

International Aircraft Materials Fire Test Working Group
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STANDARD TEST METHOD FOR FLAMMABILITY SCREENING OF AC MATERIALS



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OUTLINE



Standard Test Method for Flammability Screening of AC Materials

- **FLAMMABILITY PROPERTIES FROM MICROCALORIMETRY**
- **MICROCALORIMETRY AND:**
 - Ignitability (Radiant Panel Flame Spread)**
 - Heat Release Rate (OSU and Intermediate Scale Tests)**
 - Flame Resistance (Bunsen burner tests)**
 - Research (Product Development)**
- **SUMMARY AND CONCLUSIONS**

GOAL: PREDICT RESULTS OF AC FIRE TESTING



**FLAME
RESISTANCE**



**HEAT
RELEASE**



FLAME SPREAD



**INTERMEDIATE
SCALE
FIRE TESTS**

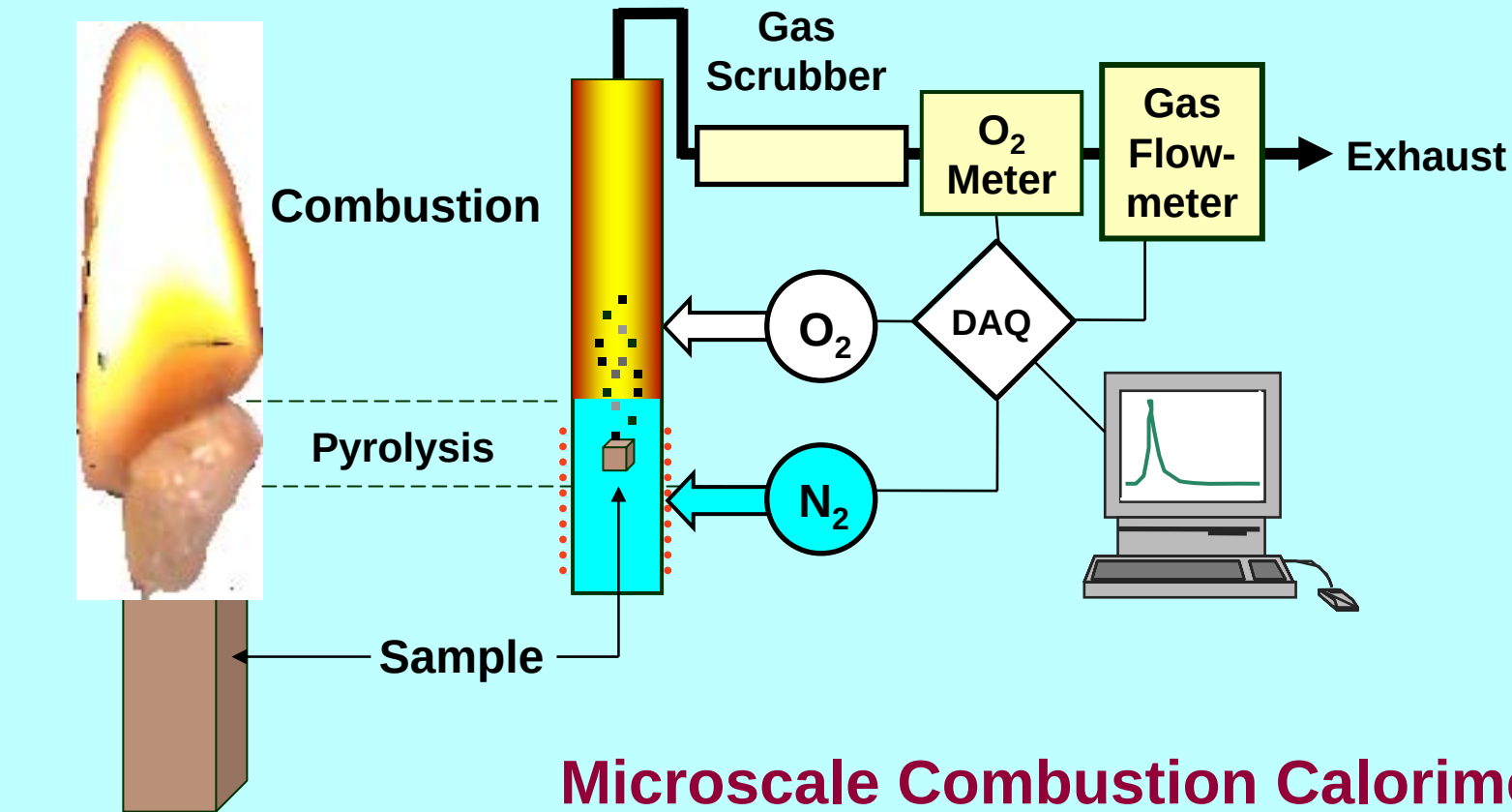


**Combustion
Properties**
 h_c^0 , T_{ign} , η_c

Semi-Empirical Correlation

$$\frac{\text{Milligrams}}{\text{Kilograms}} = 10^{-6} = \text{"Microscale"}$$

APPROACH: Simulated Flaming Combustion



**Flaming
Combustion**

Microscale Combustion Calorimetry

- Sample heated at constant rate in N₂
- Complete oxidation of fuel gases
- Oxygen consumption calorimetry to measure heat release rate

