AIT Measurement Standard Operating Procedure

Last modified on: June 11, 2020 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 and safety protocols outlined in this SOP**
  - **Sign and date this page**

By signing this, I assert that I have completed the above steps and have sufficient competence to carry out these experiments safely.

Full Name (Please Print)	Signature	Date

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1 Emergency Shutdown

In the event of an emergency do the following:

1. Close the air cylinder valve
2. Power off and unplug the furnace
3. Fully open the rotometer exhaust
4. Unplug all other electrical equipment
5. Stop the camera recording (if applicable)
6. Shutdown and unplug the camera and tablet
7. 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**

2 Measurement and Data Collection

This section enumerates the procedure for measuring AIT. Researchers should follow these procedures 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.

This method is designed to conform to the ASTM E659 Method. Therefore, any policies and procedures violate standards set forth in ASTM E659 should be rectified to maintain conformity.

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. Check the lab book to see if the flask needs to be changed before turning on the furnace. If needed, change the flask (See Section 6.5) and **indicate you did so in the lab notebook**.
3. Ensure the furnace is plugged in to the 220 V outlet on the edge of the hood
4. Power on the 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 User Interface 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
   - **CAUTION: The furnace may be hot during the start up sequence. Avoid touching anything inside the area enclosed by the aluminum ring atop the furnace including the ring itself.**
5. Ensure the vessel rupture disk is intact and positioned correctly (See Figure 1)
   - See the training on proper rupture disk installation (Section 6.3.1) if the this is not the case
6. Ensure the 4 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 1)
   - Thermocouple wires coming out of the furnace are numbered and should connect to the corresponding brown wire connected to the TADA
7. Start up computer and log on
   - Use the "AIT Research Assistant" account to log in
     - Username: aitra
     - Password: hotflame16
8. Ensure a compatible SD card is inserted securely into the TADA datalogger (See Figure 2)
9. Connect the TADA to the lab computer via the USB cable mounted under the edge of the hood (see Figure 2)
10. Plug in the wall power to the TADA 24-volt power supply (see Figure 2)
11. Open the TADA user interface program
    - There should be a shortcut to this program on the desktop
    - Path: `/home/aitra/Documents/ait_exp/tada/tada_main.py`

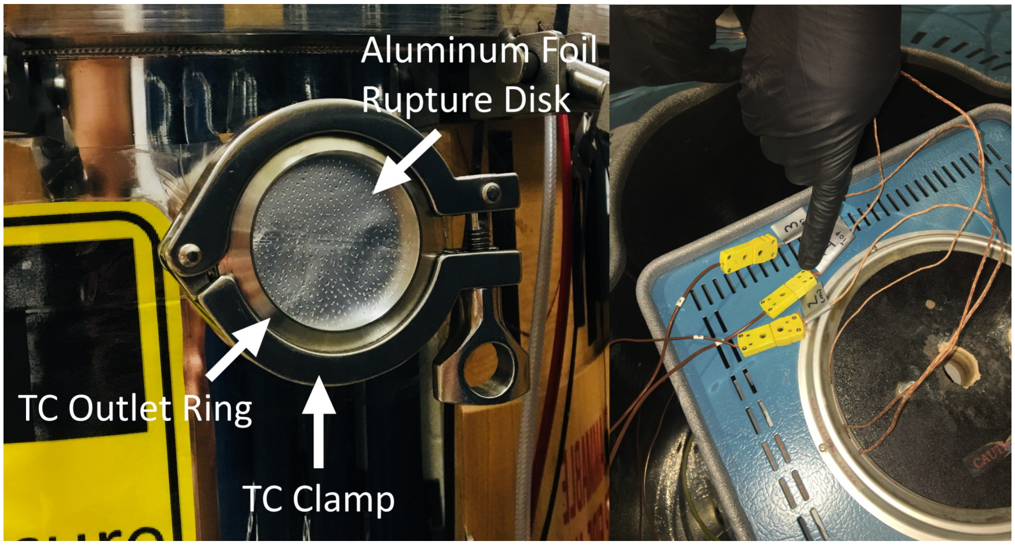


Figure 1: Ensure the rupture disk is present and intact, and the thermocouples are connected

Figure 2: TADA Connections

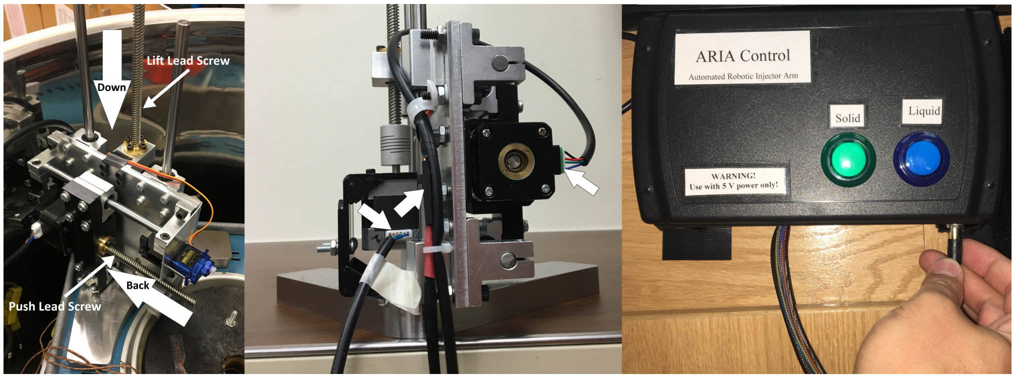


Figure 3: The ARIA shutdown position, ARIA Molex cable locations, and ARIA Power Plug (5V ONLY)

- The program will open two windows. **Ensure both windows are visible while using the program.** (The serial communication LED inside the TADA will start flashing.)

12. 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 3)

13. Ensure the three molex cables are securely plugged in to the ARIA (see Figure 3)

- Regardless of the experiments to be performed, ensure all three are plugged in and the corresponding cables are not strained

14. 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 3)

15. Test the placement of the ARIA using the funnel and ring stand
    - Use nitrile gloves when touching the ARIA
    - Ensure the ring stand is secure in the ARIA, place the funnel in the ring stand and run the solid program (Press the "Solid" Button **without gloves on**)
    - 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 4)
    - If the funnel does not go directly into the flask, adjust the placement and retest until properly positioned

16. Test the placement of the mirror using the Camera Suite app and camera
    - Connect the computer to the camera's Wi-Fi (See Section 6.2.2 on how to do this)
    - Open the Camera Suite app to use the camera's view finder
    - Mount the camera on the side of the furnace (See Section 6.2.5)
    - Using the camera's view finder, 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 and shutdown the camera for initial furnace heating

17. Check the glass wool air filter in the inlet of the rotameter replace if necessary (See Section TO BE ADDED)

18. Once the target temperature is reached, allow the system to come to equilibrium
    - The "Temp Ready" indicator in the TADA User Interface window should turn green when the system has come to an acceptable equilibrated state

