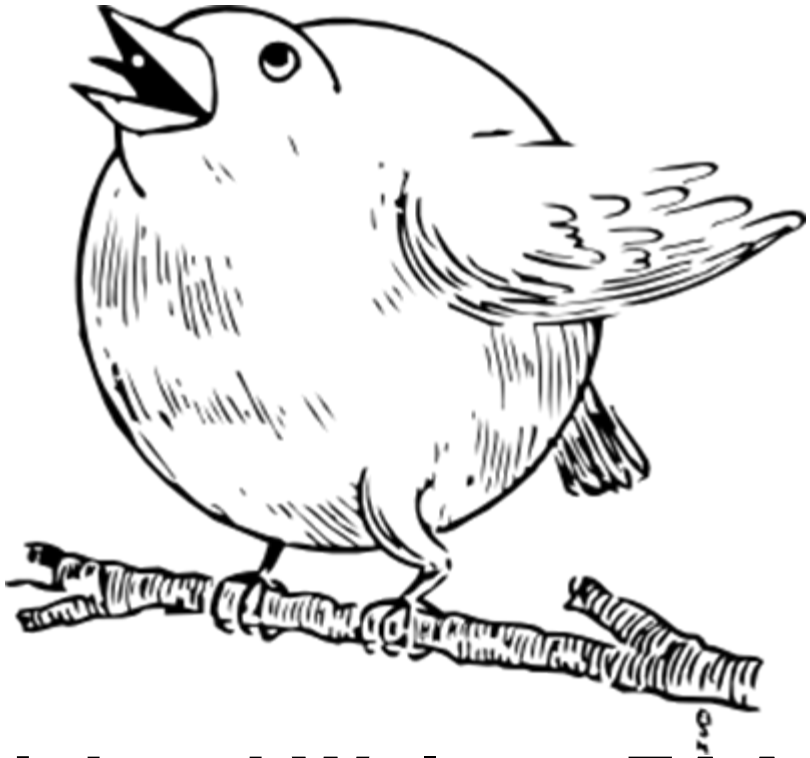
# PRELIMINARY RESULTS FROM THE MICROSCALE COMBUSTION CALORIMETER ROUND ROBIN



**Richard Walters FAA**

**Natallia Safronava TAMI**

# ASTM E691-09

**Conducting an Inter-Laboratory Study to Determine the Precision of a Method**

- **Repeatability**
  - **An action, event, or other thing that is done again**
- **Reproducibility**
  - **Create something very similar to (something else) in a different medium or context**

# Microscale Combustion Calorimeter

**Developed by FAA Fire Safety Team**

**Small scale flammability test**

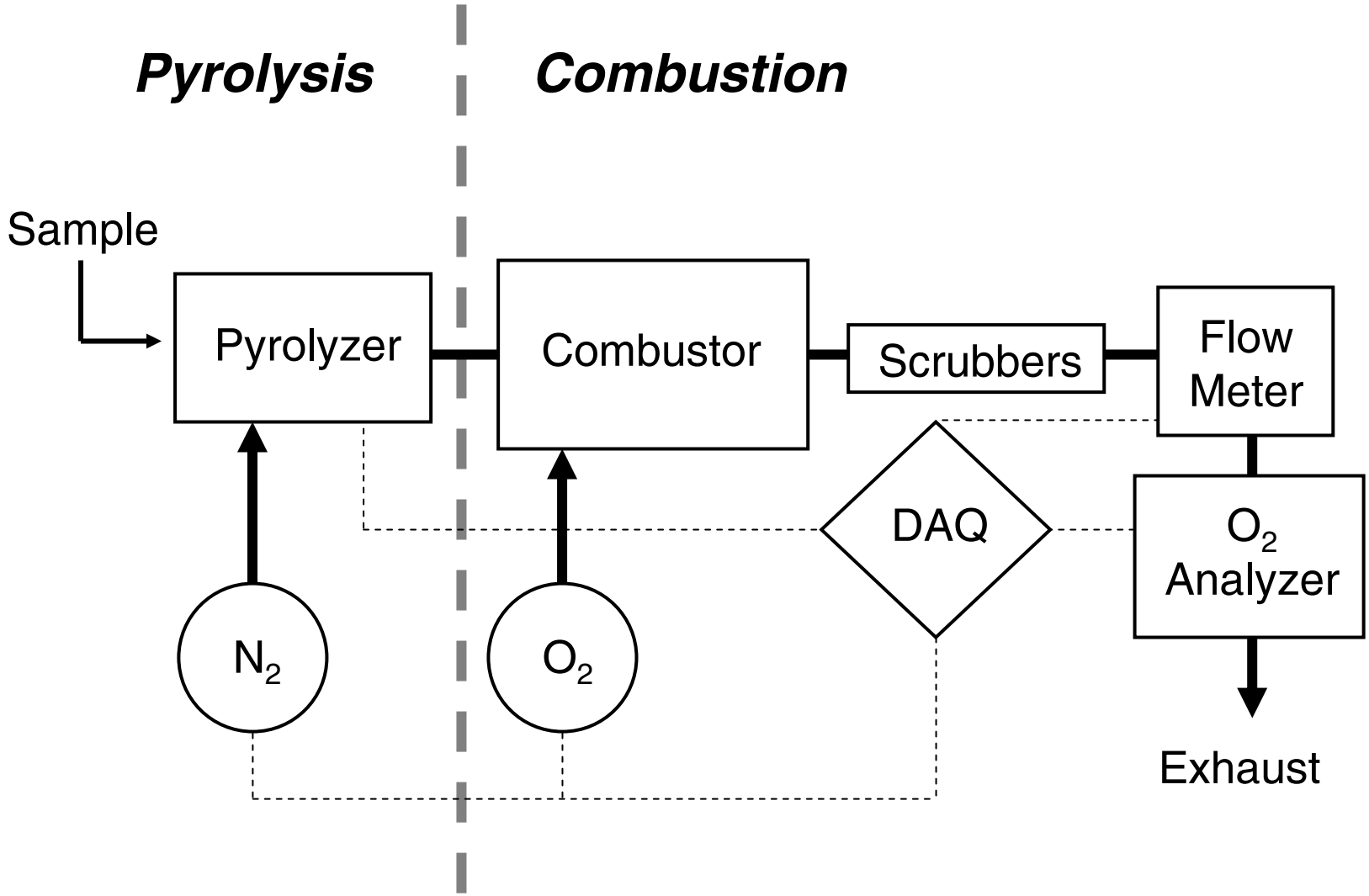
**Quantitative results**

**HRR vs Temperature**

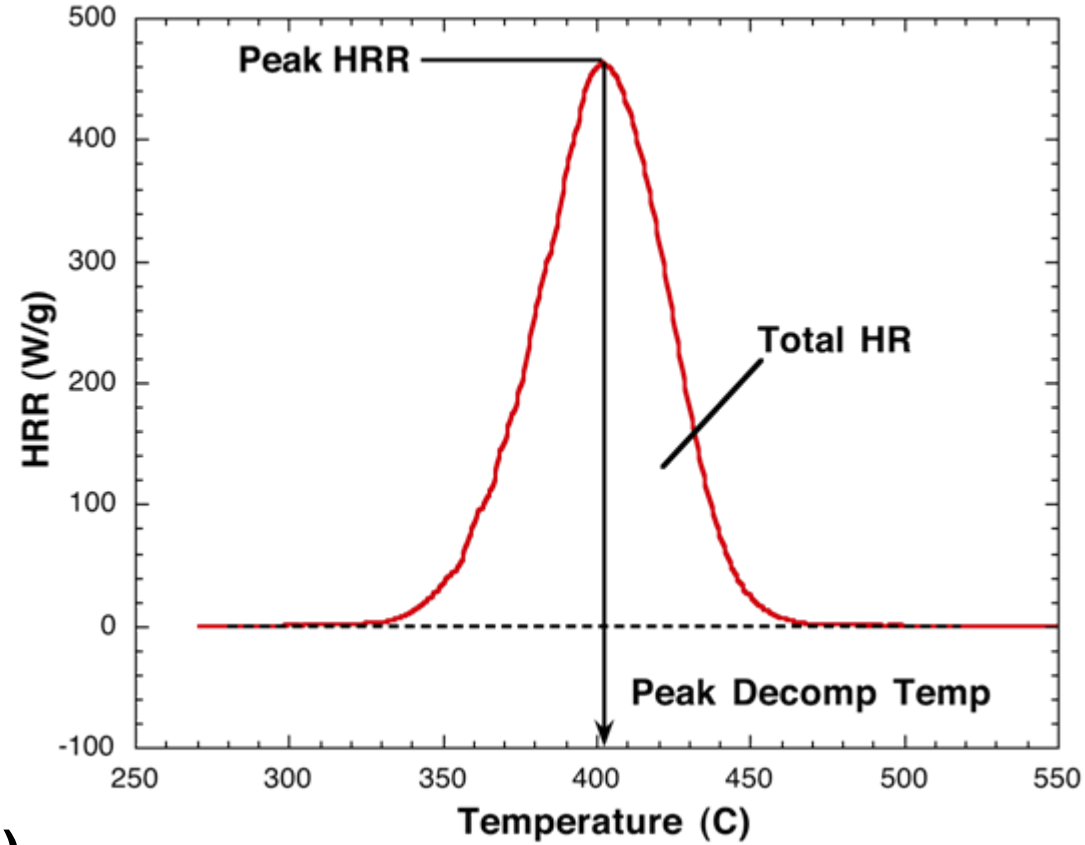
**10-15 minute test**

**Sample size of 5mg**

# MCC Schematic



# Measured Values




**Sample Weight (mg)**

**Char Yield (%)**

**Peak Heat Release Rate (W/g)**

**Total Heat Released (kJ/g)**

**Peak Heat Release Temperature (°C)**

**Heat Release Capacity (J/g-K)**

# Heat Release Capacity Measurements & Theory

## Heat Release Rate

$$\dot{Q}_c(t) = \frac{E\Delta O_2}{m_o} = h^o_{c,v}(t)\left[\frac{-1}{m_o}\frac{dm(t)}{dt}\right]$$

## Peak Heat Release Rate

$$\dot{Q}^{\max}_c = \left[\frac{E\Delta O_2}{m_o}\right]_{\max} = h^o_{c,v}\left[\frac{-1}{m_o}\frac{dm}{dt}\right]_{\max} = h^o_{c,v}\frac{\beta\left(1-\mu\right)E_a}{eR\,T^2_p}$$

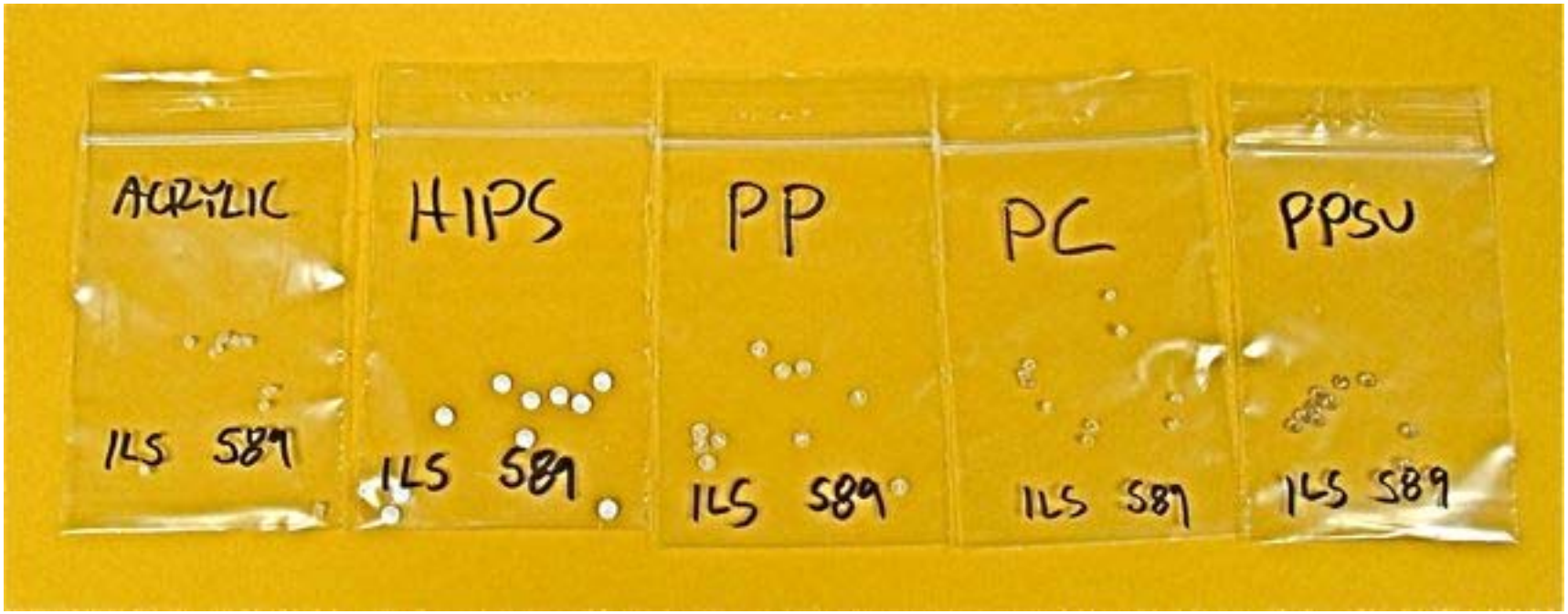
## Heat Release Capacity

$$\eta_c = \frac{\dot{Q}^{\max}_c(\beta)}{\beta} = \frac{h^o_c\left(1-\mu\right)E_a}{eR\,T^2_p}$$

