Chemistry Department Safety Office: Gilbert Hall Room 153
Emergency Medical Services: 911
Campus Student Health Center: 7-9355
Poison Control: 9-1-800-222-1222
OSU Environmental and Health Safety: 7-2273
Campus Security: 7-7000

DISCLAIMER:

This Standard Operating Procedure (SOP) was developed based on instrument manuals, manufacturer specifications, and laboratory experience to provide guidance to Subramanian Lab users in performing the activities defined in this document, in a consistent and standardized manner. This document does not contain regulatory or statutory requirements unless specified.

The authors of this document have made every attempt to present the information in a clear and concise manner for all users. However, the Subramanian Lab is not responsible for the misuse or misinterpretation of the information presented in this SOP. Under no circumstances shall the Subramanian Lab be liable for any actions taken or omissions made by users of this SOP.

In general, this document should not be used in place of the instrument manual, and should be used as a supplement to procedure and reference to safety. The Subramanian Lab reserves its right to change or suspend any or all parts of this document.

USE OF NETZCH LFA 457

1. Introduction

The NETZSCH LFA 457 MicroFlash® complies with the latest technology for modern laser flash systems. The table-top instrument allows measurements from -125°C to 1100°C using two different user-exchangeable furnaces. The innovative infrared sensor technology employed in the system enables measurement of the temperature increase on the back surface of the sample, even at temperatures of -125°C. The instrument can be used for small and large sample sizes of up to 25.4 mm diameter and, with the integrated sample changer, measurements can be run on several samples at the same time.
The vacuum-tight design enables tests under defined atmospheres.
The vertical arrangement of the sample holder, furnace and detector simplifies sample placement and, at the same time, guarantees an optimum signal-to-noise ratio of the detector signal.

2. Scope

This procedure applies to all staff, students and visitors of the Subramanian Research Group in the Department of Chemistry at Oregon State University that work in the laboratory and have the potential to use the NETZCH LFA 457.

3. Safety

- Safety handling liquid nitrogen
    - Fire and explosion hazards
        - While neither gaseous nor liquid nitrogen pose a fire or explosion risk, gases stored under pressure must not be located in areas where there is a high risk of fire of where they may be exposed to excessive heat. Vessels containing compressed gaseous nitrogen may rupture violently if overheated as a result of exposure to fire.
    - Materials Hazards
        - Certain steels, such as carbon steel, and some other materials are unsuitable for use at sub-zero temperatures because they lose impact strength and become extremely brittle. In an area where liquid nitrogen spillage can occur, care should be taken to ensure that the liquid does not come into contact with vulnerable materials.
    - Health hazards
        - Asphyxia
            - Nitrogen, although nontoxic, can constitute an asphyxiation hazard through the displacement of atmospheric oxygen. Unless adequate precautions are taken, persons can be exposed to oxygen-deficient atmospheres if they enter equipment or areas which have contained or have been purged with nitrogen.
            - Symptoms of oxygen deprivation may be apparent at an oxygen concentration of 16%, and brain damage or death may result if the concentration is 10% or less. BREATHING A PURE NITROGEN ATMOSHPERE WILL RESULT IN AN IMMEDIATE LOSS OF CONSCIOUSNESS AND ALMOST IMMEDIATE DEATH.
            - Persons showing symptoms of oxygen deprivation should be moved immediately to a normal atmosphere. Persons who are unconscious or not breathing should receive immediate first aid. Medical assistance should be immediately requested.
            - A RESCUER SHOULD NOT ATTEMPT TO ENTER AN OXYGEN-DEFICIENT ATMOSHPERE WITHOUT USING SUITABLE SELF-CONTAINED

BREATHING APPARATUS; OTHERWISE THEY THEMSELVES MAY BE OVERCOME.

- Cold burns
  - Liquid nitrogen and cold nitrogen vapors or gases can produce effects on the kin similar to a burn. Unprotected parts of the body coming in contact with uninsulated parts of the equipment may also stick, and may become torn upon removal.
  - Cold burns should receive medical attention as quickly as possible.
- Frostbite
  - Severe or prolonged exposure to cold nitrogen vapour and gas can cause frostbite.
  - Medical attention must be sought out immediately.
- Effects of cold on lungs
  - Prolonged breathing of extremely cold atmospheres may damage the lungs.
- Hypothermia
  - Low environmental temperatures can cause hypothermia and all persons at risk should wear warm clothing.