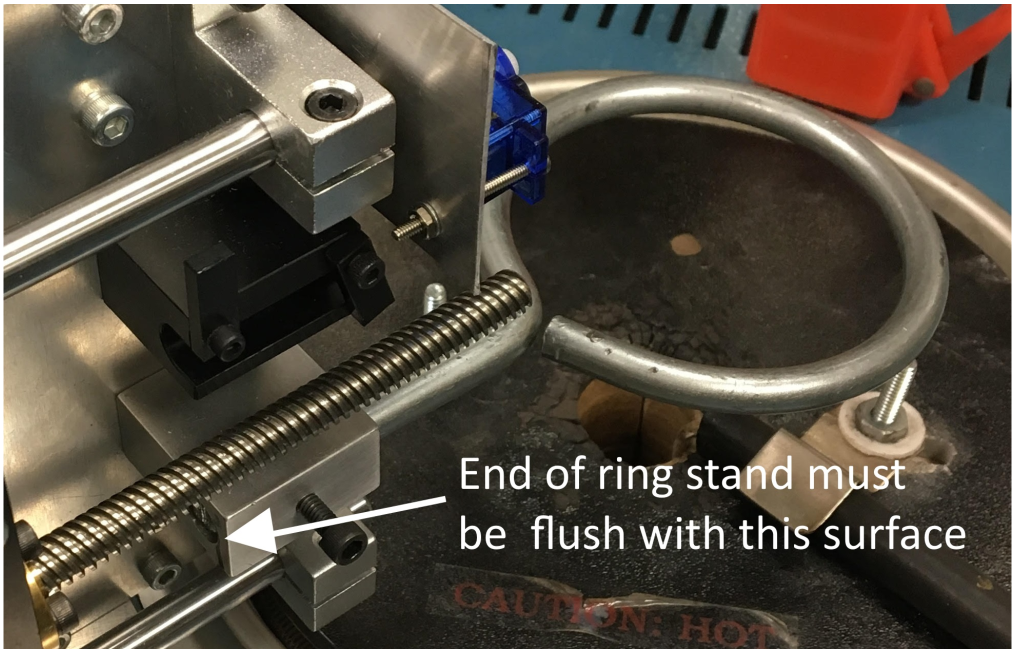


Figure 4: Place the ring stand so its end is flush with the inside surface of the mount block

- Sometimes the "Temp Ready" indicator may not turn green if the flask was not installed correctly (See Section 6.5) or there are problems with the thermocouples even when the flask has reached thermal equilibrium. Therefore, while heating up or changing temperatures, check the temperatures that are displayed in the command line window of the TADA User Interface. If the temperatures are not within about 20 K of each other the thermocouples may not be placed properly. If this is the case experiments may proceed as normal but thermal equilibrium must be verified manually by using the data on the command line window and neglecting the misplaced thermocouple.
- If any of the thermocouples in the command line window of the TADA User Interface read "NAN" (not a number) then that thermocouple is not connected. Check all thermocouple connections and reconnect the thermocouple if possible.

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.

2.2.1 Pre-Experimental Safety Checks

Before beginning experiments, ALL operators must do the following:

- Ensure your workspace, the area around the computer and both hoods are free of clutter, tripping hazards or anything which could present a hazard to you or anyone else in the lab
- Review Safety Data Sheets (SDS) and understand all hazards presented by all compounds that will be handled
  - This needs to be done only once before handling each compound for the first time but, SDS's should be reviewed periodically
  - All chemical handling should be done in the hood whenever possible. The main exceptions to this rule are for injection into the furnace and using the mass balance.
  - Refer to the compound's SDS for specific recommendations on chemical handling
- Ensure you are using appropriate PPE for handling chemicals (e.g. nitrile gloves and splash goggles)
  - 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

2.2.2 Experimental Procedure Steps

1. Measure out sample
   - Liquids
     - Before measuring a sample out for the first time, clean the syringe of any residual compound
       1. Take 2 clean 100 ml beakers and put them in the hood (these will be your sample and waste beakers)
       2. Place a small amount of compound into the sample beaker
       3. Rinse the syringe of any extraneous compounds 3 times by drawing 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
       1. Begin by drawing an excess amount of compound into the syringe
       2. Draw slowly to minimize air bubbles in the syringe
       3. Hold syringe vertically to move air bubbles to the top, gently tapping the syringe if necessary
       4. Carefully eject the syringe into the waste beaker to remove any air bubbles until the syringe reads the desired amount
     - The final sample size should not exceed 250 microliters
   - Solids
     - Measure out sample on a weigh boat
       1. Open the scale door by pressing the button with an asterisk '*' and place a new and empty weigh boat on the center of the scale plate
       2. Close the scale door with the asterisk button, and tare the scale
       3. Remove the weigh boat before measuring out sample
       4. Using a clean chemical spatula, scoop the desired amount from the compound container to the weigh boat, replacing the weigh boat on the scale to check until the desired mass is reached
       5. Take note of the initial mass of the compound sample (the mass should exceed the desired mass by about 10 mg)
       6. With the sample and weigh boat on the scale, tare the scale
       7. Remove the weigh boat from the scale
     - The initial sample size should not exceed 300 mg
   - Gases
     - Draw sample amount into a right-angle syringe
     - Sample size should not exceed 250 microliters
2. Secure the sample to the ARIA
   - Liquid/Gases Sample
     1. Place the syringe securely into the syringe holder on the ARIA, making sure the needle is in the funnel
     2. Test placement one more time to ensure the funnel is guided into the injection hole (use the **solid** button)
   - Solid Sample
     1. Carefully insert the weigh boat into the weigh boat holder sliding the securing wires out of the way as needed

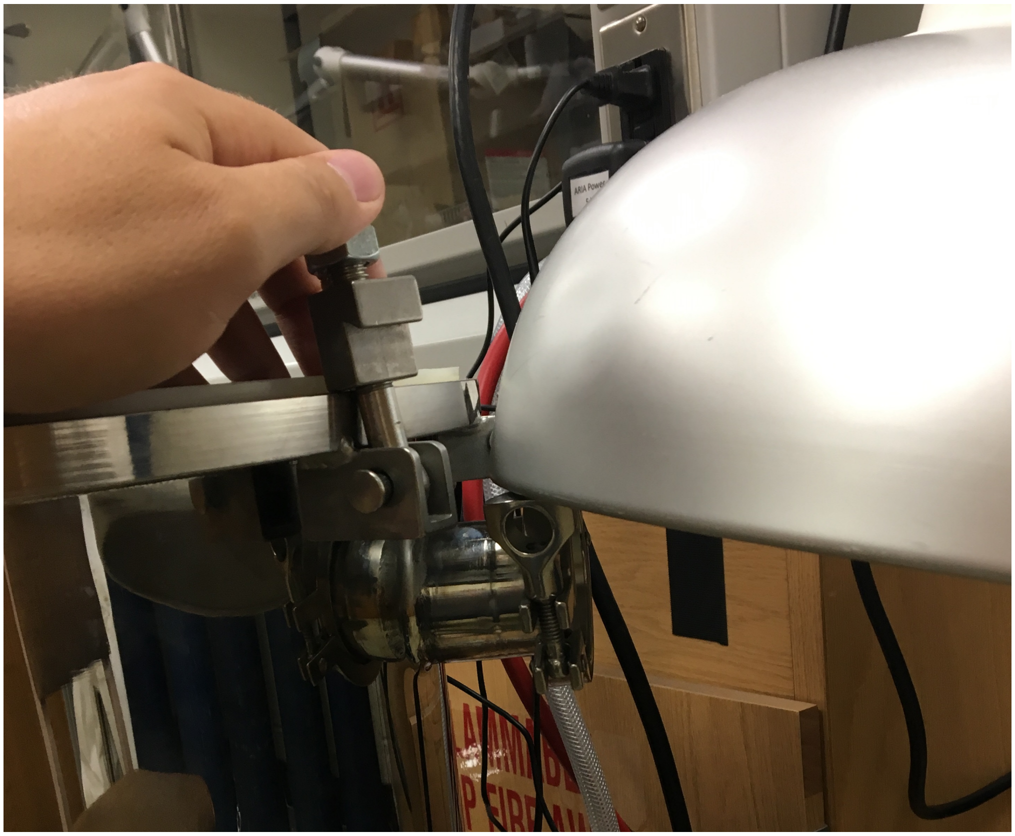


Figure 5: Snorkel placement during operation

   2. Gently tap the side of the weigh boat so that the sample collects in a pile near the leading corner of the weigh boat
   3. Press the weigh boat holder onto the servo shaft so the weigh boat is in a near-horizontal position
   4. Test placement one more time to ensure the funnel is guided into the injection hole (use the **liquid** button)

3. Remove gloves before proceeding

4. Ensure the camera battery sufficiently charged and the camera is powered on

   - Reconnect the camera to the tablet if necessary

5. Secure the camera to the side of the furnace

   - **Note for unpressurized experiments:** If you are performing unpressurized experiments (i.e. experiments with the lid off) you may safely skip the next **three** steps. Before proceeding, ensure that the vessel is being vented by the snorkel.

