around 15 minutes (not counting the 2 x 5 minutes in fresh EBR-10A below) work well.

Develop, then place the device(s) into fresh EBR-10A for 5 minutes face down and 5 minutes face up (if development times get too short you may have to adjust development time to account for this step). Move from the fresh EBR-10A into IPA for 60s with continuous agitation of the IPA. Remove promptly from the IPA and blow dry with compressed air. If there is still an odor of EBR-10A about the piece, put it back into IPA for more agitation. Repeat as necessary.

Devices should be examined under the microscope. By racking the focus of the microscope you should be able to get an idea of whether the channels are fully developed through, and whether or not any liftoff of the channels is apparent. Liftoff most likely occurs from overdevelopment, though it could also be attributable to bad lamination or under exposure of the SUEX. In the extreme cases it can lead to collapse of the mixing channels.

2.7 Dicing

At this point you should have either a set of devices in a 3x3 sheet or a set of individual pieces ready for sealing. One goal of the project was to develop a way to seal all devices in a 3x3 sheet simultaneously, but that is a ways off. If you have a 3x3 sheet, the best next step is to take it up to the laser cutter and cut out the individual devices (dice it).

Dicing out the devices is relatively straightforward:

- 1) Take a piece of relatively thick acrylic, such as 1.0 mm, and tape it down to the cutting table.
- 2) Send both the outline layer (green) and individual device layer (red) to the laser cutter.
- 3) Cut out just the outline of my baseplate (disable the individual device layer cut). Remove the cut piece, leave the acrylic on the table.
- 4) Place the 3x3 sheet into the area you just cut out, disable the outline layer and enable the device layer. Cut out the devices.

This gets the alignment pretty close to what it should be, certainly better than I have been able to do by eye.

Settings for SUEX on 0.5 mm:

22% power
10% speed
1000 PPI
1.0 mm Z

Settings for SUEX on 0.7 mm:

30% power
10% speed

1000 PPI
1.2 mm Z

As always, these cutting settings may vary a little.

2.8 Sealing

Sealing the device liquid tight while also applying a x-ray window is a significant outstanding challenge. I have tried various approaches with varying levels of success.

2.8.1 ADEX as an adhesive layer

The first approach I tried was using ADEX as an adhesive layer. ADEX is the thin version of SUEX, I tried both 5 and 10 um thicknesses. The challenge I had with this is that it did not consistently yield reliable mixers, most mixers would delaminate very quickly under even modest pressure. Additionally, the ADEX couldn't be properly hard baked, so it remained solvent sensitive (it would dissolve in IPA, for example).

It turns out to be very hard to peel full sized sheets of 5 or 10 um ADEX, usually they tear. So this bonding has to be done on pieces. To do so:

- 1) Remove one of the ADEX cover sheets.
- 2) Using tweezers or a razor to verify there is ADEX where you think it is on the coversheets (optically it can be quite hard to tell which coversheet has ADEX in which area). Usually, because it tears, each coversheet ends up with some ADEX.
- 3) Place an appropriately sized piece of kapton on your aluminum laminator sheet. Especially in the winter with low humidity this will want to stick to everything, including your gloves and the topsheet. You may have to use a little scotch tape.
- 4) Carefully place the ADEX sheet (ADEX side down!) on the kapton. Re-stretch the kapton to eliminate any creases.
- 5) Laminate using speed 1 and temperature 65 C actual.
- 6) Do a post lamination bake for 10 minutes at 65 C.
- 7) Place devices on the aluminum laminator sheet, SUEX side up. Place the kapton+ADEX on top of those, SUEX side down. Note: it may stick to the topsheet as you try to lower the topsheet. In this case, position it on the topsheet so that when you lower the topsheet it seals on the pieces correctly.
- 8) Laminate using speed 1 and temperature 65 C actual.
- 9) Do a post lamination bake for 15 minutes at 65 C

At this point, the top layer of kapton is likely weakly adhered to the SUEX via the ADEX intermediary. I've tried the following things to improve adhesion with no useful effect (but small sample sizes):

- Use UV transparent acrylic (<http://astraproducts.com/info-acrylic-uv-transmission-filters.asp>) for the substrate, and flood expose the ADEX

through the baseplate, to get it to crosslink. This really requires a post exposure bake, and typically my post exposure bakes lead to wrinkles in the kapton layer, likely due to gas evolution during the bake as moisture is driven out of the system. It maybe possible to avoid this with the right bake parameters (vacuum oven?).

- Doing a hard bake of the ADEX and SUEX at 95 C for several hours. Lead to the same crinkling problems as above.
- Using the hot press to provide heat and pressure (~200 psi, 65 C and 95 C). This actually improved adhesion in 1 of 2 test samples but also lead to collapse of the mixer channels.
- Not doing a post lamination bake after initial lamination of ADEX+kapton. Didn't seem to have a noticeable effect.

The most successful procedure was using two sheets of aluminum as a sandwich, placing ~4 pieces between them on the hotplate in the G line chem lab. That was set at 150 C (not sure what sample temperature was, but obviously less given when acrylic gets sticky), and the two small heavymet blocks were placed on top to act as weight. Over about 10 pieces this gave 2 or 3 that seemed very robust (one of which was used in January 2017 and one in November 2017). They would still delaminate with pressure greater than ~1000 mbar on all channels. Unfortunately, this was a process done late at night in desperation, so I don't have a good record of the conditions. It might be repeatable with consistency on the SB8e substrate bonder.

2.8.2 Chloroform bonding

As an alternative to the ADEX, I have successfully used chloroform to adhere an acrylic top piece with a kapton window to the SUEX. The acrylic top piece is cut from 0.5 mm acrylic, using the appropriate CorelDraw CAD file. Chloroform is available in the Class 2 room flammable's cabinet (make sure they have the appropriate waste bottle available!)

To bond:

- 1) Cut a small piece of kapton for the window, as you did in section 2.3.
- 2) Check the alignment of your cover piece with you device. If the device was cut out with a misalignment, you will need to know roughly what the misalignment was.
- 3) Align the kapton window on the SUEX piece in the hood (place all of it on a betawipe) using tweezers. Often you can get the alignment done out of the hood, and then carefully transfer the SUEX device plus kapton into the hood. It is much easier to work outside the hood, but success can depend on how dry the air is (and thus how much static there is).
- 4) Using a dropper, cover the surface of a cover sheet with a layer of chloroform.
- 5) Wait ~5 s, then place the chloroform wetted surface on the SUEX (this step could probably be optimized). While there is still a little chloroform on the device you may be able to adjust the alignment of the cover sheet with the

device. Note that the kapton usually moves with the coversheet at this point, so make sure it covers the whole window.

- 6) Apply gentle pressure for ~30 to 60 s until the piece is bonded and chloroform evaporated.

This method has several problems. If you don't wait long enough after applying the chloroform, the surface isn't sticky enough. If you wait too long, it's too sticky and acrylic can block the channels (usually the inlet channels). It also doesn't seal the kapton to the SUEX, so there is an area where there can be modest liquid leaks from the mixer channels under the kapton. In a well-sealed device these leaks are usually quite small.