# Sample Preparation

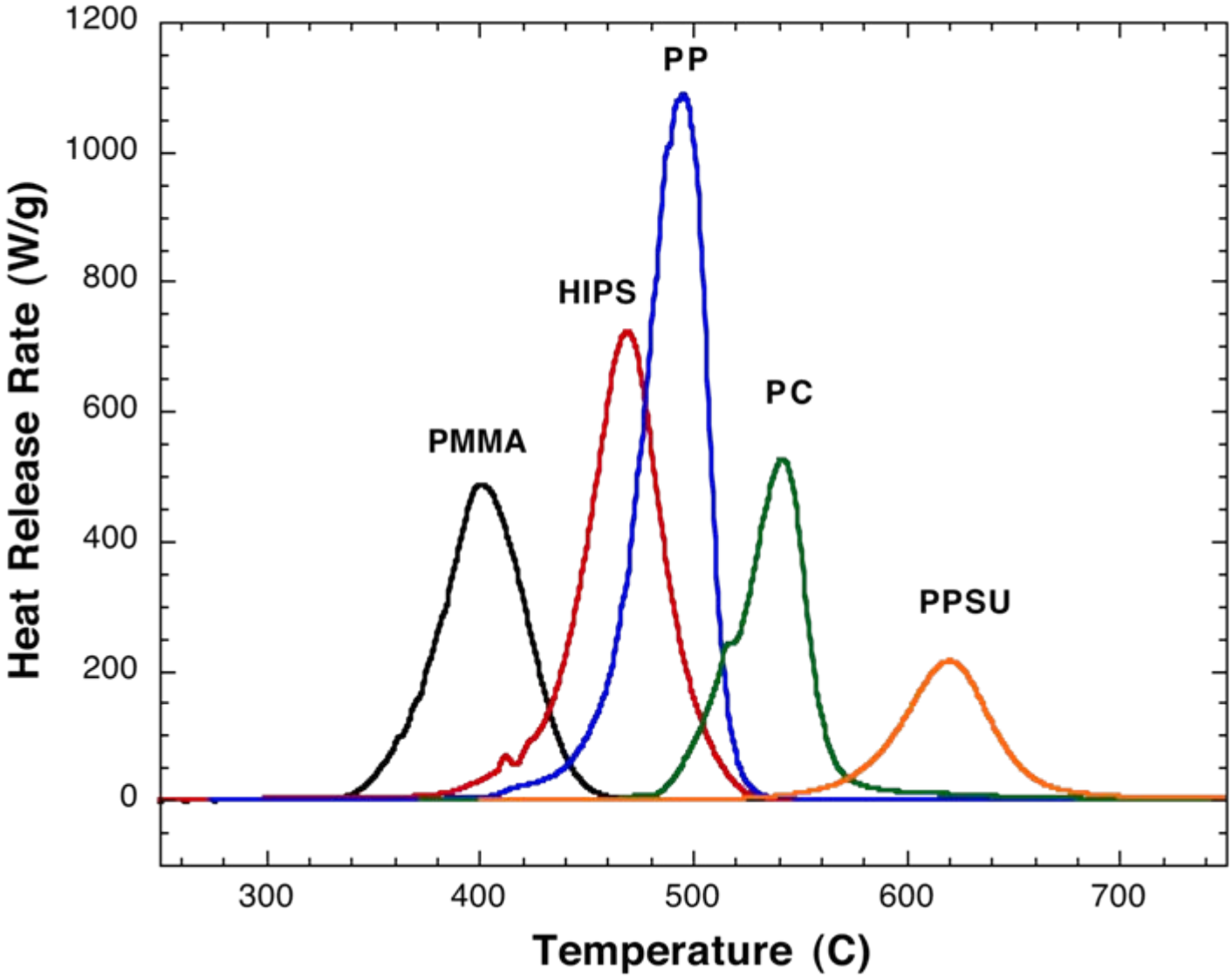
- 5 samples selected
- Hole punch used to make consistent sample sizes
- Hole size depended on film thickness and density
- Samples ranged from 3.2 mg to 6.2 mg

# Samples “as Received”




| | |
|---|---|
| **Acrylic** | **3.18 mg** |
| **Polystyrene** | **4.55 mg** |
| **Polypropylene** | **3.44 mg** |
| **Polycarbonate** | **3.47 mg** |
| **Polyphenylsulfone** | **6.20 mg** |

# Sample Heat Release Rates



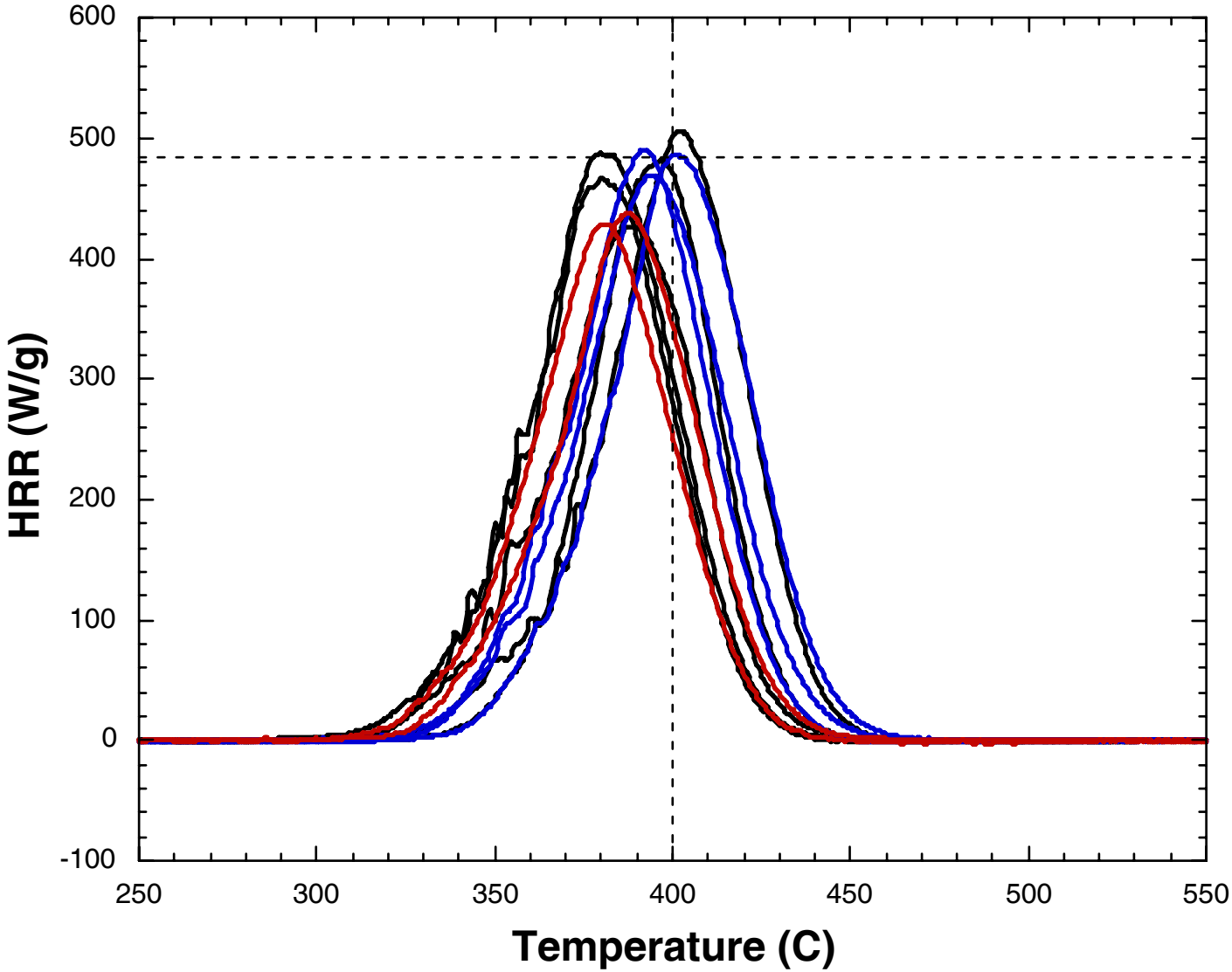
# Interlaboratory Study

- 3 manufacturers of the MCC
  - Manufacturer A
  - Manufacturer B
  - Manufacturer C

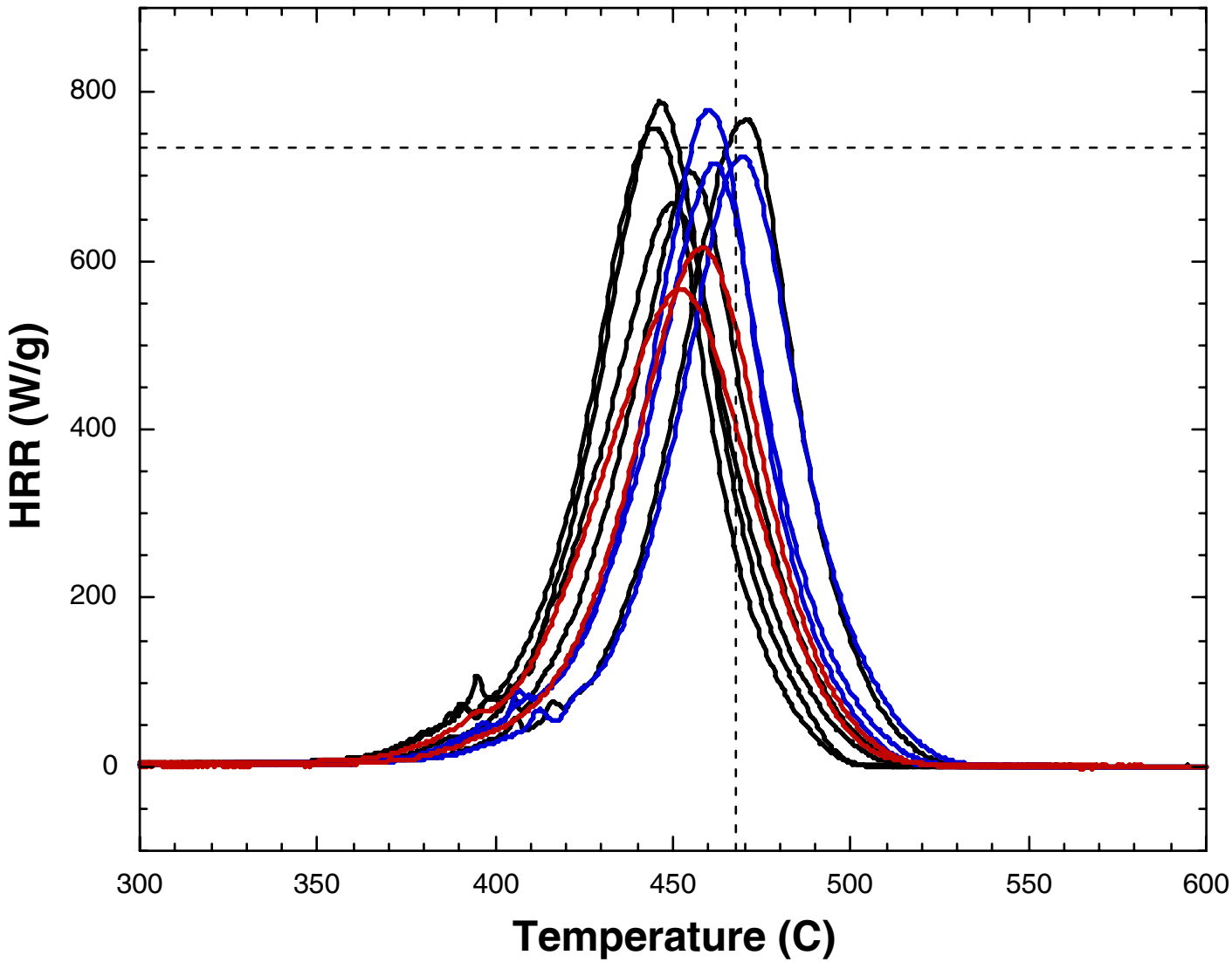
- Samples sent to 18 labs
  - Several labs could not participate due to equipment not working