6. **Simultaneously** remove the snorkel from inside the vessel and place above the rupture disk (see Figure 5) **and** place lid on pressure vessel and secure in place with the clamps and cable

   - Two people are required to perform this step. One to remove the snorkel (Person B) and the other to place the lid (Person A)

   1. Person A: open the sash on the hood vertically and carefully pull out the vessel lid ensuring the outlet hose does not catch on anything (do not pull it through the sliding doors)
      - Use caution when handling the lid; the lid is heavy
   2. Person B: Lift up the bell on the snorkel so it can swing away from the open pressure vessel

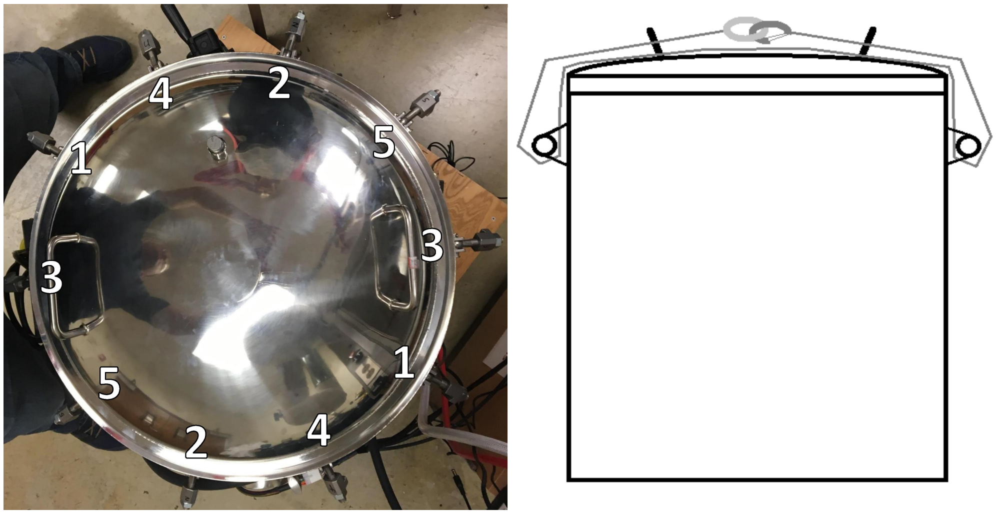


Figure 6: Securing the pressure vessel lid (Clamp numbering and Safety Cable Installation)

   3. Person A and B: Simultaneously, place the lid on the vessel and swing the snorkel away from the vessel. This should be done in one simultaneous motion.
   4. Person B: Place the snorkel over the rupture disk as seen in Figure fig:rupture_hood.
      - **The rest of the procedure requires only 1 person**
   5. 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
   6. Line up the two marks on the lid with the corresponding marks on the vessel
   7. 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
   8. Check that the lid lies flat on the O-ring and no wires or debris will break the seal
   9. Hand tighten all the pressure vessel clamps on the lip of the lid so the slack is taken out
   10. 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 6) a 3/4" box wrench, tightening each clamp about 1/4 turn
   11. Tighten each clamp another 1/4 turn with the torque wrench, this time by going around the circle
   12. 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 6)

7. Pressurize the vessel
   - **Safety glasses are required for everyone in the lab anytime the vessel is pressurized**
   - The absolute pressure in the vessel can be read at the bottom of the TADA User Interface window and should be plotted on the graph

   1. Ensure the ball valve connecting the regulators to the inlet hose is closed (valve handle perpendicular to the flow)
   2. Fully open the rotameter on the exhaust of the pressure vessel by **gently** rotating the rotameter knob counterclockwise
   3. 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
   4. Once the regulators are pressurized, ensure the cylinder has enough pressure for the next run and the low pressure regulators are not over-pressurized (adjust the regulators as needed)
   5. Slowly open the ball valve to allow air to flow into the sealed pressure vessel

    6. Slowly close the rotameter (rotate the knob clockwise) until the air flow reads 25 SCFH (The flow rate is read at the middle of the floating ball)
    7. Allow 1-2 minutes for equilibrium to be reached initially
    8. Adjust pressure in vessel using low pressure regulator until the absolute pressure reading in the TADA User Interface is highlighted green indicating that the pressure in the vessel is sufficiently close to 1 atm (760 torr +/- 2 torr)
        – This is easier with two people. One to read the pressure off the TADA User Interface and the other adjust the regulator
        – While pressurizing, make sure that the rotameter reads about 25 SCFH. This may take some adjusting back and forth.
        – Allow at least 20 secs for equilibration each time the pressure is changed
    9. Ensure that there are no leaks around the lid before proceeding
        – **Leak protocol:** If a loud, high pitched noise is heard or the pressure read on the TADA User Interface fails to rise, there is likely a leak. If this occurs, do the following:
            1. Identify where the leak is happening (using the sound or Snoop A.K.A. Soapy water)
            2. If the leak is happening anywhere besides the O-ring, immediately close the ball valve and allow the vessel to fully vent to ambient pressure to fix the leak
            3. If the leak is happening somewhere along the O- ring, verify that there is no debris or wires breaking the seal. If a seal break from debris or wires is found, immediately close the ball valve and allow the vessel to fully vent to ambient pressure to fix the leak
            4. If there is no debris or wires breaking the seal, use the wrench to slowly tighten the clamp nuts around the leak until it stops
            5. If none of these steps work or you suspect a different problem, immediately close the ball valve and allow the vessel to fully vent to ambient pressure to fix the leak

8. Look through the sight glass on the pressure vessel and ensure the vessel is sufficiently dark to see any flame from the mirror on top of the furnace

9. In the TADA User Interface 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
    - Example Path: `/home/aitra/Documents/data/compound_name/filename.csv`
    - File naming convention:
        – Filenames will be organized by the following values in order separated by underscores (‘_’)
            1. Compound name
            2. Date of experiment with the format “YYMMDD”
            3. Time of day that data collection began for that run using a 24 hour clock format “hhmm”
            4. Sample size in microliters (for liquids) or milligrams(for solids and gases)
            5. Test temperature in degrees Celsius (rounded to the nearest integer)
        – For example: The file name of an AIT experiment where 100 microliters of hexane were tested at 450 degrees Celsius on March 19, 2013 at 4:25 pm would be:`hexane_130319_1625_100_450.csv`
    - This action will reset the TADA for measurement

10. Write down everything you know in the TADA window and the lab book (i.e. date, time, sample size, temperatures etc.).

