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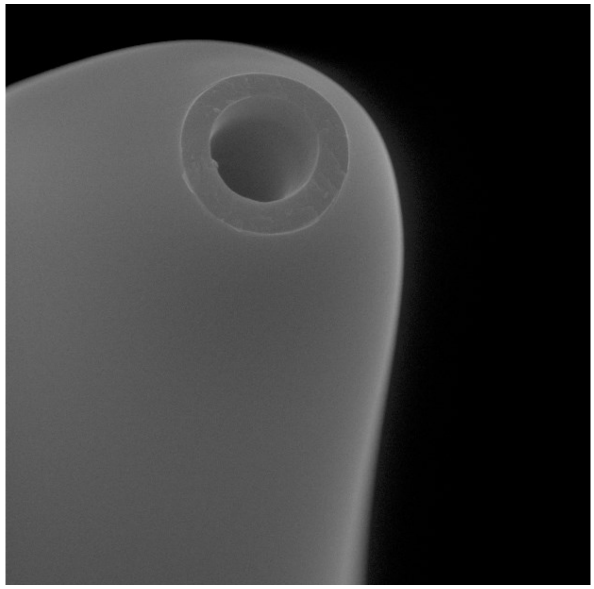

Fabricating Nano Electrospray Ionization Emitters from Fused Silica Capillary Tubing on the P-2000/F Micropipette Puller

Until recently, many investigators relied on New Objective as a commercial source for prefabricated nano electrospray ionization (nanoESI) emitters. In 2020, New Objective stopped producing their entire line of nanoESI emitters, leaving investigators without a source for these essential products. Many investigators have approached Sutter Instrument hoping to use the P-2000/F laser-based micropipette puller to produce nanoESI emitters. The SilicaTip™ emitters previously produced and sold by New Objective were fabricated in a multistep process using a P-2000/F. First, a piece of fused silica capillary tubing would be pulled using the P-2000/F. Next, the tip would be etched back using concentrated hydrofluoric acid to produce a wide range of emitters with essentially arbitrary starting and final inner diameters. While it is a critical component of this process, the P-2000/F alone cannot produce the full range of emitter geometries previously offered through New Objective. Physical factors intrinsic to the puller and the tubing preclude fabricating emitters with aperture diameters larger than approximately one-tenth the inner diameter of the starting material. That is, the largest aperture one could reasonably expect when fabricating an emitter from 360 µm OD x 75 µm ID tubing has a diameter of about 7.5 µm. In many cases, emitters which are fabricated within this limitation are suitable for experiments. However, this process is not trivial. To pull an emitter with the largest possible tip the material will support, careful programming is necessary. The purpose of this tech note is to discuss programming the P-2000/F to generate nanoESI emitters with large apertures.

The critical consideration when attempting to fabricate large diameter emitters on the P-2000/F is the number cycles a program must execute to separate the starting material. Program cycles take 2 forms:

1. Loops of a program
2. Individual lines of a multiple-line program

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Generally, the more cycles a program executes to separate the glass, the larger the tip will be and the shorter the taper. A corollary of this is that to produce a large tip, one should separate the tubing in as many cycles as possible. However, there are limitations to the maximum number of cycles to consider:

1. The stability of a program decreases as the number of cycles increases. A program with more than 6 cycles will not reliably produce the same, or even similar, emitters from one pull to the next.
2. The fused silica material will only support a tip of a certain diameter before it begins to fracture about its circumference. This size is about one-tenth the starting ID.
3. As the number of cycles increases, so does the likelihood that the pull will fail altogether. As the tubing is drawn down further, there is a chance that it will fall out of the laser beam. At this point, the program will stall as the glass fails to separate. After 50 seconds of inaction, the program will bail out to prevent damage to the laser

Taking the above into consideration along with experiments with various starting materials carried out at Sutter Instrument company, we have developed the following suggested framework for pulling large tips without further modification:

First, run a RAMP test using filament 0 on your prepared fused silica starting material to determine the starting heat. Instructions for the ramp test can be found in the P-2000 operations manual.

Next, install the following one-line program:

Heat: RAMP   Fil: 0   Vel: 15   Del: 255   Pul: 0

Change the velocity by 1 unit in each direction and pull emitters to find the entire set of velocities for which this program loops 6 times. Ideally, this is at least a 3-unit wide range, but may be smaller because of the intrinsic instability of low velocity values. Once you have the range, pick the middle value (if there are only 2 values, use the higher value), and write the program out into 6 lines using that velocity. To increase the tip size to its practical limit, lower the heat and/or velocity on the 6th line. You can lower these values until the puller starts to loop back to the start of the program (i.e. when the pull completes you will see a message reading "the program lopped 2 times"). As an example, we developed the following program to produce an emitter with an aperture of ~7 µm from 360 x 75 µm fused silica tubing with a RAMP value of 230 units (pictured above):

Line 1:	**Heat: 230**	**Fil: 0**	**Vel: 13**	**Del: 255**	**Pul: 0**
Line 2:	**Heat: 230**	**Fil: 0**	**Vel: 13**	**Del: 255**	**Pul: 0**
Line 3:	**Heat: 230**	**Fil: 0**	**Vel: 13**	**Del: 255**	**Pul: 0**
Line 4:	**Heat: 230**	**Fil: 0**	**Vel: 13**	**Del: 255**	**Pul: 0**
Line 5:	**Heat: 230**	**Fil: 0**	**Vel: 13**	**Del: 255**	**Pul: 0**
Line 6:	**Heat: 200**	**Fil: 0**	**Vel: 10**	**Del: 255**	**Pul: 0**

Keep in mind that even this approach will have a less than perfect success rate for producing consistent emitters. What is being done here pushes the limits of both the puller and the tubing, so some variability should be expected.

Some modifications can be made to this framework to bias tip sizes in one direction or another. Changing the heat by 10 units on each line can bias the aperture to be slightly larger (lower heat) or slightly smaller (higher heat). Changing the velocity by 1 unit on the penultimate line will have the opposite effect, with higher velocities leading to larger apertures and lower velocities resulting in smaller apertures. Whenever you make a change to your program, you should always ensure that the tubing still separates on the sixth line.

Following the above framework, one ought to be able to fabricate a range of emitters depending on the starting material. However, if this is insufficient to produce the materials needed for your experiments, Sutter Instrument now offers the BV-10N. The BV-10N is a new configuration of Sutter Instrument's micropipette beveler designed to polish nanoESI emitters generated using the P2000/F laser-based micropipette puller. This instrument is discussed in a separate tech note: Use of the BV-10N for fabrication of nano Electro Spray Ionization Emitters. In brief, by drawing out emitters with long tapers one can then break back the tips and polish them with the BV-10N to allow access to a larger variety of tip geometries.