APPARATUS CONSTRUCTION



Combustor

Mixing
Section

Pyrolyzer

Oxygen Inlet

Scrubber

Flow Meter

O₂ Sensor

Exhaust

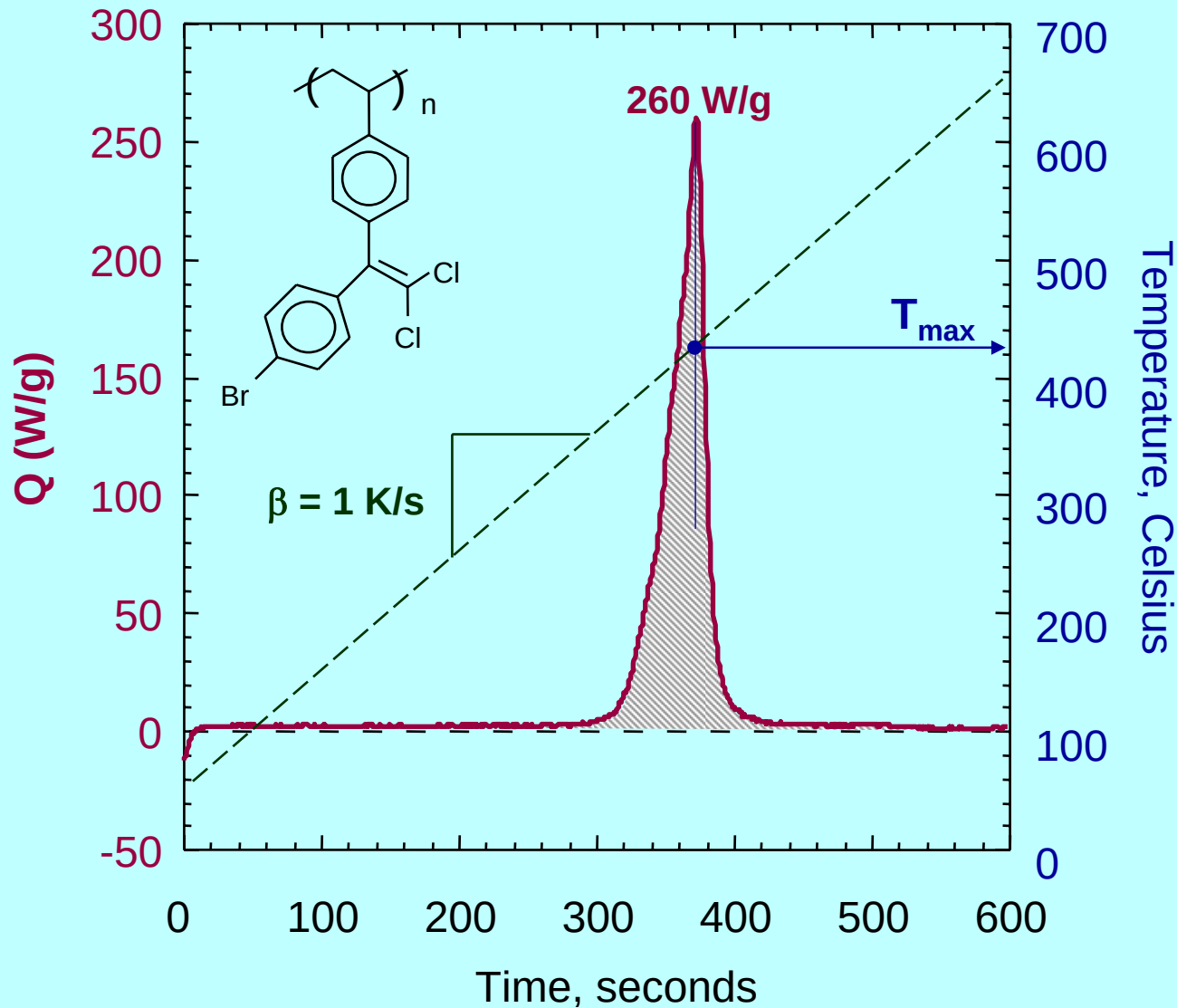
Sample Cup

Purge Gas Inlet

Sample Post &
Thermocouple

$$Q(t) = \frac{\rho_{O_2} C}{m_0} \{F[O_2]\}(t)$$

PCFC DATA: Example for Research Polymer



$$\eta_c = \frac{260 \text{ W/g}}{1 \text{ K/s}}$$

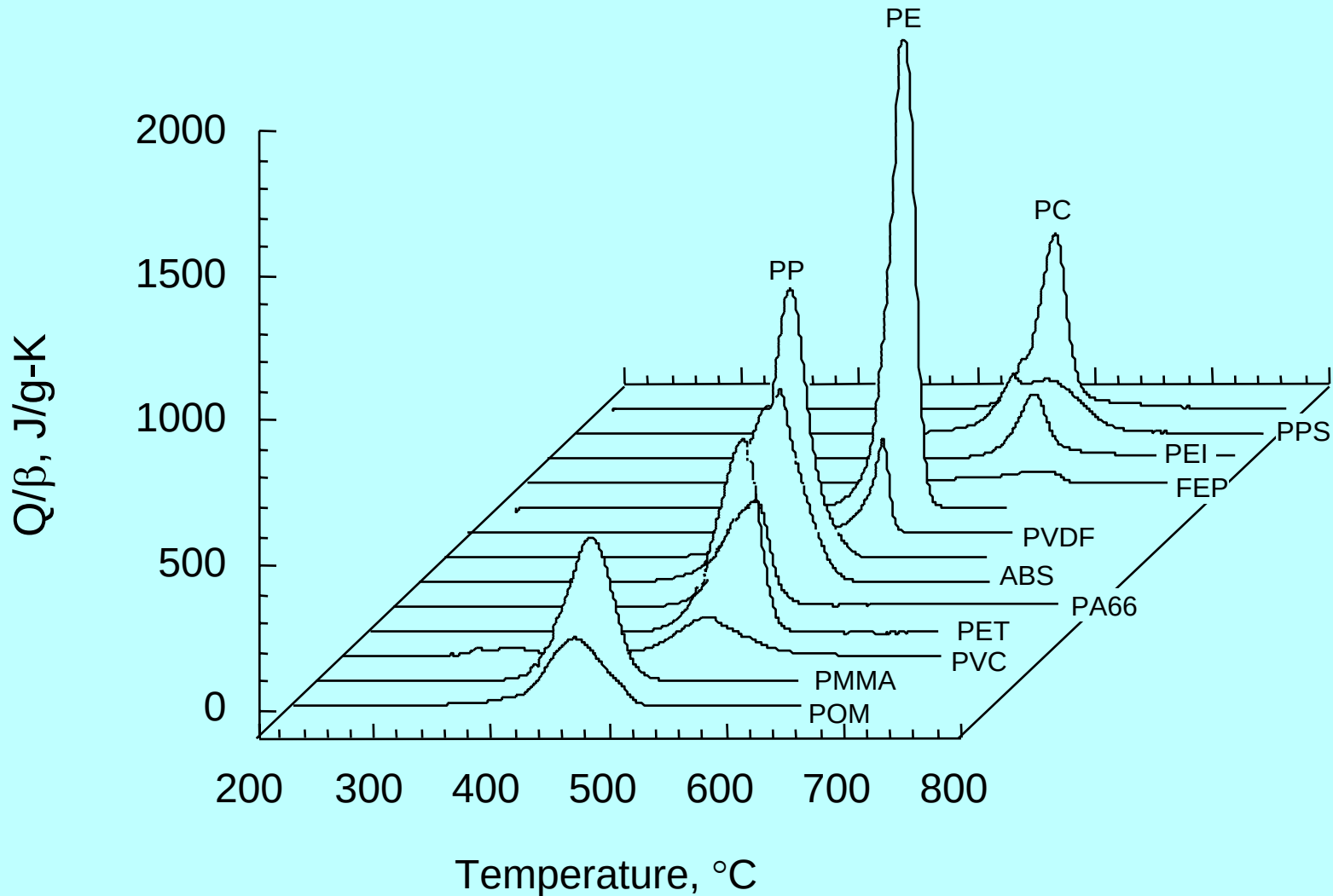
$$= 260 \frac{\text{J}}{\text{g-K}}$$

$$T_{\text{max}} = 425 \text{ }^\circ\text{C}$$

$$h_c^0 = \int_0^\infty Q(t)dt$$

$$= 13 \frac{\text{kJ}}{\text{g}}$$

MICRO HRR DATA FOR COMMON PLASTICS



MOTIVATION FOR MICRO SCALE TEST



Small Specimens (milligrams instead of kilograms)

Rapid Results (minutes instead of hours)

Reproducible ($\pm 7\%$)

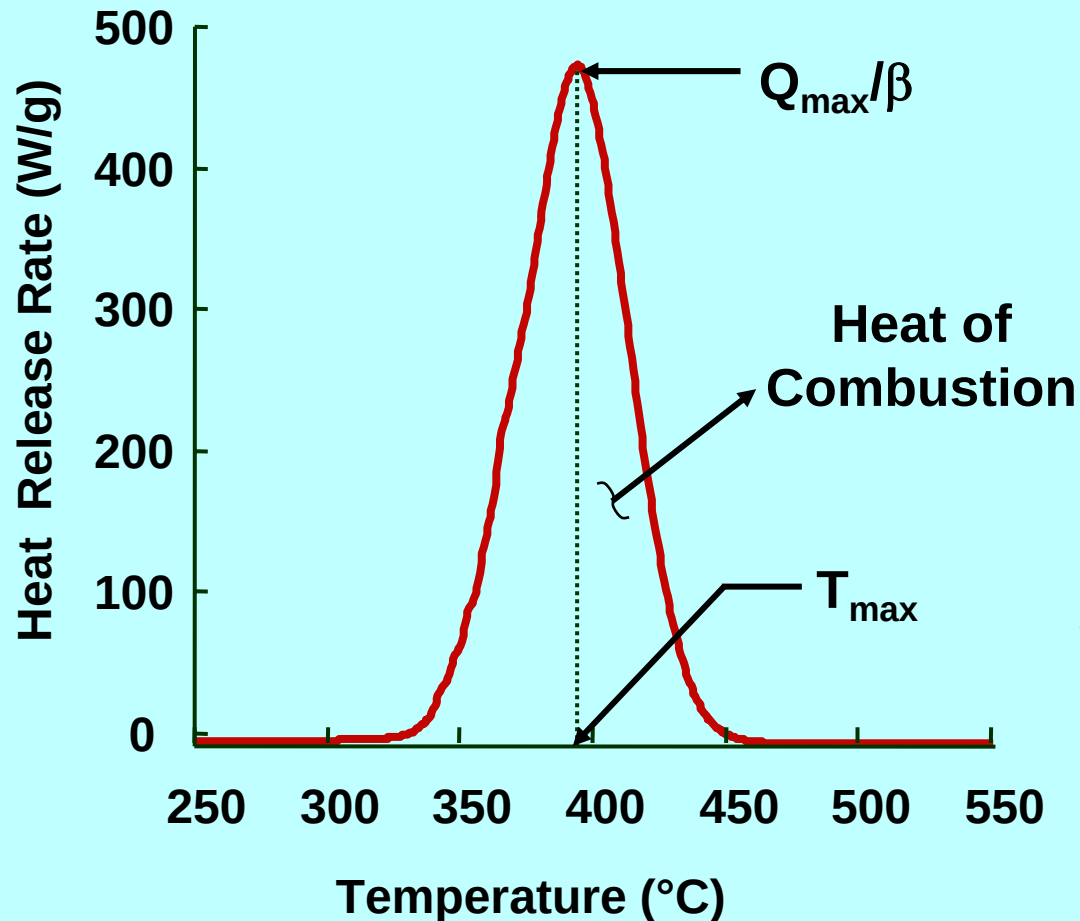
Repeatable ($\pm 5\%$)