- Precautions
  - Operations and maintenance
    - It is essential that operations involving the use of gaseous or liquid nitrogen, particularly where large quantities are involved, are conducted in well-ventilated areas to prevent the formation of oxygen-deficient atmospheres.
  - Personal Protective Equipment (PPE)
    - It is recommended that persons handling liquid nitrogen should wear goggles, lab coat, and protective gloves.

- Safety handling gas containers
  - General
    - Only trained personnel should handle compressed gas.
    - Ascertain the identity of a gas before using it.
    - Know and understand the properties and hazards associated with each gas before using it.
  - Handling and use
    - Transportation of gas cylinders must always involve the use of a gas trolley.
    - Screw caps must always be in place before transportation of a gas cylinder.
  - Storage and handling
    - Check for gas leaks using a suitable method for each gas.
    - Containers should be stored in a well-ventilated area.

- Gas containers MUST BE FIRMLY ANCHORED AT ALL TIMES to either the wall or the bench top with suitable straps, clamps, and/or chains.

- Safety Handling liquid storage containers
  - Liquid nitrogen must be obtained and transported in labelled dewar flasks. Two 4-liter flasks are provided in Gilbert 214 for such purposes.
- Laser notes
  - While in use, the LFA 457 is a laser class 1. A Class 1 laser is safe under all conditions of normal use. This means the maximum permissible exposure (MPE) cannot be exceeded when viewing a laser with the naked eye or with the aid of typical magnifying optics (e.g. telescope or microscope). Avoid radiation of eye or skin by direct or leakage radiation.
  - After dismounting the cover, the laser is upgraded to a class 4. By definition, a class 4 laser can burn the skin, or cause devastating and permanent eye damage as a result of direct, diffuse or indirect beam viewing. These lasers may ignite combustible materials, and thus may represent a fire risk.

4. System Specifications

- Temperature range: -125°C ... 500°C, RT ... 1100°C, (2 exchangeable furnace types)
- Heating- and cooling rates: 0.01 K/min ... 50 K/min
- Laser pulse energy: up to 18 J/pulse, (adjustable power)
- Contactless measurement of temperature rise with IR detector
- Measuring range: 0.01 mm2/s ... 1000 mm2/s (thermal diffusivity)
- Measuring range: 0.1 W/mK ... 2000 W/mK (thermal conductivity)
- Sample dimensions: 10 mm ... 25.4 mm diameter (also 8x8 mm and 10x10 mm, square) 0.1 mm ... 6 mm thickness
- Sample holder: SiC, graphite
- Liquid metal holder : sapphire
- Sample holder for liquids: platinum
- Atmosphere: inert, oxidizing, reducing, static, dynamic (Nitrogen gas currently in use)
- Vacuum-tight assembly up to 10-2 mbar (1 Pa)

