AIT Measurement Standard Operating Procedure

Last modified on: May 23, 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 Introduction

This SOP outlines the established procedure for performing autoignition temperature (AIT) experiments in Dr. Wilding's laboratory (CB 343A), including detailed instructions on how to perform experiments and corresponding safety protocols. As such, lab policy requires every person working on AIT to have read and become familiar with the most updated version of this document and affirm this by signing the AIT SOP Signatures Sheet.

This document is intended to outline experimental procedures that conform to ASTM Standard E659. If any part of the experimental procedure violates the standard, that part should be changed promptly to conform to established guidelines in ASTM Standard E659. The only exception to this rule is when conforming to the standard would present a significant hazard or risk.

This document will be updated continually as changes to the process are made or improvements are found. If you find an error in this document or have a suggestion to improve it please contact the last person who modified it to submit your proposed changes.

2 Safety and Hazard Mitigation

Prior to performing experimental work, all researchers should be familiar with this section, which outlines the hazards considered in these experiments as well as the steps taken to mitigate them. While reading the remainder of this SOP, researchers should note which steps in the procedure control for these hazards. If there are hazards that could be better mitigated, researchers should propose appropriate changes to the SOP.

There are three primary hazards that exist in this experiment. These hazards are listed below with corresponding general protocols that have been implemented to mitigate them.

- Electrical shock from furnace, heat gun, computer or other sources
  - Researchers will receive corresponding training about safe use of electrical equipment and follow established standards for electrical safety including lockout-tagout protocol
- Exposure to toxic chemicals via skin or eye contact, inhalation or ingestion
  - Researchers will receive corresponding training about safe handling of chemicals
  - Researchers will use appropriate PPE when handling any chemical based on recommendations from the corresponding SDS and lab policy
  - All handling of volatile chemicals will take place inside one of the ventilated hoods in the lab
  - As per ASTM Standard E659, small amounts of compound will be used in any one experiment (Maximum amounts for any one experiment are limited to 1 milliliter for liquids and 1 gram for solids and gases)
- Fire or explosion of flammable chemicals
  - Researchers will receive corresponding training about fire and explosion safety and prevention
  - Experiments will take place in a ventilated hood
  - A fire extinguisher is available in the lab in the event of a small fire

– As per ASTM Standard E659, small amounts of compound will be used in any one experiment (Maximum amounts for any one experiment are limited to 1 milliliter for liquids and 1 gram for solids and gases)

3 Experimental Setup and Maintenance

3.1 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
    - 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" below)

- 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" below); 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 hole 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 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

- Loosen the nut on top of the lid assembly and slide the corresponding half of the ceramic part of the lid assembly out
- 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 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 where researchers will be working
- 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)

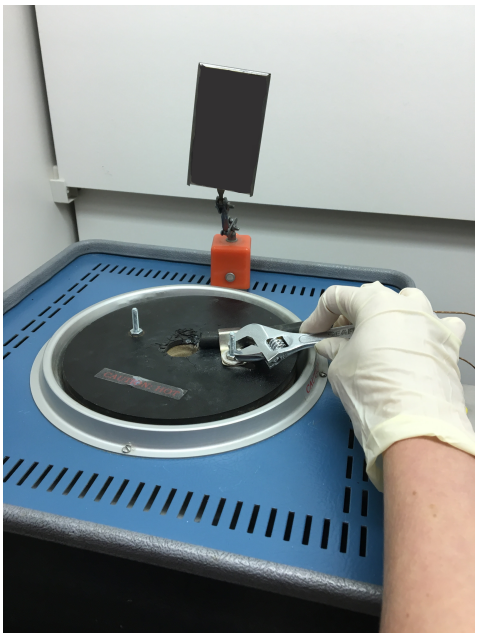

Figure 3: Position thermocouple 4 with the rubber hose and tighten

– Connect the thermocouples to the TA-DA
– The final setup should resemble Figure 4

