AIT Measurement Standard Operating Procedure

Last modified on: July 26, 2018 by Mark Redd

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

Contents

1 Experimental Setup and Maintenance

1.1 Flask and Lid

- **Latex or nitrile gloves and safety glasses are required while working with the flask/lid assembly**
- The flask in the furnace must be exchanged for a clean flask in the following situations:
  - The next experiment will be for a different compound
  - The next experiment will be for a new container of the same compound
  - There is reason to suspect that the flask has become contaminated or substantially dirty
  - The flask has been used for 10 runs without being cleaned
  - Once the AIT has been found for a compound, the final measurements should be repeated with a clean flask to verify the results
- Disassembling the Flask and Lid
  - **The furnace may be too hot to open for several hours after an experiment**
  - Unplug the thermocouples from the furnace
  - Once the furnace is cool, remove flask/lid assembly
    - Loosen (do NOT remove) the nut that secures the bracket and the rubber hose to the top of the furnace with a wrench
    - Move the bracket out of the way and remove Thermocouple 4 (along with the rubber hose) from the top of the furnace
    - Move the mirror out of the way to allow the flask/lid assembly to come out. Likewise ensure that the ARIA is out of the way
    - Grip the assembly with both hands by the screws on top and pull directly upward
    - **NOTE: The flask/lid assembly is heavy and pulling it out can be awkward. Please ask someone to help you remove it if you are at all unsure about removing the assembly**
    - The flask/lid assembly should easily come out of the furnace without catching on anything
  - **Carefully** set the assembly on a table or other stable surface with the flask on top (See Figure 2)
  - Ensure the bracket screw is loose
  - Remove the circular spring from its groove and slide the ceramic halves of the lid apart sufficiently to allow the flask to be removed
  - Remove flask from lid assembly and remove all of the aluminum foil and thermocouples from the flask
  - Discard the used aluminum foil in a normal trash can and set aside the thermocouples in the hood or on a surface where they will not catch on anything or become damaged
  - **Always store bulb flasks on the drying rack above the sink or appropriately secured to a ring stand** (see "Flask Cleaning" section)

- Assembling the Flask and Lid
  - Use the figures in this section as a reference when putting together the assembly
  - Use a **clean**, 500 ml, round bottom, long neck, bulb flask (PYREX© 500mL Long Neck Boiling Flask, Round Bottom, Tooled Mouth, Product No.: 4280-500 from Corning Inc.)
  - If dirty, wash out the flask using soap and water and dry as much as possible (see "Flask Cleaning" section); be sure to rinse thoroughly
    - 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) (Refer to Figure 1)
    - NOTE: The more reflective side of the foil should always be facing inward
    - Start by getting a long strip of aluminum foil (12" long or so)
    - Use a utility knife to poke a small hole (just big enough to poke the bead through) near the middle of the foil and insert thermocouple 3 through the foil so the bead sits at the bottom of the flask and then wrap the foil around the bottom (1 and 2)
    - Slide thermocouple 2 down to the approximate middle/equator of the flask between the flask and foil and run a couple of inches around the equator so that it stays in place
    - Use a second piece of foil to wrap further up the flask, ensuring the thermocouple wires run parallel up the side of the flask (3)
    - Place thermocouple 1 at the top of the bulb of the flask (not on the neck of the flask) and use a third piece of foil to wrap around the top starting at the middle (4)
    - Add an additional layer of foil around the flask so the wires are covered and run parallel when wrapping is finished (5)
    - Wrap additional foil around the neck of the flask to cover it completely and secure flask in lid assembly
    - The thermocouple wires should emerge from the foil covering near the top (but not at the top) of the flask neck, allowing them to run between the two ceramic halves of the lid assembly (6)

Figure 1: Steps for wrapping the flask in foil

- Ensure the bracket screw is loose
- Fit the neck of the flask in the center hole of the ceramic lid assembly with the lip of the flask fitting into the groove at the base of the center hole on both sides
- 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
- Slide the loose half of the ceramic back in to be snug around the flask neck, replace the spring, 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
- Make a "donut" of foil wrapped around the neck of the flask that will rest up against the bottom of the lid assembly
- Slide the foil "donut" up so and press it so it is flush against the ceramic and restricts air flow around 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

Figure 2: Final state of the flask/lid assembly

- See Figure 2 for the final flask/lid assembly before insertion into the furnace
- Place the prepared flask/lid assembly into the furnace by gripping the assembly with both hands by the screws on top and slowly lowering the assembly into place
- Turn the flask/lid assembly so the thermocouple wires point away from the ARIA
- 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 center of the flask, not be touching any part
  * The wire of Thermocouple 4 should run up the edge of the neck and not the middle to allow compound to be injected without making contact with the thermocouple
  * Use the bracket on one of the two screws on top of the lid to secure the rubber hose holding the thermocouple in place
  * Tighten the nut on the bracket hand tight and then give a half turn with a wrench to secure the nut (See Figure 3)

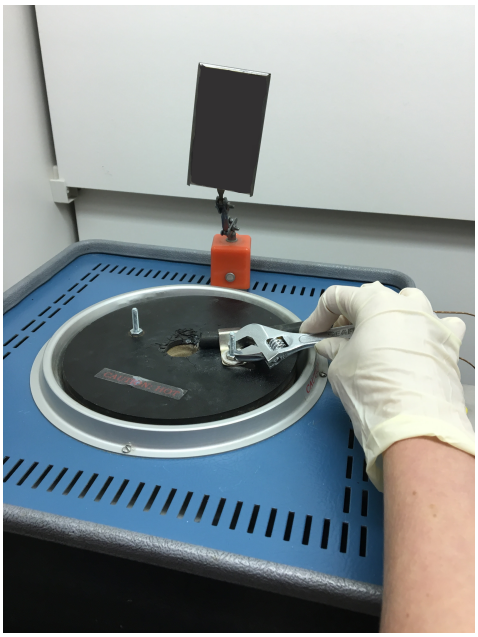


Figure 3: Position thermocouple 4 with the rubber hose and tighten

- Connect the thermocouple connectors to join the leads from the flask to the TADA, keeping the wires out of the way of the ARIA and tucked down to the side of the furnace
- Ensure the mirrors are set up correctly
- The final setup should resemble Figure 4