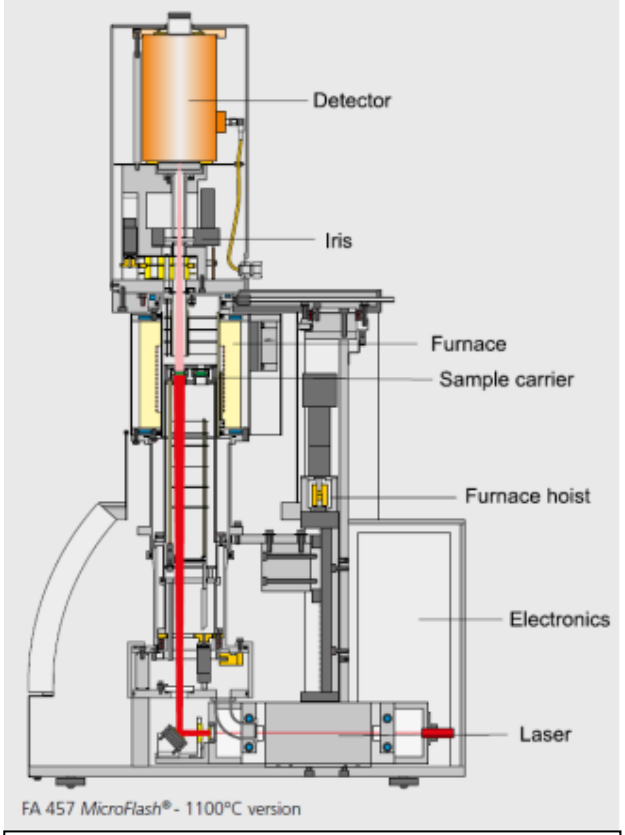

Figure 1. Schematic diagram of the NETZCH LFA 457.

5. Operating Procedures

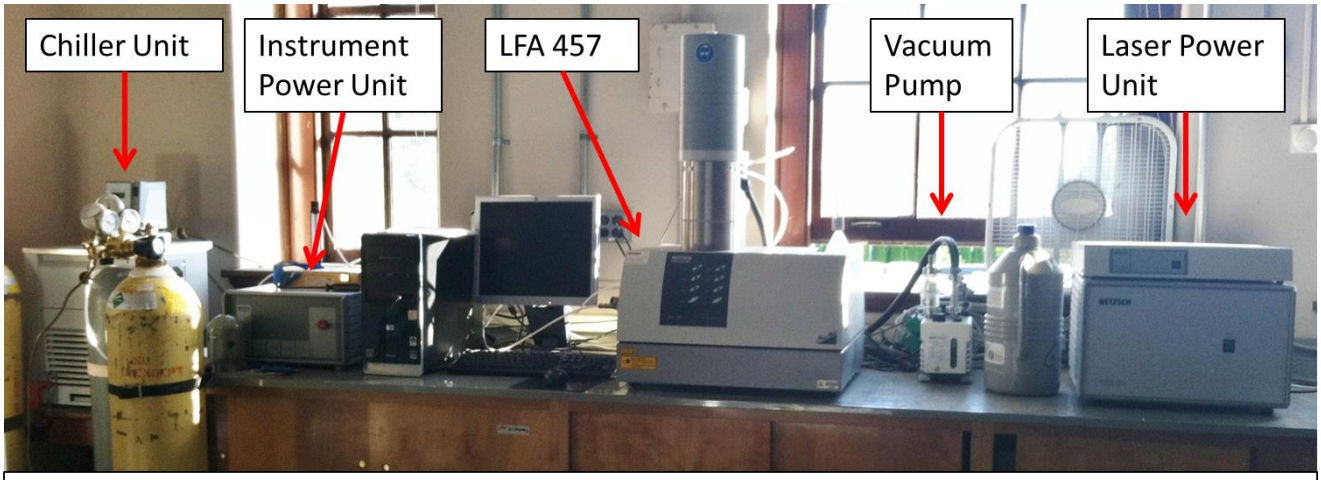

Figure 2. Layout of the NETZCH LFA 457 in the Subramanian Research Group, Gilbert Hall 214, Department of Chemistry, Oregon State University

- Filling the detector with liquid nitrogen (this should be done 1-2 hour before run):
  - Remove the white cap on the top of the detector. Place the funnel inside the opening (see Figure 3).
  - Pour a small amount of liquid nitrogen into the detector. Wait a few minutes for the temperature to equilibrate.
  - Once equilibrated, proceed to fill the detector.
  - Overflow is indicated that the detector is full.
  - Replace cap once full.

Figure 3. Liquid nitrogen fill port with funnel.

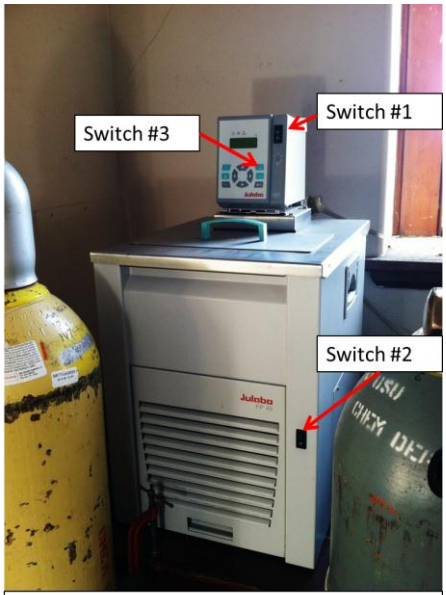

Figure 4. Chiller unit for the NETZCH LFA 457.