Fire Properties Measured (used with models to correlate other tests)

PROPERTIES RELATED TO FLAMMABILITY



PCFC Property



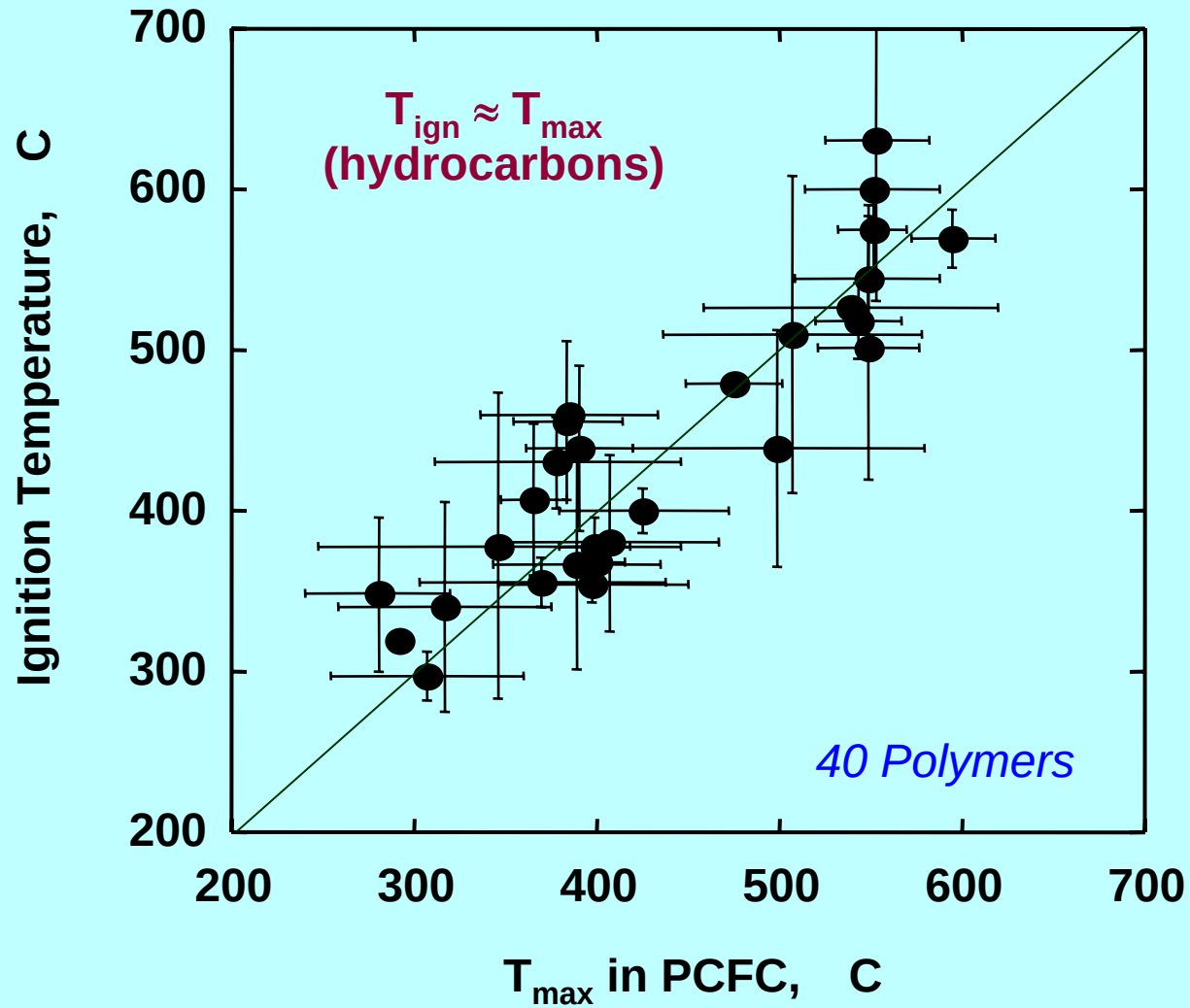
Flammability Parameter

- Heat release rate
- Flame resistance (UL94 , LOI)
- Fire Load
- Combustibility
- Ignitability (T_{ign} , CHF)
- Glow Wire Ignition