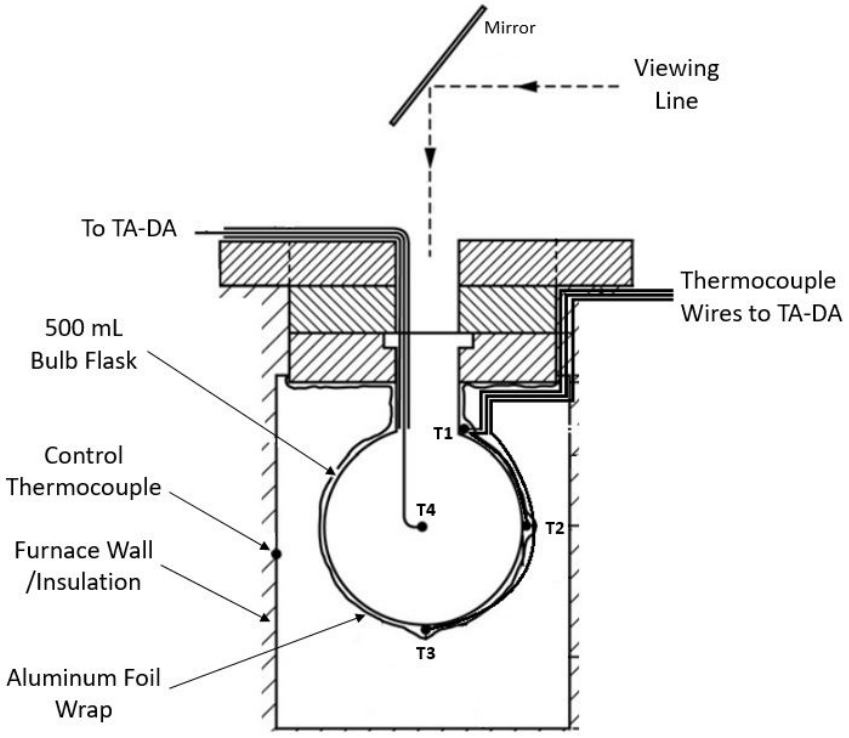


Figure 4: Diagram of the furnace when assembled

- Flask Cleaning

For consistent experimental results, flasks must be as clean as possible (See Figure 5). Dirty flasks can terminate radical reactions and artificially raise the AIT. To ensure flasks are as clean as possible before use, the following steps are required for flask cleaning:

- Always begin by soaking the inside of the flask with soapy water for 12 - 24 hours, regardless of how dirty it is
- While soaking, the flask should always be secured to a ring stand
- Wash out flask with soap and water, scrubbing the inside with tube brushes
- For difficult stains, soak the flask inside with soapy water for another 24 hours or longer if needed
    - During this process, scrub the inside and replace the soapy water on a regular basis (generally every 12 - 24 hours)
- Once all stains have been eradicated from the inside of the flask and the flask has been scrubbed in soapy water, rinse the inside and outside of the flask thoroughly
    - Using hot water for rinsing is preferred but not required
    - Rinse with tap water a minimum of 3 times, filling the flask with water, agitating the water for about 10 seconds, and then dumping the water
    - Repeat this process with distilled water available from the smaller tap on the Northeast corner of the lab sink
- If hard water spots or salt deposits appear on the inside of the flask, rinse the inside of the flask with a small amount of vinegar to remove the deposits and repeat the rinse procedure above
- Once the flask has been cleaned and rinsed thoroughly, place the clean flask on the drying rack over the sink

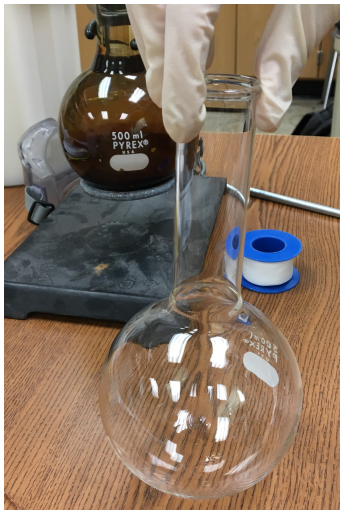


Figure 5: A clean flask (dirty flask in the background)

1.2 Furnace

1.2.1 Overview

- The furnace, shown in Figure 6, is an encased stack of ceramic insulation with cavities cut out to allow space for the heating elements and the test flask (see Figure 4 for an internal diagram of the furnace). The furnace is controlled with measurements taken at the insulated furnace wall. This design causes the furnace to have large temperature gradients while in operation. As a result, the setpoint temperature and the flask temperature will almost always differ significantly (as much as 25 K in some cases). Therefore, setpoints must be chosen between approximately 10 - 20 K above the desired temperature to reach that temperature inside the flask. **The reported AIT must be taken from the internal flask temperature (Thermocouple 4) and NOT the control thermocouple inside the furnace**

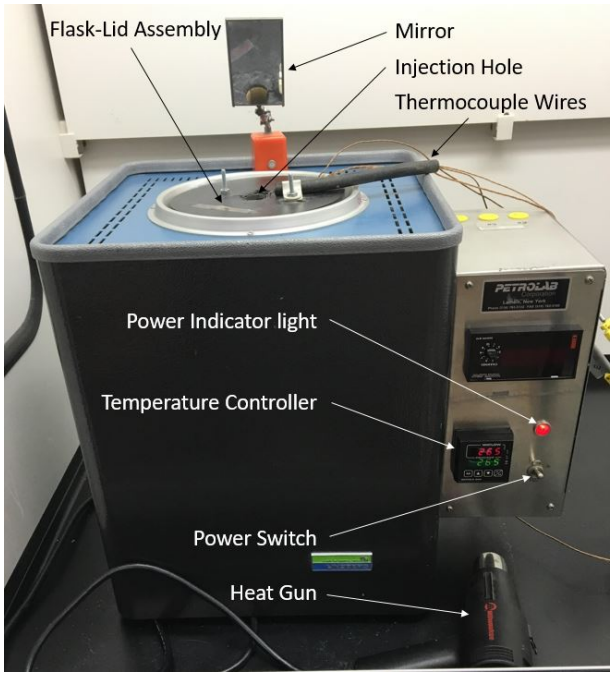


Figure 6: AIT Furnace

- When powered on initially, the furnace may take up to 2 hours or more to reach a desired temperature and thermally equilibrate
- Any time a desired temperature is reached, allow at least 30 minutes for thorough thermal equilibration in the flask; allow extra time during initial start up

1.2.2 Furnace Operation (See Figure 6)

- Power on the furnace with the power switch and use the temperature controller to choose a setpoint temperature
  - To change the set point, press the up or down arrows until the desired temperature is reached
  - The lower (green) display is the setpoint and the upper (red) display is the control thermocouple temperature

- When shutting down, turn off the power switch and unplug the furnace

1.3 Camera and Tablet

1.3.1 Overview

- Prior to using the experimental setup, all researchers must become familiar with basic use and operation of the GoPro© HERO4 Session™ camera and the Samsung Galaxy Tab A Tablet.

