Procedure Title: Fox 314 Heat Flow Meter

**Minimum Review Requirements:** Annually Creation Date: March 2014 Date of Next Review: March 2015 **Melanie Fauchoux** Creator of Procedure: Authorized by: Melanie Fauchou Table of Cont Version History..... 1. 2. Introduction ..... Definition..... 3. 4. Personnel ..... 5. Safety..... 6. Procedure ..... 6.2 Data Storage..... 7. Equipment or Materials Required......7 9. 10. 

1. Version History

Version #: <u>.1</u> Supersedes: <u>n/a</u>

Handwritten amendments to the official procedures can be made by a single line through the text, along with the date, and initialed by the authorized individual making the correction. Changes are to be noted below. Formal changes to this SOP are made on the date of revision or sooner, where required.

Section	Changes made to official copy	Date	Initials
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#### 2. Introduction

This procedure outlines the proper operation of the LaserComp Fox 314 heat flow meter, as well as the WinTherm32 software. This machine is designed to measure the thermal conductivity of a sample, by exposing the top surface and the bottom surface to different temperatures. The machine was designed according to ASTM C518 – 91 "Standard Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus".

The maximum sample size for the HFM machine is 305 mm x 305 mm x 102 mm (12 in x 12 in x 4 in). The actual measuring area is 102 mm x 102 mm (4 in x 4 in). The temperature range for the upper and lower plates is  $-18^{\circ}$ C to  $55^{\circ}$ C.

#### 3. Definition

ASTM	American Society for Testing and Materials
FE	Final Equilibrium
HFM	Heat Flow Meter
NE	No Equilibrium Conditions met
PE	Percent Equilibrium
PPE	Personal Protective Equipment
QL	Heat flux from lower plate
Q <sub>U</sub>	Heat flux from upper plate
SE	Semi-Equilibrium
SOP	Standard Operating Procedure
TL	Temperature of lower plate
Τ <sub>υ</sub>	Temperature of upper plate
TE	Temperature Equilibrium

# 4. Personnel

Persons authorized to perform this SOP:

By signing this form I acknowledge that I have read and understand this SOP, as well as the applicable MSDS's and that I will conduct myself in accordance with this SOP and the general laboratory rules.

**NOTE:** ALL SIGNATURES MUST BE PRESENT ON THE SOP LOCATED IN THE YELLOW BINDER IN ROOM 1B39.1.1 – Heat Transfer Departmental Assistant's Office. Digital copies of SOP's are made available for reference and convenience only. Printed SOP's are valid for 24 hours only, after that time their accuracy must be verified with

the **OFFICIAL HARDCOPY VERSION**. A reference copy of this SOP can be found on the bench beside the machine in room 1B39.1.

Name (Print)	NSID	Dep't	Signature	Date
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## 5. Safety

- There are no specific PPE requirements for this machine, however safety glasses and gloves may be required during the preparation of samples.
- Standard laboratory safety rules must be followed.
- Continued use of the HFM machine at temperatures below the dew point will cause problems with samples absorbing moisture, and, over time, may cause problems with electronics.
- Make sure the cooling water is turned on before the machine is turned on and turned off after the machine is turned off.

#### 6. Procedure

The procedure outlined here uses the WinTherm32 software to operate the FOX 314 HFM machine. The machine may also be operated without this software, using the buttons and LCD located on the machine. For this procedure, refer to the machine's manual.

#### 6.1 Operation of the Fox 314

- Ensure the drain hose for the machine is in the small sink on the counter in 1B39.
- Turn on the water flow to the machine (valve is located under the sink in 1B39).
- Ensure the water flow rate is set at 20 GPH (rotameter to the left of machine).
- Turn on the Fox 314 (button on back, right hand side) and the computer.
- Open the WinTherm32 program on the computer.
- A 'Calibration Stack Screen' window will appear (if the machine has just been turned on). Make sure there is no sample in the Fox 314 and then click on **Calibrate**.
  - If there is a sample in the machine, remove it.
  - If the sample cannot removed, click **Open Stack** and then remove.
- Once calibration is finished, a 'Start Test Run' window will appear. Select the following options:
  - Test mode normal,
  - o 1450b calibration,
  - Instrument's calibration,
  - Thickness if using a rigid sample choose 'auto thickness', if using a nonrigid material choose 'manual thickness',
  - To use the machine under different modes, refer to the manual.
- Place the sample in the machine. If using the manual thickness option, be sure to measure the thickness before placing the sample in the machine.
- Click Run.