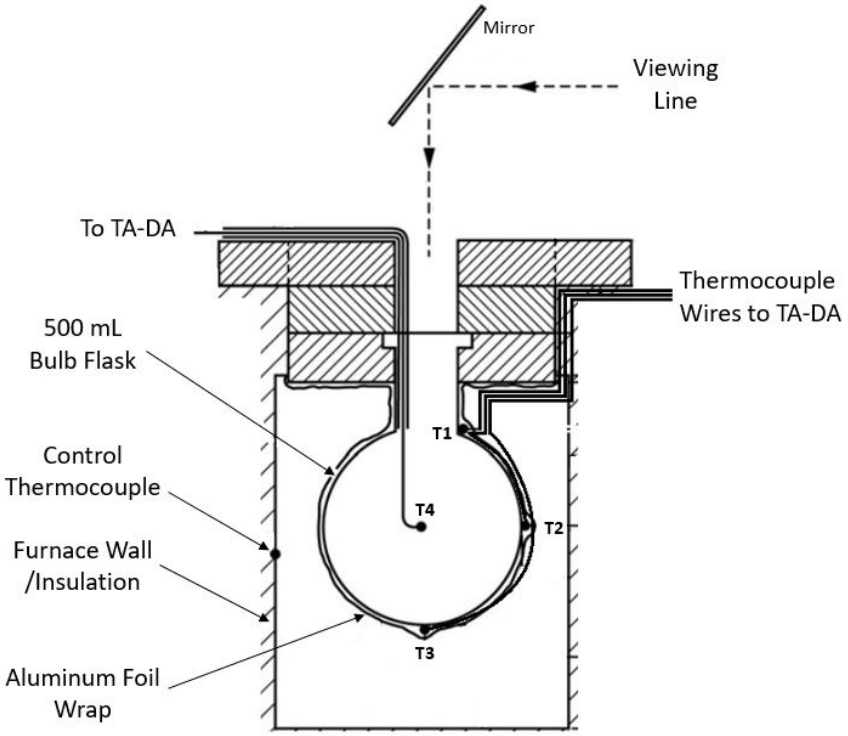

Figure 4: Diagram of the furnace when assembled

- 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)

3.2 Furnace

- 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 measurments 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**

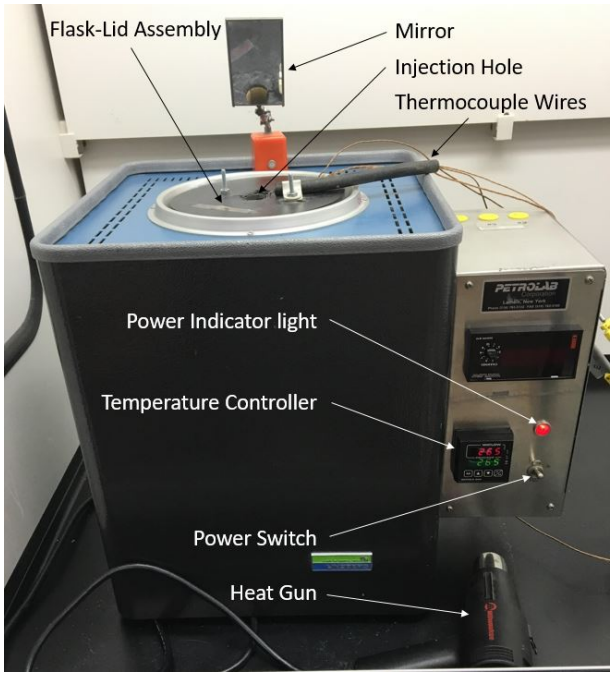

Figure 6: AIT Furnace

- When powered on initially, the furnace may take up to 2 hours or more to reach a desired temperature and thermally eqilibrate
- 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
- Furnace Operation (See Figure 6):
  - Plug in the 220 V extension cord in the corresponding outlet on the wall opposite the hood (adjacent to the DSC computer)
  - Plug in the furnace to the 220 V extension cord
  - 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, unplug the furnace and unplug the 220 V extension cord from the opposite wall