11. Begin data collection

    - **Note for unpressurized experiments:** If you are performing unpressurized experiments (i.e. experiments with the lid off) turn off the lights and ensure the laboratory is sufficiently dark to see any flame from the mirror on top of the furnace **before** beginning data collection.
    - In the TADA User Interface program, press the Enter key or click the “Collect Data” button on the TADA User Interface

- The TADA User Interface 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
- Press the "Record" button on the Camera Suite app to start recording

12. Press the green or blue 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.
    - **NOTICE:** If you need to abort the injection before it completes, immediately unplug the AIRA power.
    - If you mistakenly press the wrong button, **do NOT abort the injection**. A button mis-press is inconsequential. Simply allow the injection to complete then press the correct button.
13. Watch for an ignition event for 10 minutes
    - Upon injection, a small temperature drop is always observed. This is the start of the 10 minutes
    - A temperature rise above the initial temperature indicates an exothermic reaction has occurred
    - An ignition event is defined by the presence of a visible flame (visible to either the camera or the operator)
    - An ignition event is characterized by a large, sharp temperature rise exceeding $15\frac{K}{s}$. This is referred to as a temperature spike
    - The experiment ends when one of the following criteria is met:
      - An ignition event is observed and the temperature returns to a steady state
      - 10 minutes pass from the time of injection with no ignition event observed
    - Stop and power off the camera with the Capture app about 10 seconds after a temperature spike occurs or after a visible flame disappears
    - If temperature spike is observed, allow enough time for the temperature to return to a steady state before terminating temperature data collection
    - If no ignition event is observed nor expected after a temperature rise reaches a maximum, the camera may be stopped before 10 minutes have elapsed
    - The TADA may be used to keep track of time. A timer is also provided for that purpose.
14. If necessary, review the camera footage, looking for a flame corresponding to the temperature spike
    - This may be done by downloading the footage over the camera WiFi using the Camera Suite app
15. Record pertinent data and observations in the lab book and the TADA **BEFORE** terminating temperature data collection
    - The date of the experiment must be recorded once in the notebook
    - The following data must be present on the same row in the lab notebook, in the following order:
      1. Time of day that data collection began
      2. Compound name
      3. Sample size in microliters (for liquids) or milligrams (for solids and gases). Solids will have an exact initial and final mass included instead of nominal mass.
      4. Set-point temperature of the furnace
      5. Test temperature in degrees Celsius (NOT rounded)
         - This should be the internal flask temperature (Thermocouple 4) prior to injection
      6. Indicate the type event that took place ("h" or "hot" for hot-flame ignition, "c" or "cold" for cool-flame ignition and "n" or "none" for no ignition)
         - If the flame is bright yellow/orange, this is considered a hot-flame autoignition
         - If the flame is faint or blueish, and does not show any yellow, orange, or red color, this is considered a cool-flame autoignition
    - 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 (There is a 'notes' section in the TADA UI window for this purpose)
- Record the same data in the corresponding fields in the TADA UI **before** terminating temperature data collection
- The lot number and/or sample number of the compound container with the supplier and any other pertinent information related to the source of the compound should be recorded at least once in the lab notebook when first being used. Ensure that these data are present.
- Likewise ensure the lot number of the air cylinder being used is also recorded when it was first brought into use.

16. After the experiment ends, terminate data collection
    1. Press the Enter key again to stop data collection (the red light on the TADA should stop blinking)
    2. If you haven't already, press "Recording" button on the Camera Suite app to stop recording
    3. Shut down the camera from the Camera Suite app
17. Set furnace to next temperature
    - **Note for unpressurized experiments:** If you are performing unpressurized experiments (i.e. experiments with the lid off) you may safely skip the next **four** steps. Ensure before proceeding that the vessel is being vented by the snorkel.
18. 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
19. After the purge time has ended, depressurize the vessel
    1. Turn off the inlet air flow using the ball valve
    2. Fully open the rotameter by gently turning the knob counterclockwise
    3. Wait until the pressure vessel is **fully** depressurized (i.e. the rotameter reads zero)
20. Remove the pressure vessel lid by **first** loosening and disengaging the clamps and **secondly** removing the safety cable
    1. Loosen the clamps using a 3/4" wrench
    2. Break the seal on the lid by briefly lifting the lid with the safety cable still in place
    3. Remove the safety cable from the vessel
21. **Simultaneously** lift off the lid and place snorkel inside the pressure vessel between 6" and 10" over the furnace (this may be done with 1 person)
    1. Lift the hood sash to allow the lid to be placed back inside
    2. Pull the bell of the snorkel up to allow the snorkel to swing up once the lid is removed
    3. Swing the snorkel to a position above the vessel
    4. Pull the bell back down to the end of the snorkel and let it fall on top of the lid so the snorkel will fall into place once the lid is removed
    5. Ensure the snorkel is loose enough to fall down into the vessel upon removing the lid
    6. Remove the lid by pulling directly upward before moving laterally and allow the snorkel to fall down into the venting position inside the vessel
    7. Place the lid carefully back in the hood and close the sash, running the lid outlet hose through the small opening in the corner of the sash
    8. Ensure the snorkel is placed properly and is venting the vessel
22. Remove the camera from the vessel

- Connect it to the computer to begin video extraction if you haven't done that already
- Connect the camera to power to charge if necessary

23. Remove any syringe or weigh boats used in the previous experiment
    - **Use gloves when doing this**
    - For experiments with solid compounds:
        1. Carefully remove the funnel and the weigh boat avoiding losing any residue from both of them
        2. Using a **clean** chemical spatula, carefully scrape any compound residue from the funnel into the weigh boat, cleaning the funnel of as much residue as possible
        3. Place the weigh boat on the scale you tared earlier
            - The mass displayed should be negative, this is the approximate mass that actually entered the flask multiplied by -1
        4. Note this weight as the final sample size in the lab notebook
        5. Rinse out the funnel of any remaining residue with acetone
        6. 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 (See Section 4.2). Remember to delete the video off the camera once you have made sure that it is saved to the computer.
26. Once the next temperature is reached, allow the system to come to equilibrium
    - The "Temp Ready" indicator in the TADA User Interface window should turn green when the system has come to an acceptable equilibrated state
27. Once the system is at equilibrium, start this procedure over from step 1 (Measure Out Sample)

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. Shutdown TADA
    1. Close the TADA User Interface program
    2. Unplug TADA's USB connection
    3. Unplug the wall power from the TADA power supply
3. Shutdown ARIA and store accessories
    1. Unplug the ARIA power supply cable
    2. Remove and store any ARIA accessories used that day (leave the ring stand in place)
    3. Clean the funnel with appropriate solvents and dispose of the waste
    4. Appropriately, discard the contents of beakers and prepare them for dish washing
    5. Discard any residual sample in syringes and store them in the syringe box in the AIT drawer without rinsing
    6. Store all chemicals in the appropriate cabinets
4. Remove any organic solid residue from working surfaces (See Section 5)

5. Ensure all air systems are depressurized

    1. Ensure the ball valve is closed (the handle should be perpendicular to the flow)
    2. Slowly close the cylinder valve all the way
    3. Open the ball valve by turning the handle parallel to the flow
    4. Wait until both regulators depressurize
    5. **Leave the ball valve open**

6. Extract all data to the computer and appropriately rename them (Refer to Section refsec:data)

7. Determine if the flask should be changed, if so write this down in the lab notebook

8. Turn the camera Wi-Fi off then shut down and unplug the camera

9. Close all programs and shut down the computer

- 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 Experimental Design

When performing AIT measurements researchers will have relatively small sample sizes to work with therefore following guidelines apply to minimize the amount of compound needed to effectively ascertain the AIT.

3.1 Sample Size Procedure

There are 5 standard sample sizes specified by the ASTM method they are as follows:

- For solids: 50, 70, 100, 150, 200, and 250 milligrams (mg)
- For liquids: 50, 70, 100, 150, 200, and 250 microliters (µL)
- For gases: 50, 70, 100, 150, 200, and 250 milligrams (mg)

Acceptable errors for these sample sizes is +/- 10 mg/µL.