The advantage of this bonding is that it is quite robust, devices can withstand up to 8000 mbarr on each channel, the maximum the elveflow equipment can produce. This was the primary method used in the November 2017 beamtime.

2.8.3 SUEX as a Sealing layer

One approach I haven't explored significantly is using SUEX as a sealing layer. The idea here is that instead of having an intermediate adhesive layer, as described in section 2.8.1, you place a small piece of kapton across the observation region, and then laminate a thick sheet of SUEX (200 um or more?) across the whole device. Using standard photolithography methods (requires a new mask) remove only the SUEX above the kapton window that you just laminated. In theory you could do this quite precisely, with alignment marks both on the mixing layer mask and the sealing layer mask (http://www.cnfusers.cornell.edu/cnf5_alignmentKey.html).

This technique is challenging mostly because it's very hard to get the kapton properly aligned. Usually it sticks to one of the pieces of SUEX in a way that makes it very hard to laminate. Also, because you have the kapton between, you can't use the conventional lamination technique with a sheet between the two layers, so the lamination quality may not be as good.

Regardless, I tried this, and was able to do the masking (with a piece of the red tape for temporary masking), and second development. It more or less worked, but my kapton alignment wasn't right, and I ended up with an exposed section of the observation channel. This may be worth pursuing further.

2.8.4 Kapton Tape as a Sealing layer

Another approach explored by summer SUNRISE student Topher Flynn is the use of Kapton tape as a sealing layer. This method involves laying a piece of Kapton tape flat (adhesive side UP) on a flat surface. Then gently place the chip SUEX side DOWN on the tape. Press on the chip with a small amount of pressure. Flip the chip over and, using the tape roll itself, apply generous pressure and roll over the top of the chip to secure

the tape in place. Then, using a razor blade, cup around the perimeter of the chip to remove excess tape.

This method produced good results during the pressure testing phase, as the layer remained secure at even the highest of applied pressures of the system (8 bar). No leaks in the chip at either the inlets or mixing channel were observed. There are 2 observed issues with this method. The first is that one of the tested Kapton tape chips had a clog in the mixing channel. It is not certain if this clog comes from the channels themselves being underdeveloped or underexposed, or if the tape adhesive got into the channel itself, clogging it. This brings up the second issue which is that of the adhesive itself. Over the x-ray window, the silicone-based adhesive on the tape will interfere with the x-rays.

The next step in this process will be to investigate and test methods of either selectively applying or selectively removing the adhesive from the Kapton itself. This could be done with some sort of mask to cover the region of adhesive that needs to stay and then removing the rest of the adhesive using some sort of solvent. There also may exist some sort of light sensitive adhesive that only polymerizes when exposed to UV light. That way, a process similar to photolithography could achieve the needed results.

3 TR-SAXS at the beamline

Currently, setting up and running TR-SAXS at the beamline is not trivial. This section provides details on the set up and software controls.

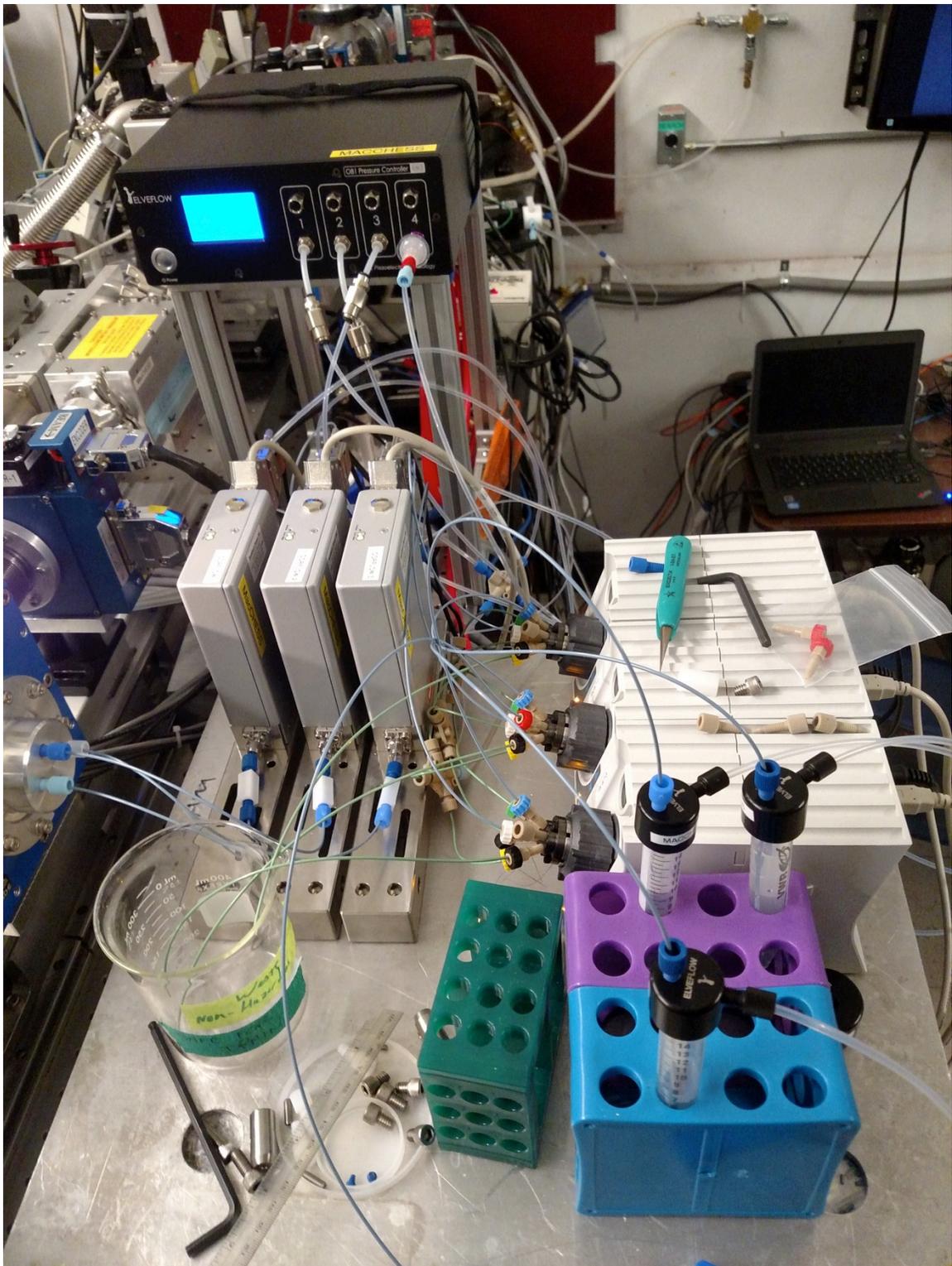


Figure 9 - TR-SAXS set up at the beamline in November 2017. Note that in this image port 5 on the sample valves are not hooked up in a normal configuration.