- Data received from 11 labs so far
  - 1 lab rejected due to bad $O_2$ sensor
  - All raw data was re-analyzed and interpreted by me

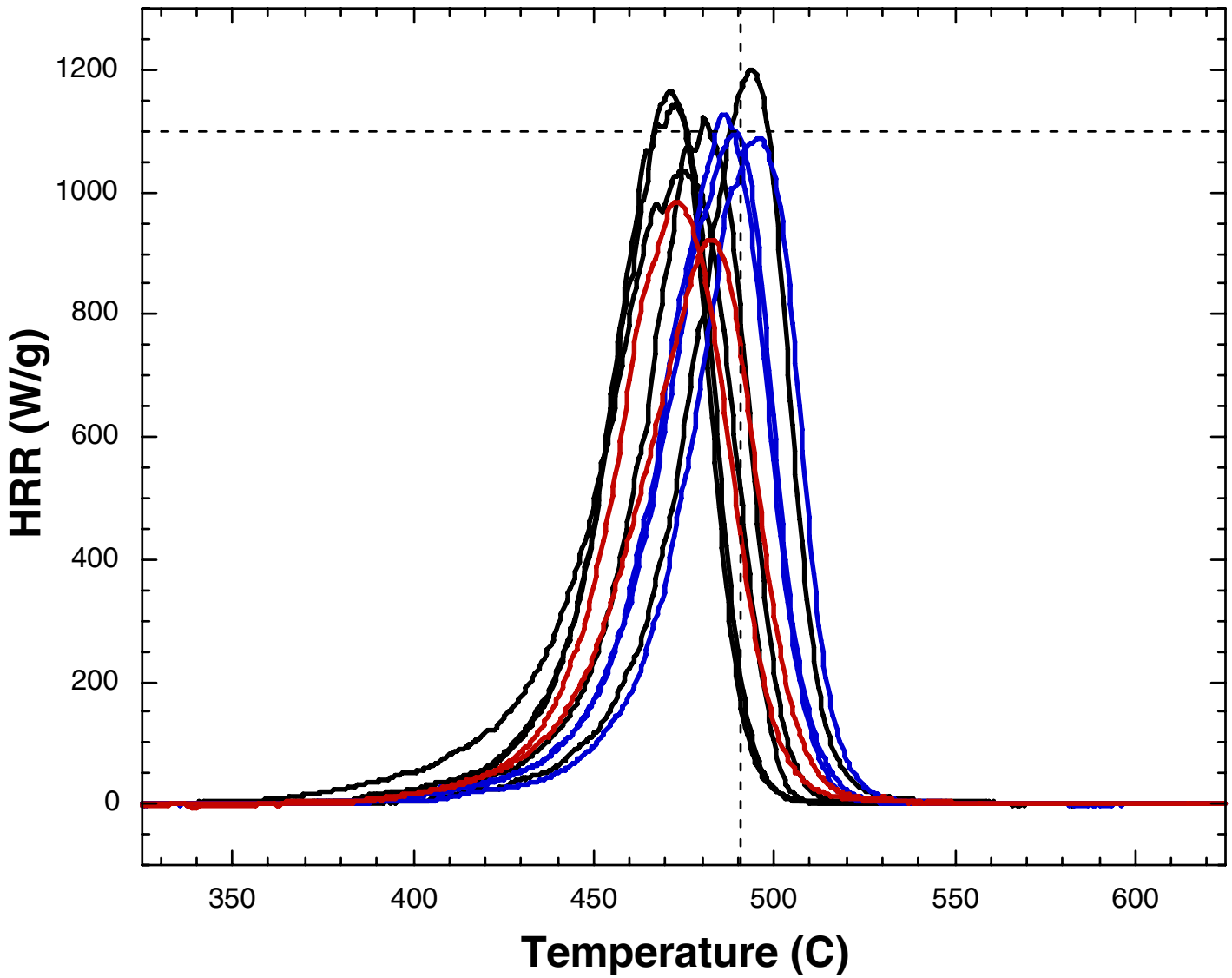
# Acrylic


600
500
400
300
200
100
0
-100
HRR (W/g)
250
300
350
400
450
500
550
Temperature (C)