- A 'Sample Information' window will appear. Type the sample's name and, if using manual thickness, enter the thickness of the sample.
- Click OK.
- An 'Enter Test Set Point' window will appear. You may enter as many as 9 set point temperatures. You may enter the temperature of the upper and lower plates or you may enter the mean temperature and the change in temperature across the sample. *The Fox 314 works best with a 25 °C temperature difference across the sample, with the lower plate being the warmer.* The default is 10°C for the upper plate and 35°C for the lower plate.
- Click **OK**. The test will begin.
  - The test will run in blocks, each consisting of 512 successive data acquisition cycles.
  - The duration of each block is approximately 5 minutes.
  - $\circ~$  Once a block is completed, the average value of each measurement (T\_U, T\_L, Q\_U and Q\_L) is obtained.
  - These values are compared to the respective average values from the previous block.
  - Once this comparison satisfies all the equilibrium criteria the sample is declared to be in thermal equilibrium and the test is ended for the current temperature set point.
  - Depending on the type of sample, the machine may need to run many blocks before equilibrium is achieved.
- The equilibrium criteria can be seen by clicking **Criteria**. The default settings are:
  - Temperature Equilibrium (TE) = 0.2°C. The average temperature of each plate in the block must be equal to the plate's set point temperature within ±TE.
  - $\circ$  Semi-Equilibrium (SE) = 49  $\mu$ V. The average of the transducers signals must be within this value for two consecutive blocks.
  - Percent Equilibrium (PE) = 2%. The average of the transducers signals must be within this value for two consecutive blocks.
  - Number of Blocks of Percent Equilibrium = 4 blocks. This is the number of successive blocks satisfying the PE criterion required to declare that thermal equilibrium has be reached and results can be calculated.
  - Number of Blocks Used to Calculate Results = 3 blocks.
  - Thicker samples need a greater number of blocks to guarantee full thermal equilibrium
- If no equilibrium criterion are met, the machine will display NE.
- Once all equilibrium criterion are met, the machine will display FE (Final Equilibrium).
- When the test is completed, the machine will automatically lower so the sample can be removed

- The program will display the results from the test (temperature, thermal conductivity and heat flux) for the upper plate, lower plate and the average. The average is the average value for the last three blocks.
- Click **OK** to exit this window.

# 6.2 Data Storage

- You can scroll through the WinTherm window to see the information for each set point.
- This file will automatically be saved to a Notepad file, located in E:\Fox 314 Data with the sample name as the file name.
- In the WinTherm window, click View -> Block Data. A 'Block Average Data' window will open. Select the highest block number from the drop down menu. Click on Display Block.
- A ' Block Data' window will open. Click **Snapshot**. This will save the data as a text file to C:\Program Files\Laser Comp. This file is tabbed, so you can easily copy and paste it into a worksheet application to find the standard deviation and average values.
- Repeat this process for the next two highest blocks (three blocks are required to calculate the same average values given by the machine).
- To view the calibration constants for the temperatures selected, Click on View -> Calibration Constants. This is necessary to convert the heat flux to W/m<sup>2</sup> from  $\mu$ V. You can cycle through the different blocks. These values are also saved in the WinTherm file.

# 6.3 Multiple Samples and Shut Down

- To run another sample, press the **Start** button and repeat the above procedure.
- To shut down the machine, close the WinTherm program, turn off the computer, turn off the machine and then shut the water flow off.

# 7. Equipment or Materials Required

- Samples
- Calipers for measuring sample thickness
- Instrument Manual optional (1B39.1.1)
- Software Manual optional (1B39.1.1)

# 8. Regulatory / Standards

ASTM C518 – 91 "Standard Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus".

ASTM C1132 – 89 "Standard Practice for Calibration of the Heat Flow Meter Apparatus"

ASTM C1045 – 90 "Standard Practice for Calculating Thermal Transmission Properties From Steady-State Heat Flux Measurements"

#### 9. Trouble Shooting

If you encounter problems that you are unsure how to deal with, consult the Departmental Assistant in the lab. The following is a list of some possible problems that might occur and how to solve them.

Problem:	Look for
Set point temperature of the plates is not	Water flow rate may be too low
reached or is unstable	Water temperature may be too high
Machine runs for a long time without stopping	Convergence criteria is not met

#### 10. References

ASTM C518, 1991. Standard Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus, American Society for Testing and Materials, West Conshohocken, PA, USA.

ASTM C1045, 1990. Standard Practice for Calculating Thermal Transmission Properties From Steady-State Heat Flux Measurements, American Society for Testing and Materials, West Conshohocken, PA, USA.

ASTM C1132, 1989. Standard Practice for Calibration of the Heat Flow Meter Apparatus, American Society for Testing and Materials, West Conshohocken, PA, USA.

LaserComp, 1999. FOX200 and FOX 300 Series Instruments Manual. LaserComp, Inc., Saugus, MA, USA. <u>www.lasercomp.com</u>.