  More detailed instructions on how to do basic tasks may be found at the following URLs:

  - `https://shop.gopro.com/softwareandapp`
  - `https://gopro.com/help/articles/Block/How-to-Pair-the-Camera-with-the-GoPro-App#HERO4 Session`
  - `https://gopro.com/help/articles/Block/Getting-Started-with-the-GoPro-App`
  - `http://www.samsung.com/us/support/owners/product/galaxy-tab-a-8-0-wi-fi`

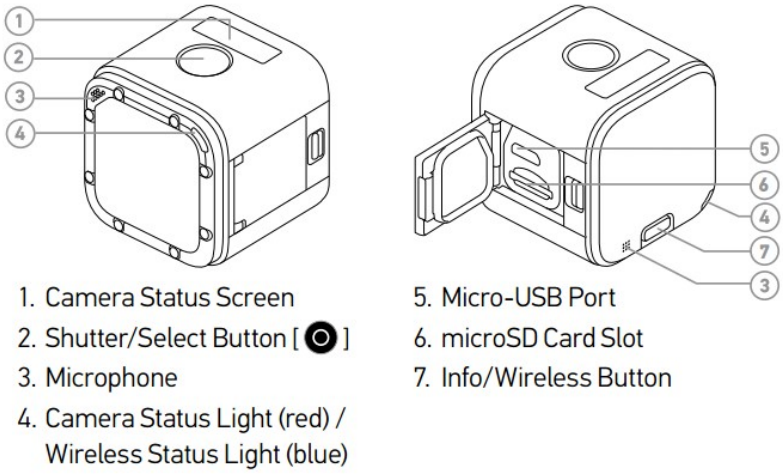


Figure 7: GoPro© HERO4 Session™ Camera Parts

Figure 8: Samsung Galaxy Tab A

- Refer to Figures 7 and 8 for camera and tablet setup

1.3.2 Connecting to the camera

- Firmly press and release the "info/wireless" button on the back of the camera (not the red circle) multiple times until you see "APP & RC" on the camera status screen
- Press the "shutter/select" button (the button with the red circle) to confirm your selection
  - The "wireless status" (blue) light will begin flashing. This indicates the camera is broadcasting a Wi-Fi signal
- Power on the tablet by holding down the power key (top, right side) until you see the splash screen indicating the tablet is booting up
- When first powering on both devices ensure they both are sufficiently charged. If not, immediately plug them in
- Once the tablet has booted, swipe to get to the home screen and select the "Settings" app
- Select the Wi-Fi settings at the top of the list on the left side of the screen
- Select the Wi-Fi network labelled "ait_cam_2016" then select "connect" on the message box that pops up (FYI: the wifi password is "hotflame16")
- Once the tablet has connected to the Wi-Fi, return to the home screen by pressing the home key
- Open the GoPro Capture App (app is labelled "Capture" on the home screen)
- Select the connect box on the top left corner of the screen to connect to camera
- Press the camera icon in the center of the screen
  - The camera will make a beeping noise and the camera view will open on the tablet

1.3.3 Shutdown

- To shutdown the camera:
  - Press the “info/wireless” button until the camera status screen reads “Turn Wi-Fi Off”
  - Press the “shutter/select” button to confirm your selection
    - The “wireless status” (blue) light will stop flashing
  - Press the “info/wireless” button until the camera status screen reads “Exit”
  - Press the “shutter/select” button to confirm your selection
    - The camera will shutdown
- To shutdown the tablet:
  - Press the “Recent” button to bring up all opened programs and close all programs by swiping on them or pressing the ’X’ in the top right corner
  - Press and hold the Power key until the option to power of pops up then press power off
    - The tablet will shutdown

1.3.4 Other Information

- Camera Operation
  - All operations may be done remotely on the tablet via Wi-Fi or directly with the “info/wireless” and “shutter/select” buttons on the camera. For experimental purposes, only basic operations will be covered. For more detail on camera operation please see the URLs above
  - In the camera’s off or normal modes the “shutter/select” button toggles recording or standby; the camera will automatically shut off after a few seconds on standby
  - If the camera is remotely controlled, the on screen red button toggles recording or standby
  - During recording, the camera will not allow viewing via the tablet. This is due to the high frame rate of our experiments
  - Captured video may be reviewed and managed remotely with the grid button on the bottom left corner of the screen
  - The camera may be powered on and off remotely with the power button on the top right corner of the screen. The camera should be powered off between experiments or when not in use
- Batteries:
  - Recharging power supplies and USB cables are available for both the tablet and camera
  - Both the camera and the tablet may be charged while in use
  - Do NOT charge tablet with the computer as it does not deliver enough current for effective charging
  - Batteries should be allowed to discharge to between 10 - 20% before recharging
  - Batteries should always be recharged to 100% capacity before unplugging
  - Do not overcharge any battery. Do not leave any battery charging overnight

1.4 Pressure Vessel

1.4.1 Changing the rupture disk

We use 1100 alloy extra thin aluminum foil as a rupture disk. This material has been shown to be effecive in preventing catastrophic failure in the vessel. These sections outline how to change and test the rupture disk.

- Remove the O-ring and outlet ring from the pressure vessel by removing the TC clamp
- place the O-ring on a piece of extra thin 1100 alloy aluminum foil
  - Do **NOT** use the aluminum foil used for wrapping the flask, it is far too strong and may lead to a catastrophic failure of the vessel
- Using your fingers, rip the foil around the O-ring so the piece is roughly the area of the O-ring
- Press the ripped foil in between the O-ring and the outlet ring
- Place the combined rings and foil on the rupture outlet on the pressure vessel and fold any excess foil over the rupture outlet on the pressure vessel
- Secure the assembly on the pressure vessel using the TC clamp
- The foil should appear smooth across the rupture surface without any folds or crumpled spots
- The studded surface may still be apparent but this is normal