3.3 Camera and Tablet

- 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`

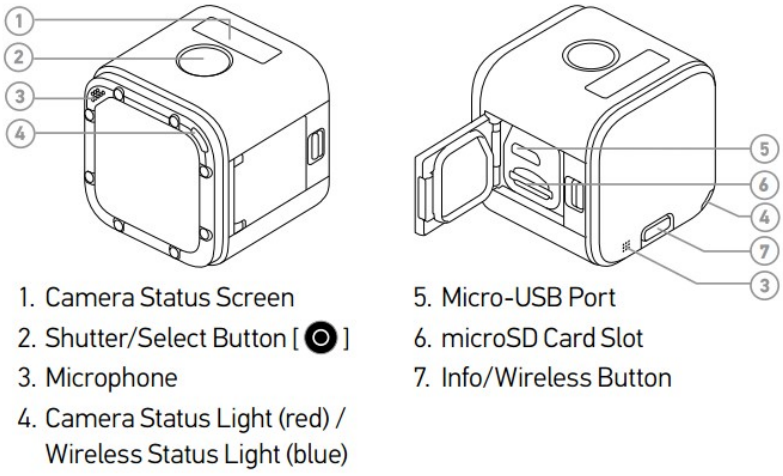

Figure 7: GoPro© HERO4 Session™ Camera Parts

Figure 8: Samsung Galaxy Tab A

- Refer to Figures 7 and 8 for camera and tablet setup
- Connecting to the camera:
  - Press the "info/wireless" button on the back of the camera until you see "APP" on the camera status screen
  - Press the "shutter/select" button 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 until you see splash screen indicating the tablet is booting up
  - 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 labeled "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 labeled "Capture" on the home screen)
  - Select the connect box on the top left corner of the screen
  - 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
- 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 remotly 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 framerate 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

- 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

- Batteries:
  - Recharging power supplies and usb cables are availible 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

4 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.

4.1 Startup

1. Plug in the 220 V extension cord in the corresponding outlet on the wall opposite the hood (adjacent to the DSC computer)
2. Plug in the furnace to the 220 V extension cord
3. Power on furnace and set furnace temperature between 20 - 30 degrees above your initial target flask temperature
4. Reduce the set point temperature when the internal flask temperature exceeds your initial target temperature by 5 - 10 degrees
    - Wait a minimum of 90 minutes before changing the temperature
    - When powered on initially, the furnace may take up to 2 hours or more to reach a desired temperature and thermally eqilibrate
5. 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 me know and I 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*
6. Ensure a compatible SD card is inserted securely into the TA-DA datalogger
7. Ensure the thermocouples are connected to the TA-DA
8. Connect the TA-DA to the lab computer via the USB cable mounted under the edge of the hood
9. Open the TA-DA user interface program
    - Path: *C:\Users\Public\Documents\AIT\tools\TADA_UI.py*
    - You may wish to make a shortcut to this location and put it on your CAEDM desktop.
    - Upon opening the program, a yellow LED in the TA-DA should begin flashing (See Figure 9)