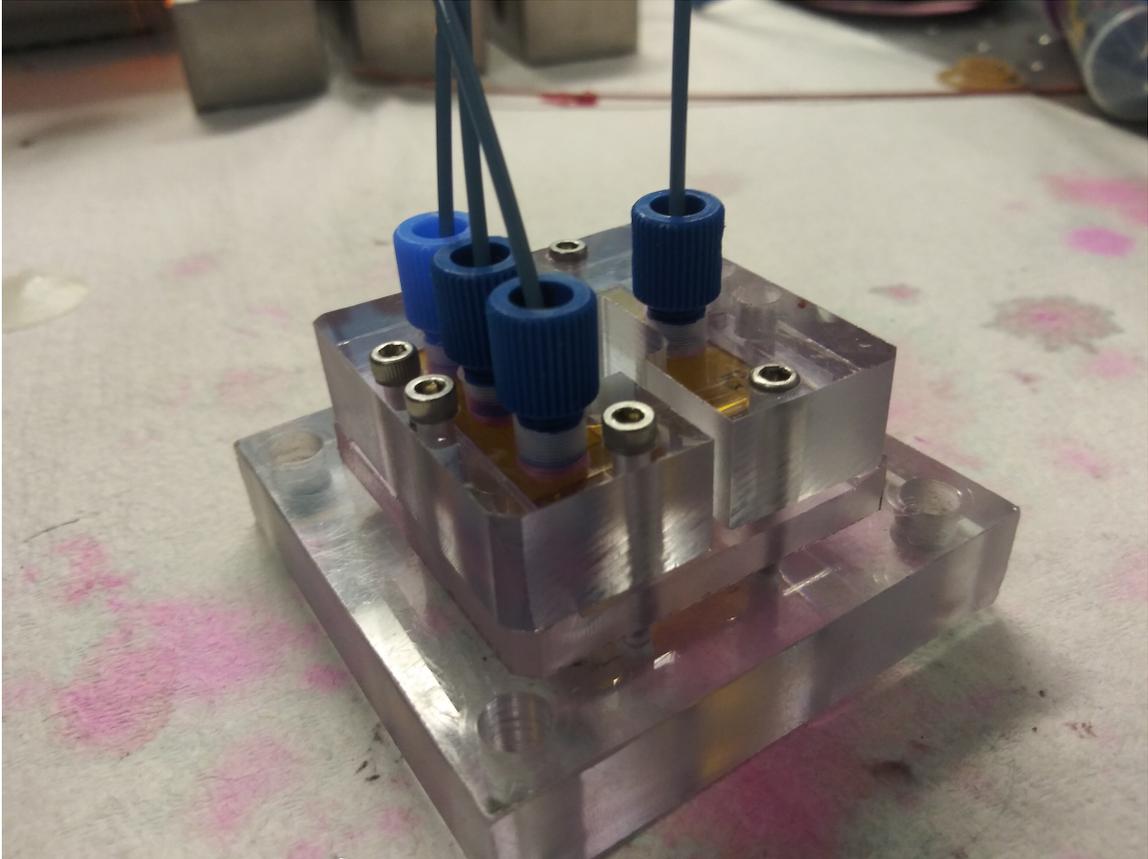


Figure 10. Chip holder and fluidic connections ready for testing.

3.1 Hardware overview

The time resolved system is comprised of the following pieces of hardware:

- Elveflow OB1 for pressure driven flow
- 4x Evleflow corflow flow sensors for measuring flow
- 3x IDEX 6/7 multiple input selector valves
- 3x IDEX 2 um or 0.5 um inline filters
- Elveflow sample and buffer reservoirs
- Mixing chip and holder, and appropriate interconnects

The mixer consists of three input channels and one outlet channel. Each input channel should have the follow items, connected in order:

- 1) Selector valve for sample, buffer, cleaning inputs
- 2) Inline filter
- 3) Flow sensor
- 4) Input into mixer chip

The outlet channel should also have a flow meter on it, this is very useful for diagnosing leaks.

The IDEX selector valves have six inputs. I have used the following consistent hook ups for each valve. On the designated channel:

- 1) Sample (if any, or blank)
- 2) Buffer
- 3) Water
- 4) IPA
- 5) Air
- 6) Blank

Note that the way the mixer is designed, all of the input channels take buffer, but only the center channel (channel 2) takes protein.

3.2 Hardware set up

3.2.1 Fluidic connections

The set up from the G-line chem lab should be able to be translated more or less directly to the beamline. It is useful to use the robot table for extra space to set up the equipment. When reconnecting all of the fluidic connections, make sure that valve 1 is connected to flow meter 1, and that flow meter 1 is connected to inlet 1 in the mixer (by convention I think this was the 'bottom' inlet in our last experiment, but it doesn't really matter), and so on for channels 2 and 3. The selector valve outlet is the center port, and the flow meters have a direction of flow indicated on them.

Each selector valve should be hooked up to the inputs given above. All selector valves use the same water, IPA, and air input, using the cross junctions in the setup to split the flow from the appropriate supplies. The water and IPA should be in the 100 mL glass bottles with the appropriate Elveflow caps. **Note: these can only take 1 bar of pressure!**

Buffer and sample for each line is controlled independently, so each selector valve should be hooked up to separate buffer and sample reservoirs.

Typically I use the 250 μm (blue) PEEK tubing for all fluidic paths except the water, IPA, and air inputs into the selector valves. If extra flow resistance is needed in the system, we have some smaller (100 μm , black) tubing that can be used. This might be needed when you want to run at very low flow rates on a channel, and need to move the control pressure for those low flow rates up to somewhere the pressure controller is more stable.

Note: It is important to have the in-line filters after the selector valves but before the flow meters.

3.2.2 Pressure connections

The OB1 should be hooked up to pure Nitrogen from a gas bottle, at a pressure of ~ 9 bar (maximum pressure for the system is 10 bar). This should always go through

the in-line air filter to make sure it is as clean as possible. **Note: I believe one of the filters has a small leak, so be sure to turn off the nitrogen at the gas bottle when you aren't using it.**

Channels 1-3 on the Elveflow are 0-8 bar channels, and should be hooked up to the buffer and sample reservoirs for fluidic paths 1-3, respectively. Note that for channels 1 and 3, there is usually only a buffer input, so the pressure supply is hooked up to just one reservoir. Channel 2 uses a Y junction to send the pressure to a sample and a buffer reservoir.

Channel 4 on the Elveflow is a -1-1 bar channel (-1-0 only available if a vacuum pump is hooked up). It should always have a backflow regulator on it, so that liquid cannot get into the electronics. This should be hooked up to the water, IPA, and air supplies. To do so, it uses a cross junction to split the pressure to the water reservoir, the IPA reservoir, and directly to the cross junction that provides air input to each of the selector valves.

Note: It is important to have the OB1 elevated above the fluidic reservoirs to help prevent backflow.