1.4.2 Testing the rupture disk

- Put on safety glasses and use earplugs
- Take the O'ring and place it on a piece of tin foil
- Using your fingers, rip the tin foil around the O'ring
- Take the piece of tin foil and place it in between the O'ring and the cap
- Fold the excess tin foil over so that it is not in the way
- Secure the cap on using the clamp
- Open the air tank by screwing the cap counterclockwise
- Plug in the pressure regulator (you will plug this in and unplug it for each experiment)
- Slowly turn the pressure regulator clockwise so and observe the increase in pressure (PSI)
- Observe at what pressure the tin foil failed
- Turn the pressure regulator counterclockwise to decrease the pressure
- Unplug the pressure regulator
- Close the air Tank by screwing the cap clockwise
- Record the Pressure at which it failed, whether the shiny or dull side was facing outwards, if the foil was original smooth or crumpled, and where the hole appeared

2 Measurement and Data Collection

This section enumerates the procedure for measuring AIT. Researchers should follow these procedures every day and for every experiment performed to ensure consistent results. The first priority should always be safety. Therefore, if any step of this process is found to be unsafe or pose an unacceptable risk it should be changed. Furthermore, changes should be made if any step of the process violates the ASTM E659 Method to conform to the requirements of the method.

2.1 Startup

1. Ensure the lid is off the pressure vessel and the vessel is being vented by the snorkel
    - Under normal operation, the vessel should be vented with the snorkel any time the vessel is open
    - The only exception to this rule is when the experimental setup has been shut down for an extended period of time for maintenance purposes
2. Ensure the vessel rupture disk is intact and positioned correctly (See Figure 9)
    - See the training on proper rupture disk installation if the this is not the case

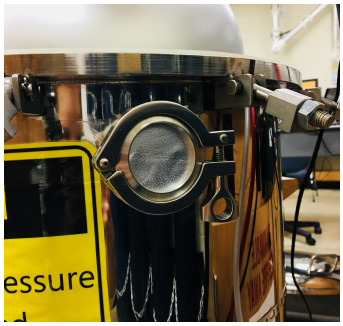


Figure 9: Ensure the rupture disk is present and intact

3. Start up computer and log on
    - Use your CAEDM account to log in
        - You should be able to access all the needed tools and programs from your account
        - If you cannot access a program or file from your account, let Mark know and he will give you administrator access as needed
    - You may need to specify the domain you are logging into. If that is the case enter your CAEDM credentials in as follows:
        - Username: CAEDM_AD*your_caedm_username*
        - Password: *your_caedm_password*
4. Ensure a compatible SD card is inserted securely into the TADA datalogger (see Figure 10)

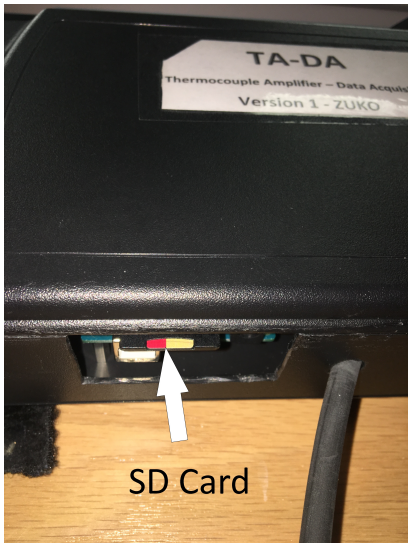


Figure 10: SD card slot location

5. Ensure the furnace thermocouples are connected to their corresponding connectors inside the vessel and ensure that the wires are tucked down between the side of the furnace and the wall of the vessel and are out of the way (see Figure 11)

    - Thermocouple wires coming out of the furnace are numbered and should connect to the corresponding brown wire connected to the TA-DA

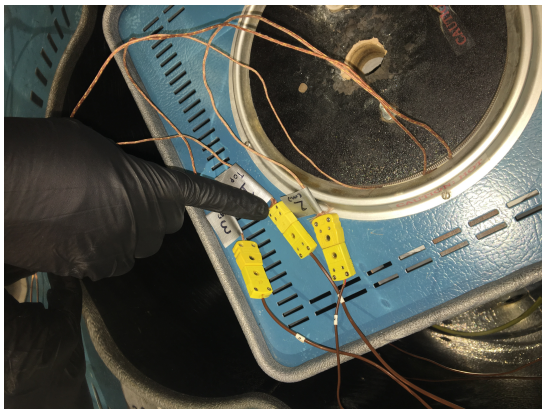
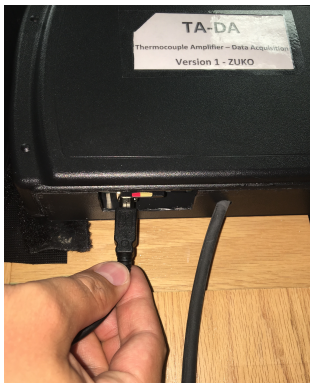


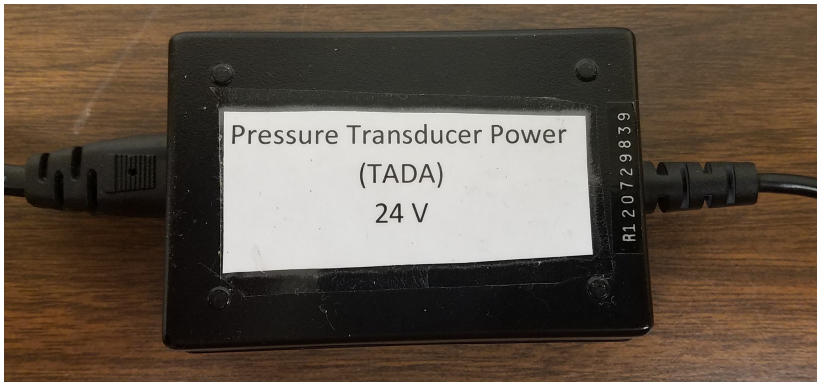
Figure 11: Thermocouple connections

6. Connect the TADA to the lab computer via the USB cable mounted under the edge of the hood (see Figure 12)



(a) USB Connection

(b) 24V Power Connection



(c) 24V Power Supply

Figure 12: TADA Connections

7. Plug in the 24 volt power supply to the TADA (see Figure 12)
    - The connection is on the bottom right hand side of the box
    - Do NOT plug the 24 volt power supply into the Arduino as this will destroy the Arduino
8. Open the TADA user interface program.
    - Path: *C:\Users\Public\Public Documents\AIT\ait_exp\TADA\TADA_UI.py*
    - You may wish to make a shortcut to this location and put it on your CAEDM desktop.
    - The program will open two windows. Ensure both windows are visible while using the program
9. Return to the computer and press the "Sync Time" button on the bottom left corner of the TADA_UI window to synchronize the Arduino clock to the computer time
    - This must be done at least once every work day

