AIT Measurement Apparatus Standard Operating Procedure

Last modified on: August 2, 2016 by Mark Redd

1 Preparation

- Flask and Lid
  - Use a 500 mL, round bottom, long-neck flask
  - If dirty, wash out the flask using soap and water and dry as much as possible
    - Any leftover water will boil away when the furnace heats up and before any measurements are taken
  - Wrap entire flask in aluminum foil with thermocouples at the bottom, side and top of the round part of the flask (thermocouples should be touching the glass directly)
    - Start by getting a long strip of aluminum foil (8" wide or so) and wrapping it around the middle of the flask
    - Poke thermocouple #3 through the foil near the bottom so the bead sits at the bottom of the flask and then wrap the foil around the bottom
    - Slide thermocouple #2 down to the middle of the flask between the flask and foil and start to wrap the foil up the flask
    - Place thermocouple #1 at the top of the bulb of the flask and wrap the rest of the foil up around the top
    - Wrap additional foil around the neck of the flask to cover it completely and secure flask in lid assembly
    - Make a "donut" of foil that will rest up against the bottom of the lid assembly
  - Loosen the nut on top of the lid assembly and slide the corresponding half of the ceramic part of the lid assembly out
  - Place the flask in the ceramic part of the lid assembly with the lip of the flask fitting into the groove of the ceramic
  - Slide the loose half of the ceramic back in to be snug around the flask neck and tighten the nut on the top to hold it in position
    - The two halves nearest to the top of the assembly should meet or very nearly meet; if they don't then some foil should be removed from the neck of the flask
    - Use a circular spring to help hold the halves together
  - Slide the foil "donut" up so it is flush against the ceramic and basically seals the opening
  - Carefully turn the flask/lid assembly over making sure the flask doesn't fall out
    - The flask will fit into the lid assembly somewhat loosely, but it shouldn't fall out
    - If the flask falls out, remove it and add more foil around the neck
  - Guide the thermocouple wires in the gap between the two ceramic halves so they are out of the way when the flask/lid assembly is inserted into the furnace
  - Place the prepared flask/lid assembly into the furnace
- Furnace
  - Power on furnace and set temperature for initial measurement
    - To change the set point, press the up or down arrows until the desired temperature is reached
  - Insert flask interior thermocouple (#4) carefully down the flask neck, making sure it goes straight in and the bead doesn't get caught anywhere

    - The bead of thermocouple #4 should be suspended in the approximate middle of the flask, not be touching any part
    - Use the bracket on one of the two handles on top of the lid to secure the thermocouple in place
  - Connect the thermocouples to the DAQ

2 Measurement and Data Collection

- Start up computer and log on
  - Username: McKay
  - Password: asdfghjkl (Home row on a QWERTY keyboard)
- Open "AIT Data Collection.vi" (Shortcut Located on Desktop)
- Press the "run" button to start the program
- Measure out sample
  - Draw sample amount into a right-angle syringe
  - Sample size:
    - Initially use a sample size of 100 microliters
    - Once AIT is measured for 100 microliters, go to 150 microliters
    - If the AIT decreases for 150 microliters, go to 200/250 microliters
    - If the AIT increases for 150 microliters, go to 50 microliters
- Enter the filename in the textbox
  - Path: "$C:\AIT\compound\_name\filename.txt$"
  - Filename naming convention:
    - Filenames will be organized by the following values in order separated by underscores ("_")
      - Compound name
      - Sample size in microliters
      - Temperature in degrees Celsius
      - Date of experiment with the format "YYMMDD"
    - For example: The filename of an AIT experiment where 100 microliters of hexane were tested at 450 C on March 19, 2013 would be: "hexane_100_450_130319.txt"
  - Make sure to press enter to save the filename in the LabVIEW program
- Set a timer for 10 minutes but don't start it yet
- Depress the pedal marked "D" to initiate data collection
- Introduce your sample about 3 to 5 seconds after initiating data collection
  - Immediately begin the 10 minute timer and depress the pedal marked "L" to turn off the small light in the hood
- Watch the mirror above the furnace for any flame/glow for 10 minutes
  - If a flame or glow is observed, document it (color, size, brightness, sound) and then continue data collection for 1 minute after the flame or glow has disappeared, then terminate data collection

        * If the flame is bright yellow/orange, this is the hot-flame auto-ignition and the temperature should be decreased for the next test
        * If the flame is faint and blueish, this is the cool-flame auto-ignition and the temperature should be increased for the next test
        * The reported AIT is the minimum temperature at which hot-flame ignition occurs
        * If no flame or glow if observed by the 10 minute mark, increase the temperature for the next measurement
        * The bracket size goal for AIT measurement is +- 3 deg C

- Prepare for the next measurement
    - Set furnace to next temperature
    - Clean out the flask between measurements by blowing hot air into the flask for 5 minutes using the heat gun
    - Wait a minimum of 10 minutes between measurements for the furnace to equilibrate at the next temperature (5 min w/heat gun, 5 min to equilibrate)
- Start this procedure over from the third step (measuring out a sample)

3 Clean-up/Shut-Down

- The furnace may be too hot to open for several hours
- Once the furnace is cool, remove flask/lid assembly
- Remove flask from lid assembly and remove the aluminum foil
- Wash out flask with soap and water (scrubbing stains if necessary) and place on drying rack
- Do not rinse out needles