- Turning on the instrument (See Figure 2):
  - Turn on the chiller unit by turning on first switch #1, then switch #2, and finally switch #3 (see Figure 4).
  - Turn on the instrument power unit by turning the red switch on the front (see Figure 5)
  - Turn on the instrument with the switch located on the back (see Figure 6).

Figure 5. Power Unit for the NETZCH LFA 457.

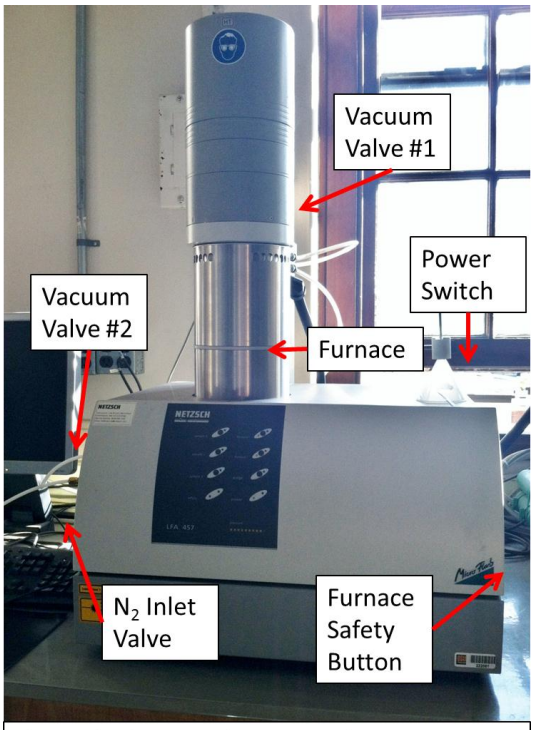

Figure 6: The NETZCH LFA 457 instrument.

- Turn on the laser power unit by first turning on the switch located on the back, and second by turning on the green switch located on the front (see Figure 7).

- Loading the sample:
  - If sample is not black, may require a graphite coating.
  - Choose the diameter of sample holder that fits your sample.
  - Place sample in bottom of the holder and replace seated cap.
  - Open the furnace by simultaneously holding the side safety button (see Figure 6) and the furnace down button on the front display panel (see Figure 8).

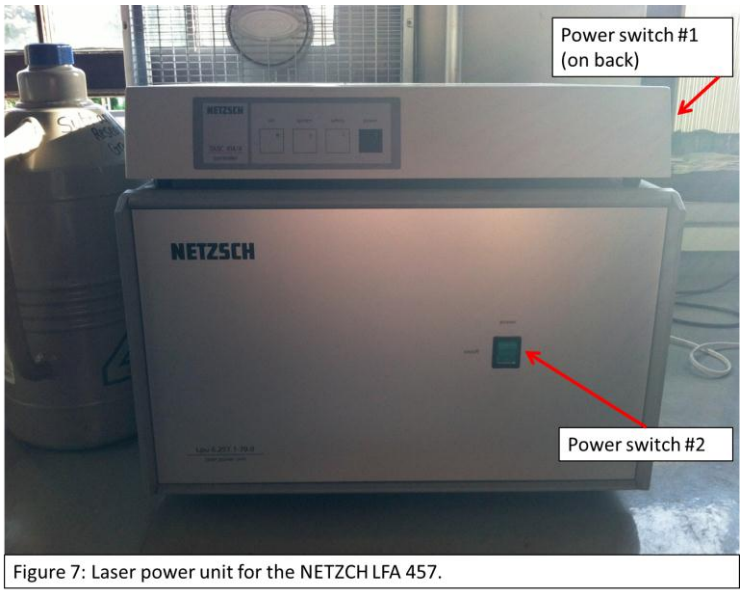

Figure 7: Laser power unit for the NETZCH LFA 457.