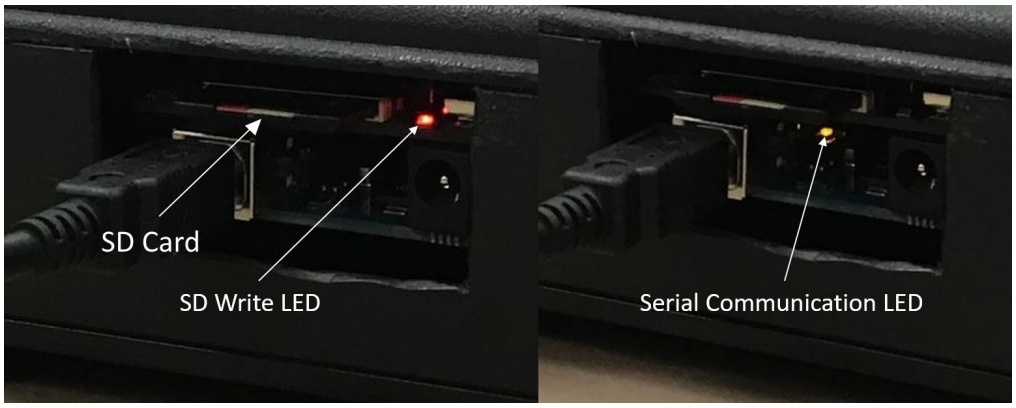


Figure 13: Control LEDs inside the TADA

10. Using the TADA_UI, check the temperature of the furnace to ensure safe handling for startup
    - The temperature should be close to ambient lab temperature
    - Do not perform maintenance or change the flask unless the internal temperature of the furnace is below $40°C$
11. Twist both of the ARIA lead screws by hand such that the mounting plate is all the way down and touching the base plate and the push block all the way back and is touching the horizontal stepper motor. (This is the shutdown position. See Figure 14)

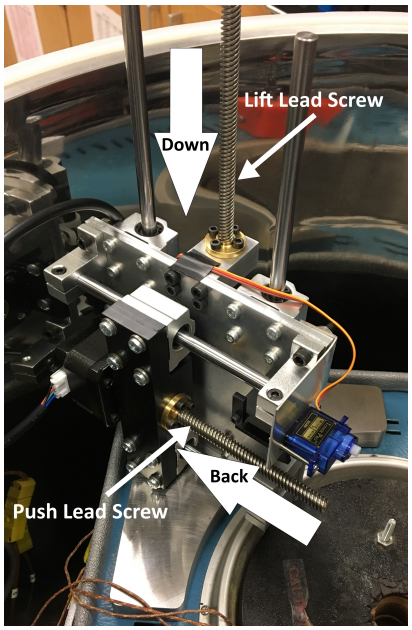


Figure 14: The shutdown position for the ARIA

12. Ensure the three molex cables are securely plugged in to the ARIA (see Figure 15)
    - Regardless of the experiments to be performed, ensure all three are plugged in and the corresponding cables are not strained

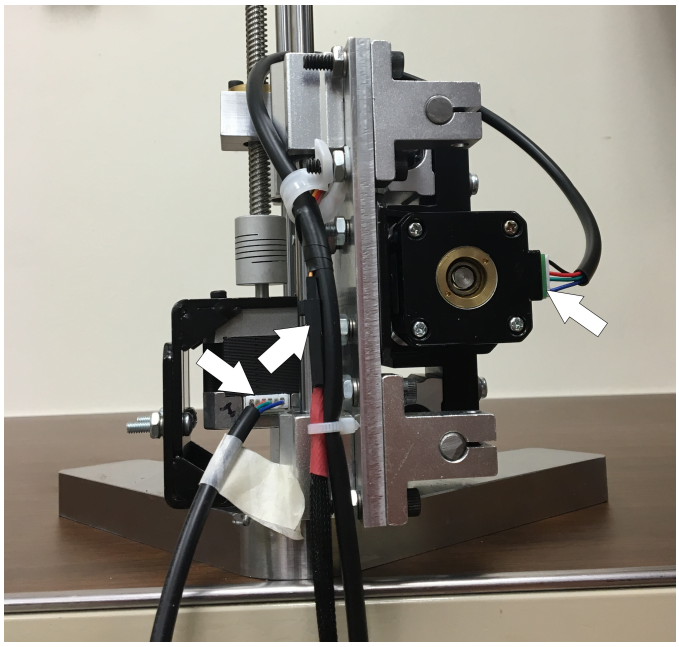


Figure 15: Molex cables secured and out of the way

13. Plug 5 volt power supply into the ARIA control and wait for initial setup sequence to complete (The two button lights on the ARIA control will come on when the sequence is complete) (See Figure 16)

    - **WARNING: Do NOT plug the 24 V power supply into the ARIA Control. This WILL destroy the ARIA Control.**

Figure 16: Plug in ARIA; 5V ONLY

14. Test the placement of the ARIA using a blank sample

- Place the ring stand in the ARIA, then secure the funnel in the ring stand, then run the solid program (Press Solid Button)
- Note that the end of the ring stand rod must be nearly flush with the inside surface of the ring stand mount block See Figure 17
- If the funnel does not go directly into the flask, adjust the placement and retest until properly positioned

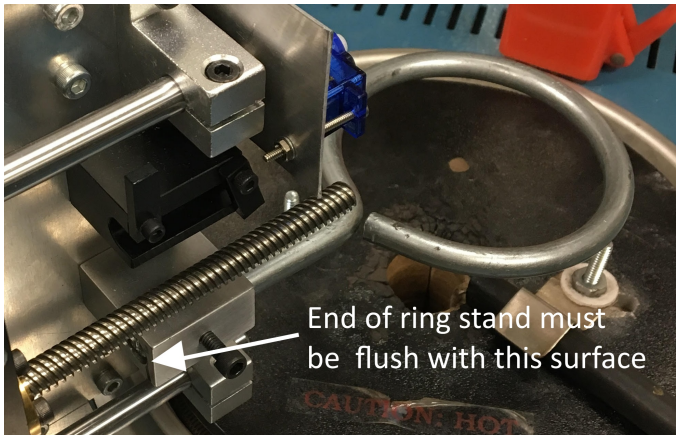


Figure 17: Place the ring stand so its end is flush with the inside surface of the mount block