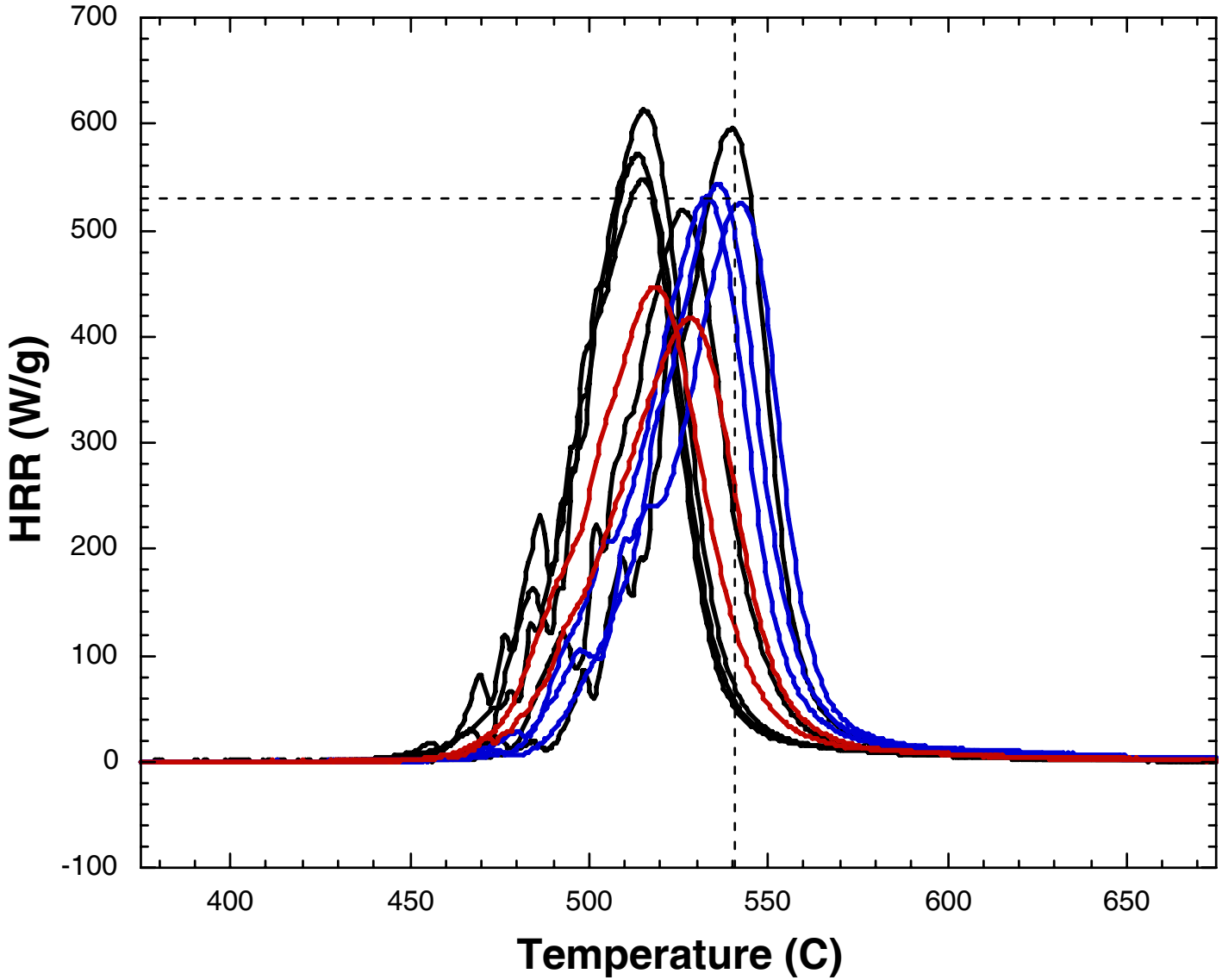
# Polystyrene



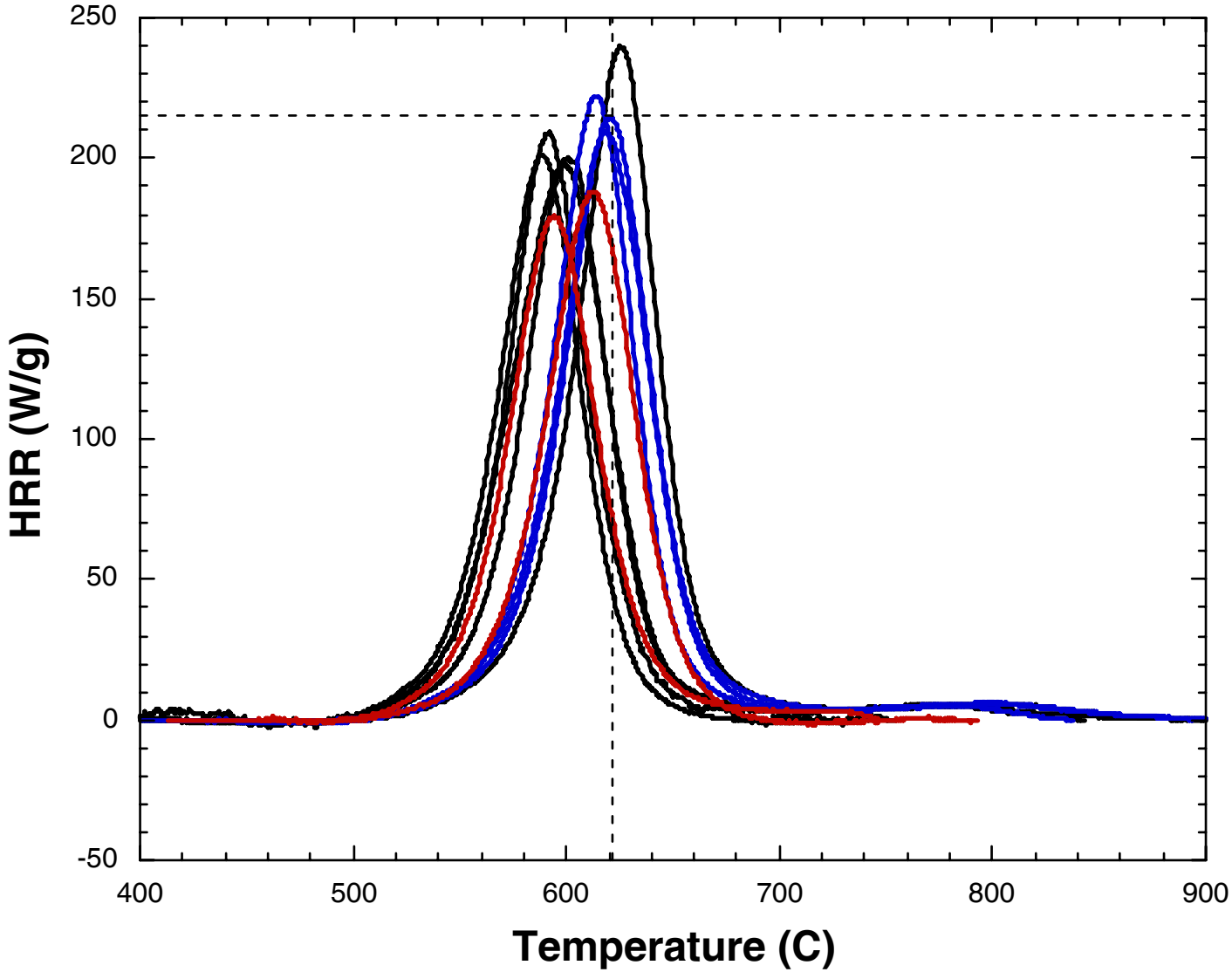
# Polypropylene



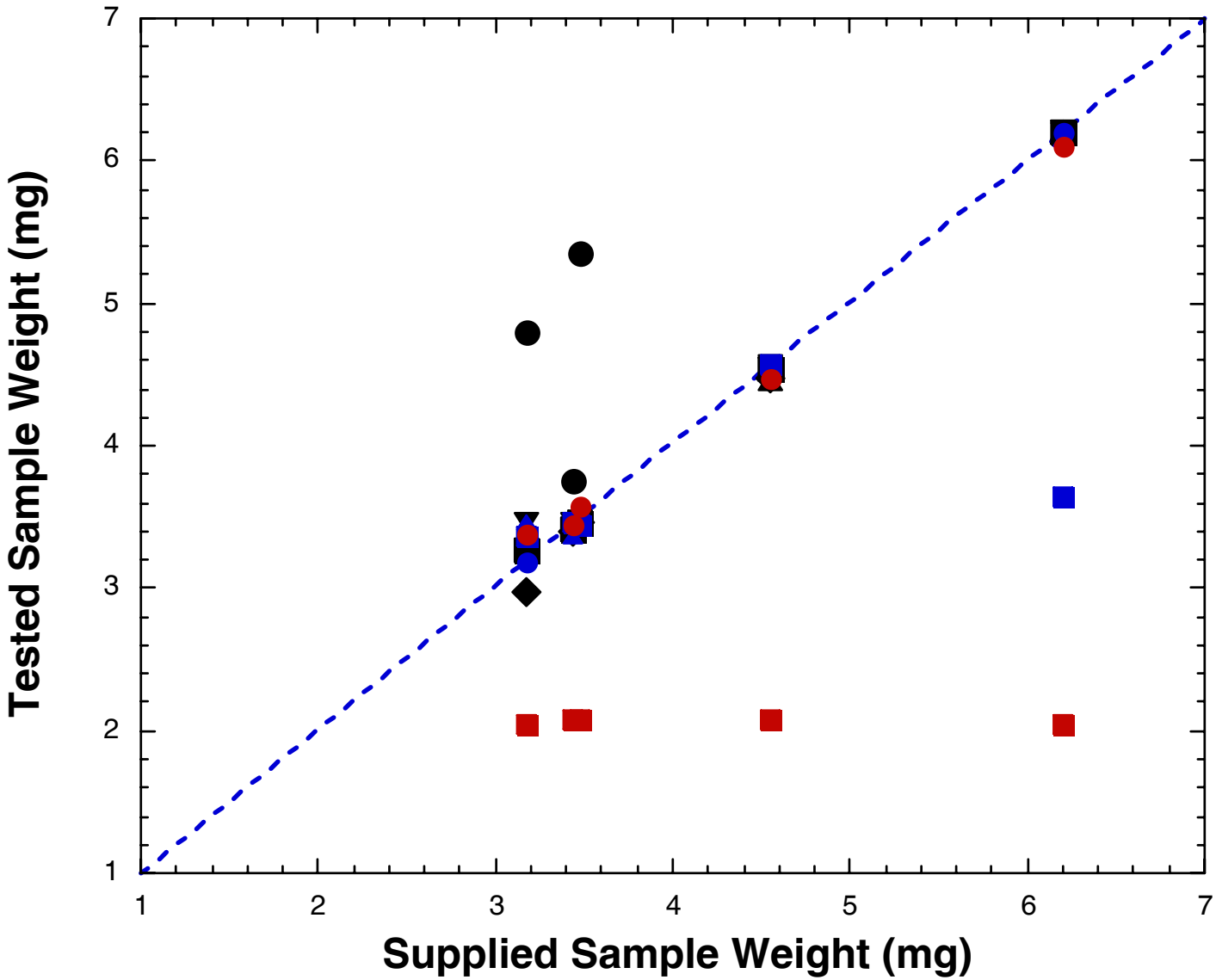
# Polycarbonate



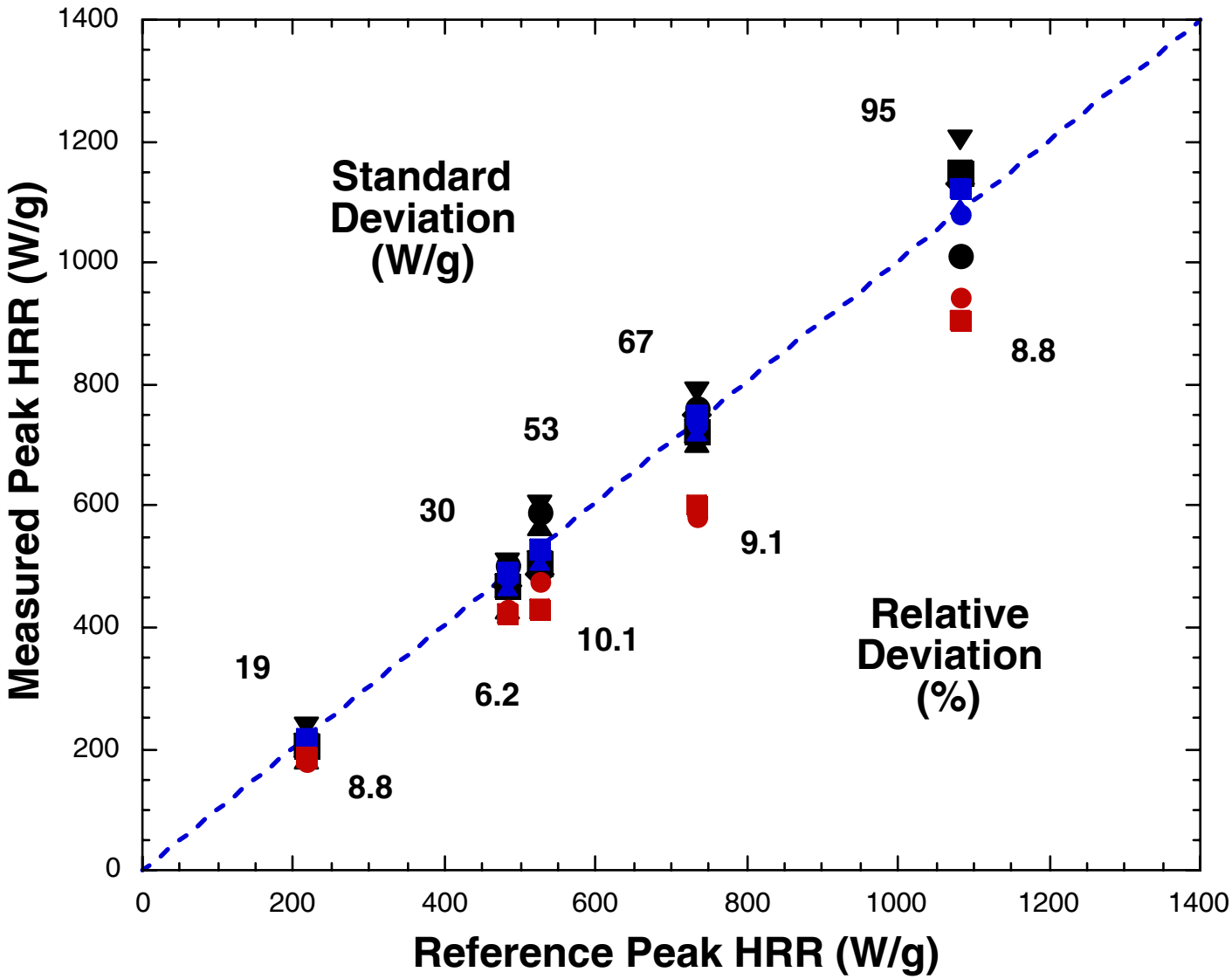
# Polyphenylsulfone



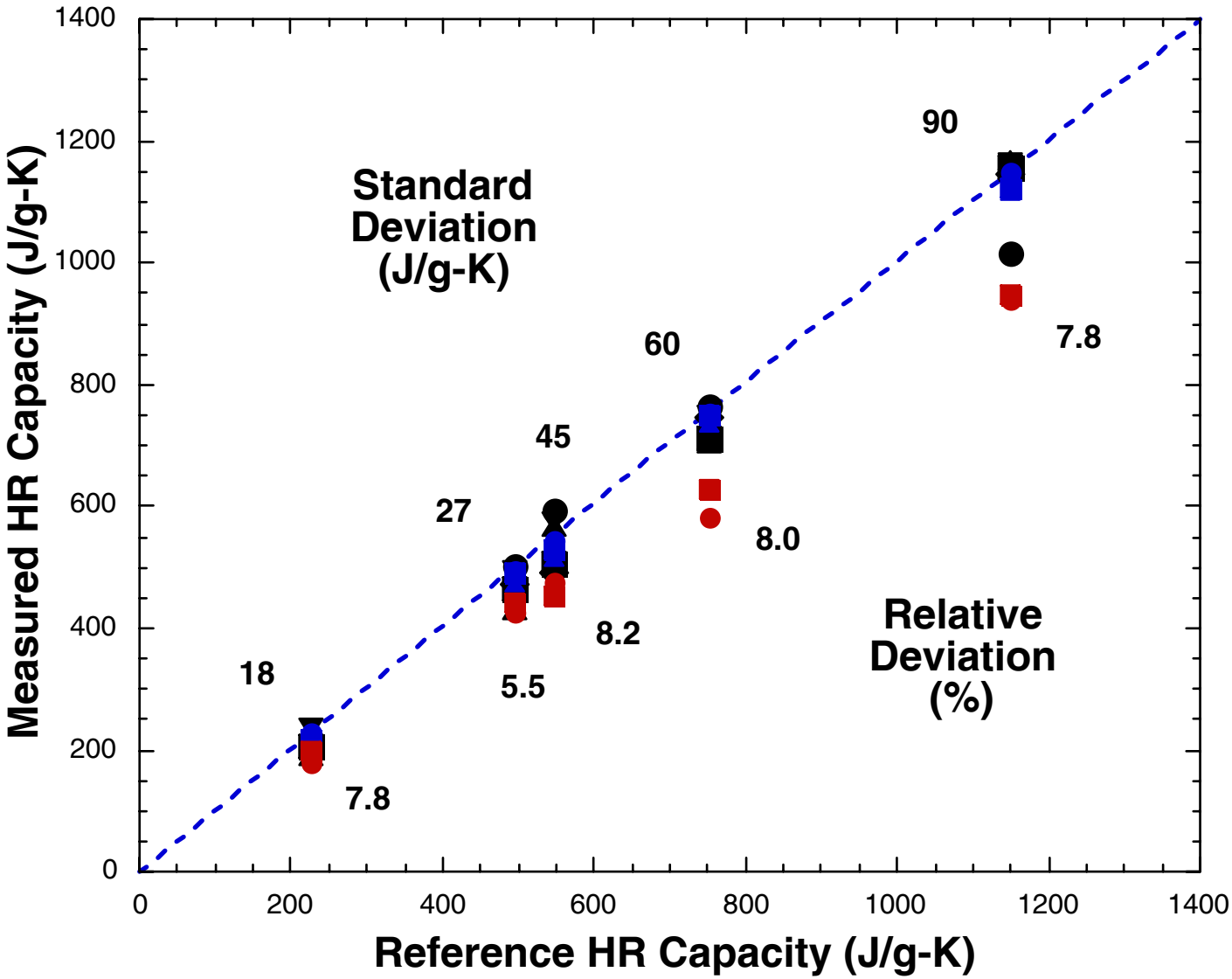