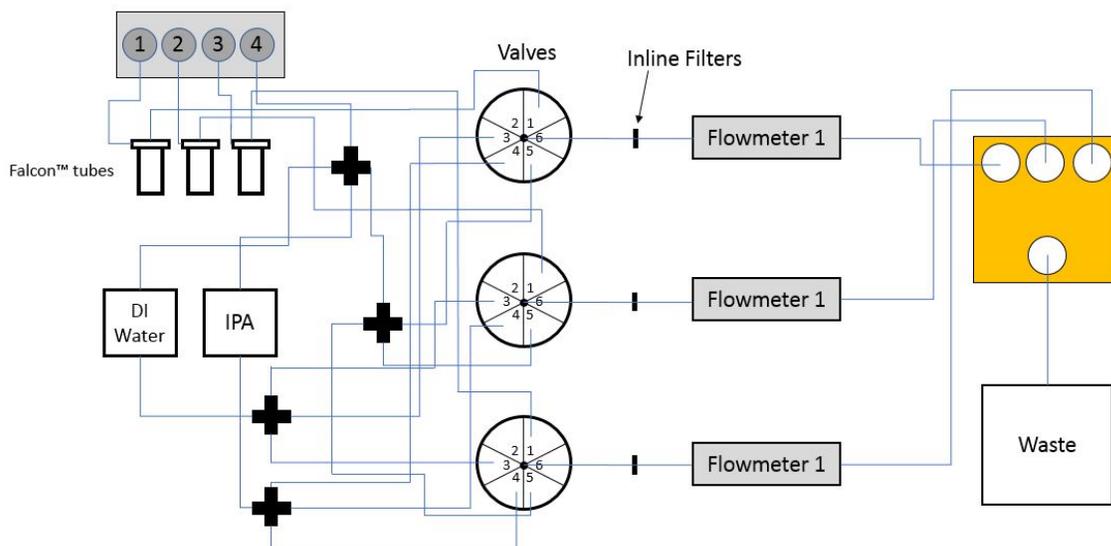


Figure 11. Schematic diagram of fluidic and pressure experimental setup.

3.3 Software setup

The Elveflow software is currently installed on the Wyatt control computer. The latest version can also be downloaded from the Elveflow website. Open it using the ESI shortcut on the desktop.

3.3.1 Check naming

I've found that when the setup is moved (devices powered down and unplugged from the computer), they can sometimes show up in the software with the wrong

name. To check this, you can do two things. One approach is to unplug all of the devices of a given type but one (for example, unplug all of the flow meters except Corflow 1). Check that the name in the Elveflow control panel corresponds to the label on the hardware. Alternatively, and this works best for valves, just open the hardware control panel in the elveflow software and change a parameter, such as the selected port for a valve, then verify that the appropriate valve changes ports.

If the naming convention is wrong in the elveflow software, click on the configuration for the device:



Then go to the General panel, and change the name.

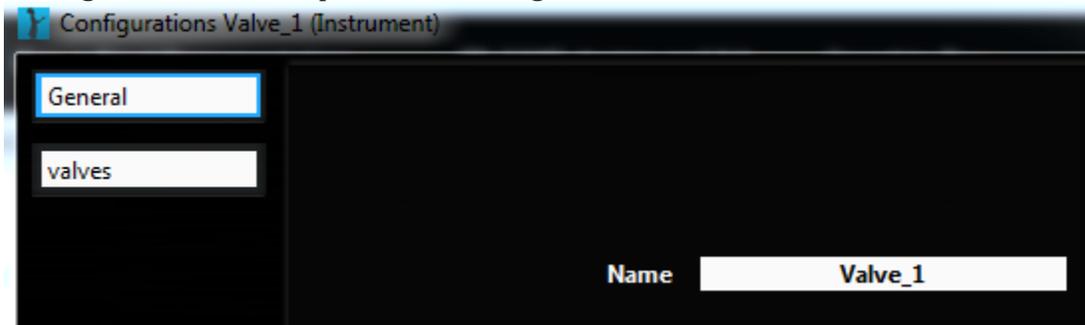


Figure 12 - Changing the name of an instrument in the elveflow software

Note: two devices cannot have the same name, so if, for example, the names of Valve 1 and Valve 2 were switched, you would need to rename Valve 1 to, for example, Valve 10, then rename Valve 2 to Valve 1, then rename the (now named) Valve 10 to Valve 2.

If a device completely fails to show up in the Elveflow software, double check the connection cable and power cycle the device (check the device manager to see how many devices are connected, and if one disconnects when you disconnect or power cycle the device). The flow meters are particularly sensitive to connection issues because the serial to usb converters can fail. If none of these are at fault, you need to use the 'Add Instrument' (or 'Add Sensor') function in the Elveflow software. Click on 'Add Instrument' and then select the device type. If it's connected and not already in the software it should automatically detect the device. If it doesn't, you'll see an error message. Give the device an appropriate name, and click okay.

3.3.2 Recalibrate the OB1

It is not strictly necessary to recalibrate the OB1 when you move it. However, I've found it useful to do so, both to verify that it is in good working order, and that the channels are set correctly. If you notice that the pressure values are significantly different from 0 when you have the set point at 0, or you have trouble stabilizing at a certain pressure, you need to recalibrate. I've found this to be particularly

important if you switch the OB1 to a new computer or installation of the Elveflow software.

To recalibrate, plug all of the output ports on the OB1, and make sure you have ~9 bar of pressure going into the device. Plugs for all the ports are currently stored in a 50 mL tube on the small optics table in the g-line chem room, labeled 'important fittings/plugs'.

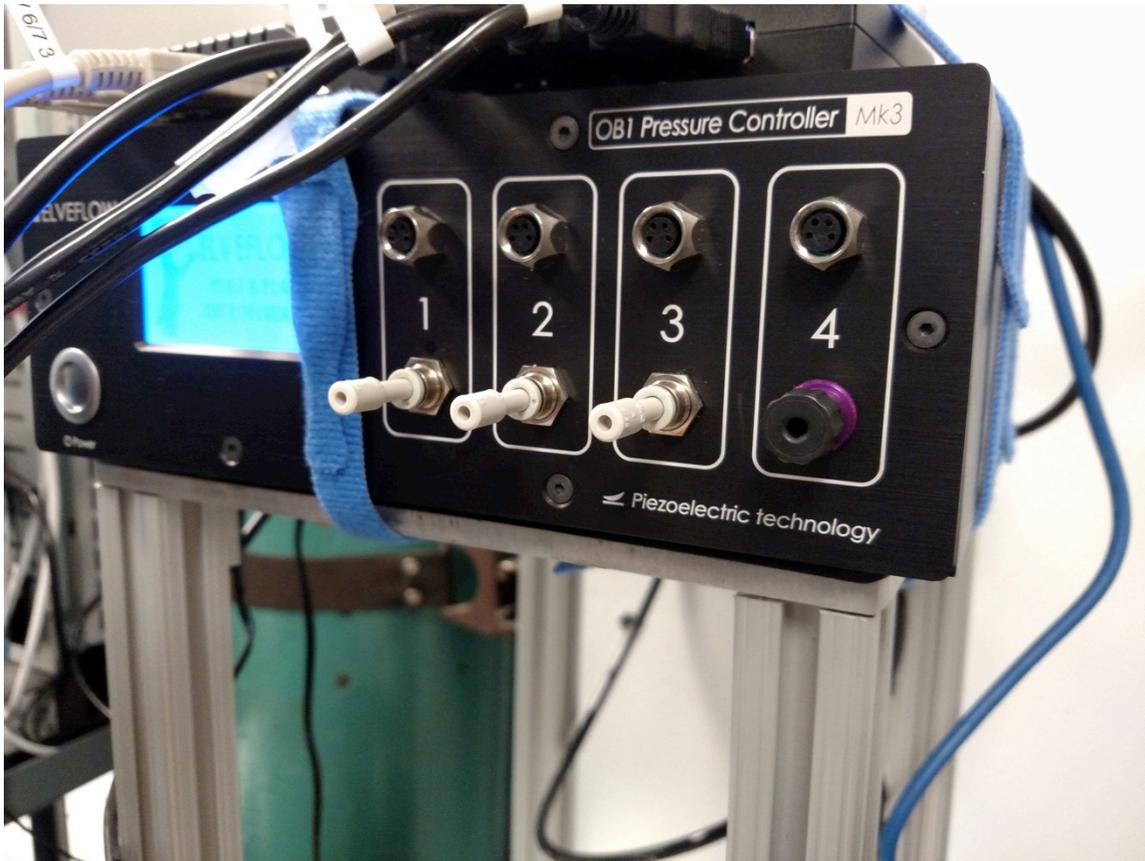


Figure 13 - Elveflow with plugs inserted for each of the four channels.