15. Test the placement of the mirror using the tablet and camera
    - Power on the tablet and connect it to the camera's Wi-Fi (See Section 1.3 on how to do this)
    - Open the Capture app to view the camera's view finder
    - Mount the camera on the side of the furnace
        - Lift the rubber flap on the base of the camera buckle out of the way to allow the buckle to slide into the camera mount
        - Insert the plastic buckle into the camera mount on the side of the furnace until it snaps securely into place
        - Press the rubber flap back into place to lock the camera buckle into the camera mount
    - Using the camera's view finder on the tablet, adjust the position of the mirror on the furnace to align the camera's view to see directly down the center of the flask
        - The camera should be positioned so that the hole in the furnace and the mirror are visible
    - Once you have aligned the mirror, remove the camera for initial furnace heating
        - Lift the rubber flap on the base of the camera buckle out of the way to allow the buckle to slide out of the camera mount
        - Squeeze the two catches on the camera buckle and pull the buckle out of the camera mount
16. Ensure the furnace is plugged in to the 220 V outlet on the edge of the hood
17. Power on furnace and set furnace temperature between 10 - 20 degrees above your initial target flask temperature
    - When powered on initially, the furnace may take 2 hours or more to reach a desired temperature and thermally equilibrate
    - Use the TADA_UI to track the internal temperature of the flask

- Once the internal temperature starts to reach equilibrium, you may adjust the set point temperature until the target temperature is reached

18. Once the target temperature is reached, allow 30 minutes for thermal equilibration, then begin experiments

2.2 Experimental

This section outlines the steps for experimental runs. Each experiment should be performed following these steps exactly (insofar as that is possible). Doing so will ensure consistent results with the lowest uncertainty possible.

1. In the TADA_UI program, press the "Choose Target File" button and choose where to save your file
   - Save all temperature data files in comma separated values (.csv) format
   - Path: *C:\Users\Public\Documents\AIT\data\compound_name\filename.csv*
   - File naming convention:
     - Filenames will be organized by the following values in order separated by underscores ("_")
       * Compound name
       * Phase of the compound ('g' for gases, 'l' for liquids, 's' for solids)
       * Date of experiment with the format "YYMMDD"
       * Time of day that data collection began for that run using a 24 hour clock format "hhmm"
       * Sample size in microliters (for liquids) or milligrams (for solids and gases)
       * Test temperature in degrees Celsius (rounded to the nearest integer)
     - For example: The file name of an AIT experiment where 100 microliters of liquid hexane were tested at 450 °C on March 19, 2013 at 4:25 pm would be: "hexane_l_130319_1625_100_450_.csv"
   - This action will reset the TADA for the next measurement
2. **Safety: Ensure you are using proper PPE and have minimized hazards in the lab environment 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
   - Appropriate PPE (e.g. nitrile gloves and splash goggles) are required when handling chemicals
   - 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 handing 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.
   - Unless an SDS states otherwise, lab coats are recommended but not required when handling chemicals

- All chemical handling (except for injection into the furnace) should be done in the hood to avoid a potential fire hazard

3. Measure out sample
    - Liquids
        - Take 2 clean 100 ml beakers and put them in the hood (these will be your sample and waste beakers)
        - Place a small amount of compound into the sample beaker
        - Rinse the syringe of any extranous compounds 3 times
            * Draw approximately 300 microliters into the syringe from the sample beaker and eject it into the waste beaker
        - Draw sample amount into a right-angle syringe
            * Begin by drawing an excess amount of compound into the syringe
            * Draw slowly to minimize air bubbles in the syringe
            * Hold syringe vertically to move air bubbles to the top, gently tapping the syringe if necessary
            * Gently eject the syringe into the waste beaker to remove any air bubbles until the syringe reads the desired amount
            * The desired amount should not exceed 250 microliters
    - Solids
        - Tare the lab scale with the weigh boat and measure out sample
            * This should not exceed 250 mg
    - Gases
        - Draw sample amount into a right-angle syringe
            * This should not exceed 250 microliters

4. Secure the sample to the ARIA
    - Liquid/Gases Sample
        - Place the syringe securely into the syringe holder on the ARIA, making sure the tip of the syringe is aligning down the denter of the hole in the funnel needle is aligned down the center of the hole in the furnace
        - Test placement one more time with the opposite program (for liquid sample use the solid button)
    - Solid Sample
        - Carefully insert the weigh boat into the weigh boat holder and press the weigh boat holder onto the servo shaft so the weigh boat is in a near-horizontal position
        - Test placement one more time with the opposite program (for solid sample use the liquid button)

5. Remove gloves before proceeding

6. Ensure the camera has sufficient battery and is powered on

7. Secure the camera to the side of the furnace

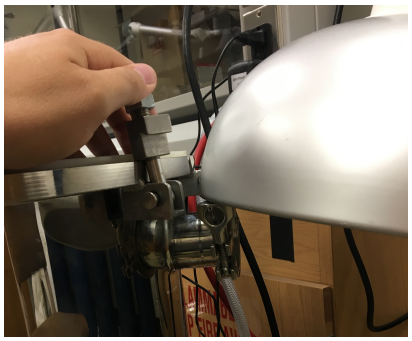


Figure 18: Snorkel placement during operation

8. **Simultaneously** remove the snorkel from inside the vessel and place above the rupture disk (see Figure 18) **and** place lid on pressure vessel and secure in place with the clamps and cable

    - Two people should perform this step. One to remove the snorkel and the other to place the lid
    - Open the sash on the hood and carefully pull out the vessel lid ensuring the outlet hose does not catch on anything (do not pull it through the sliding doors)
    - When placing the lid, hold the lid directly above the vessel and carefully lower it straight down on the vessel to avoid hitting the ARIA with the lid. Make sure to avoid crimping the outlet hose and keep it as smooth and straight as possible
    - Line up the two marks on the lid with the corresponding marks on the vessel
    - Ensure the lid is centered on the vessel by running your fingers around the edge to ensure the edge of the vessel and the lid are flush
    - Hand tighten all the pressure vessel clamps on the lip of the lid so the slack is taken out
    - Using your other hand to keep the pressure vessel from rotating, tighten the clamps in opposing pairs following the numbering on the back of each clamp (see Figure 19) using a torque wrench, tightening each clamp until the torque reads 60 inch pounds

Figure 19: Clamp numbering

    - Tighten each clamp again with the torque wrench, this time by going around the circle, ensuring that torque is 60 inch pounds on each.