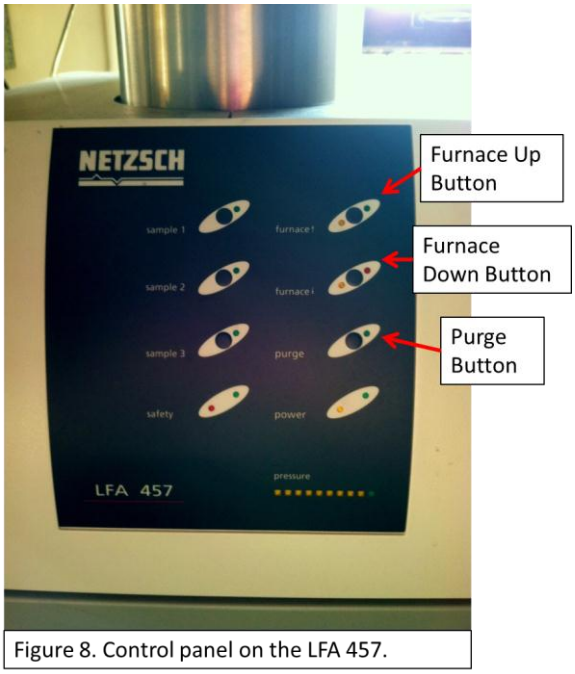

Figure 8. Control panel on the LFA 457.

- Swivel the detector to the left to expose the sample stage (see Figure 10). Be very careful with the IR detector fins (see Figure 9).
- The sample holder numbers are labelled by dots on the samples stage (see Figure 11). The Pyroceram standard will always be loaded in position #1.
- Gently place sample into position #2.
- Swivel the detector back into place.
- Raise furnace by pressing the safety button on the side and the furnace up button on the front control panel (see Figure 6 and Figure 8).

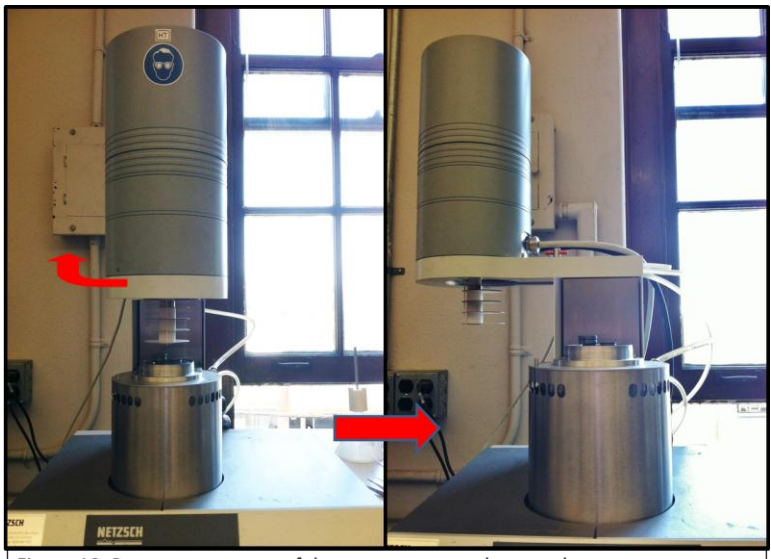

Figure 10. Proper movement of detector to access the sample stage.

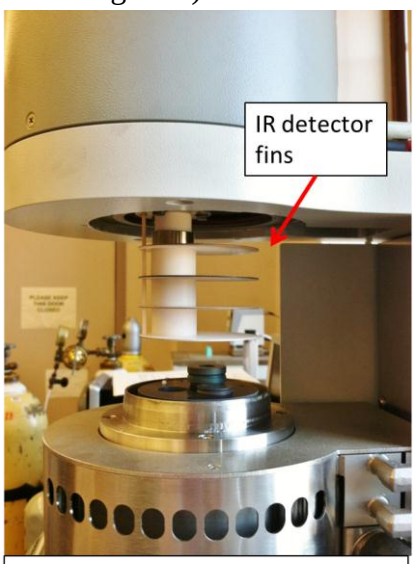

Figure 9. Side view after the furnace has been lowered.