In the Elveflow software, open the OB1 device configuration, and click on the 'Calibrate' tab, then on the 'Start Calibration' button.

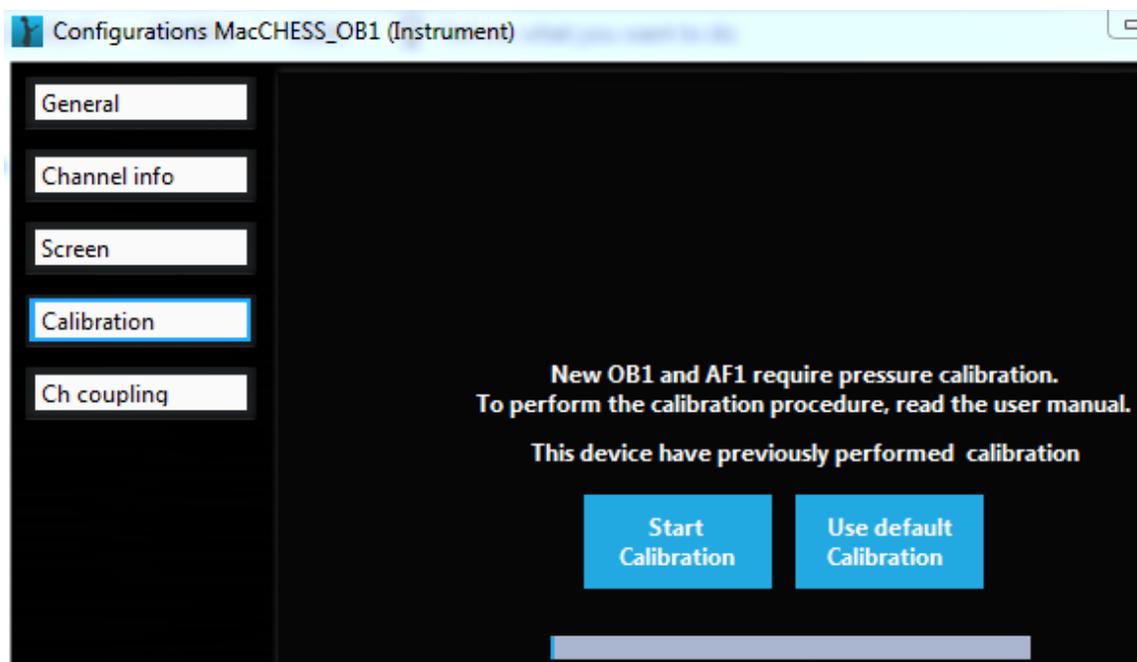


Figure 14 - Calibrating the OB1

3.3.3 Adding sensors to the OB1 control.

The OB1 control can display (and potentially feed back on) the output from one flow sensor per channel. To add these sensors, click on the configuration button for the sensor in the sensor panel (bottom of the 2 panels in the main ESI window). Then go to the 'Connected To' panel and make the appropriate changes. Note that to add a Corflow flow meter, do not use the “ADD SENSOR” button. Use the “ADD INSTRUMENT” button and select the BFS instrument. Name it “Corflow_1” (or whatever corflow meter you are connecting) and you will see that a sensor with the same name is automatically added under the sensor list. Open the configuration menu for these sensors to make sure they are connected to the correct channels.

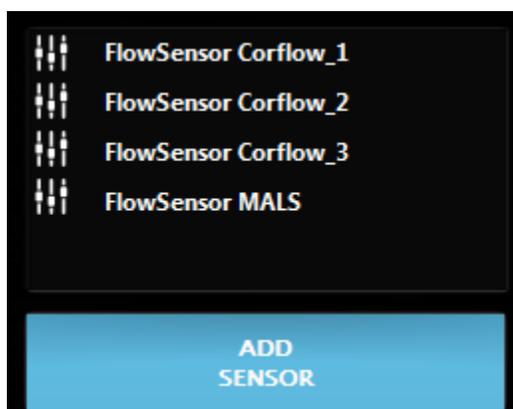


Figure 15 - Elveflow sensor panel

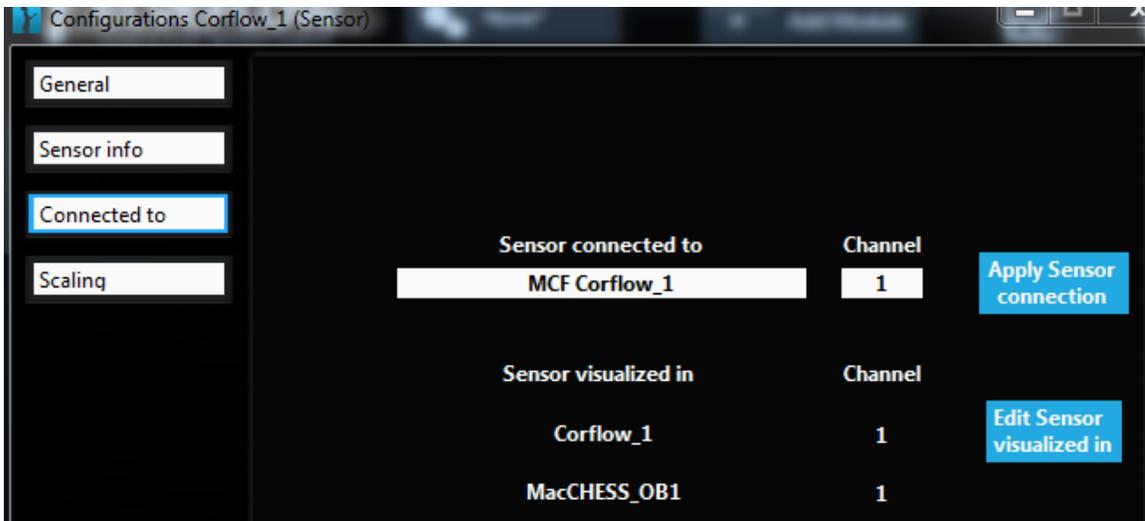


Figure 10 - Elveflow sensor connection to the OB1

3.3.4 Checking Bronkhurst flow meter mode

The Bronkhurst corflow flow meters can run in two modes, continuous and punctual. The continuous mode continuously measures fluid density in addition to the usual measurements. The density is required to get the flow rate. Punctual doesn't measure density. The punctual mode is faster, but has to be recalibrated whenever you change fluids. For our purposes, the continuous mode is more than fast enough. Thus, you want the sensors always set to continuous mode otherwise you might change fluid density and not get a correct flow rate.

