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 of the Subramanian Lab. 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 a manufacture's instrument manual, warnings and instructions pertaining to use and safety of the specified equipment. This specific document may be used as a supplement to procedure and quick reference to safety but does not replace the information found with the manufactures documentation for the specified equipment. The Subramanian Lab reserves its right to change or suspend any or all parts of this document.

USE OF ULVAC ZEM-3

1. Introduction

Thermal power generation is a method of generating power based on the thermoelectric effect which was discovered by J. T. Seebeck, German physicist in 1821. In the face of recent global warming caused by carbon dioxide and depletion of fossil fuels, thermoelectric conversion devices are attracting attention because of its effective utilization of waste heat energies. To meet these pressing requirements, ULVAC-Riko has developed a characteristic evaluating instrument for these materials and devices.

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 ULVAC ZEM-3.

3. Safety

Before operation, have a good knowledge of components, safety information, and cautions. Incorrect operation of this instrument may cause fire or electric shock. Familiarize yourself with the operation manual before operation.

- 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.

- Recommended PPE to be used while operating the ULVAC ZEM-3 are:
    - Safety Glasses
    - Gloves
    - Lab Coat

4. System Specifications

Temperature range	- 80 to 100°C (L type) Room temperature to 800°C (M8 type) Room temperature to 1000°C (M10 type)
Temperature setting range	Number of measurement temperature steps and number of temperature sample measurement difference steps: Maximum 125
Measurement method	Seebeck coefficient : Static dc method Electric resistance : Four-terminal method
Atmosphere	Low pressure helium gas
Sample size	2 to 4 mm square or diameter x 6 to 22mm long (maximum)
Lead interval	8 mm
Power requirements	200 VAC, single phase, 40 A (M8 type & M10 type) 100 VAC, 20 A (L type & M8 type & M10 type) Transformer for European use available
Cooling water requirements	Water pressure 1.5 kgf/cm2 or more Flow rate 7 L/min or more Cooling water chiller is recommended

5. Operating Procedure

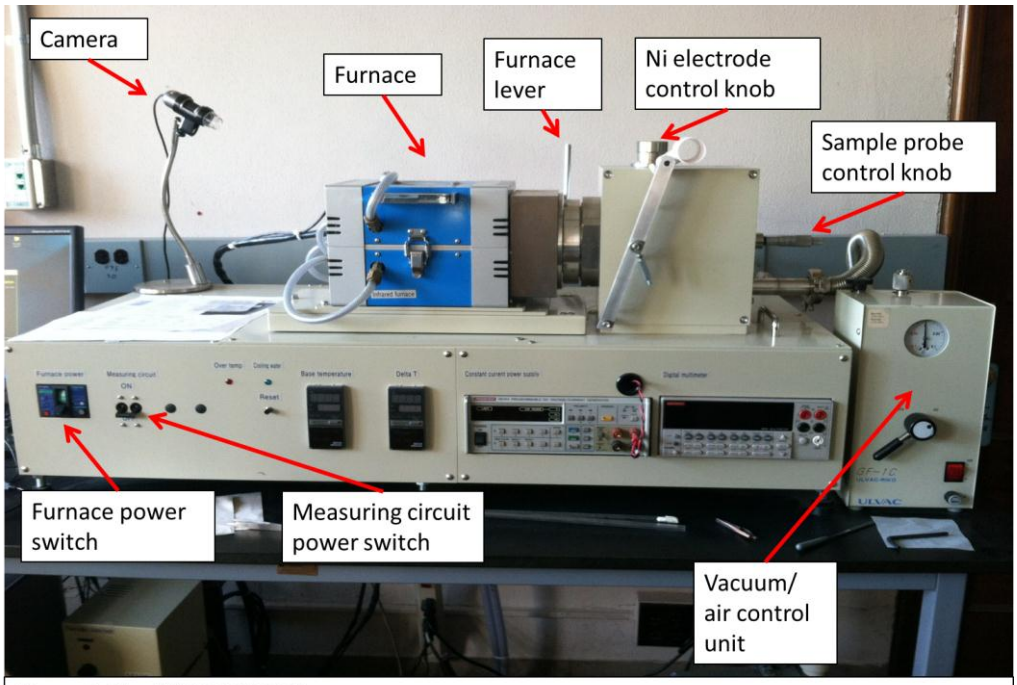

Figure 1. The ULVAC ZEM-3 instrument.

- Before beginning, make sure everything is powered off, including the computer.
- Powering on the instrument:
  - Turn on the chiller unit.
  - Turn on the furnace power switch located on the front panel (see Figure 1).
  - Turn on the measuring circuit power switch located on the front panel (see Figure 1). Hit the reset button on the front panel to stop the buzzing noise.
- Loading the sample:
  - Before opening the furnace chamber, bleed the vacuum on the chamber using the air bleed valve (see Figure 2).