- Loop the safety cable through both lid handles and through the handles on both sides of the vessel and then back through the lid handles so the two ends meet then secure the two ends together (See Figure 20)

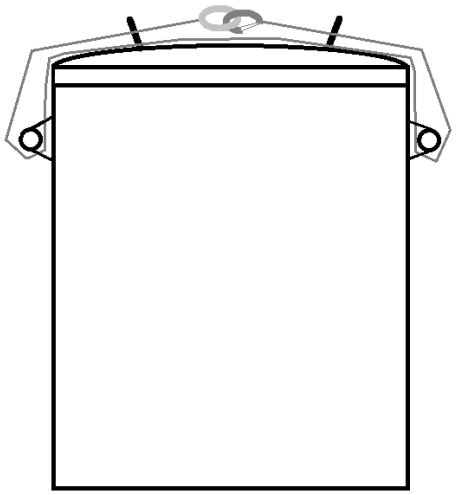


Figure 20: Security Cable Installation

9. Pressurize the vessel
   - **Safety glasses are required anytime the vessel is pressurized**
   - The absolute pressure in the vessel can be read at the bottom of the TADA_UI window
   - Ensure the ball valve connecting the regulators to the inlet hose is closed (valve handle perpedicular to the flow)
   - Fully open the rotometer on the exhaust of the pressure vessel by **gently** rotating the rotometer knob counterclockwise
   - If it has not been done earlier in the day, slowly open the cylinder valve all the way and then turn back one quarter turn
   - Once the regulators are pressurized, slowly open the ball valve
   - Slowly close the rotometer (rotate clockwise) until the air flow reads 25 SCFH (The flow rate is read at the middle of the floating ball)
   - Allow a 1 - 2 minutes for equilibrium to be reached initially
   - Adjust pressure in vessel using low pressure regulator until the absolute pressure reading in the TADA_UI is highlighted green indicating that the pressure in the vessel is sufficiently close to 1 atm (760 torr)
     - Do this with two people. One to read the pressure off the TADA_UI and the other adjust the regulator
     - While pressurizing, make sure that the rotometer reads about 25 SCFH. This may take some adjusting back and forth.
     - Note: If a loud, high pitched noise is heard, there is a leak. Immediately close the ball valve and allow the vessel to fully vent to ambient pressure and check all seals
     - Ensure that there are no leaks around the lid before proceeding
     - Allow at least 20 secs for equilibration each time the pressure is changed
10. Ensure the vessel is sufficiently dark to see any flame from the mirror on top of the furnace

11. Begin data collection

    - In the TADA UI program, press the Enter key
    - The TADA UI will keep track of the elapsed time since data collection began at the bottom of the window. This may be used to time the experiment

12. Press the red button on the tablet screen to start recording

13. Press the LED button on the ARIA control box that corresponds to the physical state of the sample (green for solid, blue for liquid) to initiate ARIA sample injection.

14. Watch through the view port for an flame or a large temperature spike for 10 minutes

    - A temperature rise or a visible flame indicates a combustion event has occured
    - If a combustion event is observed, allow enough time for the temperature to return to a steady state before terminating temperature data collection
    - The experiment ends when one of the following criteria is met:
        - An ignition event is observed and the temperature returns to steady state
        - 10 minutes pass with no ignition event observed
    - Stop the camera at least 10 seconds after a large temperature spike is observed or after a visible flame disappears
    - If the UI is used to keep track of time and no flame is observed, continue collecting temperature data until 600 seconds have passed since injection

15. Record pertinent data and observations in the lab book and the TADA_UI

    - The following data must be present on the same row in the lab notebook, in the following order:
        - Time of day that data collection began for that run
        - Compound name
        - The lot number and/or sample number of the container (This only needs to be recorded in the lab book and only once for every compound container)
        - Phase of the compound upon injection ('g' for gases, 'l' for liquids, 's' for solids)
        - Sample size in microliters (for liquids) or milligrams (for solids and gases)
        - Set-point temperature of the furnace
        - Test temperature in degrees Celsius (rounded to the nearest integer)
            * This should be the internal flask temperature (Thermocouple 4) prior to injection
        - Indicate whether an ignition event was observed (i.e. Did the camera see a flame?)
        - Indicate whether a hot flame or cold flame was observed (if applicable)
            * If the flame is bright yellow/orange, this is considered a hot-flame autoignition
            * If the flame is faint and blueish, this is considered a cool-flame autoignition
        - Indicate if any sound was heard upon ignition (if applicable)
    - If any item is not applicable write down N/A in its place
    - If any item is unknown, leave it blank until it can be determined
    - Optionally, leave any pertinent comments about the experiment next to or directly under this row of data
    - Record the same data in the corresponding fields in the TADA UI **before** terminating temperature data collection

16. After the experiment ends, terminate data collection
    - Press the Enter key again to stop data collection (the red light on the TADA should stop blinking)
    - If you haven't already, press the red button on the tablet screen to stop recording
17. Review the camera footage, looking for a flame corresponding to the temperature spike
    - This may be done on the capture app on the tablet
18. Set furnace to next temperature
    - When changing temperature, always approach your target temperature from 5 - 10 degrees Celsius above and then descend slowly to your target temperature
19. Wait about 20 minutes after ignition to allow for the pressure vessel to be purged of the combustion products. If there was no ignition, only wait 10 minutes for the pressure vessel to purge after the experiment ends
20. After the purge time has ended, depressurize the vessel
    - Turn off the inlet air flow using the ball valve
    - Fully open the rotometer by gently turning the knob counterclockwise
    - Wait until the pressure vessel is **fully** depressurized (i.e. the rotometer reads zero)
21. Remove the pressure vessel lid by **first** loosening and disengaging the clamps and **secondly** removing the safety cable
    - Loosen the clamps using a 3/4" wrench instead of a torque wrench
    - Break the seal on the lid by breifly lifting the lid with the safety cable still in place
    - Remove the safety cable from the vessel
    - **Simultaneously** Lift off the lid and place snorkel inside the pressure vessel between 6" and 10" over the furnace
        - Two people should perform this step One to remove the lid and one replace the snorkel inside the vessel
22. Remove the camera from the vessel
23. Remove any syringe or weigh boats used in the previous experiment
    - Dispose of any weigh boats in the solid waste
24. Clean out the flask between measurements by blowing hot air into the flask for 5 minutes using the heat gun on the low setting
    - The heat gun should **only** be plugged in to the outlet when in use
    - Do not point the heat gun towards the ARIA at any time
25. Extract, save and appropriately rename the video data between experiments
26. Wait a minimum of 30 minutes to reach the new temperature and allow the furnace and flask to thermally equilibrate
27. Start this procedure over from step 1 (choose the target file)