To check/change this, In the top section of the main panel, click on the configure button for a BFS Corflow instrument. In that window, select the BFS Config panel, and make sure that continuous mode is selected.

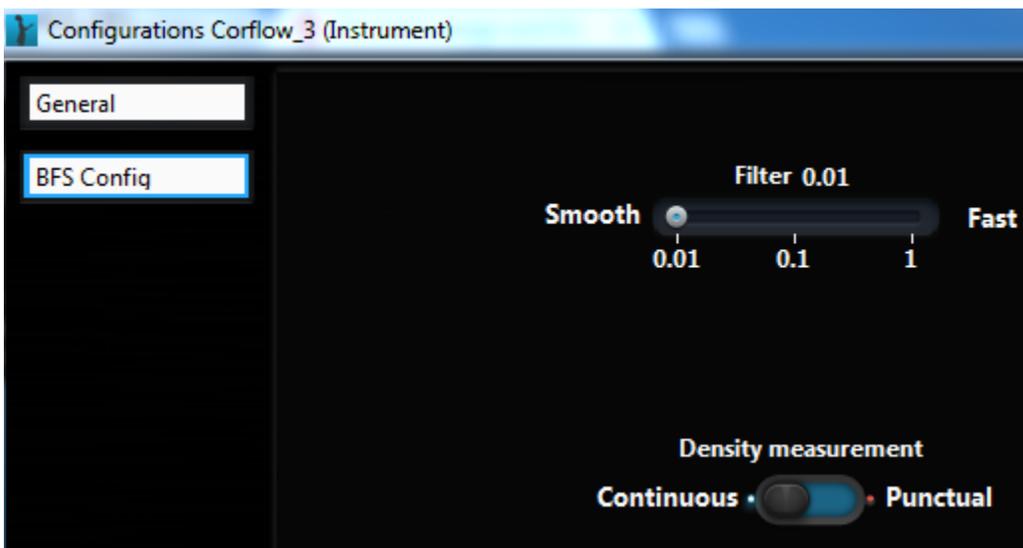


Figure 17 - Elveflow BFS configuration to select continuous density measurement.

3.4 Running the system

The Elveflow software is quite point and click. Click on the play button



to open an instrument control. In the OB1 control panel set appropriate pressures, or run in flow control mode (I haven't used this, but the Pollack group has and it seems to work for them) and set the appropriate flow rates. Use the selector valves to switch appropriate inputs. **Note: IPA can cause the SUEx bonded chips to delaminate. While important for cleaning the flow meters, only use it with a dummy (all acrylic) chip in the flow path.**



Figure 18 - The Elveflow software interface to the OB1 pressure controller.

Use the sample camera and stage motors to scan in the mixer channel, and index the position of the beam relative to the mixer. It is important to know how far along the channel you are to achieve different time points. This part hasn't been perfected yet. Also note that the software somewhere gets confused and mixes up channels (sensors you have connected to channel 2 may show up in channel 3, for example). I found that this can sometimes be fixed by restarting the software, power cycling the OB1, or disconnecting all of the sensors, then one-by-one connecting and routing them.

Also note that noise coming from the OB1 during testing is expected, especially when increasing, decreasing, or turning on/off the pressure in the channels.

4 Appendix – Streamlines in COMSOL

I tested the effect of number of streamlines (10, 100, 1000, 10000) and streamline quality (corase, normal, fine, finer) on the calculated mixing time of the simulated mixers. After calculating the streamlines, the python script previously described was used to calculate the time it took for each streamline to reach $c > 0.4$ (or any other threshold). Note that $c = 0.5$ represents fully mixed. The median time for every streamline was then calculated, as were the 25% and 75% times (times which 25% and 75% of all mixing times are less than). These were used as the 'average' mixing time and the 'error' in the mixing time.

The effect of streamline number on the mixing times seemed to stabilize at 1000 streamlines (no noticable difference between 1000 and 10000 streamlines). A small effect was seen between 100 and 1000 streamlines. In general, I recommend using 1000 streamlines to calculate mixing times.

The plot below shows mixing times for different streamline numbers. There was no significant change in the error bars either, but those plots are not shown as they are quite hard to read.

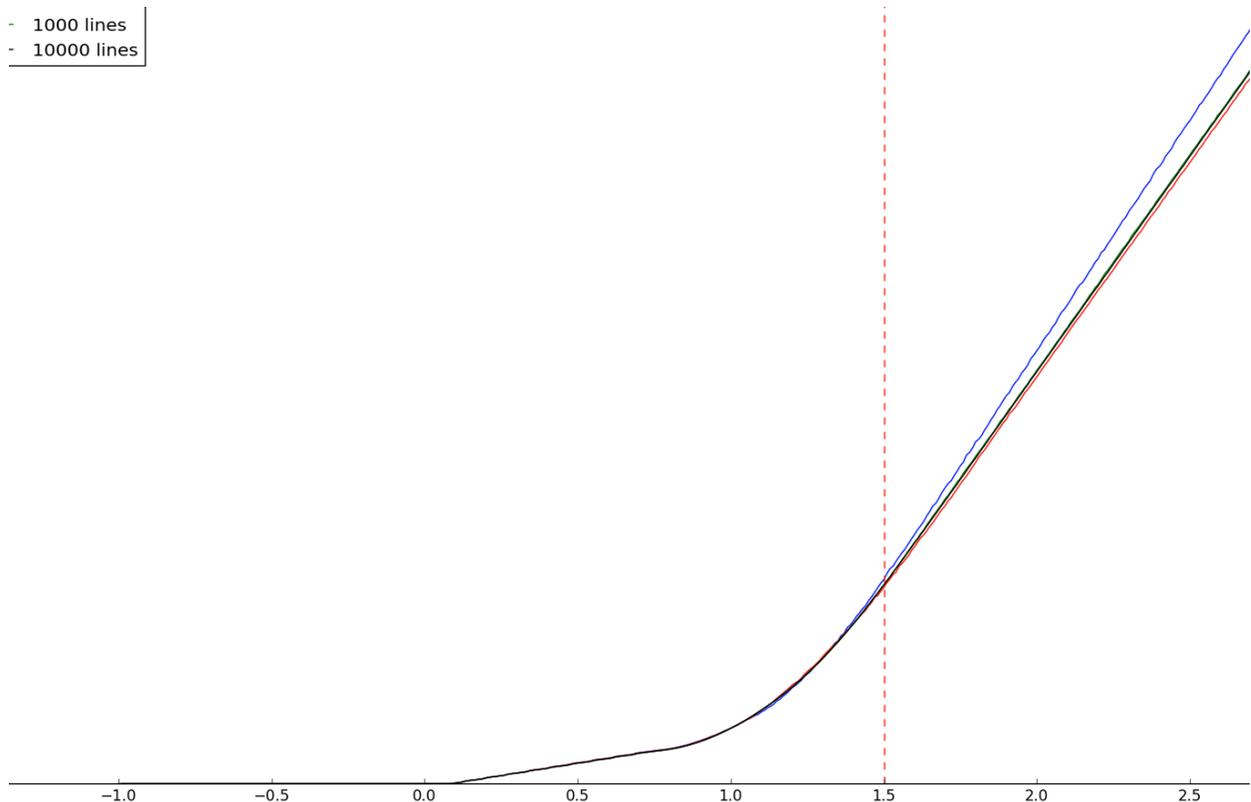


Figure 11 - Effect of number of streamlines on calculated mixing time.