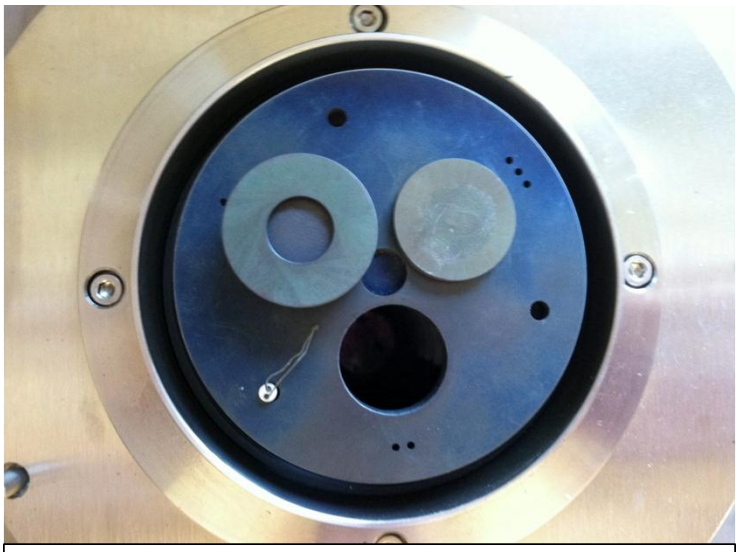

Figure 11. Sample holder stage. The number of dots indicate sample holder position.

- Preparing the sample chamber:
  - Turn on the vacuum pump for the system (see Figure 2).
  - Open the sample chamber the vacuum by first turning the magenta vacuum valve #1 (see Figure 6 and Figure 12) and then by very slowly opening the black vacuum valve #2 (see Figure 6 and Figure 13). Open this knob slowly until the pressure gauge is so low no more light are visible. Completely open the valve. Wait five minutes.
  - Close vacuum valve #2. Open the brass Nitrogen gas inlet valve (see Figure 13) 45° and slowly fill with nitrogen until the pressure gauge indicates the chamber is full (green light on gauge). Close brass nitrogen gas inlet valve.

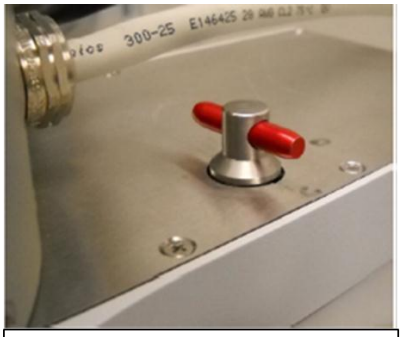

Figure 12. Vacuum Valve #1.

- Once again, open vacuum valve #2 slowly until the pressure gauge is so low no more light are visible. Completely open the valve. Wait five minutes.
- Once again, close vacuum valve #2. Open the brass Nitrogen gas inlet valve 45° and slowly fill with nitrogen until the pressure gauge indicates the chamber is full (green light on gauge). Close brass nitrogen gas inlet valve.
- One last time, open vacuum valve #2 slowly until the pressure gauge is so low no more light are visible. Completely open the valve. Wait five minutes.
- Finally, close vacuum valve #2. Open the brass Nitrogen gas inlet valve 45° and slowly fill with nitrogen until the pressure gauge indicates the chamber is full (green light on gauge). Close brass nitrogen gas inlet valve.
- Hit the purge button on the LFA 457 front control panel (see Figure 8).
- Close vacuum valve #1 and turn off the vacuum pump.
- Check that the nitrogen flow on the gauge above the instrument power unit is flowing around 80.

Figure 13. Vacuum Valve #2 (black knob) and gas inlet valve (brass lever).

- Setting up the experimental parameters on the computer:
  - Open the Density Calculator excel file on the desktop and enter sample parameters to calculate the sample density. Write this down for later use.
  - Open the LFA457 Measurement icon on the desktop.
  - Click on "Measurement"→ "New"
  - On panel, enter "Mas Lab" into the "identity" section.
  - Enter your name into the "operator" section.
  - Enter "Mas Lab" in the "Lab" section
  - Select "InSb" under "detector".
  - Click "Next".
  - Select sample one and sample two.
  - Click "next".
  - Enter information for sample 1 (remember this is the Pyrocerm standard).
    - Customer→ Mas Lab
    - Remark→ Pyroceram Standard
    - Sample holder is already set
    - Click next
    - Sample coating→ graphite
    - .Model→ Cowen + pulse correction
    - Baseline type→ linear
    - Material→ Pyroceram standard
    - Standard dimensions are on sample bag
    - Next