2.3 Shutdown

- The following should be done before leaving the lab at the end of every work day or any time the setup is not in use:
    1. Power off the furnace
    2. Close the TADA_UI program
    3. Unplug TADA USB connection
    4. Unplug the ARIA and TADA power supply cables
    5. Shut down and unplug the tablet
    6. Turn the camera Wi-Fi off then shut down and unplug the camera
    7. Extract all data to the computer and appropriately rename them (Refer to Section 3)
    8. Close all programs and shut down the computer
    9. Remove and store any ARIA accessories used that day
    10. Clean the funnel with appropriate solvents and dispose of the waste
    11. Discard the contents of both beakers and prepare them for dish washing
    12. Discard any residual sample in syringes and store them in the syringe box in the AIT drawer without rinsing
    13. Store all chemicals in the appropriate cabinets
    14. Remove any organic solid residue from working surfaces (See Section 4.2)
    15. Ensure all air systems are depressurized
        - Ensure the ball valve is closed (the handle should be perpendicular to the flow)
        - Slowly close the cylinder valve all the way
        - Open the ball valve by turning the handle parallel to the flow
        - Wait until both regulators depressurize
- A hot furnace may be left with the pressure vessel open and the snorkel venting it without waiting for it to cool
- Under normal use, disposable gloves may be thrown into the normal trash receptacle instead of solid chemical waste

3 Data Extraction

During experiments data are being recorded on the lab computer, the datalogger and the camera. Both the camera and the datalogger on the TADA have SD cards with a 32 GB storage capacity that allows multiple runs to be recorded without extraction. The following policies are in place to ensure ease of use, efficiency and avoid common mistakes.

- For the AIT setup, do not exceed 10 runs without extracting temperature data to the computer and deleting the data from the SD cards.
- All data should be extracted at least *daily*
- Video data should be extracted and properly renamed as often as possible (i.e. between every run or every other run) to ensure the correct filenames are assigned to their corresponding video files
- File naming convention:
  - Filenames will be organized by the following values in order separated by underscores ("_")
    - Compound name
    - Phase of the compound ('g' for gases, 'l' for liquids, 's' for solids)
    - Date of experiment with the format "YYMMDD"
    - Time of day that data collection began for that run using a 24 hour clock format "hhmm"
    - Sample size in microliters (for liquids) or milligrams (for solids and gases)
    - Test temperature in degrees Celsius (rounded to the nearest integer)
  - For example: The filename for temperatures from an AIT experiment where 100 microliters of liquid hexane were tested at 450 °C on March 19, 2013 at 4:25 pm would be: "hexane_l_130319_1625_100_450.csv"
- Video and datalogger data must be processed (i.e. parsed, edited, timestamped etc.) before being organized and therefore will be saved to a different path initially
- After processing, all data should be organized according to the following path convention:
  - Path: *C:\Users\Public\Documents\AIT\data\compound_name\filename.ext*
  - All data, including videos, from the same run should have the same filename and path but different extensions except data from the datalogger
  - The datalogger filename convention should also have '_dlog' at the end of the name to distinguish it from the UI generated file (e.g. "hexane_l_130319_1625_100_450_dlog.csv")
  - When processing is finished all runs should have the following 4 files with the same name preceding them
    - A .xlsx file (for temperature data with graphs and analysis)
    - A .csv file (UI generated)
    - A _dlog.csv file (datalogger)
    - A .avi/.mp4 file (video)
- The camera may be plugged in via USB and video extracted with GoPro© Quik software
  - Connect the camera to the computer via a micro USB cable (See Figure 7)
  - Press the "info/wireless" button on the camera to connect the camera to the computer

  - Quik should be configured to open automatically extract video and erase the microSD card when the camera connects to the computer
  - Video files should be extracted to the DIPPR legacy server and organized by date:
    * Path: *\\dipprlegacy.et.byu.edu\aitra\video_import*
    * Username: dipprleg\aitra
    * Password: hotflame16
  - If Quik is not configured to do this refer to the Quik manual for how to configure this (or ask me and I will configure it)
    * *C:\Users\Public\Documents\AIT\docs\GoPro_App_for_Desktop_User_Manual.pdf*
  - Once extracted to the DIPPR legacy server, video data may be timestamped and converted to .avi format on the server

- To extract data from the datalogger
  - Unplug the TADA from the computer
  - Pull out the SD card from the datalogger and use the USB SD card adapter to copy the"DATALOG.CSV file into the "raw_data" path and rename it to the original filename with the date tagged on in "YYMMMDD" format (e.g. "DATALOG_130319.CSV")
    * Path: *C:\Users\Public\Documents\AIT\data\raw_data*
  - Open the "DATALOG.CSV" file on the SD card, erase all data from it and save it, making sure to not change its name, extension or file path
  - Close all windows with the USB SD card adapter open (i.e. Excel files, Windows Explorer etc.)
  - Pull out the SD card without ejecting the unit from the computer

- Ensure all files from the camera and datalogger are deleted after they have been properly saved in the data folder

4 Spill Clean-up

In the event of any spill, appropriate PPE specified in the corresponding SDS should be used in clean-up. Always check the SDS for special considerations when cleaning up any compound.

4.1 Liquids

- 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 under the counter west of the sink 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 under the counter on the left-hand side of the lab sink 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**
- Spills involving compounds that are particularly toxic or unstable should always be considered large spills

4.2 Solids

We will generally work with organic solids that readily dissolve in simple organic solvents (e.g. acetone). Researchers must always check chemical compatablity with solutes and solvents before dissolving any compound.

- Small amounts of organic solids may be dissolved in a small amount of solvent and put in organic liquid waste
- Larger amounts of solids should be transferred to solid waste and the residue should be dissolved in solvent and discarded in liquid waste

5 Emergency Shutdown

- In the event of an emergency do the following:
  - Close the air cylinder valve
  - Power off and unplug the furnace
  - Fully open the rotometer exhaust
  - Unplug all other electrical equipment
  - Stop the camera recording (if applicable)
  - Shutdown and unplug the camera and tablet
  - Close all programs and shutdown the computer
- If an emergency requires you to evacuate the lab, do only the first 2 steps
- **DO NOT perform any steps that present a danger to you**