The effect of the quality of refinement in COMSOL on the calculated mixing time was also measured. The same python scripts as above were used to analyze the data. The

end result was that no level of refinement beyond coarse had any significant effect on the calculated mixing times.

The plot below shows the calculated mixing times.

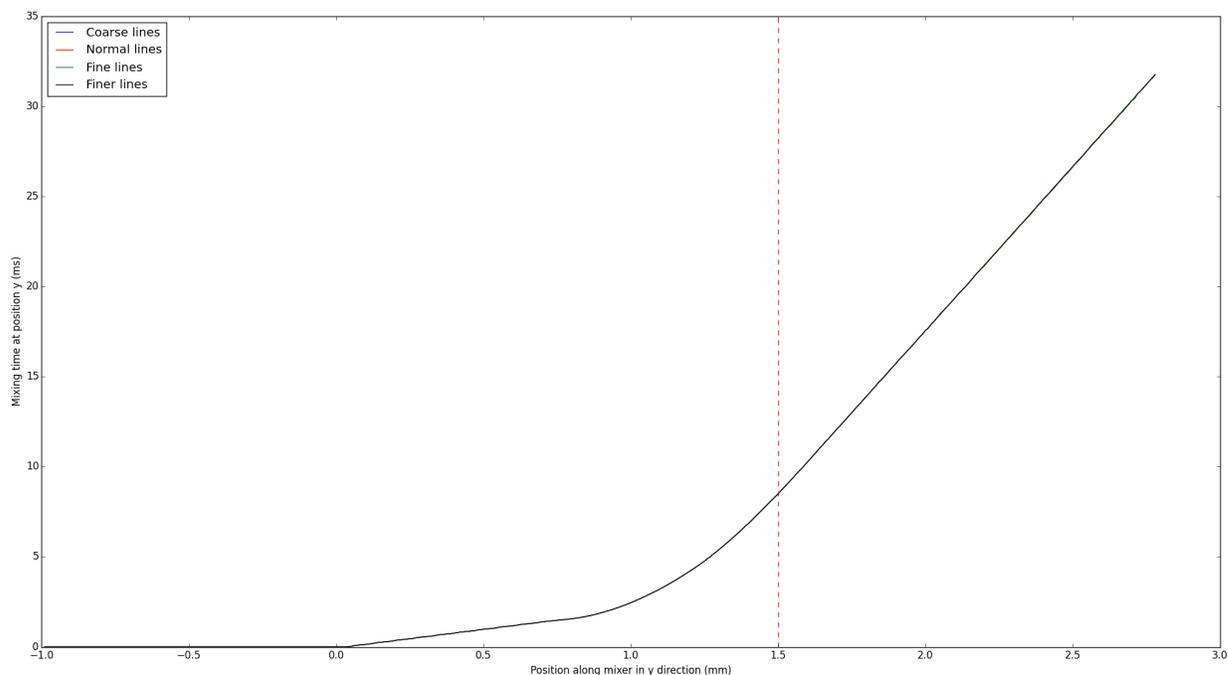


Figure 12 - Effect of quality of streamlines on calculated mixing time.

5 Appendix – APTMS/GPTMS bonding

One bonding procedure I've spent a reasonable amount of time investigating but had not luck with is using APTMS/GPTMS to chemically bond kapton and SUEX. The idea is fairly simple, you functionalize APTMS on the katpon and GPTMS on the SUEX (or vice versa), then press them together and a chemical crosslink happens between the APTMS and GPTMS which bonds the substrates quite strongly. There is some literature suggesting this type of bond is possible and quite strong (Tang & Lee, 2010; Cortese et al., 2011; Hamdi et al., 2014; Ren et al., 2015).

The general procedure goes like this:

- 1) Activate the surfaces with an oxygen plasma
- 2) Functionalize the surfaces with APTMS/GPTMS
- 3) Stick them together with a bit of pressure and heat
- 4) Wait a bit

Unfortunately, all of the papers above use liquid chemistry to functionalize the surface, whereas CNF is very reluctant to let me do that. Lacking a way to activate

the surfaces in our own lab, we're stuck using vapor deposition to functionalize the surface. The MVD100 has APTMS (standard) and can have GPTMS (upon request to the tool manager).

Using the MVD100 is straightforward. My usual procedure is:

- 1) Prime chamber by running a 5 minute oxygen clean and 1 minute deposition of APTMS/GPTMS with an empty chamber
- 2) Insert sample and use 60-120s oxygen plasma to activate the surface followed by functionalization. For the APTMS I use the standard recipe. For the GPTMS I use a pressure of 1.5 instead of 1.2

I will do the APTMS and GPTMS back to back, then take the samples directly to the SB8e substrate bonder. This bonder allows piece bonding with carefully controlled force, temperature, and chamber pressure. In the SB8e, use a silicon wafer on top and bottom to make a stack, and then have your kapton and SUEX piece in the middle of this stack. This seems to require loading the bonder for pieces, I haven't tired using the full wafer loading (which would be much faster), that might be something to talk to the tool manager about.

I have yet to get a successful bond from this procedure. I have verified that some surface functionalization is happening, as the contact angle on the substrates changes dramatically after functionalization. I have also been able to get one strongly bonded substrate, but it was using a very large force, which collapsed the mixer channels. I have demonstrated good bonding between two bare silicon wafers using this procedure, so bonding is possible.

My supposition, in discussion with Tom Pennell (CNF staff who's been helping me with this) is that the interface between the SUEX and kapton isn't flat enough, so we're not making the contact needed for functionalization. It's possible that smoothing out the surface might help. I have made some very initial attempts including:

- 1) Wet sanding down the acrylic baseplate using fine grit sandpapers (1000, 1500, 2000, 2500), to remove burrs from laser cutting
- 2) Using chloroform bonding to apply kapton across the entire acrylic piece, to remove distortions from epoxy and finite kapton edges. Note that in this case you then have to cut out the kapton from the mixer inlets/outlets.
- 3) Smoothly laying down the kapton on a silicon wafer substrate before functionalizing. This is rather involved:
 - a. Spray a pool of IPA covering the silicon wafer,
 - b. Gently lay the kapton down so it floats on the IPA
 - c. Smooth out the kapton so it is flat on the wafer using your glove or a plastic spatula or tweezers (metal will scratch the kapton)
 - d. Carefully blow dry the IPA off of the kapton and wafer by keeping the air nozzle facing directly down onto the wafer and above the kapton at all times.
 - e. Smooth out the kapton again, to eliminate any bumps.