IGNITABILITY

IGNITION TEMPERATURE AND $T_{\max}$



CRITICAL HEAT FLUX (CHF) FOR IGNITION FROM $T_{\max}$

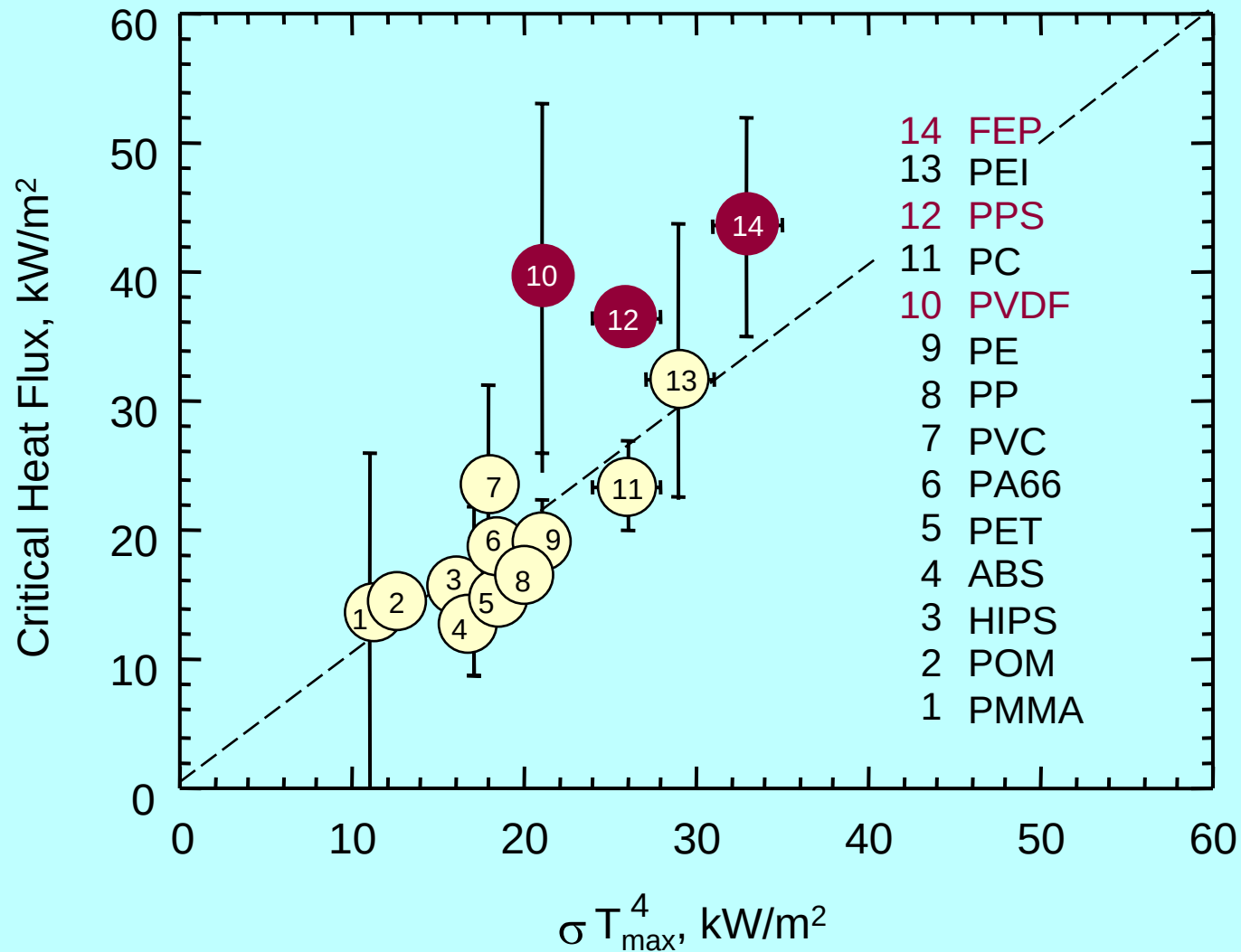


POLYMER	$T_{\max}$ (°C)		CHF, kW/m ²	
			$\sigma T_{\max}^4$	Measured*
PMMA	401	8	11 - 12	6 - 23
POM	409	10	11 - 13	13
HIPS	463	10	15 - 17	15
ABS	467	12	16 - 18	9 - 15
PET	471	12	16 - 18	10 - 19
PVC**	478	8	17 - 18	15 - 28
PA66	482	11	17 - 19	15 - 21
PP	493	10	18 - 20	15 - 16
PVDF**	510	2	21	30 - 50
PE	514	10	20 - 22	15 - 20
PC	556	9	25 - 28	15 - 20
PPS**	551	18	24 - 28	35 - 38
PEI	576	10	28 - 31	25 - 40
FEP	600	10	31 - 34	38 - 50

***Note poor agreement
for halogen (Cl, F) and
heteroatom (S)
polymers for which
 $T_{\text{ign}} > T_{\max}$*

*D. Drysdale, A. Tewarson, R.E. Lyon

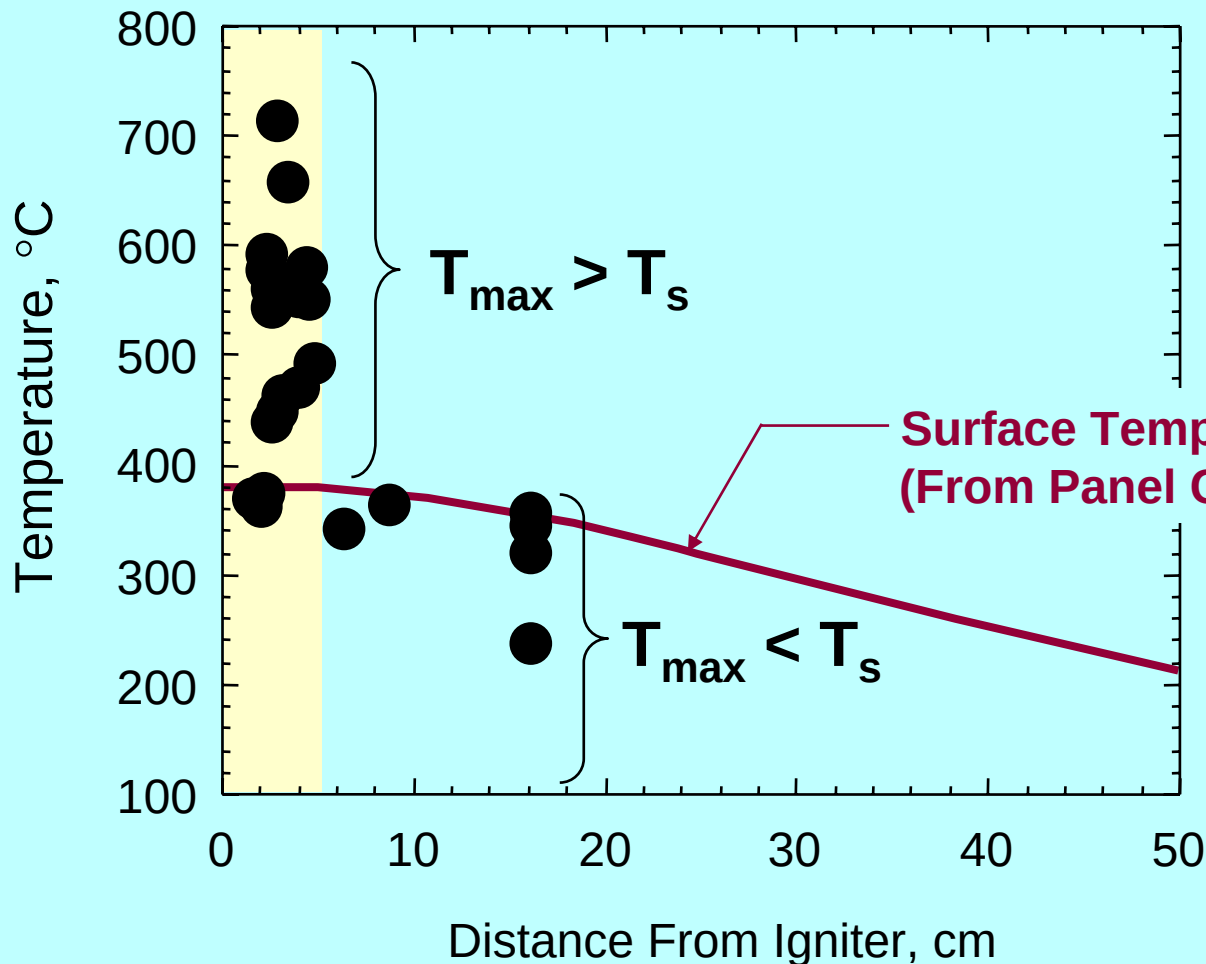
CRITICAL HEAT FLUX: Graphical Comparison