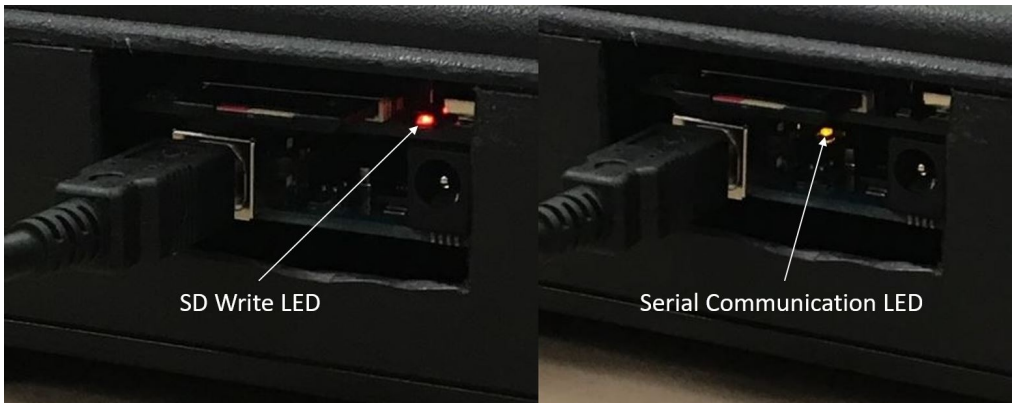

Figure 9: Control LEDs inside the TA-DA

10. Press the "Sync Time" button on the bottom left corner of the TADA_UI window to syncronize the arduino clock to the computer time
    - This must be done at least once every work day
11. Use the TADA_UI program to track the internal temperature of the flask as it heats up initially
12. Prepare the tablet and the camera
    - Power on the tablet and connect it to the camera's Wi-Fi (See Section 3.3 on how to do this)
    - Open the capture app to view the camera's view finder
    - Mount the camera on the tripod in the hood using the quick-release plastic camera mount on top of the tripod
    - Using the camera's view finder on the tablet, adjust the position of the camera, tripod and mirror on the furnace to align the camera's view directly down the center of the flask
      - The camera should be positioned approximately level with the top of the furnace looking slightly upward into the mirror
      - For the best view, the camera should be as close to the furnace as the tripod will allow and pointed directly into the mirror
    - Gently tighten the knobs on the tripod to fix the camera in place
    - Close the hood sash
13. Place a piece of cardboard or other opaque sheet on the right side of the hood against the outer sash and tuck it into the edges of the space between the sash and the hood wall so that it blocks light from entering the hood
14. Once the target temperature is reached, allow 30 minutes for thermal equilibration then begin experiments

4.2 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. Measure the absolute pressure in the lab with the mercury barometer mounted on the west wall by the sink and write down the pressure in the lab notebook

2. Measure the relative humidity and ambient temperature in the hood with the hygrometer and write down those values in the lab notebook

    - Remove the cap from the hygrometer probe and set the probe inside the hood away from the furnace
    - Press the "ON/OFF" button on the hygrometer to turn the unit on
    - Press "MODE" until the unit displays the relative humidity
    - Wait 1 minute to allow the unit to equilibrate
    - Write down the value for the relative humidity in the lab notebook
    - Press "MODE" again until the temperature is displayed
    - Write down the value for the ambient temperature in the lab notebook
    - Turn off the unit by pressing the "ON/OFF" button, remove the probe from the hood and put the probe cap back on and store the unit in its case
    - For more information on the proper use of the hygrometer, refer to the instructions in the hygrometer case
    - Do **NOT** leave the probe sitting in the hood for an extended period of time without its cap on. The hygrometer probe is sensitive to dust and debris and can be easily damaged if left in the hood

3. 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 filename 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 TA-DA for the next measurement

4. **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 left hood to avoid a potential fire hazard

5. Measure out sample
   - Liquids
     - Draw sample amount into a right-angle syringe
   - Solids
     - Measure out sample in a weigh boat on the lab scale
     - Use a powder funnel to inject the sample into the furnace
     - Keep the funnel as close to vertical as possible during injection to minimize the amount of compound that does not enter the flask
   - Gases
     - Draw sample amount into a right-angle syringe
6. Set the measured sample aside in the left hood
7. Remove any glove from your LEFT hand and use your left hand for touching any non-hazardous surfaces
8. Ensure the lab is sufficiently dark to see any flame from the mirror on top of the furnace
   - Use the lights in the hoods for preparation before your run
9. With your LEFT hand, press the Enter key to initiate data collection (a red LED in the TA-DA should begin blinking)
   - The TA-DA 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
10. With your LEFT hand, press the red button on the tablet screen to start recording
11. With your LEFT hand, open the sash horizontally just enough that your RIGHT hand can inject the sample
12. With your RIGHT (gloved) hand, retrieve your sample and center the end of the syringe or funnel in the hole at the top of the furnace for injection, ensuring that the sample will go straight down and not hit the sides of the flask
13. Simultaneously turn off the light in the hood (with your LEFT hand) and introduce your sample (with your RIGHT hand) (the light turning off indicates to the camera that the sample has been injected)

- Immediately withdraw the syringe or funnel, close the sash (with your LEFT hand) and place the syringe in the adjacent hood
- Begin the 10 minute timer
- **NOTE ON EXPERIMENTS WITH SOLIDS:** When experimenting with solids, it is difficult for one person to simultaneously introduce a sample, hold the funnel in place and turn off the light. Therefore for solid experiments, have a second person there to turn off the light as the sample is injected. If a second person is present, he/she must also wear appropriate PPE for the experiment. (i.e. The second person must wear PPE corresponding to the compound SDS but must NOT be wearing gloves when switching off the light.)