# Sample Weight



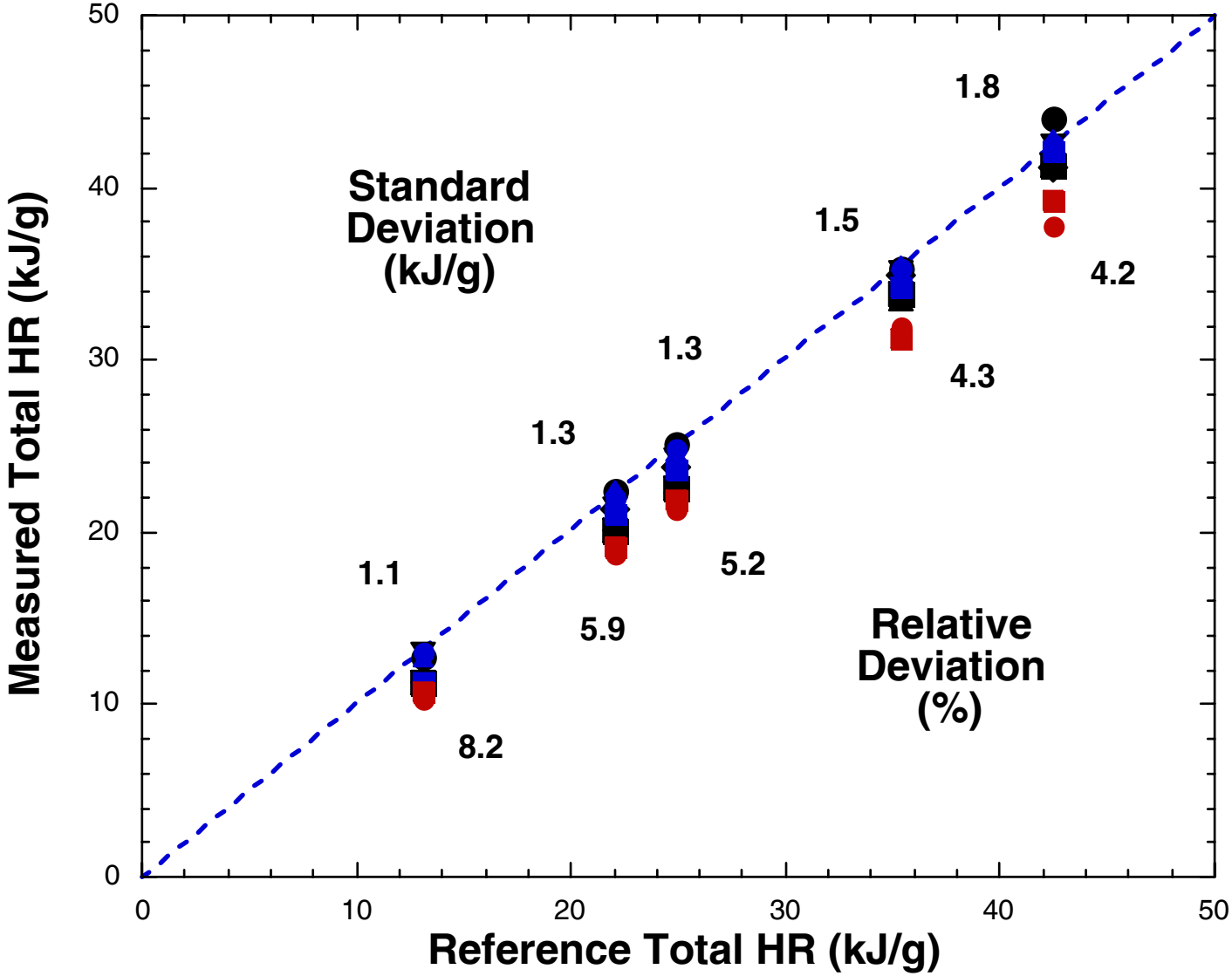
# Peak Heat Release Rate



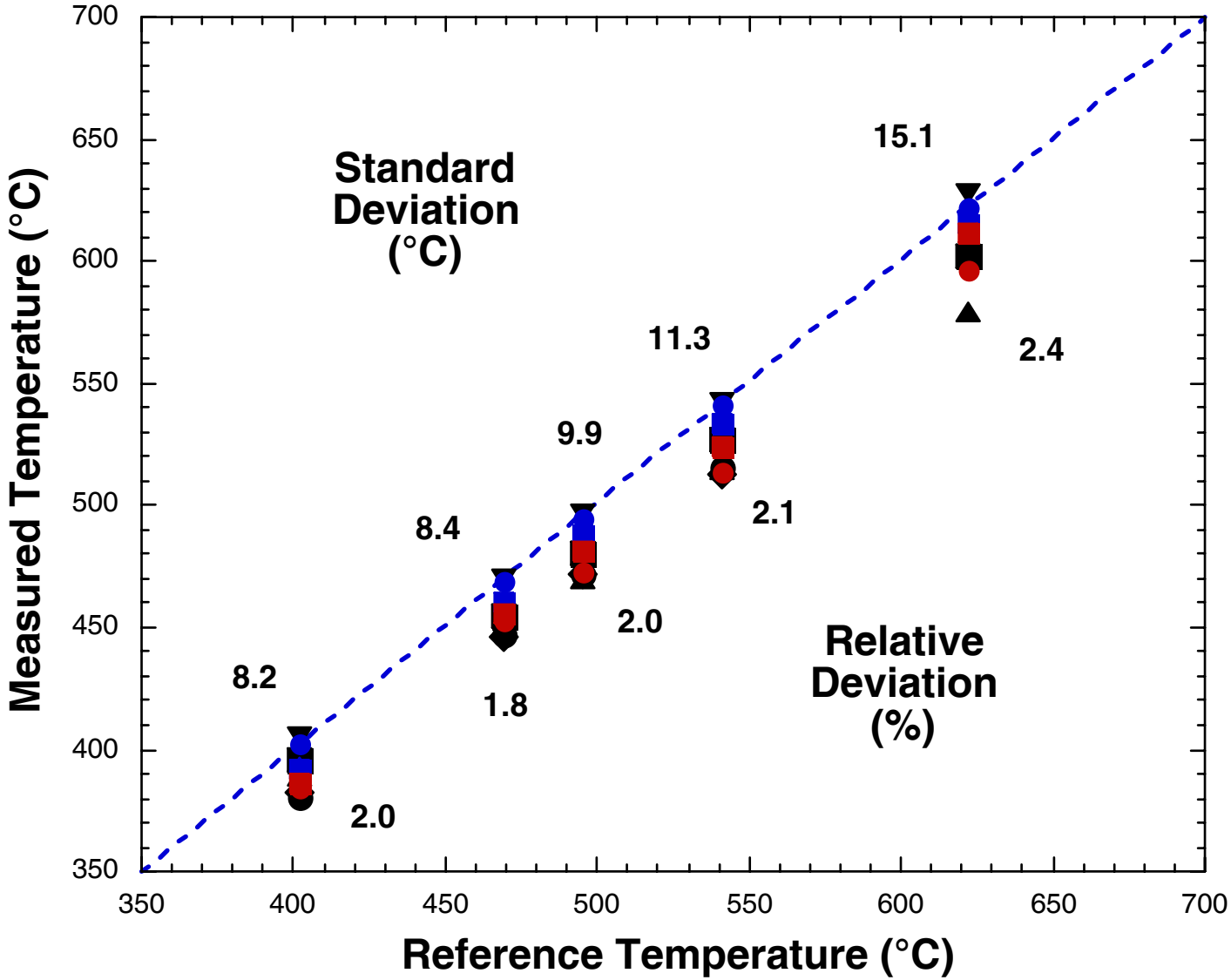
# Heat Release Capacity



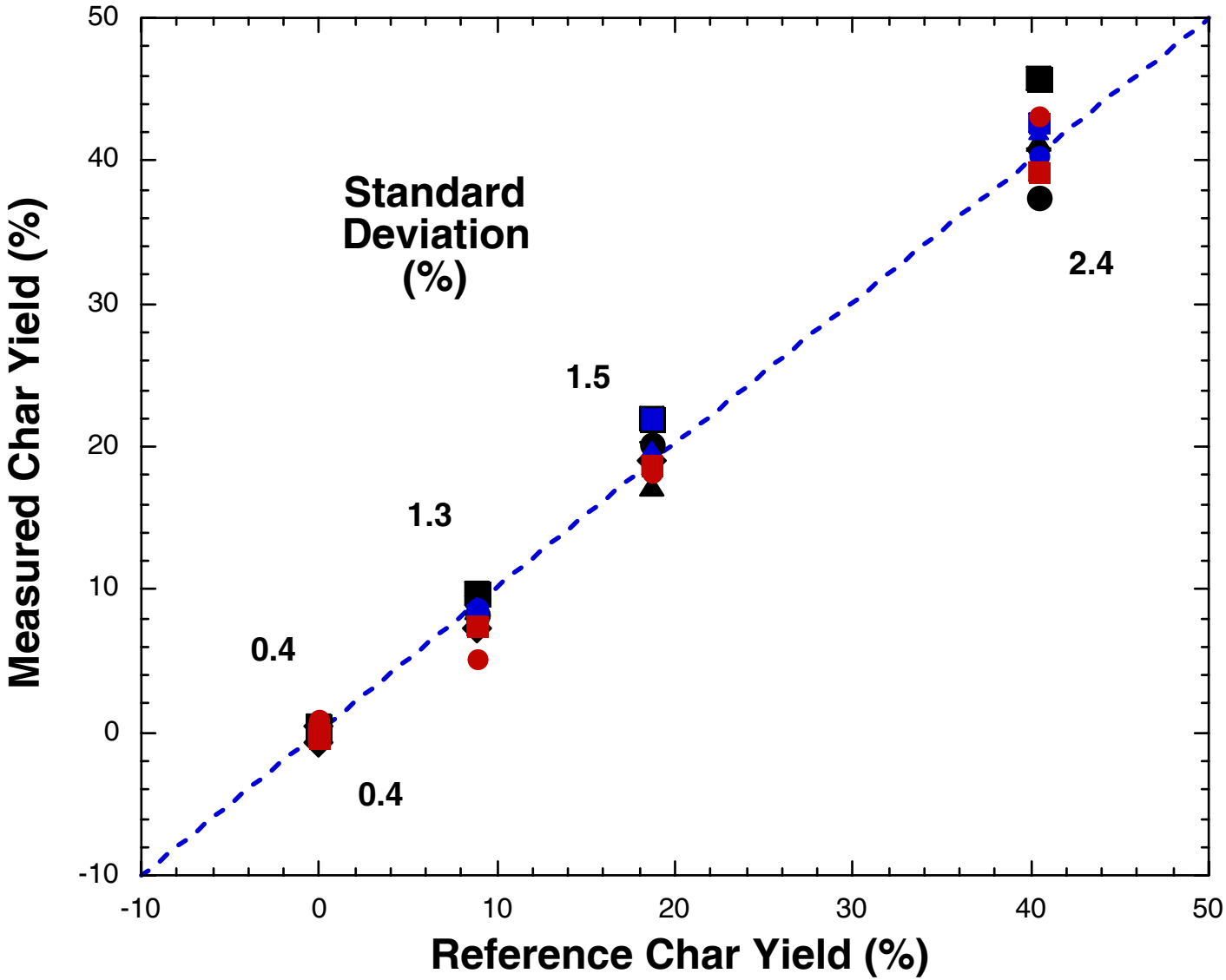
# Total Heat Release



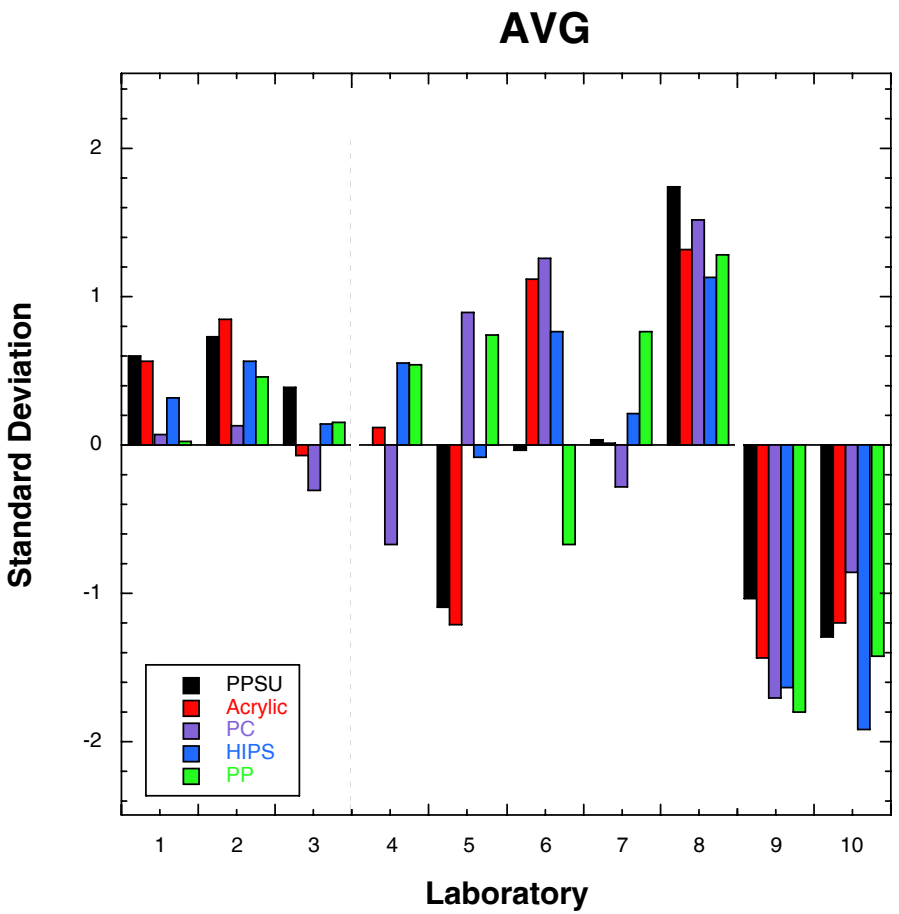