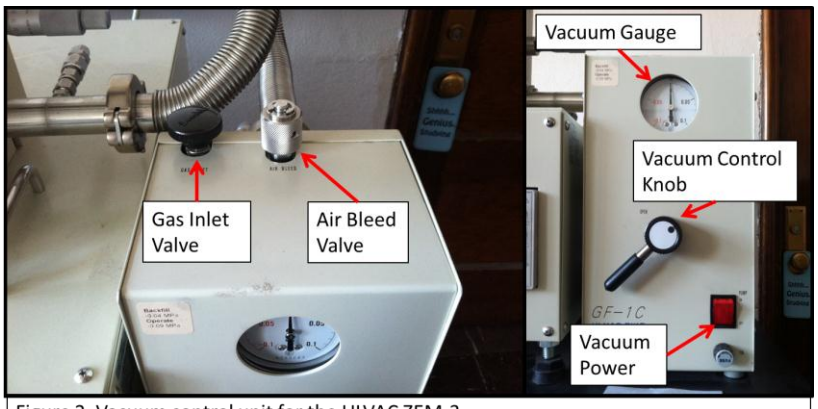

Figure 2. Vacuum control unit for the ULVAC ZEM-3.

  - Open the furnace by pushing the furnace level back and sliding the furnace open (see Figure 3).

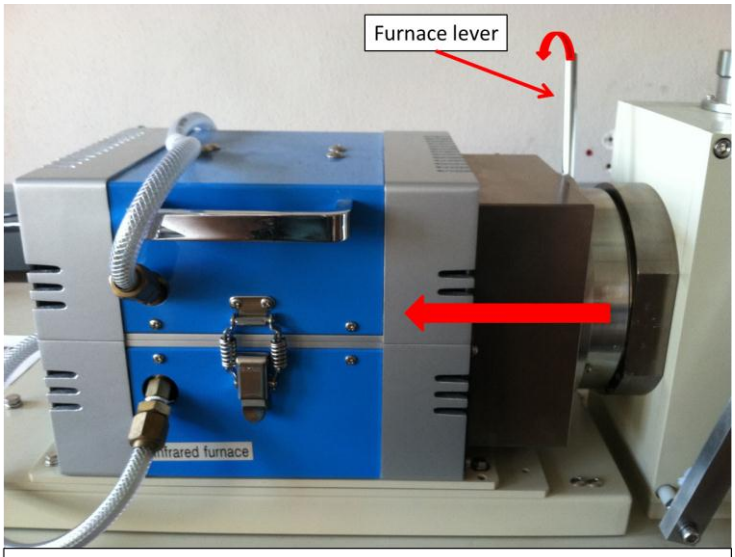

Figure 3. Up-close view of the furnace.

- To expose the sample holder, carefully remove the outer thermocouple with tweezers from the nickel sheath, and then carefully remove the nickel sheath (see Figure 4).

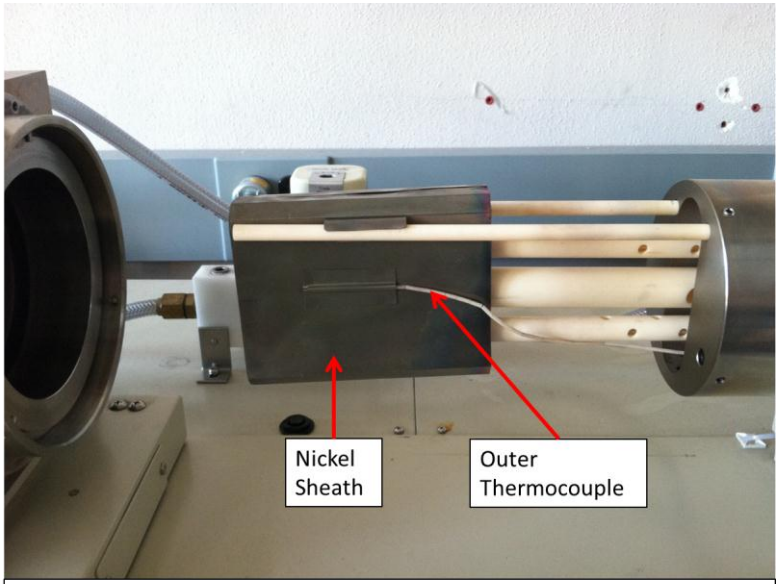

Figure 4. View once opening the ULVAC ZEM-3 furnace.