For any compound measurement, there must be a minimum of 3 sample sizes tested. The following steps must be observed:

1. Start with as sample size of 100 mg/µL and find the minimum AIT (explained below).
2. Always do 150 mg/µL next and find the minimum AIT for that sample size
3. Compare the minimum AIT's from the first two sample sizes:
   - If 100 mg/µL gives a lower AIT, do 50 mg/µL next.
   - If 150 mg/µL gives a lower AIT, do 250 mg/µL next.
4. Find the minimum AIT for the third sample size.
5. Compare the minima from all three experiments and determine if further tests are needed:
   1. Find the % error between the lowest and the other two using the following formula $\%Error_i = \frac{|AIT_{lowest} - AIT_i|}{AIT_{lowest}} * 100\%$ where AIT_{lowest} is the lowest AIT between the three and AIT_i is the AIT of one of the other two. You should get two error values.
   2. If both error values are $\leq 2.0\%$ then report the lowest value
   3. If either error values are $> 2.0\%$ then further tests are needed
      - If you did the 50 mg/µL sample size, find the minimum AIT with a sample size of 70 mg/µL
      - If you did the 250 mg/µL sample size, find the minimum AIT s with a sample size of 200 mg/µL
6. Report the minimum AIT found between all of the sample sizes

3.2 Finding the minimum AIT

The methodology for finding a minimum AIT for a particular sample size involves a bisection method described as follows:

1. Do the first experiment at a reasonable temperature.
   - Usually this involves choosing a starting temperature based on a predicted value or looking at family plots of the compound.
2. Bracket the minimum AIT by finding a temperature where ignition was observed and one lower temperature where no ignition was observed.
   - If the initial temperature produced a hot-flame ignition, decrease the temperature.
   - If a cold-flame ignition was observed, decrease the temperature to find a non-ignition then increase to find a hot-flame ignition.
   - Begin by changing the temperature by at least 10 K. If this new temperature does not successfully bracket the minimum AIT, double the temperature change until a bracket is found.

    - E.g. Suppose you have no ignition at 450 K. Increase to 460 K produces no ignition. Increase to 480 K produces no ignition. Increase to 520 K produces a hot-flame ignition. Your bracket is $480K < AIT \leq 520K$.

3. Once the minimum has been bracketed, bisect the temperature space until the bracket size is $\leq 3.0K$.

    - E.g. if you have an ignition at 450 K, a non-ignition at 430 K and no measurements between the two, your next experiment should be at 440 K. Suppose that experiment ignites. The next temperature should be at 435 K. If that does not ignite, the next temperature should be at 437.5 K. If that ignites, then you have successfully bracketed the minimum between 437.5 K and 440 K with a bracket size less than 3 K.

4. The last step is to confirm the minimum. To ensure the minimum has been found, at least 3 experiments must confirm that there is no ignition observed within $3.0K$ below the minimum ignition temperature. If lower ignition temperatures are observed, continue doing confirmation experiments until there are at least 3 non-ignitions within $3.0K$ below the lowest observed ignition value **that were NOT part of the bisection process**. If cold ignitions are observed, they may be considered the same as non-ignitions for the purposes of finding the hot-flame AIT.

5. The lowest temperature where a hot-ignition was observed is the reported AIT for that sample size.

4 Data Extraction

During experiments data are being recorded on the lab computer, the data logger and the camera. Both the camera and the data logger 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.

4.1 General Policies

- All data, including video and raw temperature 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
- The datalogger data is there as a redundant backup to the UI data in case of data loss. Therefore it will be mainly archived and used only when the original data cannot be found
- After processing, all data should be organized according to the following conventions:
  - Path: `$PREFIX/compound_name/filename.ext`
    - `$PREFIX` for video data: `smb://pgl6ed.byu.edu/aitra/video`
    - `$PREFIX` for all other data: `/home/aitra/Documents/data`
  - All experiments should have a unique filename associated with them according to convention
  - All data from the experiment run should have the same filename but different extensions
  - The corresponding data from the datalogger will be archived and accessed as needed
    - Path: `/home/aitra/Documents/Data`
  - When processing is finished, all experiments should have the following 3 files with the same name preceding them
    - A .png/jpg) file (for temperature data with graphs and analysis)
    - A .csv file (TADA-generated)
    - A .avi/.mp4 file stored on the DIPPR Legacy Server (this video file will have a different path and extension but the same filename)
- File naming convention:
  - Filenames will be organized by the following values in order separated by underscores (‘`_`’)
    1. Compound name
    2. Date of experiment with the format “YYMMDD”
    3. Time of day that data collection began for that run using a 24 hour clock format “hhmm”
    4. Sample size in microliters (for liquids) or milligrams(for solids and gases)
    5. Test temperature in degrees Celsius (rounded to the nearest integer)
  - For example: The file name of an AIT experiment where 100 microliters of hexane were tested at 450 degrees Celsius on March 19, 2013 at 4:25 pm would be: `hexane_130319_1625_100_450.csv`
  - The corresponding video file would be named: `hexane_130319_1625_100_450.MP4"`

4.2 Video Extraction

The following procedure is necessary only if you have not or cannot extract and delete video via WiFi (See Section 6.2.3).

1. Connect the camera to the computer via a micro USB cable (See Figure **??**)
2. Press the “info/wireless” button on the camera to connect the camera to the computer
3. A new icon should appear allowing you to access the SD card as if it were a USB drive.

4. Video files should be copied to the DIPPR Legacy Server (a.k.a. The Properties of Gases and Liquids 6th Edition Server) and organized as explained above
   - Path: `smb://pgl6ed.byu.edu/aitra/video`
   - Domain: `dipprleg`
   - Username: `aitra`
   - Password: `hotflame16`
5. Once you have ensured all video data have been have been properly saved in the appropriate data folder, delete all files from the camera

If you have trouble with the above method, you may remove the SD card and copy the video to the server using a similar method as below.

4.3 Datalogger Extraction

To extract data from the datalogger:

1. Unplug the TADA from the computer
2. Pull out the SD card from the data logger 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`)
3. Path: `/home/aitra/Documents/data/raw_data/`
4. Once you have ensured the data log file has been copied and renamed successfully, delete the `DATALOG.CSV` file on the SD card (the SD card should be empty)
5. Close all windows with the USB SD card adapter open (i.e. Windows Explorer etc.)
6. Pull out the SD card without ejecting the unit from the computer

5 Spill Clean-up

In the event of any spill, appropriate PPE specified in the corresponding SDS should be used in clean1.up. Always check the SDS for special considerations when cleaning up any compound.

5.1 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 left1.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 refsec:e_shtdn), 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

5.2 Solids

We will generally work with organic solids that readily dissolve in simple organic solvents (e.g. acetone). Researchers must always check chemical compatibility 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