- f. Put the kapton in a 65 C oven. Remove after several minutes, lift up one part and peel back until half the kapton is off the wafer to release IPA trapped under the kapton. Gently smooth that half back onto the wafer. Repeat with the second half.
- g. Place back in the 65 C oven. Remove occasionally to smooth out the kapton.
- h. Remove from the oven and let cool to room temperature. Smooth out the kapton again, and carefully tape down the corners. Note: if you don't tape it down it may get sucked in by the MVD vacuum pump!

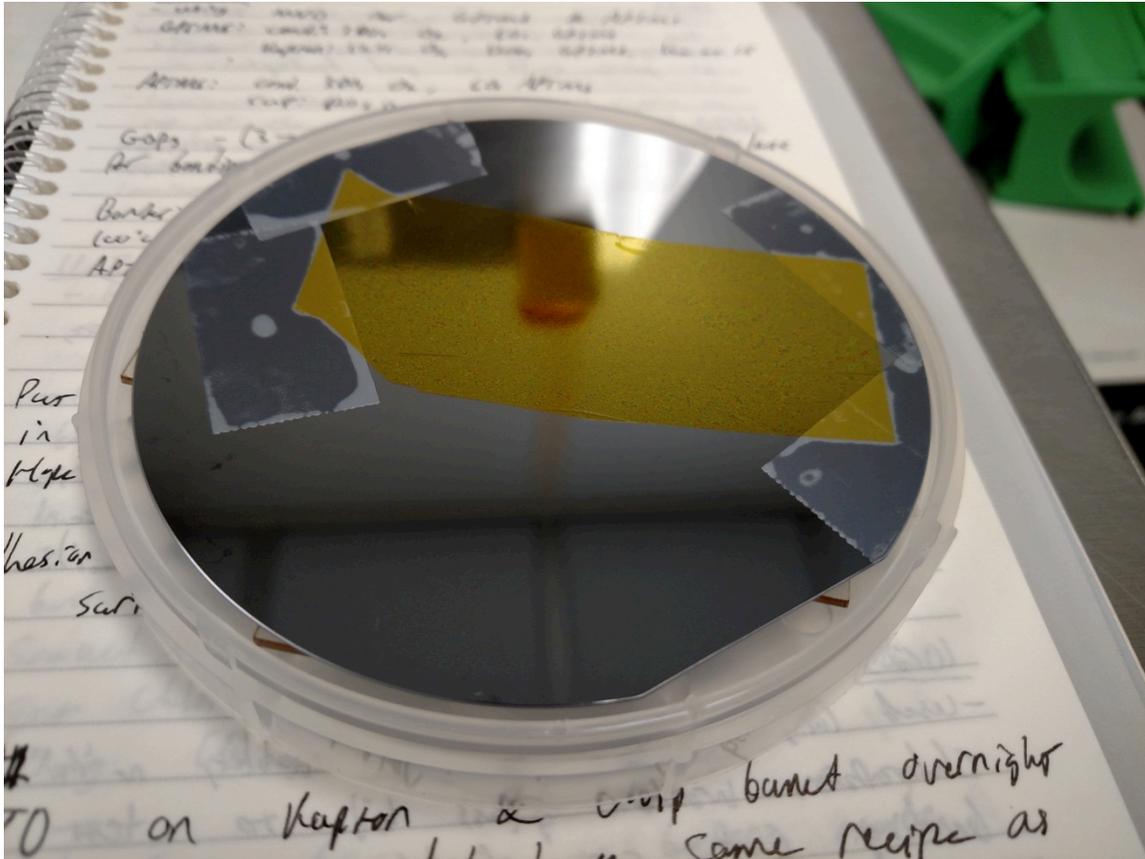


Figure 13 - Kapton flat and taped to a wafer, ready for the MVD.

I have found that the maximum force a single chip change take in the SB8e is ~50 N before the mixer channels start to collapse. This is best achieved by setting the SB8e to ~200 mbarr (gives ~150 N of force), and having three chips in your wafer stack.

6 Appendix – Previous Fabrication

My first attempts to fabricate the mixer used PDMS. The hope was that I could fabricate the mixer out of PDMS and manage to get channels all the way through the

PDMS, which I could then bond with windows (thin glass, perhaps, using standard oxygen plasma treatment). This didn't work. I did the following:

- Made a master in silicon using the Unaxis deep etcher (that was the actual device I wanted)
- Coated the master with FOTS
- Cast PDMS on the silicon master to get a PDMS secondary master (inverse of the actual device)
- Coat PDMS secondary master with FOTS
- Cast PDMS on the PDMS secondary master to get a PDMS device

Using the secondary master has several advantages: It preserves the primary master longer (which is expensive to make) and it allows the primary master to have channels rather than ridges (which could be quite fragile in the aspect ratios used).

In the secondary casting I tried two ways of obtaining the right thickness of PDMS to have through channels, including:

- Careful calibration of amount of PDMS used
- Using a top piece (bare silicon wafer coated with FOTS) to press against the secondary master during curing.

Neither of these worked particularly well, and because the device is so thin (~500 um), the PDMS lacked stability, even if there were channels through it. I decided that this approach, though it let me test the mixer idea in the lab initially, was not the right approach.

7 Appendix – Future Fabrication

I have several ideas for other possible ways to fabricate devices that I haven't had time to test yet. A brief overview is provided here, in the hopes that it may be useful to others in the future.

7.1 Deep etching in silicon

Overview: Deep etch devices directly into silicon, then bond windows to the silicon wafer, for example anodic bonding for glass. Use aluminum to mask the Si wafer for deep etching.

Advantages: Deep etching is reliable, easy to do. Anodic bonding is well established.

Possible pitfalls: Deep etching is expensive, each device likely costs >\$500 in fab alone. Can you anodically bond thin enough glass for x-ray windows without it cracking?

7.2 Use hot embossing

Overview: Several groups have reported making microfluidic x-ray devices out of COC, most recently this (Denz et al., 2017), but also various devices from, among others, Sarah Perry and her collaborators (Dhouib et al., 2009; Khvostichenko et al., 2013).

Advantages: Reportedly COC can be sealed against itself pretty easily, and against other materials relatively easily as well see the previous citations and: (Cortese et al., 2011; Azouz et al., 2014). It's cheap. Hot embossing is also a cheap process.

Possible pitfalls: I measured COC as a window material and it had extremely variable scattering, mostly due to ridging in the materials I have. If the ridging is a persistent feature of COC that is hard to remove (Perry's group, for example, claims they can remove it by pressing it in the hot press between two flat surfaces like glass slides, I had mixed luck with that), it is not going to be a very suitable SAXS window. Additionally, Steve Meisburger has tried hot embossing, and says that because the device shrinks as it cools, it can be hard to get consistent feature sizes and structures.

7.3 Moving from kapton to another window material, like glass

Overview: Kapton in general is quite chemically resistant, and can be hard to bond to. It may be possible to bond to glass with other methods and use that for windows.

Advantages: Relatively rigid, may have better scattering properties, allows use of different chemistry for bonding, may be flatter (better for bonding). Many of the papers linked the APTMS/GPTMS section also describe processes for bonding with glass.

Possible pitfalls: Thin glass is fragile and challenging to work with. It might not survive processes like lamination, and may require lower pressures for substrate bonding. It's also expensive.

8 References

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