TA Instruments Nano ITC – Quick Start Guide
Polymer Facilities - MRL @ UCSB
Rachel Behrens (rachel@mrl.ucsb.edu)

Reservations: Reserve with FBS, login to FBS to access instrument computer. Record time on Log sheet.

Sample Preparation:

Do not carry out any reaction that will create a precipitate, as it will damage the instrument!

1. Concentrations: Binding solution A and binding solution B should be at least 10 fold difference in concentration
2. Quantity: You can choose to use either the 100µL or 250µL sample syringe. The sample and reference cells each have a maximum capacity of 1200 µL, so bring according quantities of solutions.
3. Preparation/Degassing: Degas sample solutions and milliQ water **for at least 20 minutes** using the degassing station (unless it will damage your sample). *Any gas bubble formation in ITC cell will create noisy data!*
    a. Place open vial in degassing chamber and pull a vacuum of ~0.5atm

Starting the ITC:

1. Turn on the ITC using the switch on the back of the instrument
2. On the computer, open ITCRun software
    a. Popup box will say to home the buret→ ok
        i. Carefully remove buret handle from calorimeter by pressing down and turning CCW
        ii. Remove syringe from buret handle if present
        iii. Reinstall empty buret handle into instrument (Make sure the white notches are facing you)→ ok
        iv. Instrument will now home buret settings
    b. After homing, settings will say idle, can now prepare cell

Preparing Sample and Reference Cells:

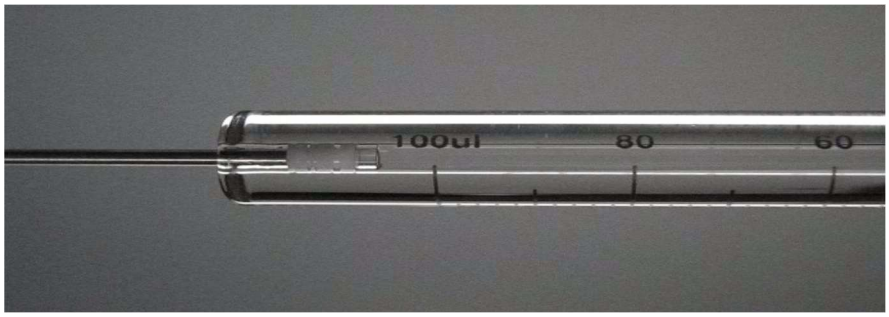
The sample cell (also referred to as the reaction vessel) will contain the titrant that will be used for your experiment, and is on the left side of the calorimeter (centered in bore). The reference cell typically contains milli-Q water and a dummy needle and is on the right side of the calorimeter.

1. Flush Sample & Reference Cells: Remove buret and insert cleaning adapter into sample cell until the silicone stopper forms a seal. Ensure tubing from side port (inlet) of adapter is connected to beaker of MilliQ water, and the top port (outlet) tubing is connected to the vacuum flask to collect waste. Start cleaning mode on the degasser, let ~500mL-1L water flow through cell. Repeat on reference side if needed.
2. Rinse & Fill Reference Cell: On the reference side, use the filling syringe (syringe with long needle) to rinse the cell several times with degassed MilliQ water(~3x 1mL). Fill clean empty reference cell with 1200 µL of milliQ water and use forceps to install the dummy reference needle.
3. Rinse & Fill Sample Cell: Using the filling syringe, rinse the sample cell several times with the same degassed buffer solution in which the sample is prepared (~3x 1mL). The aim is to end experiment with equal volumes in sample and reference cells, so adjust starting sample volume according to planned # and volume of injections. (i.e. 1200 µL capacity – 25 x 10µL injections = 950 µL starting volume)

Fill Sample Syringe and Load Buret:

1. Rinse the 100µL or 250µL sample syringe with the degassed buffer solution
2. Load the sample syringe with the titrant, taking care to remove any bubbles from the barrel of the syringe, but leaving a small, 5 to 10 µL air gap between the plunger tip and the liquid in the barrel, as shown in the picture below. Fill to slight excess of intended volume.

TA Instruments Nano ITC – Quick Start Guide
Polymer Facilities - MRL @ UCSB
Rachel Behrens (rachel@mrl.ucsb.edu)



3. Verify that the position indicator on the handle of the buret is in the fully raised position
    a. Partially filled syringes can be used, but buret position must be set accordingly in the software. See Appendix A in the Getting Started Guide for info.
    b. A separate PEEK buret is available for samples using organic solvents.
4. Hold sample syringe by knurled knob and insert the plunger end into the buret handle and thread until hand tight. Use kimwipe to dab the excess drop from tip of syringe.
5. Carefully insert buret and needle first into the Nano ITC, with gradations on buret facing towards you. Push down and rotate CW to lock in place

Wait for Equilibration:

In ITCRun, turn on stirring at 250RPM and set to desired temperature. Leave running for ~30 minutes to allow equilibration, or until baseline is steady.

Running a Sample:

1. Use SETUP tab to adjust experimental parameters.
2. Press the GO icon, enter filename when prompted. Instrument will verify signal stability at equilibrium and then autostart the experiment. Use MONITOR tab to view progress.

Cleaning the Nano ITC:

1. When experiment is over, stop stirring and, if applicable, let temperature go down to below 30C.
2. Very carefully remove buret and syringe by rotating CCW and lifting directly upwards. *The o-rings make it so the buret is difficult to remove, and if the handle is tilted at all during removal the stirring needle will be bent and made unusable. PLEASE REPORT BENT SAMPLE SYRINGE NEEDLES TO LAB STAFF IMMEDIATELY!*
3. Withdraw sample cell contents using the filling syringe. Flush with milli-Q water and degassing assembly in cleaning mode as before.
    a. *If you will be running another experiment in the next few days*, you may leave milliQ water in the sample and reference
    b. *If you will not be using the instrument again soon,* fill sample and reference cells with 50/50 mix of milliQ water and methanol
4. Reinstall empty buret into Nano ITC and clean sample and filling syringes.
5. Stop timer in FBS

Processing Data:

Data processing can be done using NanoAnalyze software, which is installed on the instrument computer and the processing computers in MRL 1050. See the Software Guide built into the software for processing options.