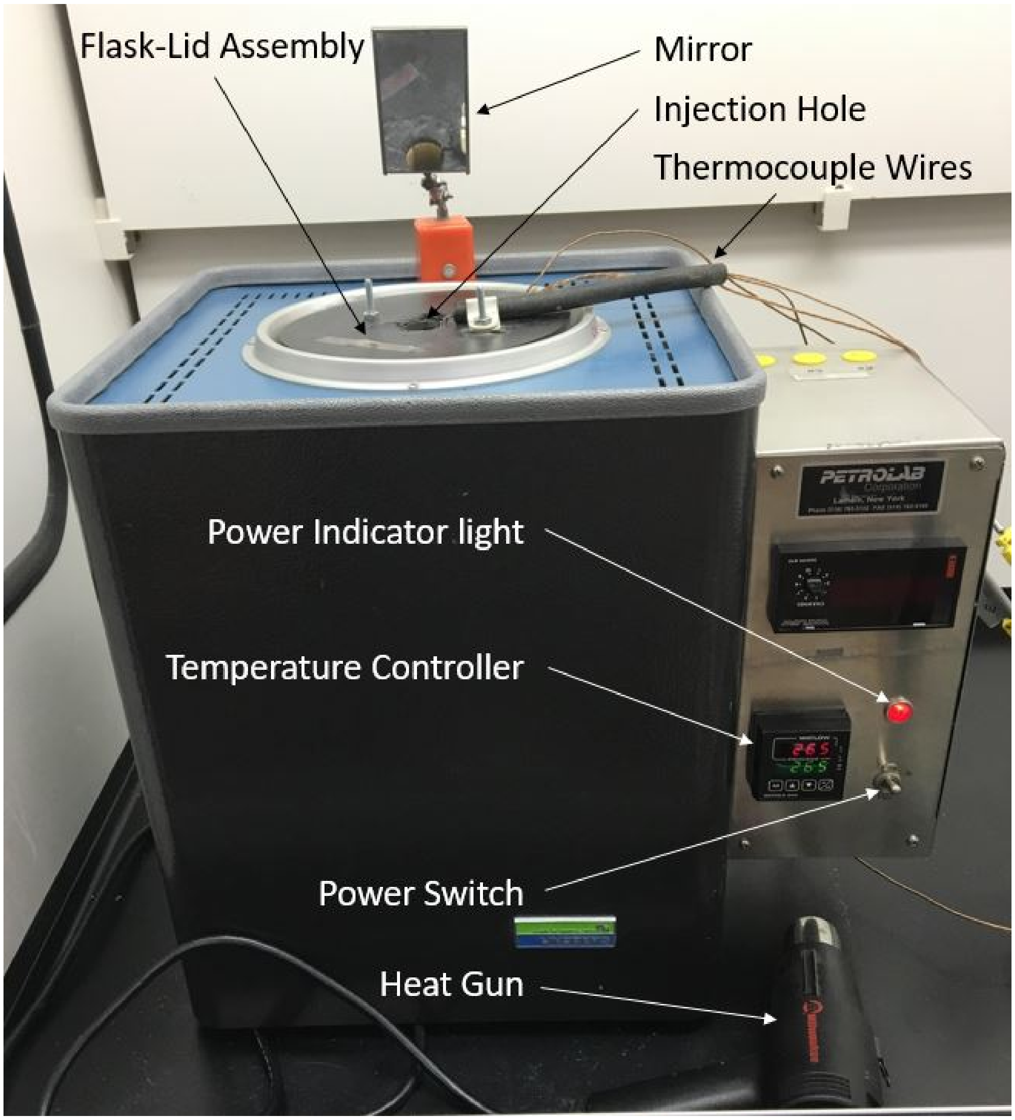


Figure 7: AIT Furnace

6 Experimental Setup and Maintenance

6.1 Furnace

6.1.1 Overview

The furnace, shown in Figure 7, is an encased stack of ceramic insulation with cavities cut out to allow space for the heating elements and the test flask (See the Figure 11 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 set point temperature and the flask temperature will almost always differ significantly (as much as 25 K in some cases). Therefore, set points 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.** 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 enough time for thorough thermal equilibration in the flask; allow extra time during initial start up.

6.1.2 Furnace Operation

See Figure 7 for reference on how to operate the furnace

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

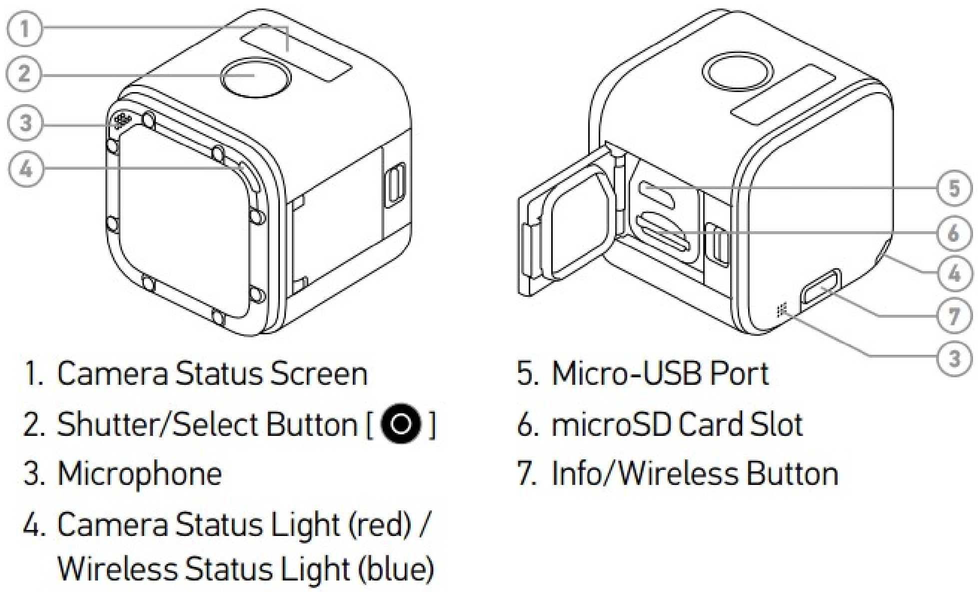
6.2 Camera

6.2.1 Overview

Prior to using the experimental setup, all researchers must become familiar with basic use and operation of the GoPro© HERO4 Session™ camera and Camera Suite© software.

GoPro Login:

- **username/email:** dipprlab.ait@gmail.com
- **password:** hotflame16



6.2.2 Connecting to the camera

1. Firmly press and release the "info/wireless" button on the back of the camera (not the red circle) multiple times until you see `APP` or `APP & RC` on the camera status screen
2. Press the "shutter/select" button (the button with the red circle) to confirm your selection
3. The "wireless status" (blue) light will begin flashing. This indicates the camera is broadcasting a Wi-Fi signal
4. When first powering on the camera, ensure it is sufficiently charged. If not, immediately plug it in to charge it.
5. In the lower-right-hand corner of the desktop you will see an icon for internet connections. The computer should automatically connect to the Wi-Fi but if it does not, connect to the Wi-Fi using the following credentials:
   - Network Name: `ait_cam_2020`
   - Password: `hotflame16`
6. Open the Camera Suite© app (There should be a shortcut on the desktop.)
7. If the app does not immediately try to connect to the camera, go to the pop up dialogue box and select `Hero 4` from the drop-down menu and click on `connect to camera`.

- The app should attempt to connect to the camera.

8. Press the “info/wireless” button to make the camera accept the connection from the computer.

9. On the right side of the Camera Suite© app, the camera info along with control buttons should appear. This means the camera is connected.

6.2.3 Camera Operation

All operations may be done remotely 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 GoPro© HERO4 Session™ camera operation manual in the `docs` section of the `ait_exp` repository.

1. The “shutter/select” button toggles recording or standby; the camera will automatically shut off after a few seconds on standby

2. If the camera is remotely controlled, the on screen red button toggles recording or standby

3. During recording, the camera will not allow viewing via preview mode. This is due to the high frame rate of our experiments

4. Captured video may be reviewed, downloaded and managed remotely with camera browser button on the top left corner of the screen (make sure you hit “refresh” to update the contents in the camera).

5. 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

6.2.4 Shutdown

1. Press the “info/wireless” button until the camera status screen reads “Turn Wi-Fi Off”

2. Press the “shutter/select” button to confirm your selection

    - The “wireless status” (blue) light will stop flashing

3. Press the “info/wireless” button until the camera status screen reads “Exit”

4. Press the “shutter/select” button to confirm your selection

5. The camera will shutdown

6.2.5 Camera Placement and Removal

To mount the camera on the furnace:

1. Lift the tab on the corner of the camera cage

2. Insert the camera from the front of the cage with the “shutter/select” button facing up.

3. Snap the tab back to lock the camera into place.

To remove the camera from the furnace:

1. Lift the tab on the corner of the camera cage

2. Slide the camera out of the cage.

6.2.6 Batteries

1. USB chargers and cables are available for the camera

2. Charge batteries as needed. A charge above 80% is required before running an experiment to ensure the battery doesn’t die during an experiment.