# Temperature at Peak HRR

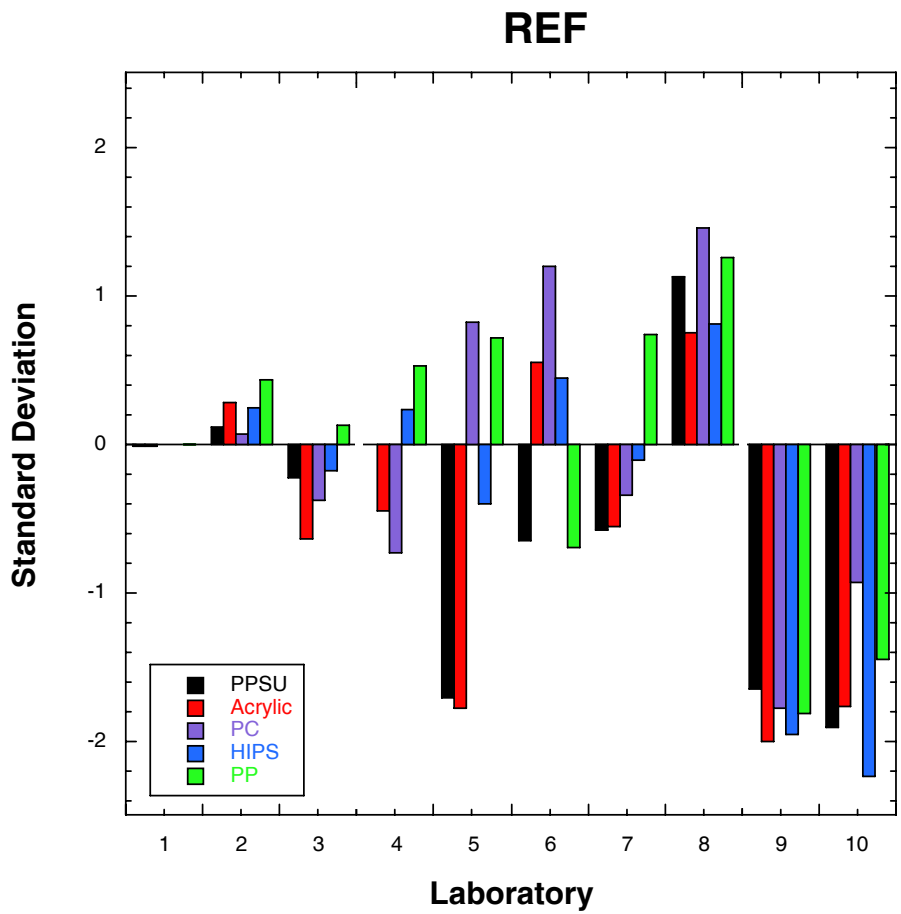


# Char Yield

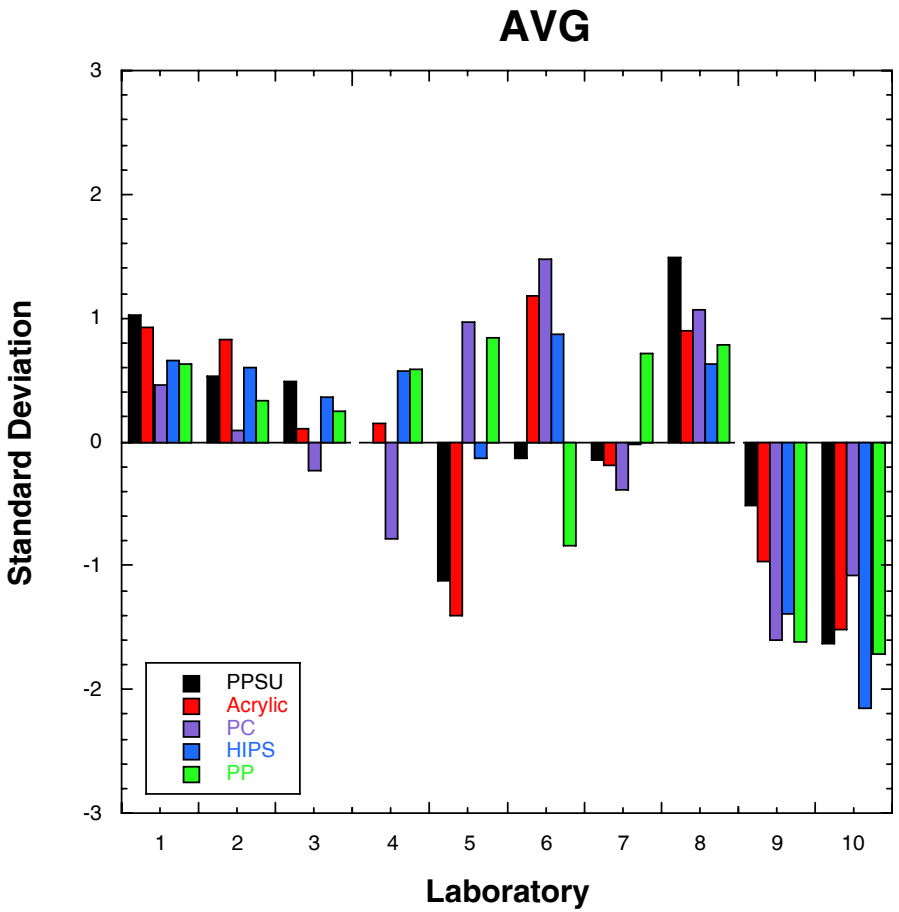

Standard Deviation (%)
0.4
0.4
1.3
1.5
2.4
Measured Char Yield (%)
Reference Char Yield (%)
50
40
30
20
10
0
-10
-10
0
10
20
30
40
50

