

### • Preparation

#### – Flask (See picture guide if these instructions are unclear)

- \* 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
  - 3 · Poke the thermocouple through the foil near the bottom so the bead sits at the bottom of the flask and then wrap the foil around the bottom
  - 2 · Slide the middle thermocouple down to the middle of the flask between the flask and foil and start to wrap the foil up the flask
  - 1 · Place the top thermocouple at the top of the round part 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 in the ceramic
- \* Slide the loose half of the ceramic back in to be snug around the flask neck and tighten the nut on top to hold it in position
  - The two halves nearest the top of the lid assembly should meet or very nearly meet, and 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 thermocouples 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

#### – Furnace

- \* Place prepared flask/lid assembly into furnace
- \* Power on Furnace and set temperature for initial measurement
  - To change set point, press the up or down arrows until the desired temperature is reached
- \* Insert flask interior thermocouple carefully down flask neck, making sure it goes straight in and the bead doesn't get caught anywhere
  - Use the bracket on one of the two handles on top of the lid to secure the thermocouple
- \* Connect thermocouples to DAC

### • Measurement and Data Collection

- Start up computer and log on
  - \* Username: McKay; Password: asdfghjkl
- Open "AIT Data Collection.vi" (Located on Desktop)
- Press the play button to start the program

## AIT Measurement Apparatus Standard Operating Procedures

- Measure out sample
  - \* Draw sample amount into right-angle syringe
  - \* Sample size
    - Initially, use a sample size of 100  $\mu$ L
    - Once AIT is measured for 100  $\mu$ L, go to 150  $\mu$ L
    - If the AIT decreases for 150  $\mu$ L, go to 200  $\mu$ L **250**
    - If the AIT increases for 150  $\mu$ L, go to 50  $\mu$ L
- Enter the filename in the text box
  - \* Path: C:\AIT\"Compound Name\"filename.txt"
  - \* Naming convention: COMPOUND\_SAMPLESIZE( $\mu$ L)\_TEMP(C)\_YYMMDD.txt
    - 100  $\mu$ L of hexane at 450 C on 3/19/13 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 sample into flask about **2** seconds after initiating data collection
  - \* Immediately begin 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 hot-flame autoignition and the temperature should be decreased for the next test
    - If the flame is faint and bluish, this is cool-flame autoignition 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 is observed after 10 minutes, increase the temperature for the next measurement
- Prepare for next measurement
  - \* Set furnace to next temperature
  - \* Clean out the flask between measurements by blowing hot air into the flask using the hot air gun
  - \* Wait a minimum of 10 minutes between measurements for the furnace to equilibrate at the next temperature
- Start this procedure over from the third step (measuring out sample)

### • Clean-up/Shut-down

- The furnace may be too hot to open for several hours
- Once furnace is cool, remove flask/lid assembly
- Remove flask from lid and remove aluminum foil
- Wash out flask with soap and water and place on drying rack
- Don't rinse out needles

AIT  
SOP