3. Do not overcharge the camera battery. Do not leave any battery charging overnight.

Figure 8: Foil Sandwiches between the O-ring and the TC outlet

6.3 Pressure Vessel

6.3.1 Changing the rupture disk

We use 1100 alloy extra thin aluminum foil as a rupture disk. This material has been shown to be effective in preventing catastrophic failure in the vessel. This section outlines how to change and test the rupture disk. (See Figures 1 and 8 for reference)

1. Remove the O-ring and the TC outlet ring from the pressure vessel by removing the TC clamp (See Figure 1)
2. 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**
3. Rip the foil around the O-ring so it roughly matches the area of the O-ring
4. Sandwich the ripped foil in between the O-ring and the outlet ring
5. 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
6. Secure the assembly on the pressure vessel using the TC clamp
7. The foil should appear smooth across the rupture surface without any folds or crumpled spots
8. The studded surface may still be apparent; this is normal

6.4 Rotameter

6.4.1 Disassembling the Rotameter

1. gently squeeze plastic cover and pull off
2. using a small Allen wrench (~ 0.060") loosen the set screw on the rotameter dial and remove it
3. remove ring around the valve
4. use a 7/16" wrench to remove the valve assembly at the bottom of the rotameter
5. use 2 3/8" wrench and a 7/16" wrench to remove the inner part of the valve assembly
6. Unscrew the needle valve by hand so the valve assembly is completely disassembled (there should be 3 parts)
7. (DO NOT REMOVE THE O-RINGS)
8. use a 5/8" wrench to remove the brass hose fitting at the inlet to the rotameter
9. use a small screwdriver to pry off the plastic cap at the top of the rotameter
10. use a 1/8" Allen wrench to loosen the top screw over the rotameter sight glass to allow the sight glass to be removed
11. carefully remove the sight glass by tipping the top away from the rotameter and pulling up out of the bottom portion

6.5 Flask and Lid

6.5.1 Notes about Flask and Lid

- **Latex or nitrile gloves and safety glasses are required while working with the flask/lid assembly**
- **Always store bulb flasks on the drying rack above the sink or appropriately secured to a ring stand (See Section 6.5.4)**
- Using the TADA_UI, check the temperature of the furnace to ensure safe handling before changing the flask
  - 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 $40^{c}ircC$
- 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 "confirmation" measurements should be repeated with a clean flask

6.5.2 Disassembling the Flask and Lid

NOTE: The furnace may be too hot to open for several hours after an experiment

1. Unplug the thermocouples from the furnace
2. Once the furnace is cool, remove flask/lid assembly
   1. Loosen (do NOT remove) the nut that secures the bracket and the rubber hose to the top of the furnace with a wrench
   2. Move the bracket out of the way and remove Thermocouple 4 (along with the rubber hose) from the top of the furnace
   3. 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
   4. Grip the assembly with both hands by the screws on top and pull directly upward
   5. **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**
3. The flask/lid assembly should easily come out of the furnace without catching on anything
4. **Carefully set the assembly on a table or other stable surface with the flask on top (See Figure reffig:f_lid_done)**
5. Ensure the bracket screw is loose
6. Remove the circular spring from its groove and slide the ceramic halves of the lid apart sufficiently to allow the flask to be removed
7. Remove flask from lid assembly and remove all of the aluminum foil and thermocouples from the flask
8. 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
9. Check the thermocouples and thermocouple wiring for damage or fraying that may affect thermocouple performance. If needed, replace the thermocouples before assembling the flask and lid.

6.5.3 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.)

1. If dirty, wash out the flask using soap and water and dry as much as possible (See Section 6.5.4); be sure to rinse thoroughly
2. Any leftover water will boil away when the furnace heats up and before any measurements are taken
3. 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 9) **NOTE: The more reflective side of the foil should always be facing inward**
    1. Start by getting a long strip of aluminum foil (12" long or so)
    2. Use a utility knife or pin 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)
    3. 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
    4. 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)
    5. 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)
    6. Add an additional layer of foil around the flask so the wires are covered and run parallel when wrapping is finished (5)
    7. Wrap additional foil around the neck of the flask to cover it completely and secure flask in lid assembly
    8. 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)
4. Ensure the bracket screw is loose
5. 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
6. 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
7. 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
8. Use a circular spring to help hold the halves together
9. Make a "donut" of foil wrapped around the neck of the flask that will rest up against the bottom of the lid assembly
10. Slide the foil "donut" up so and press it so it is flush against the ceramic and restricts air flow around the opening
11. 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

Figure 9: Steps for wrapping the flask in foil

- If the flask falls out, remove it and add more foil around the neck

12. See Figure 10 for the final flask/lid assembly before insertion into the furnace
13. 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
14. Turn the flask/lid assembly so the thermocouple wires point away from the ARIA
15. Insert flask interior thermocouple (#4) carefully down the flask neck, making sure it goes straight in and the bead doesn't get caught anywhere
16. The bead of Thermocouple 4 should be suspended in the approximate center of the flask, not be touching any part
17. 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
18. Use the bracket on one of the two screws on top of the lid to secure the rubber hose holding the thermocouple in place
19. Tighten the nut on the bracket hand tight and then give a half turn with a wrench to secure the nut (See Figure 10)
20. 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
21. Ensure the mirrors are set up correctly
22. The final setup should resemble Figure 11

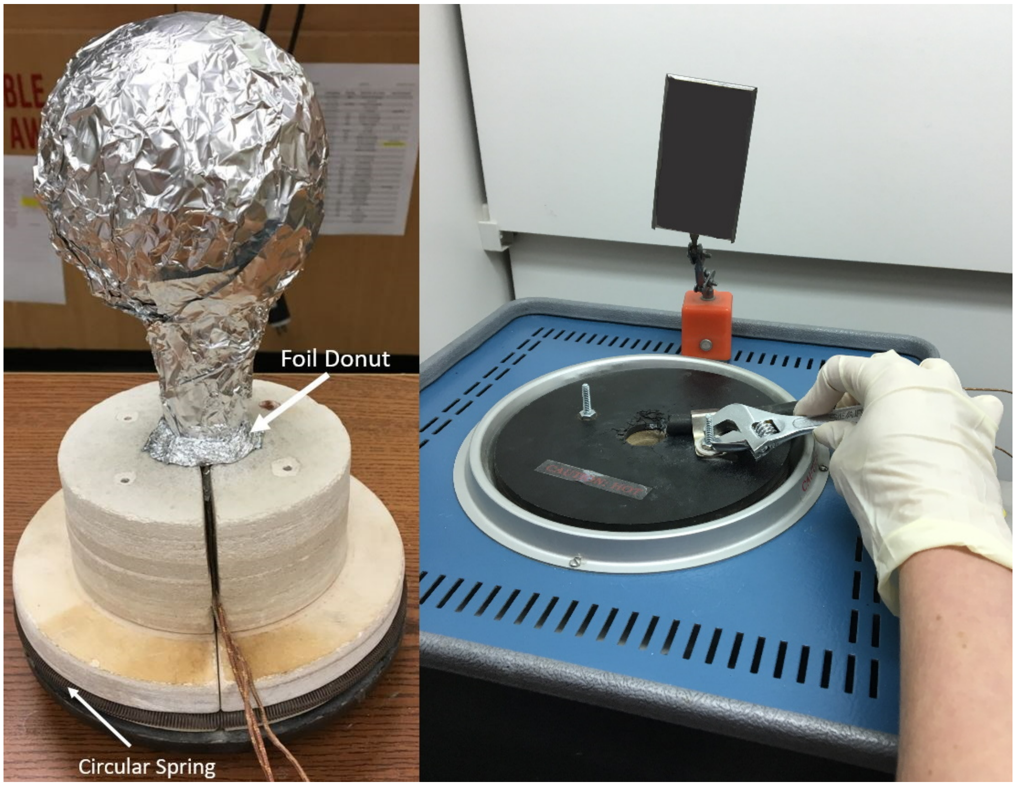


Figure 10: Final state of the flask/lid assembly; Tighten hose clam with a wrench