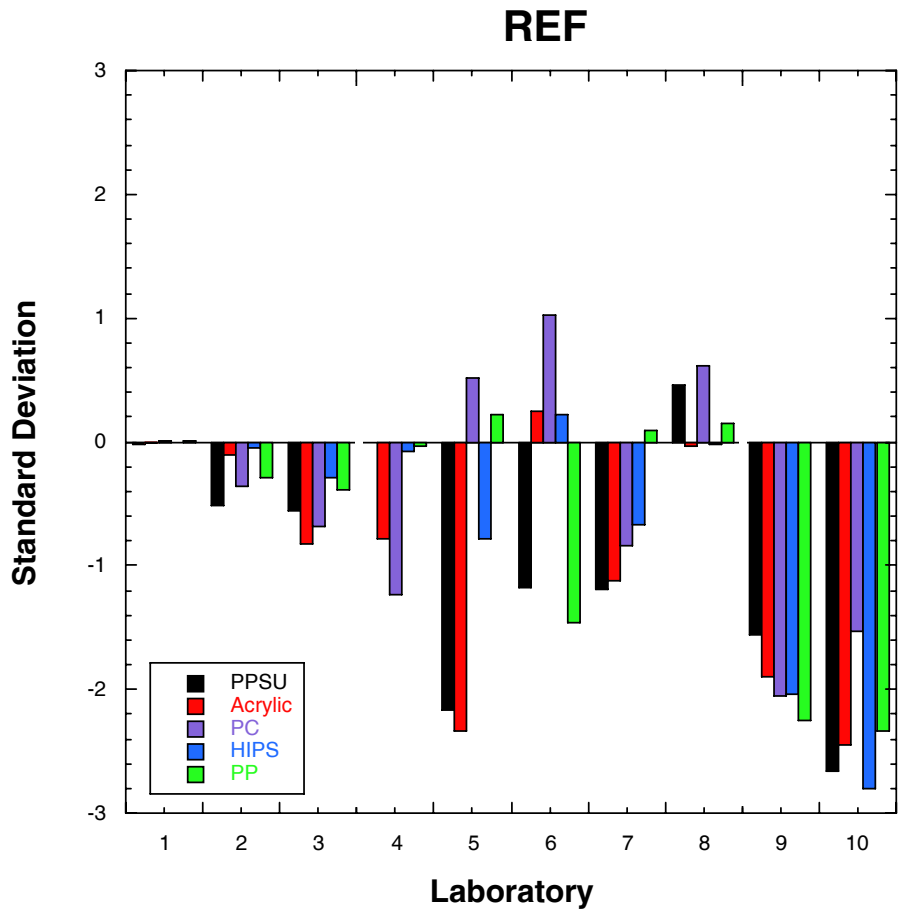
# Lab Comparison – Peak HRR



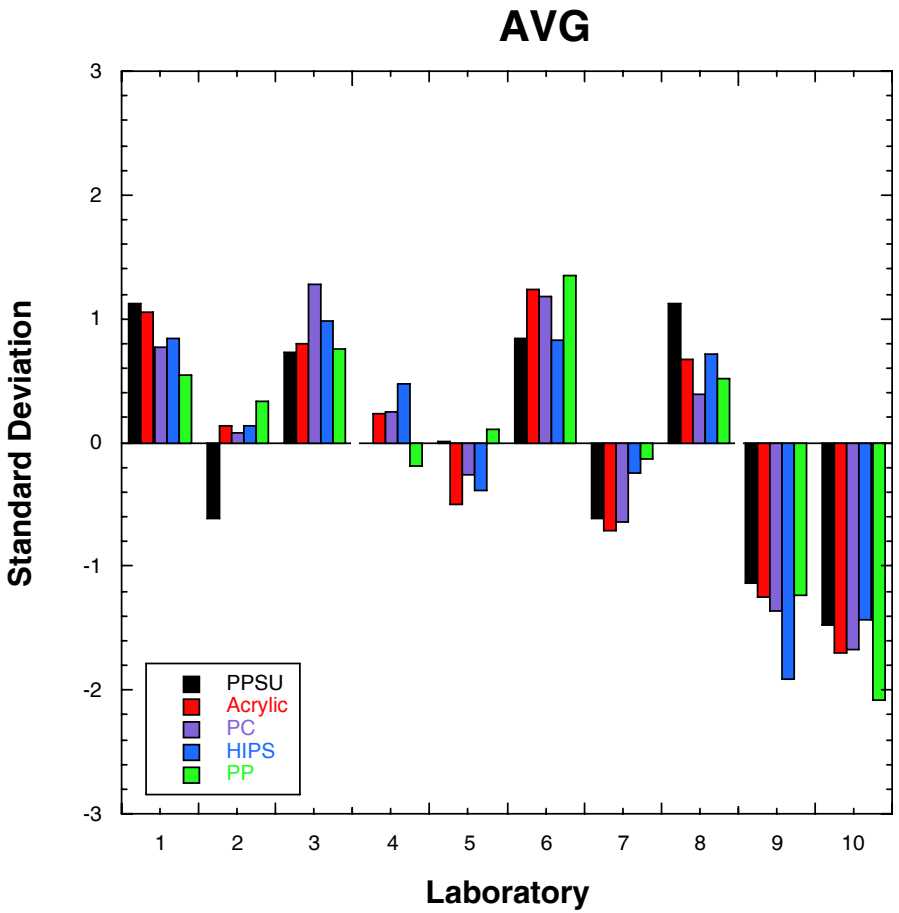



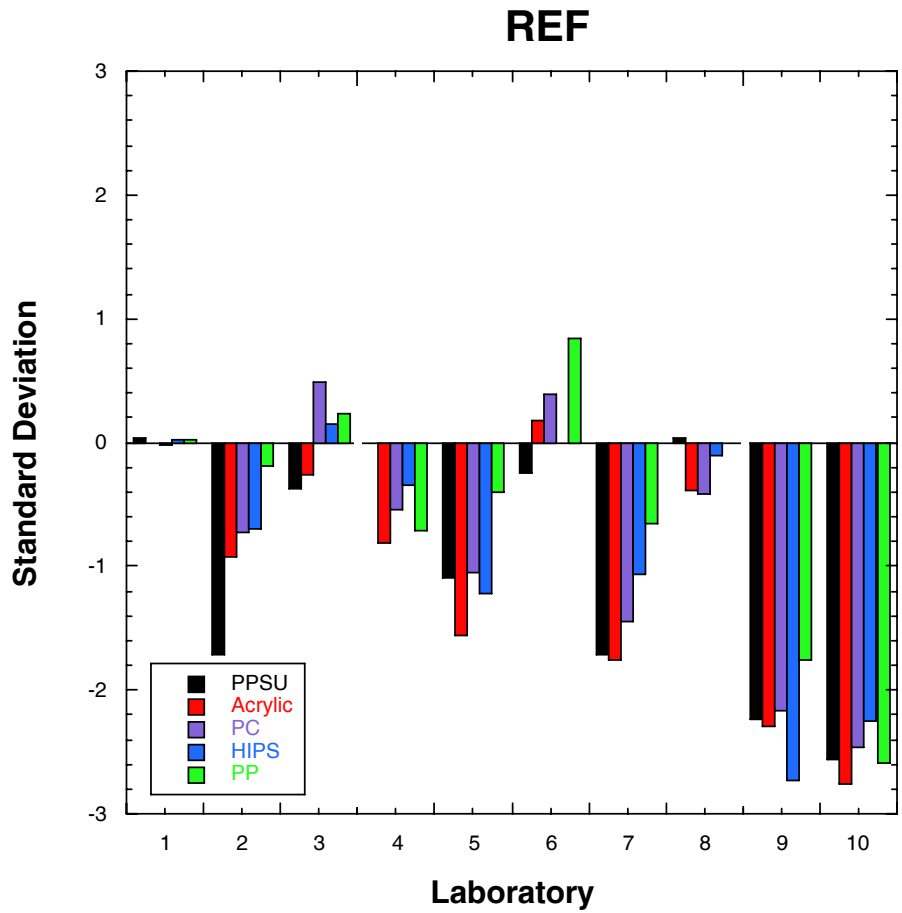
# Lab Comparison – HR Capacity



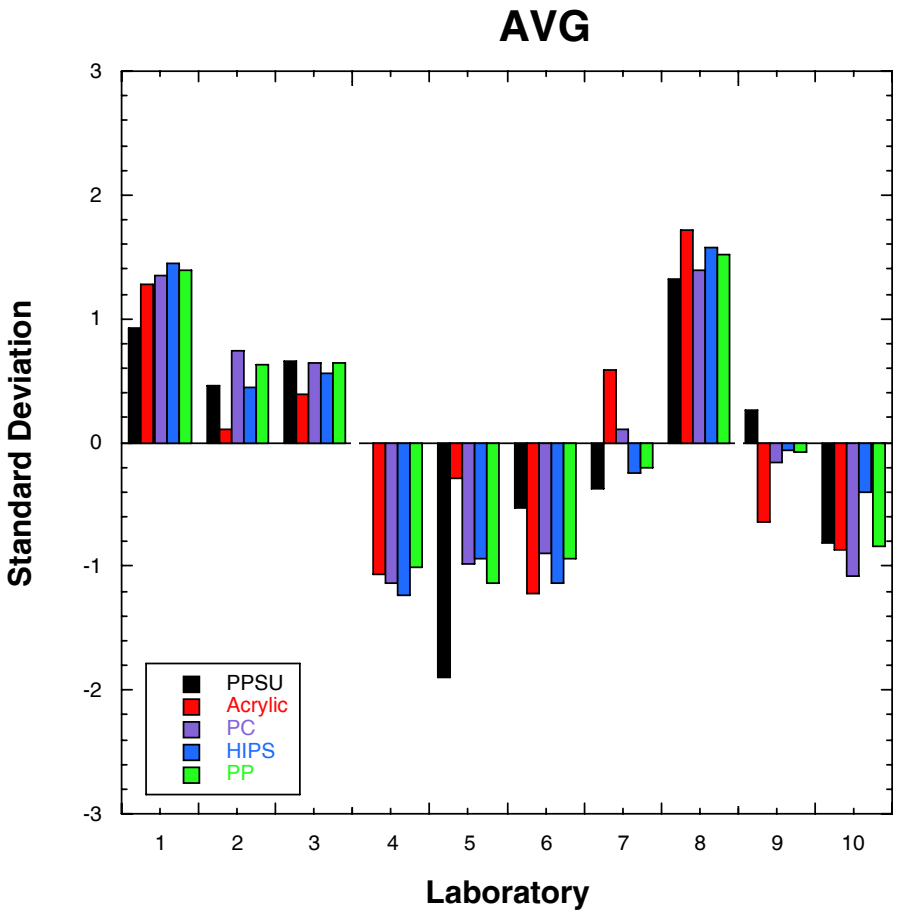



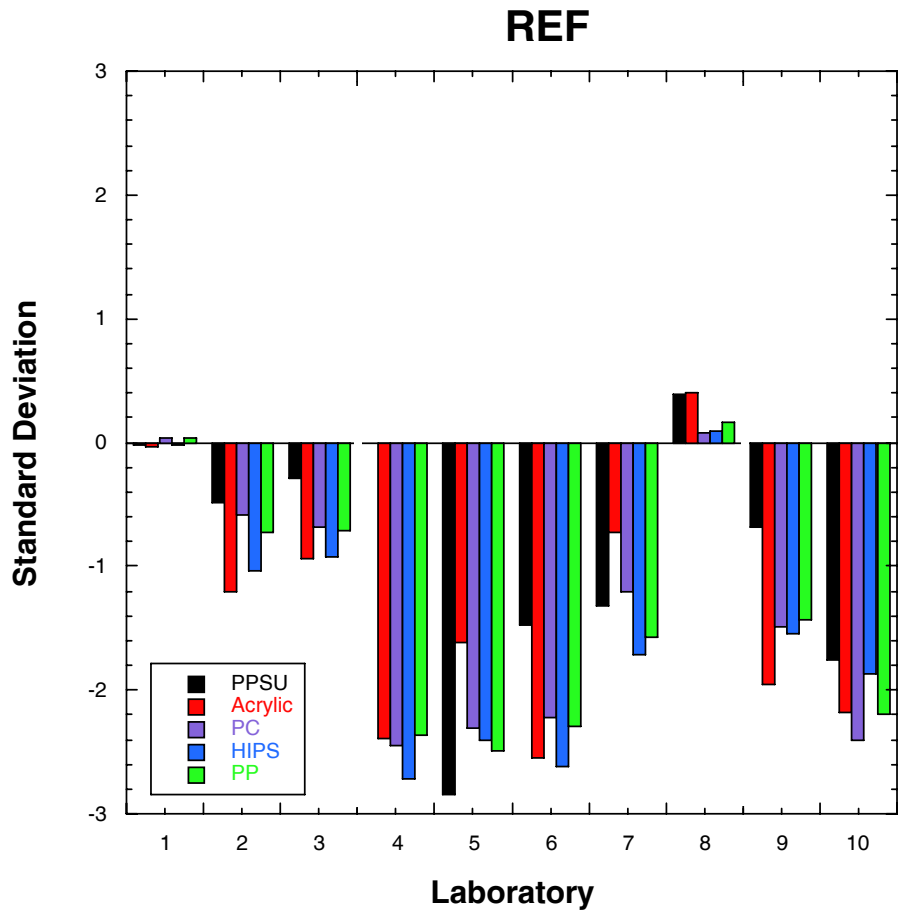
# Lab Comparison – Total HR



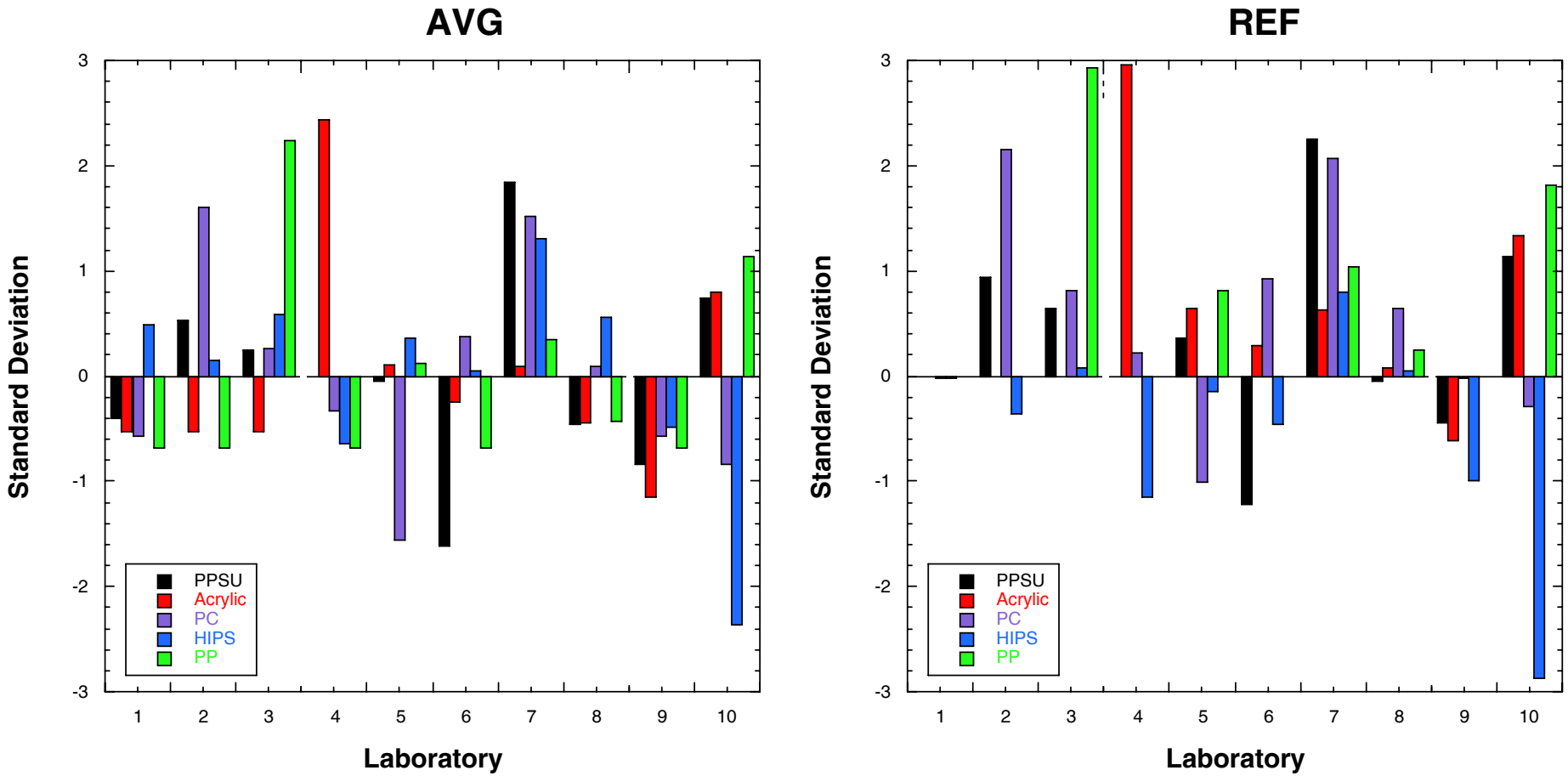
# Lab Comparison – Temperature




- **Reference temperature obtained from calibrated theromgravimetric analyzer (TGA) measurements**

# Lab Comparison – Char Yield




- **Char values show a lot of scatter due to different maximum temperature settings**

# Sources of Error

- **Heat Release Rate**
  - **Bad $O_2$ sensor calibration**
  - **Leak in system**
  - **Sample size**
  - **Bad weight measurements**
  - **Flow meter/controllers out of calibration**

- **Temperature**
  - **Thermal lag**
  - **DAQ board calibration**
  - **Time shift**

# Temperature Calibration

- **Why is it needed?**
  - **Reproducibility between laboratories**

- **What does it do?**
  - **Corrects for thermal lag when making dynamic measurements**