PERFORMANCE OF HIDDEN MATERIALS



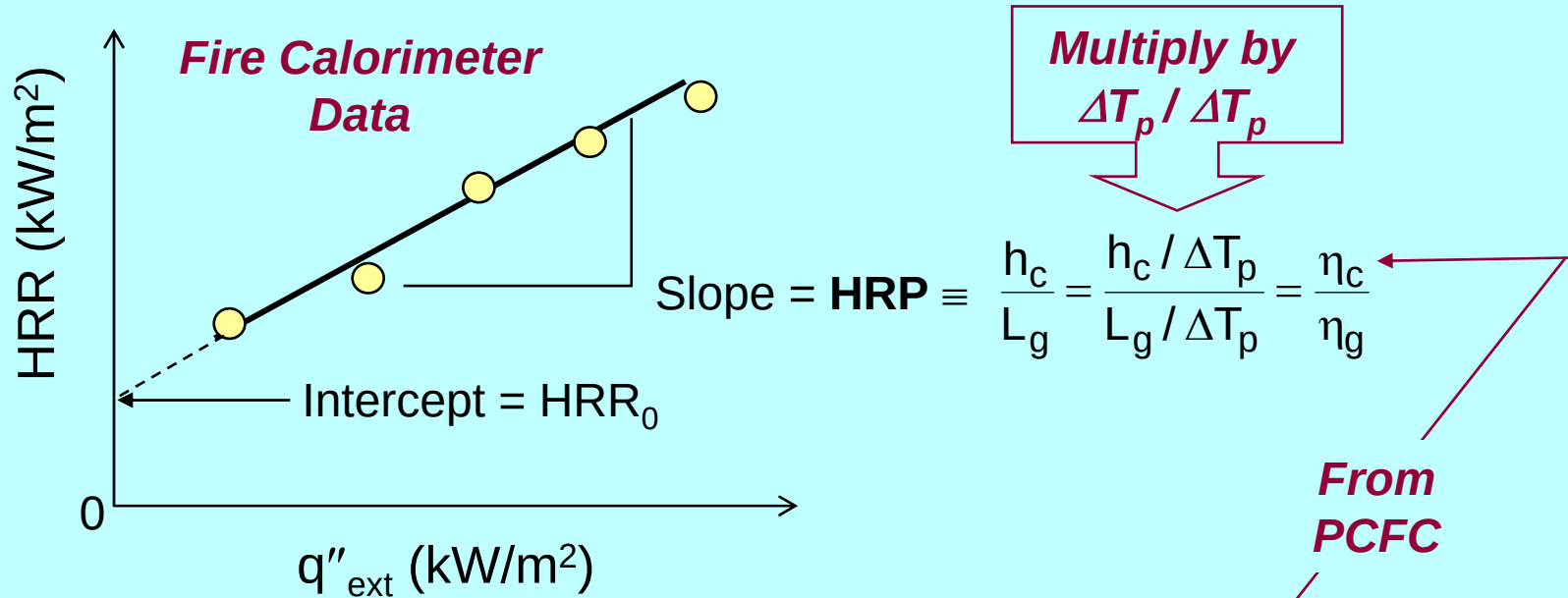
Heat Resistance / T_{max} is a Good Predictor of Flame Spread in FAA Radiant Panel Test





HEAT RELEASE RATE

HEAT RELEASE RATE: Macro / Micro

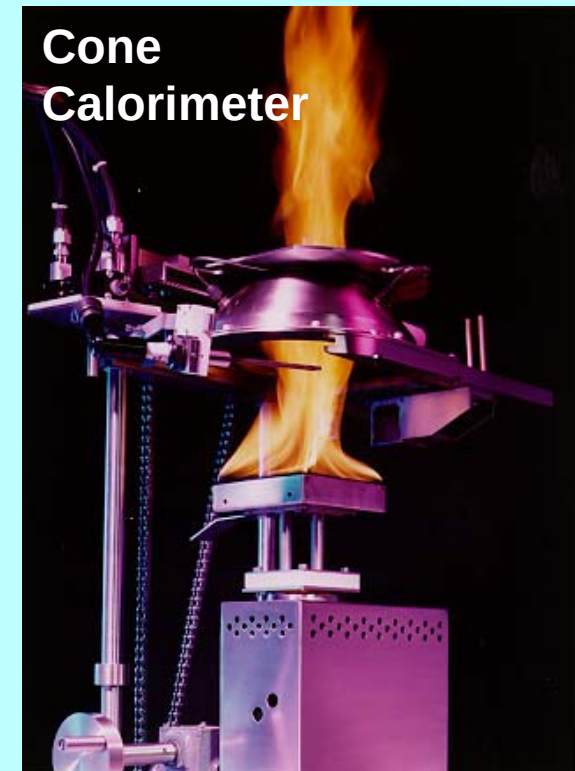
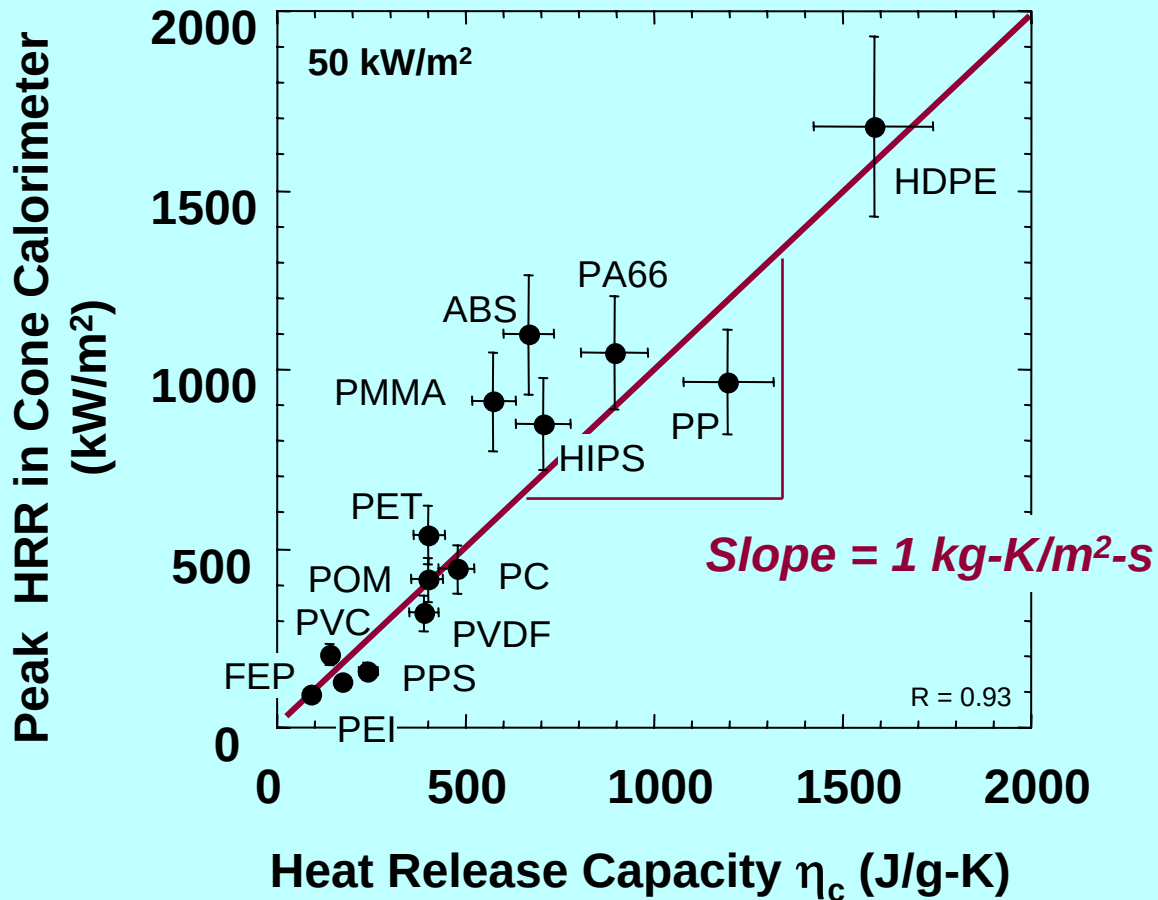