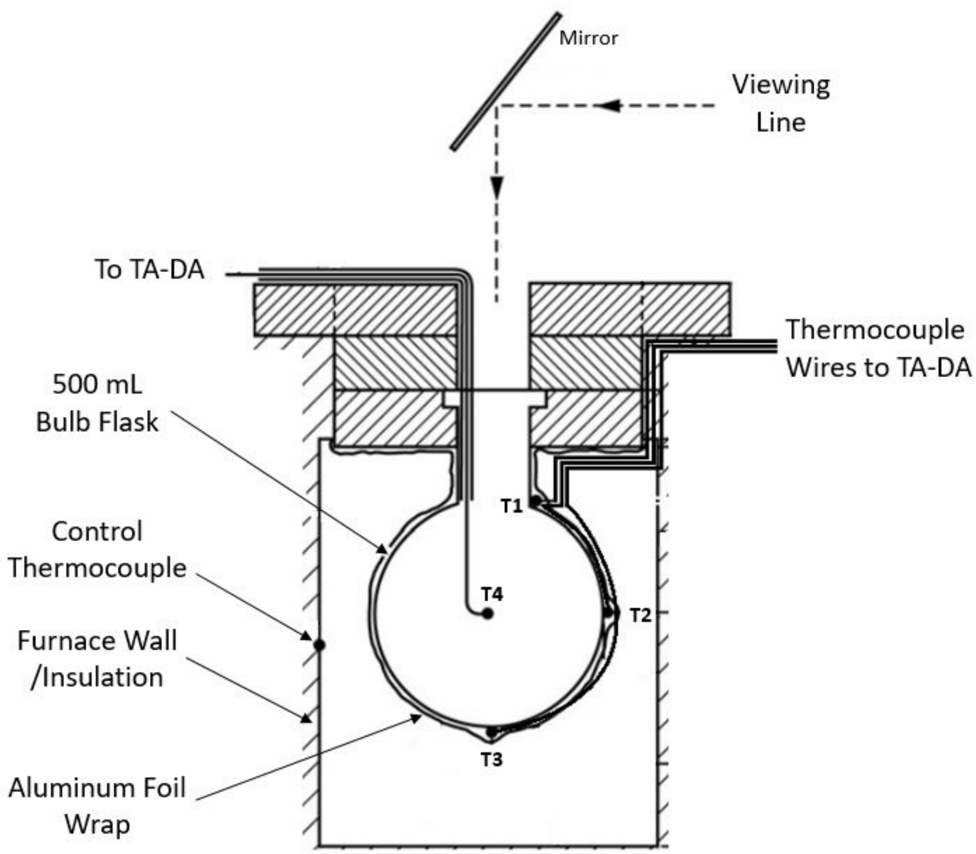


Figure 11: Diagram of the furnace when assembled

6.5.4 Flask Cleaning

For consistent experimental results, flasks must be as clean as possible (See Figure 12). 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:

1. Always begin by soaking the inside of the flask with soapy water for 12 - 24 hours, regardless of how dirty it is
2. While soaking, the flask should always be secured to a ring stand
3. Wash out flask with soap and water, scrubbing the inside with tube brushes
4. For difficult stains, soak the flask inside with soapy water for another 24 hours or longer if needed
5. During this process, scrub the inside and replace the soapy water on a regular basis (generally every 12 - 24 hours)
6. 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*
    1. 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
    2. Repeat this process with distilled water available from the smaller tap on the Northeast corner of the lab sink
    3. 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
7. Once the flask has been cleaned and rinsed thoroughly, place the clean flask on the drying rack over the sink

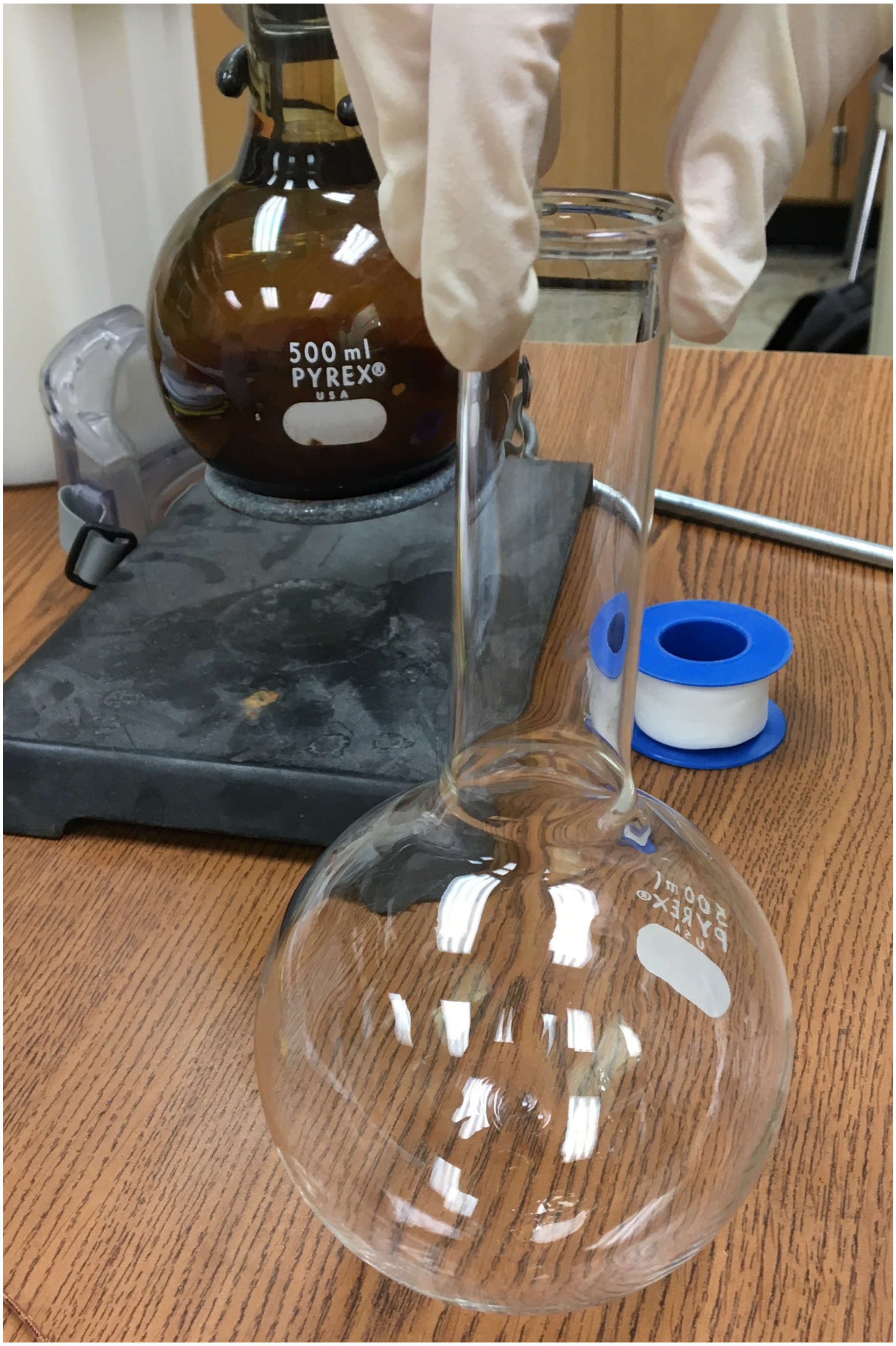


Figure 12: A clean flask (dirty flask in the background)