- If another sample is already in the sample holder, remove by first opening the sample probe, and then open the nickel electrodes (see Figure 5 and Figure 6).
- Load the sample; place the sample on its end and close the nickel electrodes. Once the electrodes are touching the sample, turn the knob two additional full turns.
- Close the sample probes. Once again, once the probes are touching the sample, turn the probe knob two additional full turns.
- Replace the nickel sheath and outer thermocouple.
- Close furnace chamber

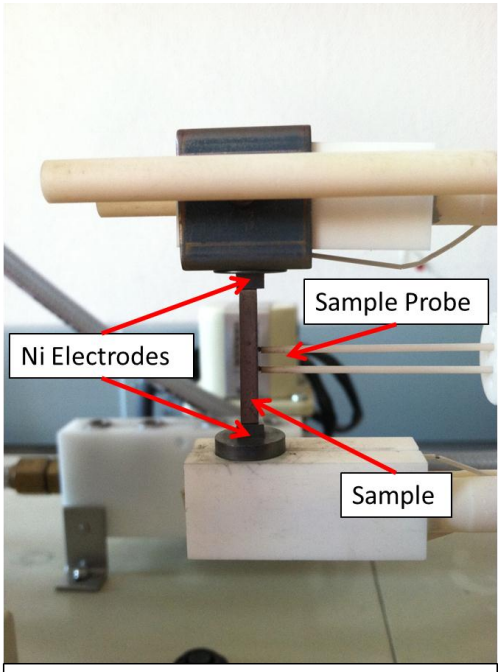

Figure 5. Close-up of the nickel electrodes, sample probe, and sample arrangement.

Figure 6. Control knobs for the nickel electrodes, left, and the sample probe, right.

- Preparing the furnace chamber:
  - Turn on the vacuum pump and open the vacuum to the chamber by using the vacuum control knob located on the vacuum control unit (see Figure 2). Wait five minutes while chamber is under vacuum.
  - Close the vacuum to the chamber by using the vacuum control knob.
  - Backfill the chamber with Helium to -0.04 MPa by using the gas inlet valve located on the vacuum control unit.
  - Repeat vacuum on chamber.
  - Repeat Helium backfill.
  - Repeat vacuum on chamber.
  - Repeat Helium backfill.
  - Vacuum the chamber down to -0.09 MPa; this will be the sample running atmosphere.
- Setting up the program parameters:
  - Turn on the computer.
  - Open the ZEM-3 Operation Program on the desktop.
  - Screen with “Seebeck Coefficient & Electrical Resistance Measurement” will appear.
  - Click “Input Sample Parameters”
  - Enter sample name and comment.
  - Enter sample width and depth in centimetres (sample should be rectangular column).

- Click "replace".
- Click "Return to Main Menu".
- Click "Input Temperature Program".
  - A standard temperature profile increases every 50°C, with delta temperatures of 10 °C, 20 °C, and 30 °C.
- Click "set the new program". A Plot of your temperature program should appear.
- Click "Return to main menu".
- Click "Check V-I Plot".
- Under "Select. Config", select "Manual".
- Under "Mode", select "Step(V/I)mode".
- Click "Start".
- V-I Plot should yield a straight line. Once this is obtained, reset "Mode" to "REV(+-)mode" and click "exit".
- Click "Start Measurement".
- Click "Start Measurement".
- Once program has run, select "end program" to exit software.

- Data Analysis
  - Open ZEM-3 Data Analysis icon on the desktop.
  - Open data file.
  - Click "analysis".
  - Save file to export data.

6. Controls and Calibration

The Constantan Standard sample is located in the clear plastic box underneath the ZEM 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 ULVAC ZEM-3 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.

8. Equipment and Maintenance

- No person shall operate the instrument unless it is in good repair.
- Users are not to make repairs. The ULVAC ZEM-3 shall be maintained and repaired by qualified persons.

9. Relevant Documents / References

- Model ZEM-3 Power Conversion Efficiency Measuring Instrument Instuction Manual (available in Rm. 214, Gilbert Hall)
- ZEM series Power Conversion Characteristics Evaluation System Instruction Manual (available in Rm. 214, Gilbert Hall)

- Oregon State University Department of Chemistry-Safety Web

10. Signage / Summaries / Templates

Competency Training Records Form – Attached, see page 8.

- Copies of this form are to be stored and filed and in the Subramanian Research Group ULVAC ZEM-3 Instruction book.

SUBRAMANIAN RESEARCH LABORATORY

COMPETENCY TRAINING RECORDS – ULVAC ZEM-3

Date	Name	University/Dept.	Trainer	Competency		
				Date	Trainee	Trainer