$$\text{HRR} = \text{HRR}_0 + \frac{\eta_c}{\eta_g} q''_{\text{ext}}$$

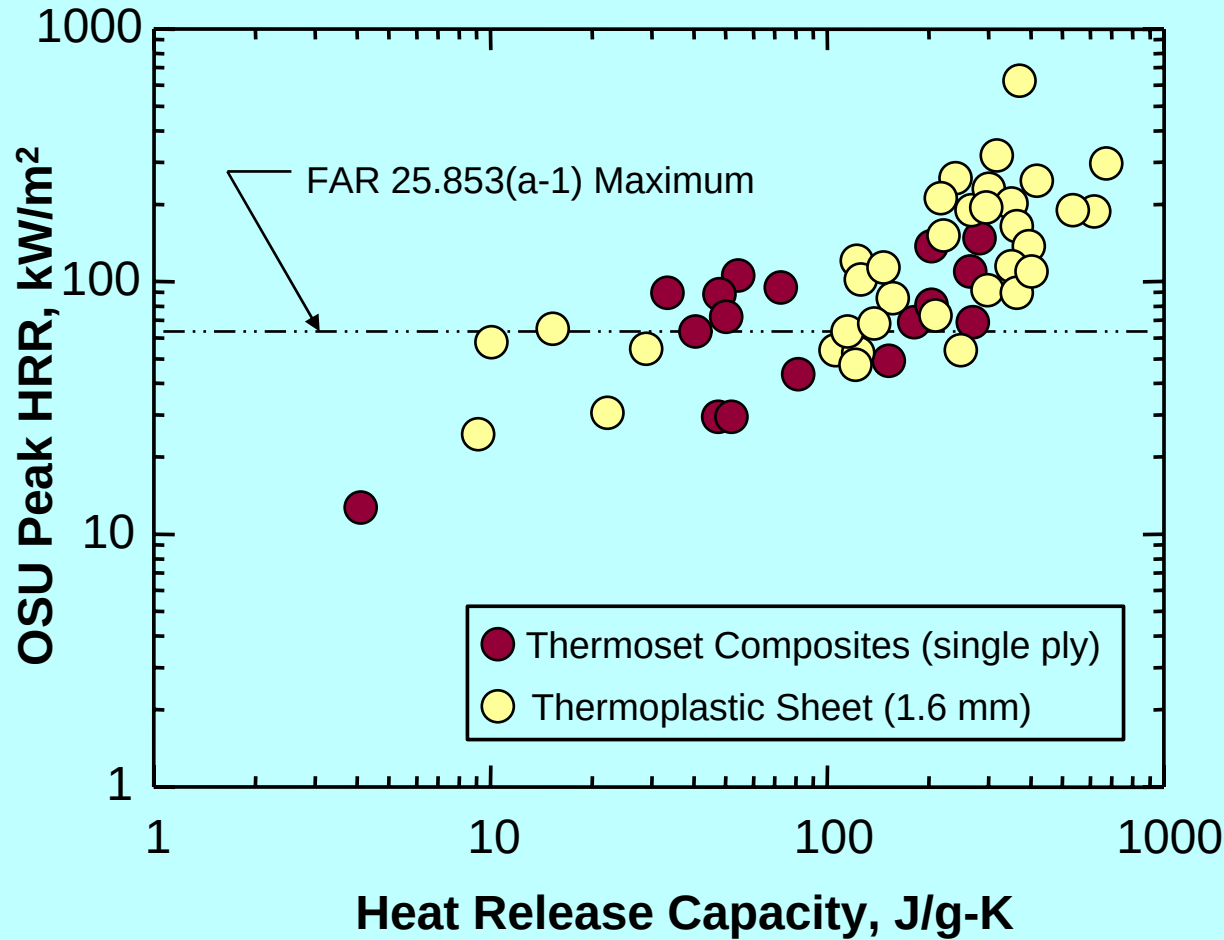
CONE HRR AND MICROCALORIMETER η_c



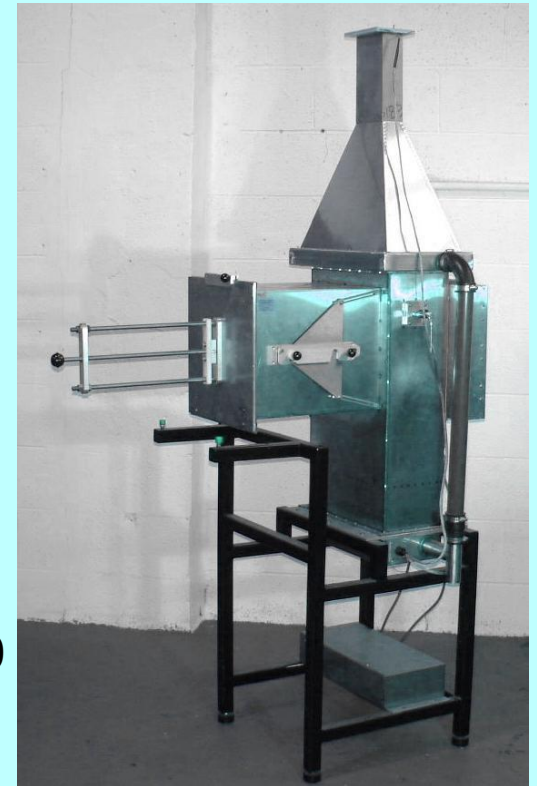
$$\text{HRR} \approx (q''_{\text{ext}}/\eta_g) \eta_c = (50 \text{ kW/m}^2)/(50 \text{ kJ/kg-K}) \eta_c$$
$$= (1 \text{ kg-K/m}^2\text{-s}) \eta_c$$



OSU HRR AND MICROCALORIMETER η_c



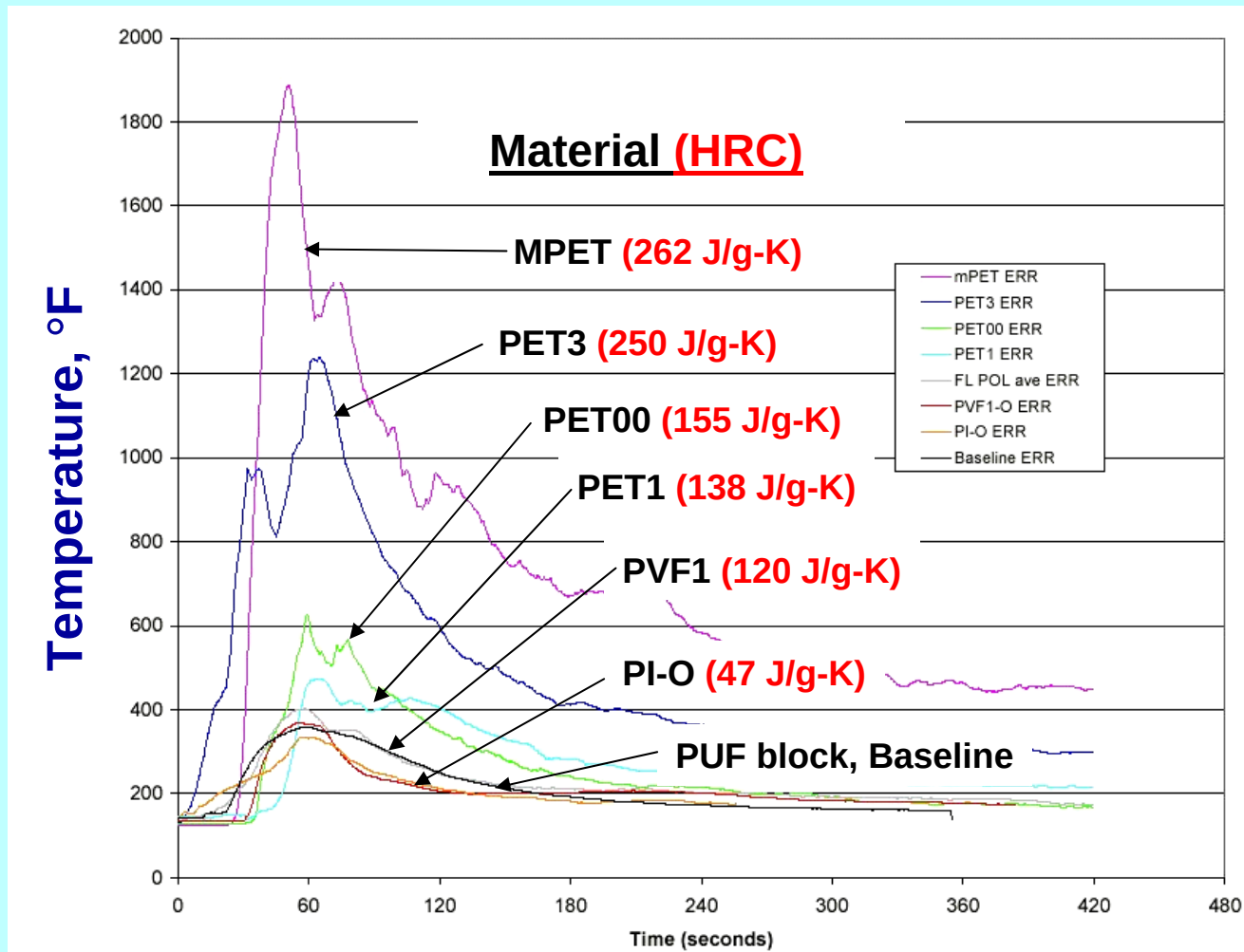
ASTM E 906/OSU
Rate of Heat
Release Apparatus