        - Amplify gain→ 127
        - Signal acquisition→ 15000 ms
        - Laser voltage→ 1922
        - Filtration→ 100%
        - Click next until you reach the panel for sample #2
    - Enter information for sample 2 (your sample)
        - Customer→ Mas Lab
        - Remark→ Sample Name
        - Sample holder is already set
        - Click next
        - Sample coating→ graphite or none
        - Model→ Cowen + pulse correction
        - Baseline type→ linear
        - Material→ Click "add"
            - Enter composition
            - Enter calculated density
            - Click "ok", "yes", and "yes"
        - Select sample you just entered from the drop down menu
        - Enter sample thickness
        - Next
        - Amplify gain→ 127
        - Signal acquisition→ 15000 ms
        - Laser voltage→ 1922
        - Filtration→ 100%
        - Click next
    - Enter flow rate→ 80
    - Select "purge"
    - Setup temperature program
        - On the first line, enter 35°C
        - On the second line, enter 50°C for temperature, 5°C/min for rate, 1 shot for position one, 2 shots for position two, and 1 for dwell time.
        - On the third line, enter 100°C for temperature, 10°C/min for rate, 1 shot for position one, 2 shots for position two, and 1 for dwell time.
        - Increase temperature to desired maximum by every 50°C, keeping the rest of the information on each line the same as before.
            - Furnace can go up to 1100°C, but it routinely ran up to 600°C.
        - Scroll through using the "next" button and make sure the temperature profile is set up correctly.
        - Enter emergency shut off temperature (this should be 100°C above the maximum temperature in your profile).
    - Once complete, exit the setup page.
- Testing to see if the detector is cooled:
    - Click yellow triangle "test shot" to give sample shot.
    - If working correctly, should hear beeps which indicate laser is about to shoot.
    - Once finished, diffusivity signal should have intensity between 2 and 8.
        - Intensity can be increased by the following

- Decreasing sample thickness
- Coating sample with graphite
- Increasing laser voltage

  - Once the desired intensity is achieved, you are ready to run the sample.

- Press "play" button.
- After approximately two hours, detector needs to be refilled with liquid nitrogen. Be sure to fill during a "heating" phase to avoid laser exposure.
- When shutting down the instrument, turn components off in the reverse order of the turn on process.

6. Controls and Calibration

The Pyroceram Standard sample is located in the green box underneath the NETZCH LFA 457 for any calibration and investigative purposes.

7. Training and Competency

The trainee must have already mastered an understanding of and have been given the instruction in the use of the NETZCH LFA 457 by an approved trainer (the instrument supervisor or any trained member of the Subramanian Research Group). Competency will be assessed by close observation of the trainee by the instrument supervisor or an approved trainer. The training records are attached at the end of this SOP.

7. Equipment and Maintenance

- No person shall operate the instrument unless it is in good repair.
- Users are not to make repairs. The NETZCH LFA457 shall be maintained and repaired by qualified persons.

8. Relevant Documents / References

- NETZCH LFA 457 Instrument Manual (available in Rm. 214, Gibert Hall)
- Software Analysis Instruction Manual (available in Rm. 214, Gilbert Hall)
- NETZCH website: http://www.netzsch-thermal-analysis.com/en/products/detail/pid,26.html
- Liquid nitrogen MSDS: http://www.airgas.com/documents/pdf/001040.pdf
- Laser safety information: http://en.wikipedia.org/wiki/Laser_safety
- Oregon State University Department of Chemistry-Safety Web

9. Signage / Summaries / Templates

Competency Training Records Form – Attached, see page 11.

- Copies of this form are to be stored and filed and in the Subramanian Research Group NETZCH LFA 457 Instruction book.

SUBRAMANIAN RESEARCH LABORATORY

COMPETENCY TRAINING RECORDS – NETZCH LFA 457

Date	Name	University/Dept.	Trainer	Competency		
				Date	Trainee	Trainer