

AIT Measurement Apparatus Standard Operating Procedure

Last modified on: August 16, 2016 by Mark Redd

- **NOTICE:** Lab policy requires that any person performing experiments with the AIT Measurement Apparatus must have done the following before performing any experimental work:
 - Complete pertinent laboratory safety training which includes becoming familiar with all the steps outlined in this SOP
 - Read this SOP in its entirety
 - Sign and Date the AMA SOP Signatures Sheet

1 Preparation

- Flask and Lid
 - **Latex or nitrile gloves and safety glasses are required while preparing the flask**
 - 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 (see Section 3: Clean-up/Shutdown)
 - * 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
 - * **Do this over a table or close to a level surface to avoid accidental breaking of the flask**
 - * 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 2016.vi" (Shortcut Located on Desktop)
- Press the "run" button to start the program
- Enter the filename in the textbox or use the browse dialog to save to the right path
 - 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"
 - Time of day that data collection began for that run using a 24 hour clock format "hhmm"
 - * For example: The filename of an AIT experiment where 100 microliters of hexane were tested at 450 C on March 19, 2013 at 4:25 pm would be: "hexane_100_450_130319_1625.txt"
 - Make sure to press enter to save the filename in the LabVIEW program
- **Safety: Ensure you are using proper PPE and hazards in the lab environment are minimized before continuing**
 - Ensure your workspace, the area around the computer and both hoods are free of clutter, tripping hazards or any object which could present a hazard to you or anyone else in the lab
 - Refer to the SDS for the chemical you are working with when determining appropriate PPE

- * NOTE: Some SDS's will recommend using a face shield in addition to splash goggles when handling their respective chemicals. In our lab we will use ventilation hoods which, when used properly, serve as better protection than face shields. Therefore, any time an SDS recommends using a face shield you may safely ignore that recommendation provided you are using the hood properly by positioning the sash between your face and the work being performed in the hood.
- Appropriate hand protection (e.g. nitrile gloves) and splash goggles are required when handling chemicals
- Lab coats are recommended but not required when handling chemicals
- All chemical handling (except for injection into the furnace) should be done in a hood other than the hood containing the furnace to avoid a potential fire hazard
- 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
- Ensure the lab is sufficiently dark to see any flame from the mirror on top of the furnace
 - Use the lights in the hoods for preparation before your run
- Set a timer for 10 minutes but don't start it yet
- Center the end of the syringe in the hole at the top of the furnace for injection, ensuring that the sample will go straight down and not hit the sides of the flask
- Depress the pedal marked "D" to initiate data collection
- Turn off the lights
- Introduce your sample about 5 seconds after initiating data collection
 - Immediately withdraw the syringe and place it in the adjacent hood
 - Begin the 10 minute timer
- 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 by pressing the "D" pedal again
 - * 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 is observed by the 10 minute mark, increase the temperature for the next measurement
 - * *The bracket size goal for AIT measurement is $\pm 3^\circ C$*
- 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/Shutdown

- Under normal use, disposable gloves may be thrown into the normal trash receptacle instead of solid chemical waste
- 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
 - For difficult stains, soak the flask inside with soapy water for 24 hours or longer if needed
 - For best results, flasks must be as clean as possible
- Do not rinse out needles

4 Spill Clean-up

- In the event of any spill, appropriate PPE specified in the corresponding SDS should be used in clean-up
- In the event of a small spill (i.e. less than 100 ml), the following protocol should be followed:
 - If the spill occurs in or out of the hood, use absorbent clay that can be found in the lab to soak up the bulk of the liquid and wipe up the rest with a paper towel
 - Dispose of the clay, any disposable gloves and towels in the solid waste container
- In the event of a large spill (i.e. greater than 100 ml), the following protocol should be followed:
 - If the spill occurs in the hood, use absorbent clay that can be found in the lab to soak up the bulk of the liquid and wipe up the rest with a paper towel
 - Dispose of the clay, any disposable gloves and towels in the solid waste container
 - If the spill occurs outside the hood or the spill is particularly large (e.g. an entire bottle of a flammable material breaks) **perform the Emergency Shutdown Procedure (Section 5), evacuate the lab and call: BYU Risk Management and Safety - (801)-422-4468**

5 Emergency Shutdown

- In the event of an emergency do the following:
 - Power off the furnace
 - Unplug the furnace
 - Close all programs and shutdown the computer
- If an emergency requires you to evacuate the lab do only the first 2 steps