  If no one is available to do this, the light may be turned off quickly after introducing the sample and removing the funnel. In this case, the camera should be able to view the sample entering the flask and have an accurate time of injection.

14. Watch the mirror above the furnace for any flame/glow or the TA-DA UI for a large temperature spike for 10 minutes

    - If a flame, glow or spike is observed, stop the camera after the flame disappears and then allow enough time for the temperature to return to a steady state before terminating temperature data collection
      - 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
    - The experiment ends when one of the following criteria is met:
      - An ignition event is observed (i.e. a temperature spike or seeing a flame) and the temperature returns to steady state
      - 10 minutes pass with no ignition event observed
    - If the UI is used to keep track of time and no flame is observed, continue collecting temperature data until 700 seconds have passed to ensure the full 10 minutes of data have been captured

15. Record pertinent data and observations in the lab book and the TA-DA UI

    - The following data must be present on the same row, 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 you or the camera see a flame?)
      - Indicate whether a hot flame or cold flame was observed (if applicable)
      - Indicate if any sound was heard upon ignition (if applicable)
      - The barometric pressure at the time of the experiment (in mmHg)
      - The relative humidity (%) and the ambient air temperature in the hood
    - If any item is not applicable write down N/A in its place

- If any item is unknown, leave a 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 TA-DA 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 TA-DA should stop blinking)
    - Press the red button on the tablet screen to stop recording
    - Turn the lights back on
    - Video recording may be stopped as soon as a flame or glow disappears
17. Prepare for the next measurement
    - Set furnace to next temperature
      - When changing temperature, always approach your target temperature from 5 - 10 degrees Celsius above and then descending slowly to your target temperature
      - This practice is intended to control for hysteresis effects (See Section 6.2)
    - Clean out the flask between measurements by blowing hot air into the flask for 5 minutes using the heat gun
      - The heat gun should **only** be plugged in to the outlet when in use
    - Extract, save and appropriately rename the video data between experiments
    - Wait a minimum of 30 minutes to reach the new temperature and allow the furnace and flask to thermally equilibrate
18. Start this procedure over from step 1 (measure the pressure)

4.3 Shutdown

- The following should be done before leaving the lab at the end of every work day or anytime the setup is not in use:
  - Power off the furnace
  - Unplug the furnace from the 220 V extension cord
  - Unplug the 220 V extension cord from the wall opposite the hood
  - Unplug TA-DA from the computer
  - Tuck the end of the USB cord into the mounted section under the edge of the hood so it does not present a tripping hazard
  - Extract all data to the computer and appropriately rename them
  - Shut down and unplug the tablet and the camera
  - Remove the cardboard from the hood and stow it between the hood and the toolbox
  - Remove the camera from the tripod using the plastic quick-release lever and store the camera next to the lab computer
  - Put all chemicals and syringes away in their proper places

  - Remove any organic solid residue from all working surfaces (See Section 9.2)
  - Close all programs and shutdown the computer
- A hot furnace may be left in the hood without waiting for it to cool
- Do not rinse out needles
- Under normal use, disposable gloves may be thrown into the normal trash receptacle instead of solid chemical waste

5 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 TA-DA 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 w/ 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: $\backslash\backslash dipprlegacy.et.byu.edu\backslash aitra\backslash video\_import$
    * 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)
    * $C:\backslash Users\backslash Public\backslash Documents\backslash AIT\backslash docs\backslash GoPro\_App\_for\_Desktop\_User\_Manual.pdf$
  - Once extracted to the DIPPR legacy server, video data may be timestamped and converted to .avi format on the server (See Section 7)

- To extract data from the datalogger
  - Unplug the TA-DA 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: $C:\backslash Users\backslash Public\backslash Documents\backslash AIT\backslash data\backslash raw\_data$
  - 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

6 Experimental Design

This section outlines basic principles for finding the AIT of a compound from perspective of experimental design. This section must be read at least once to be authorized to work on AIT. However, understanding all the steps in this section is optional to begin experimental work. It is expected that researchers will become familiar with and understand these steps over time after some experience measuring AIT.

