How to Make Small Drops with the FTÅ4000

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Note: This information applies to the first generation FTA4000. The second generation FTA4000 with NanoDispense technology automates much of the procedure.

1. Choose Syringe. If you are using the piezo pump, choose a 500 or 1000µl syringe in order to have sufficient volume to fill the piezo pump and the hub of the needle easily. Smaller syringes may be used when the piezo pump is removed and not used. If the piezo pump is to be used, it is in place before the syringe is filled.

2. Load Syringe. Fill the small syringe to be used by first filling a *second*, large syringe equipped with a long flexible needle. WPI MicroFil™ fused silica needles are supplied with the instrument. Filters are available to remove particles in the liquid Luer hub filters attach to the large syringe after it is loaded and before its needle is attached. The small syringe is oriented upwards and the long needle inserted down through the central bore of the piezo pump until the needle contacts the plunger of the small syringe. Fluid is *slowly* inserted into the small syringe from the large. The liquid level rises from the bottom towards the top of the small syringe. In this way, no air is trapped in the syringe. It is most important that no bubbles be present, or else it will be very difficult to control drops of small radius. It is normally best to fill the syringe only to its tip, and not try to fill the piezo pump also while the filling needle is present. Instead, remove the filling needle and second syringe, then, while still holding the small syringe in the same upright position, *slowly* push its plunger upwards so as to fill the piezo pump until liquid appears at the needle port. The syringe should always be held vertical and the filling operations performed slowly to prevent trapping air. The needle will be attached after the syringe is installed in the instrument.

3. Choose Needle. While counter-intuitive, it is often possible to make smaller drops with larger needles. This can not be carried to extremes, of course, but smaller needles develop much higher pressures during drop formation and these high pressures make drop control more difficult. The best application for the smallest needles is in expanding drop experiments where no distinct pendant drop is ever formed. Start with the FTÅ 36GA fused silica needle. This needle has an outside diameter (OD) of 90µm and an inside diameter (ID) of 20µm. The fused silica needles are more rugged than the drawn glass capillary needles which have smaller diameters. Check that the small glass capillary tips are not blocked by blowing air through them using a second, dry, syringe equipped with a particular filter.

4. Attach Needle. The needle can be attached with the syringe already in place. Move the sample stage and the pump stage so a clear path is available from below. The needles are subject to breakage and splitting because of their small size. While dirt can be blown off the outside with compressed air, small needles can not be wiped clean without damaging their tip. The needle Luer hubs screw into place. A tight seal is necessary.

5. Fill Needle. Run main syringe pump at 1µl/s to slowly fill Luer hub and main body of needle. Reduce rate to 0.05µl/s as liquid nears tip. Pumping rapidly will split tips.

6. Dispense Without Piezo Pump. Run syringe at 0.1 or 0.05µl/s until liquid appears at bottom of tip. Visibly check for any air bubbles in needle. Pendant drops can be formed by pumping slowly. Because pressure builds in the syringe, liquid may continue to dispense a second or two after the syringe is stopped. Pendant volume can be read in real time by enabling Acquisition/Live Focus in the main menu. Pendant volume can be reduced by reversing the syringe, but this will take a few seconds to respond because of backlash. Drops can be touched off using the Pump Z control (see discussion in #8 below).

7. Dispense With Piezo Pump. Enable pressure readings if the pressure sensor is installed. Pressure will build in the pump as liquid is initially forced through the small radius tip. This "Laplace" pressure is inversely proportional to tip radius. It may be as low as 1kPa for larger silica needles and 10kPa for smaller capillary needles. You must learn at what pressure liquid can initially escape the tip, but be aware the formation process occurs quickly and the pressure sensor reads slowly, so the peak pressure may be missed. The displayed pressure is only a guide to when a drop is ready to emerge. Use the piezo voltage ramp to slowly increase pressure to, perhaps, 50-75% of the rate of 0.5V/s. Use the piezo pulse to then force liquid through the tip. Start with a 20V pulse lasting 0.5s, then stepping back 20V. Change the amplitude and duration to control the volume which emerges. You may step back further than you stepped forward to limit the volume. You may ramp down voltage after the drop is formed in order to reduce the outstanding volume. In general, the initial drop will be too large and must be reduced back to the desired size. The piezo can be controlled from 0 to 96 volts. When the maximum voltage is reached, set back to 0 and run syringe pump forward to restore pressure. Split or dirty tips will cause the drop to climb up the tip.

8. Drop Touch-Off. A pendant drop can be automatically touched-off onto the specimen, once the Z distances have been measured. The correct touch-off height depends on drop radius. It is OK for the tip to go a little below the height at which the drop bottom first touches the specimen, but if it goes all the way down it will artificially spread the drop and the contact angle will be low. Carefully watch in the movie to see if the drop base spreads as the tip comes down further *after* initial contact and sessile drop formation. If it appears to do so, then limit the tip movement further. It has been shown that the correct contact angle *is* achieved under the above conditions because additional liquid flows from the tip to replace the volume occupied by the tip itself as the tip rises.

9. Expanding Drop. This is an alternative to first forming a pendant drop and then touching-off. Place the tip very close to sample, say 3 to 5µm. Setup to capture images *before* the trigger and start Run. Now pump. If using piezo pump, start slow ramp. When liquid emerges from tip and touches sample, it will form capillary around tip. Trigger movie when capillary forms; this can be used to stop further pumping. Images which are spherical in shape correctly represent contact angle. This method will form the smallest sessile drops and analyze the smallest physical areas. It is no matter if drop ultimately expands to a drop larger than desired--the images captured when the drop was still small can be used. It is often

possible to back the drop up by ramping the piezo voltage down. This method will work with the tip actually touching the sample as long as the tip is not damaged.

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