# Temperature Calibration - Metals


**Indium: 156.6°C**

**Tin: 231.9°C**

**Zinc: 419.5°C**

**Antimony: 630.7°C**

**Aluminum: 660.4°C**

**Sodium Chloride: 801°C**

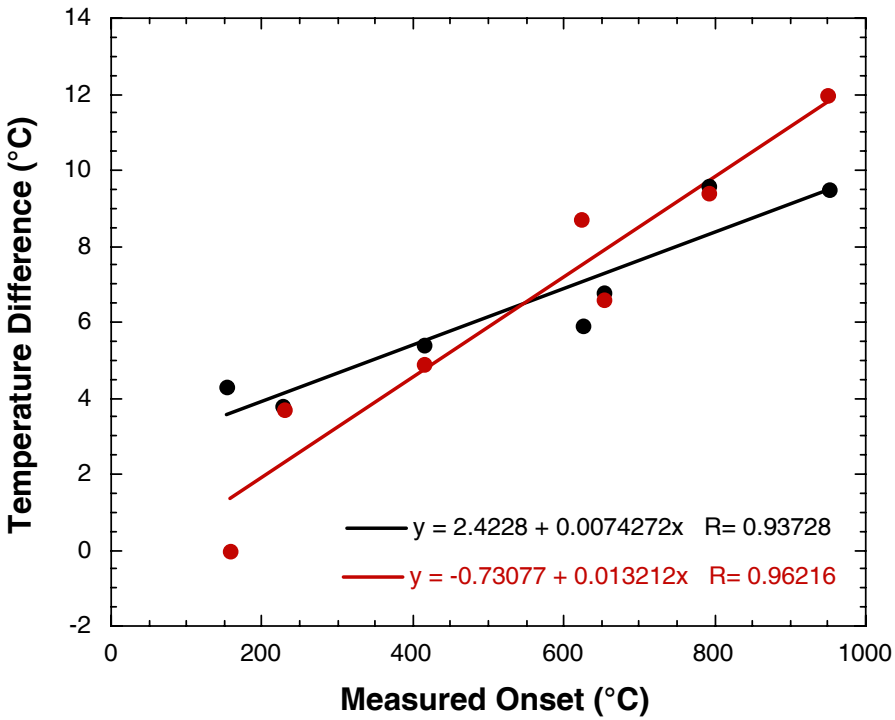
**Silver: 961°C**

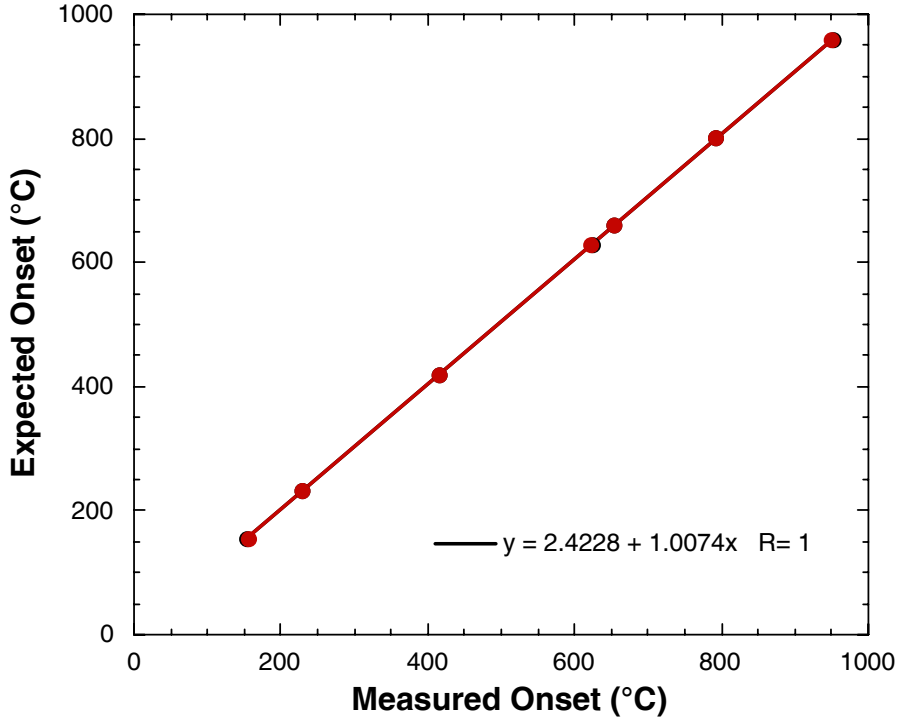
**Calibration Kit**

**99.999% Pure**

**New Dedicated Pans**

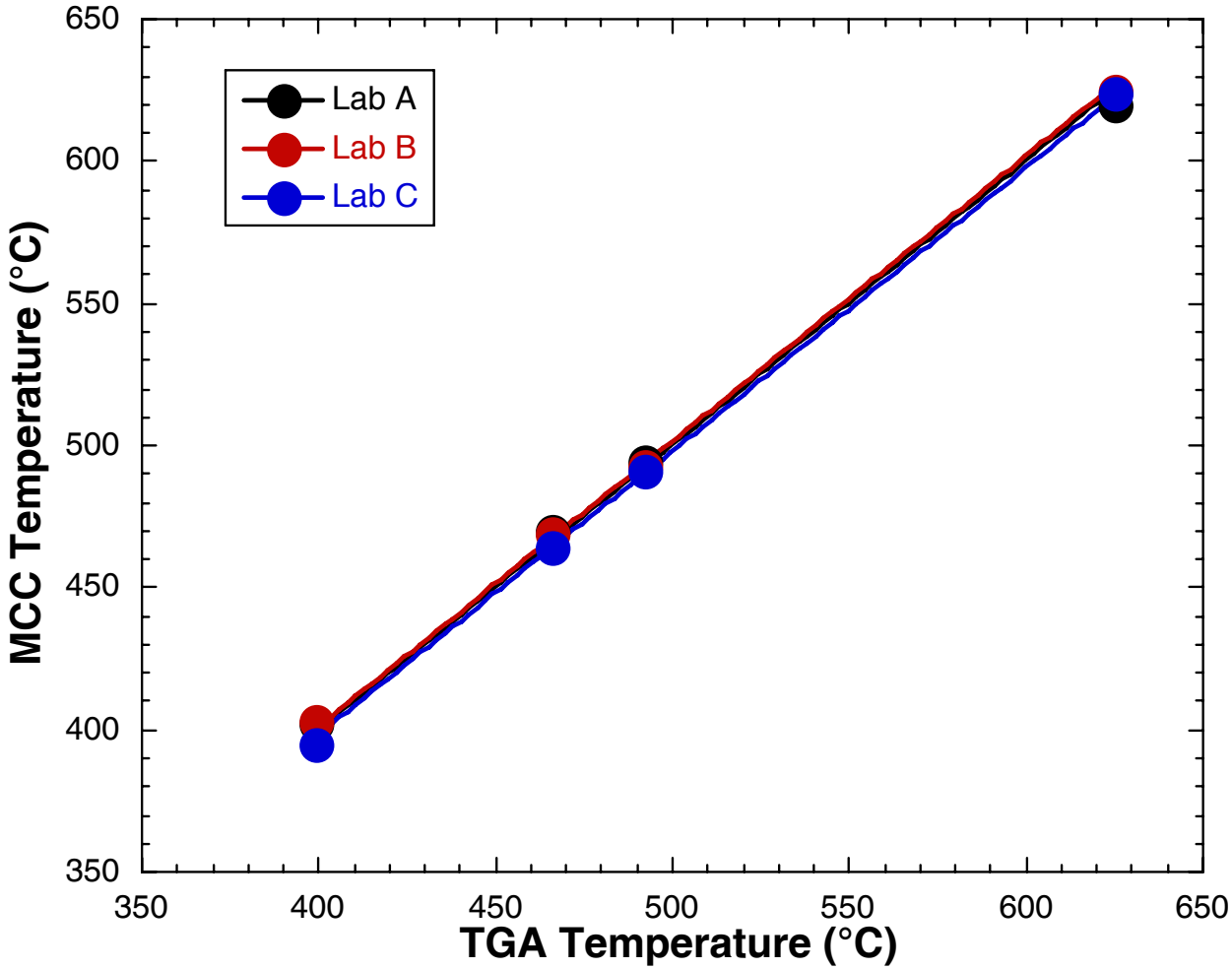
# Temperature Calibration – Metal Foils



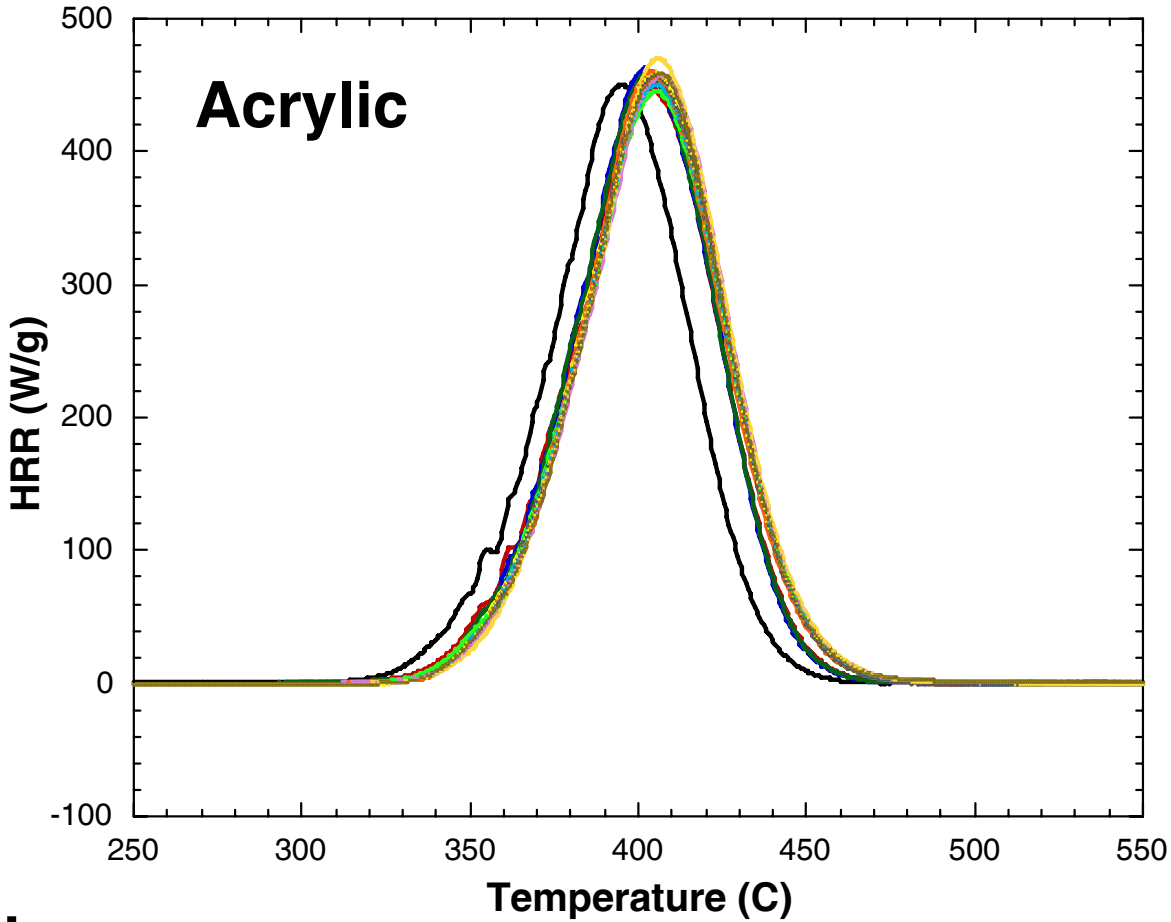

- Linear deviation
- Largest deviation at high temperature
- “Melting” observed with little to no discoloration
- Correlates with TGA

# MCC vs TGA




**MCC T calibration brings measured temperature within 4 degrees of TGA and 1 degree of other MCC**

# Repeatability & Reproducibility




- 13 Tests
- 3 Laboratories
- 4 Operators

# Conclusions & Future Work

- More labs will be added to study as the data is received
- Temperature calibration will be implemented
- Comprehensive users guide will be available soon
- More explicit instructions will be given to the participants in the future
- One of the manufacturers has issues that need to be dealt with
- Inter-laboratory study – round 2

# WANTED: More Participants

**Contact Information:**

**Richard Walters**
**FAA William J Hughes Technical Center**
**Fire Safety Team AJP-6320**
**Atlantic City International Airport, NJ 08405**
**609 485-4328**
**richard.walters@faa.gov**