The other sections of this SOP concern matters related to saftey and precise, consistent measurement and should be followed exactly to ensure the best results. This section, however, deals with a much more nuanced view of AIT and how to find the minimum value. Because of this, the steps and procedures in this section may be taken as flexible guidelines rather than imperative rules. They should inform decisions made by researchers rather than perscribe them. With this in mind, these are the guidelines for some specifics on how to measure AIT.

The following principles should guide all decisions regarding AIT measurement:

- **The autoignition temperature is defined as the minimum temperature at which hot-flame ignition occurs in the absence of an ignition source**
  - Hot-flame ignition is defined as seeing a yellow/orange/red part of a flame or glow associated with the ignition event
  - Cool-flame ignition is defined as seeing an entirely blue flame or glow associated with the ignition event
- **The reported AIT must be the internal flask temperature (Thermocouple 4) and NOT the temperature of the furnace**
  - The best measure available for AIT in the ASTM method is having the AIT be the air temperature prior to injection inside the flask
- **The bracket size goal for AIT measurement is ± 3 °C**
  - This means that if a hot-flame ignition can be consistently measured at a certain temperature and a cold flame can be consistently measured < 3 degrees below it, the hot-flame temperature should be reported as the measured AIT

6.1 Guidelines for Finding AIT

- A more complete and precise definition of AIT as per ASTM E659: The minimum temperature of a fuel/air mixture at which hot-flame ignition occurs, in the absence of an ignition source, for a system that meets the following criteria:
  - The fuel/air mixture is contained in a 500 ml borosilicate bulb flask
  - The bulb flask is open to the atmosphere
  - The bulb flask is at a uniform temperature
  - Both the bulb flask and the air inside are at steady state temperatures immediately before the fuel is introduced
  - The composition of the air in the flask is 20.95% oxygen with the balance being inert gases

  - The barometric pressure of the system is 1 atmosphere
  - The fuel to air ratio is optimized to minimize the AIT

- The experimental setup has been designed to conform to these criteria insofar as it is practical to do so. Changes will be made continually to the setup to better match these criteria.

- To choose temperatures to be tested the following guidelines may be useful:

  - If there are any data that indicate where the AIT is, start there
  - If there are no data for a compound look at the compound's family and interpolate from those data to choose a starting temperature
  - If there are no data for a compound or its family find a compound with data that most resembles your compound and start at that compound's AIT
  - Once a starting point is found, begin with a broad strokes approach by jumping up and down by 30 degrees Celsius or more until you bracket the minimum AIT with hot-flame and cold flame ignitions
  - Shrink that bracket with a bisection method until the bracket is < 3 degrees Celsius

- Once a minimum AIT is found for a baseline sample size, vary the sample size (See Section 6.3) and repeat the procedure above until you find a minimum AIT with an optimum sample size

6.2 Furnace/Flask Temperatures

- We have observed some hysteresis effects in how we approach the temperature to be tested. We have noticed if we start high and drop down to the desired temperature the AIT tends to be lower than if we start low and heat up to the desired temperature.

- To control for these effects, we strongly recommend always overshooting higher than the temperature needed and then backing off the heat to allow the temperature to fall to the desired value. Doing so should control for the hysteresis effects observed.

- The flask temperature (Thermocouple 4) should be monitored while temperatures change to check for steady state. From tests on the response curve of the furnace, we have determined that the time constant of the system is roughly 10 minutes and the dead time is about 2 minutes which would mean that within 30 - 35 minutes the flask should come close to steady state. If the temperature changes less than 3 degrees Celsius over 10 minutes researchers may always consider the system at steady state.