THERMO ACOUSTIC INSULATION



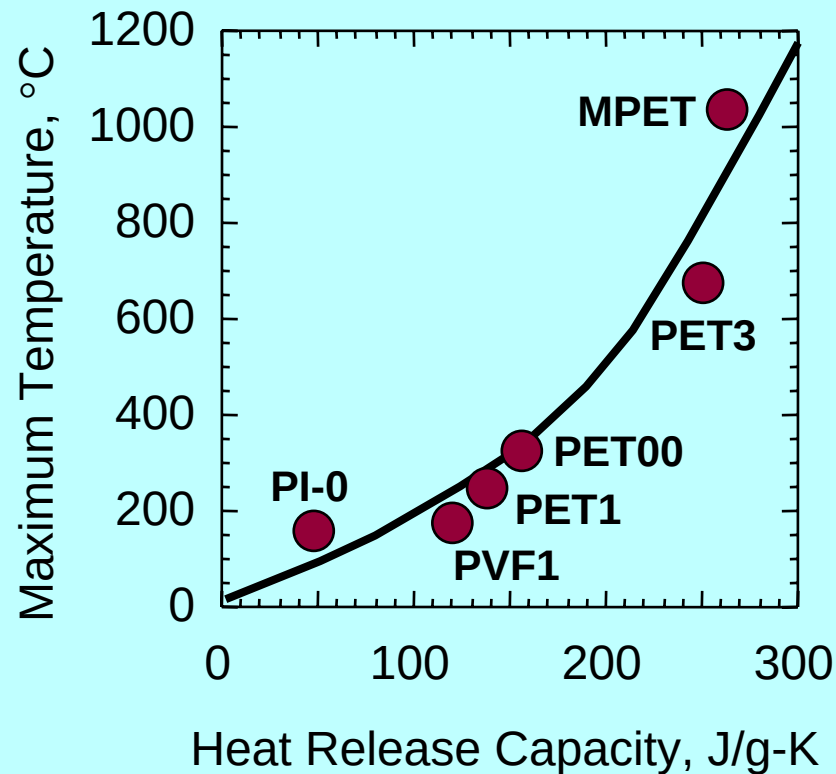
Intermediate Scale and Micro Scale Results



TEMPERATURE RISE versus HRC



Intermediate Scale Test of Thermo-Acoustic Insulation





FLAME RESISTANCE

FLAME RESISTANCE AND MICROSCALE HRR



Isolated flame / Unforced Combustion

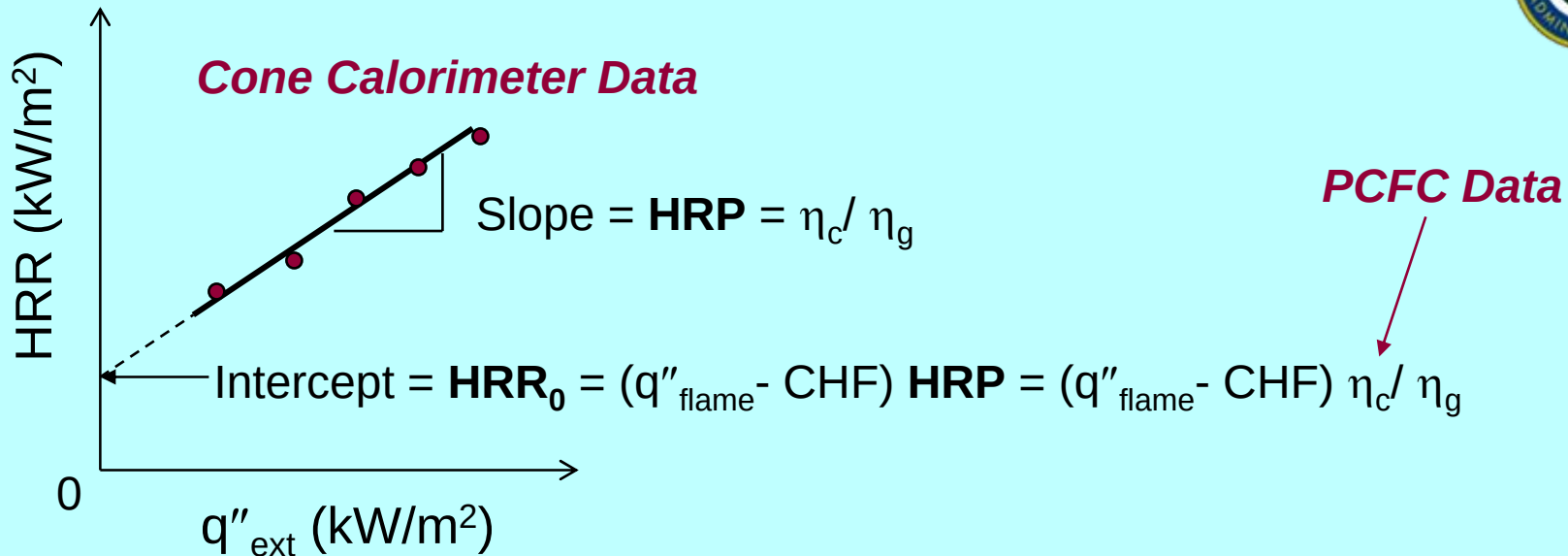


Bunsen Burner Tests



**Limiting Oxygen
Index**

FLAME RESISTANCE AND MICROCALORIMETRY



Extinction in flame tests ($q''_{\text{ext}} = 0$) when $\text{HRR}_0 \leq \text{HRR}^* \approx 50\text{-}100 \text{ kW/m}^2$,

