Surface Tension Measurements
Polymers, Waxes, and Solders

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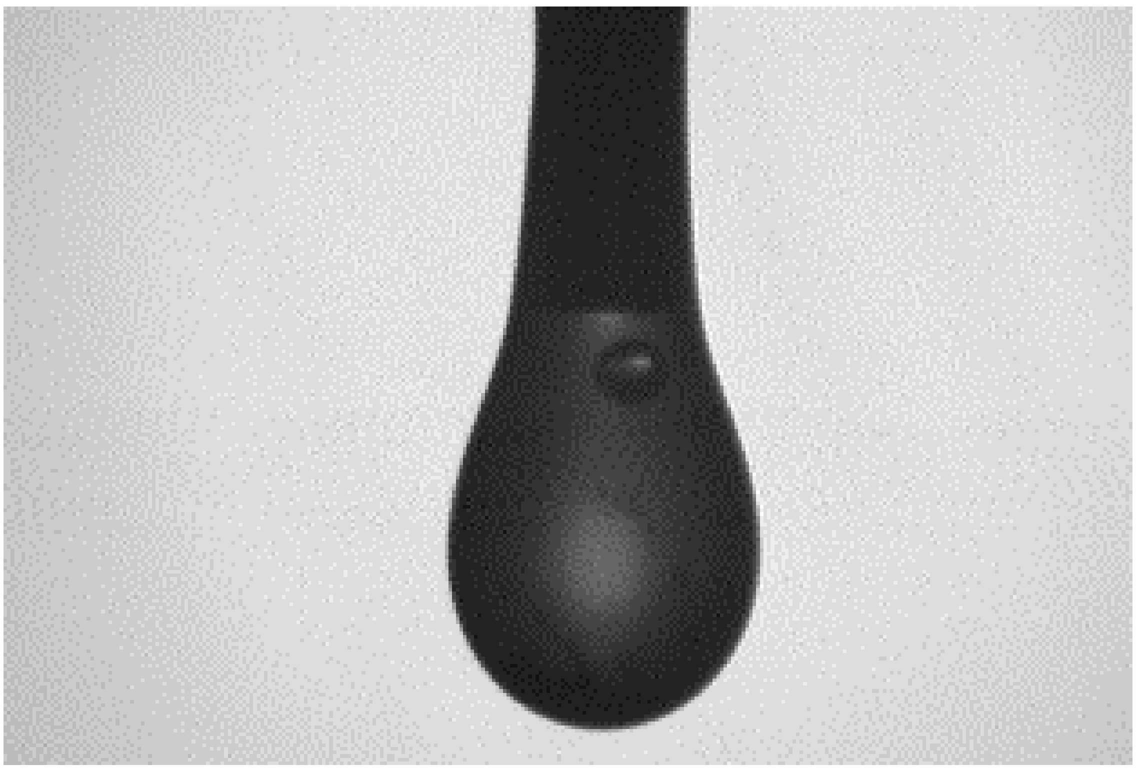
Pendant drop surface tension measurements of substances which are solids at room temperature but melt below, say, 250°C, can easily be made with the FTÅ200 Dynamic Contact Angle Analyzer and its Environmental Chamber using the following protocol.



Pendant Drop of Molten Polymer

Generally speaking, it is difficult to load a syringe with a material which is liquid only at high temperatures. One of the problems is expelling all of the air, and another is heating the syringe sufficiently so all of the material is in the liquid state during measurement.

A much easier method is to load only the dispensing needle, with no material actually in the syringe. Inexpensive disposable stainless steel needles may be used, so no cleaning is involved. The sample is melted in a container on a hot plate. The container and sample should provide a liquid depth of about 5mm. If the sample oxidizes at these temperatures, it may be kept under a "blanket" of nitrogen. In any event, the sample needs to be hot enough that its viscosity is lowered to the point it can be picked up by the syringe with its needle partially immersed in the liquid. We set the depth of liquid, say 5mm, to be how much of the needle we want to immerse so the needle can be dipped in until it hits bottom. Since we do not want to actually draw sample into the syringe, we can take advantage of the fact that the lower portion of the needle is heated to the liquid's temperature, but the top of the needle will be much cooler, even below the melting point. When we apply suction with the syringe, sample will flow up into the needle until it reaches the cooler portion, where it will freeze and stop. With one sample which melted at 150°C, we found that 15 seconds of needle immersion to 5mm, then 10 seconds of suction filled the needle nicely but did not bring material into the syringe. The exact timing for any particular sample must be determined by trail and error.

We have been able to use Luer-type plastic hubbed stainless steel needles at high temperatures because the top of the needle stays cool relative to the bottom. One trick is to use 2 inch needles, but fill only the lower 1 inch. This ensures the top of the needle can remain cool while the portion inside the chamber is at the chamber temperature.

Typical dispense needles will only hold a drop or two, but this will be sufficient for measurement. A 22 gauge needle 1 inch long (25mm) will hold 4.6µl, a 18 gauge needle, 19.3µl, and a 15 gauge needle, 45.6µl. For reference, a typical pendant drop for surface tension measurements might contain 10µl, so a 22 gauge needle is sufficient only for a small drop.

In preparing sample needles, it is a good idea to load a number of needles at one time, setting them aside and letting the sample freeze. Once the environmental chamber is up to the desired temperature, insert the needle into the chamber. It will come up to the chamber temperature within a minute. Then one can apply either pressure or suction with the syringe to make the droplet on the tip larger or smaller. The figure on the reverse side shows a molten polymer with a surface tension of about 15 dynes/cm. Notice there is a small bubble just below the needle tip. This came from drawing some air into the tip during loading. This will lower the true density and make the reading high, so try to obtain measurements on droplets containing no bubbles. (This image was specifically chosen to illustrate the issue.) Heating the original sample enough so its viscosity is lowered and having enough liquid in the container make it easier to avoid air bubbles in the aspirated liquid.

Remember you will need the density of the material at the measurement temperature in order to compute surface tension.

Plots of surface tension versus temperature can be made by ramping the chamber temperature and taking a movie of the drop. First determine how long it takes the chamber to change from one temperature to the other. With some experimentation, you can relate time to temperature in your movie, where each image will be analyzed for surface tension and represent one particular temperature.

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