6.3 Sample Size

In Section 6.1 the guidelines for finding AIT were covered with respect to temperature. This section lays out guidelines for how to choose a sample size and how to store and inject. Before varying sample size, researchers should generally find an AIT for the current sample size

- Liquids

  - Draw sample amount into a right-angle syringe
  - Sample size:

        * Initially use a sample size of 100 microliters
        * Once AIT is measured for 100 microliters, go to 150 microliters
        * If the AIT decreases for 150 microliters, go to 200/250 microliters
        * If the AIT increases for 150 microliters, go to 50 microliters

- Solids
    - Measure out sample in a weigh boat on the lab scale
    - Use a powder funnel to inject the sample into the furnace keeping the funnel vertical to minimize the amount of compound not entering the furnace
    - Sample size:
        * Initially use a sample size of 100 milligrams
        * Once AIT is measured for 100 milligrams, go to 150 milligrams
        * If the AIT decreases for 150 milligrams, go to 200/250 milligrams
        * If the AIT increases for 150 milligrams, go to 50 milligrams

- Gases
    - Draw sample amount into a right-angle syringe
    - Sample size:
        * Initially use a sample size of 100 milligrams
        * Once AIT is measured for 100 milligrams, go to 150 miligrams
        * If the AIT decreases for 150 miligrams, go to 200/250 miligrams
        * If the AIT increases for 150 miligrams, go to 50 miligrams

6.4 Flame and Glow

The following are guidelines for flame observation:

- If the flame is at least partly bright yellow, orange or red, this is the hot-flame autoignition and the temperature should be decreased for the next test
- If the flame is faint and blueish, this is the cool-flame autoignition and the temperature should be increased for the next test
- If no flame or glow if observed by the 10 minute mark, increase the temperature for the next measurement

7 Data Processing and Analysis (Section is in ALPHA)

NOTE: This section is in ALPHA stage of development and does not outline any procedures that are necessary for safety in the lab. Therefore, until this section is taken out of ALPHA stage the material in this section is optional and not considered part of the SOP. This means that you may skip this and any sections in ALPHA stage when reading the entirety of the SOP as required for lab work.

8 Temperature Calibration (Section is in ALPHA)

NOTE: This section is in ALPHA stage of development and does not outline any procedures that are necessary for safety in the lab. Therefore, until this section is taken out of ALPHA stage the material in this section is optional and not considered part of the SOP. This means that you may skip this and any sections in ALPHA stage when reading the entirety of the SOP as required for lab work.

This section outlines the procedure for temperature calibration of the TA-DA. It also includes guidelines for maintaining thermocouples. The practices in this section are intended to ensure minimum uncertainty in temperature measurements. Therefore to ensure consistentcy, researchers should follow procedures outlined here as rigourously as the experimental procedures.

8.1 VA710 Thermocouple Calibrator

The VA710 Thermocouple Calibrator, sold by ThermoWorks, is calibrated to NIST standards by ThermoWorks before shipping and recommends recalibrating the VA710 on an annual basis. The instrument was originally put into service on June 15, 2017. Every June, ThermoWorks should be contacted to recalibrate the instrument to NIST standards. Associated documentation gives specific data on the most recent calibration.

When up-to-date on its calibration schedule, the VA710 should accurately measure and simulate thermocouple voltages. This ensures thermocouple measurement uncertainty remains below the uncertainty inherent in the thermocouples. The uncertainty inherent in the thermocouples is usually specified by the thermocouple manufacturer (usually Omega in the case of the AIT setup).

For detailed instructions on how to use the VA710, please refer to the User's Manual in the inside pocket of the VA710 case.

8.2 Calibration Procedure

The TA-DA should be calibrated to output correct temperatures

9 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.

9.1 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 in the lab to soak up the bulk of the liquid and wipe up the rest with a paper towel
  - Dispose of the clay, any disposable gloves and towels in the solid waste container
  - If the spill occurs outside the hood or the spill is particularly large (e.g. an entire bottle of a flammable material breaks) **perform the Emergency Shutdown Procedure (Section 10), 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

9.2 Solids

We will generally work with organic solids that readily dissolve in acetone. Researchers must always check chemical compatablity with acetone before dissolving any compound in acetone.

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

10 Emergency Shutdown

- In the event of an emergency do the following:
  - Power off the furnace
  - Unplug the furnace
  - 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