Limiting Oxygen Index = $[O_2^*]$

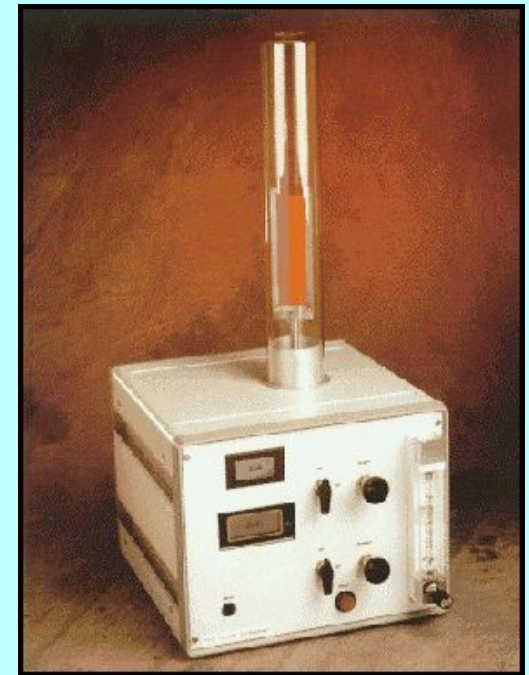
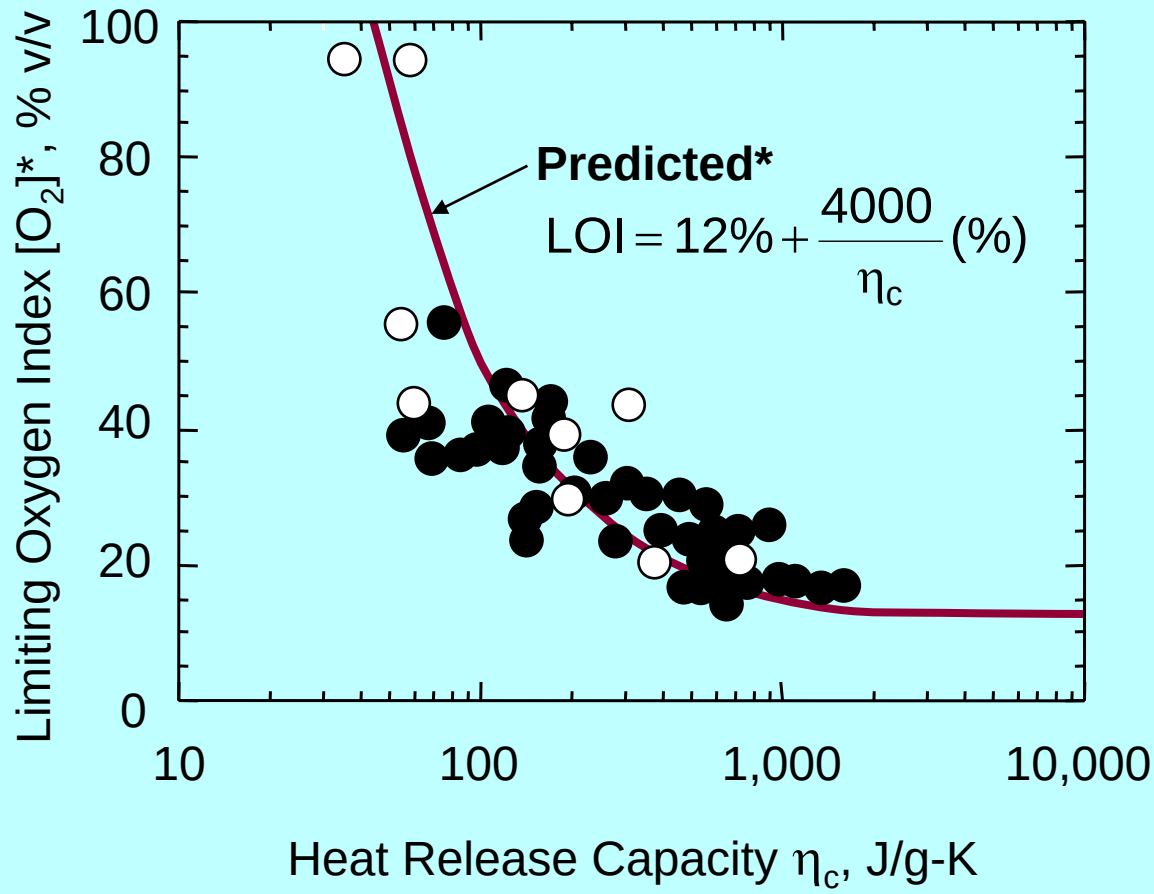
$$q''_{\text{flame}} \propto [O_2] = a [O_2] = 1.4 \text{ kW/m}^2\text{-}\%[O_2]$$

$$[O_2^*] = \frac{\text{CHF}}{a} + \frac{\text{HRR}^* \eta_g / a}{\eta_c} \approx 12\% + \frac{4000}{\eta_c} (\%)$$

UL 94 V Criterion

$$\eta_c^* \leq \frac{\text{HRR}^* \eta_g}{(q''_{\text{flame}} - \text{CHF})}$$

FLAME RESISTANCE (LOI) AND MICROCAL (η_c)

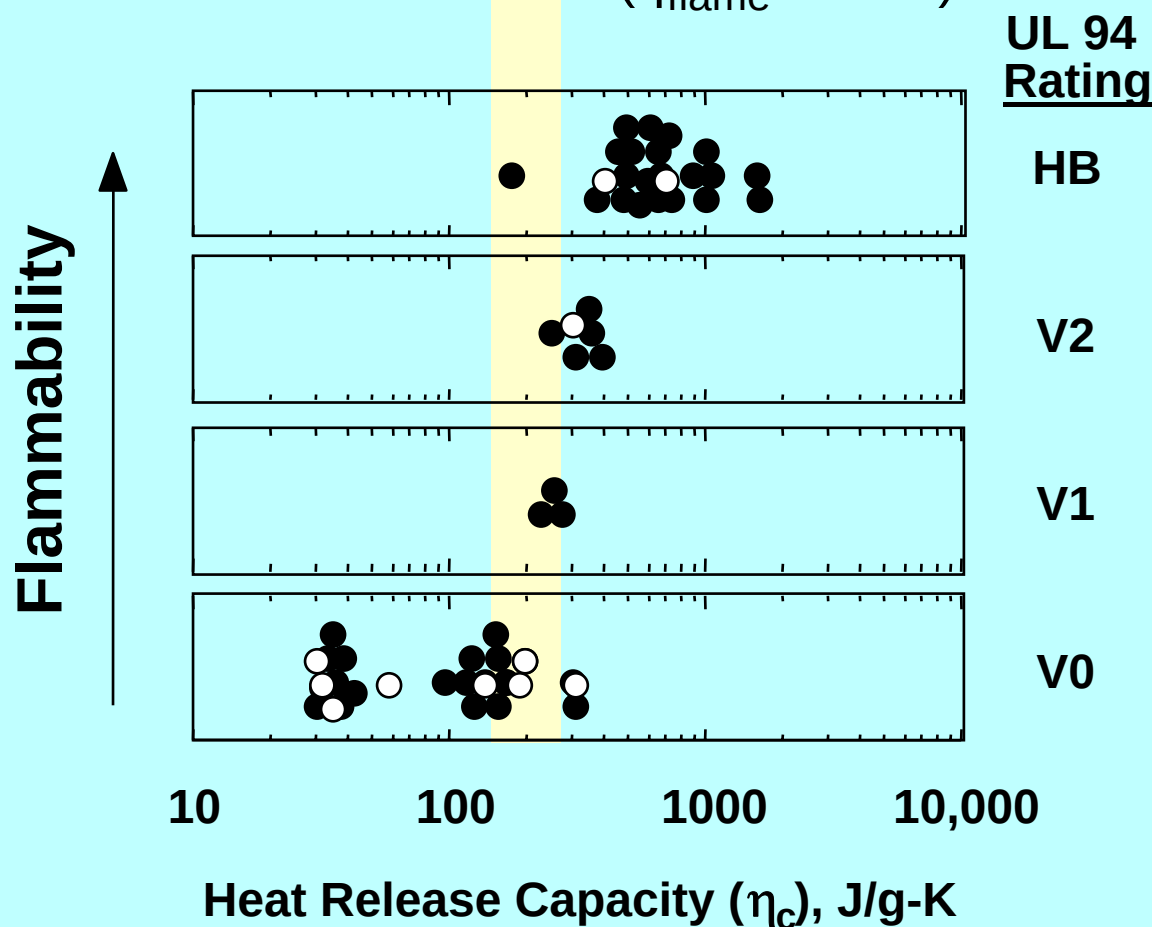


○ Halogen containing

FLAME RESISTANCE (UL 94) AND MICROCAL (η_c)

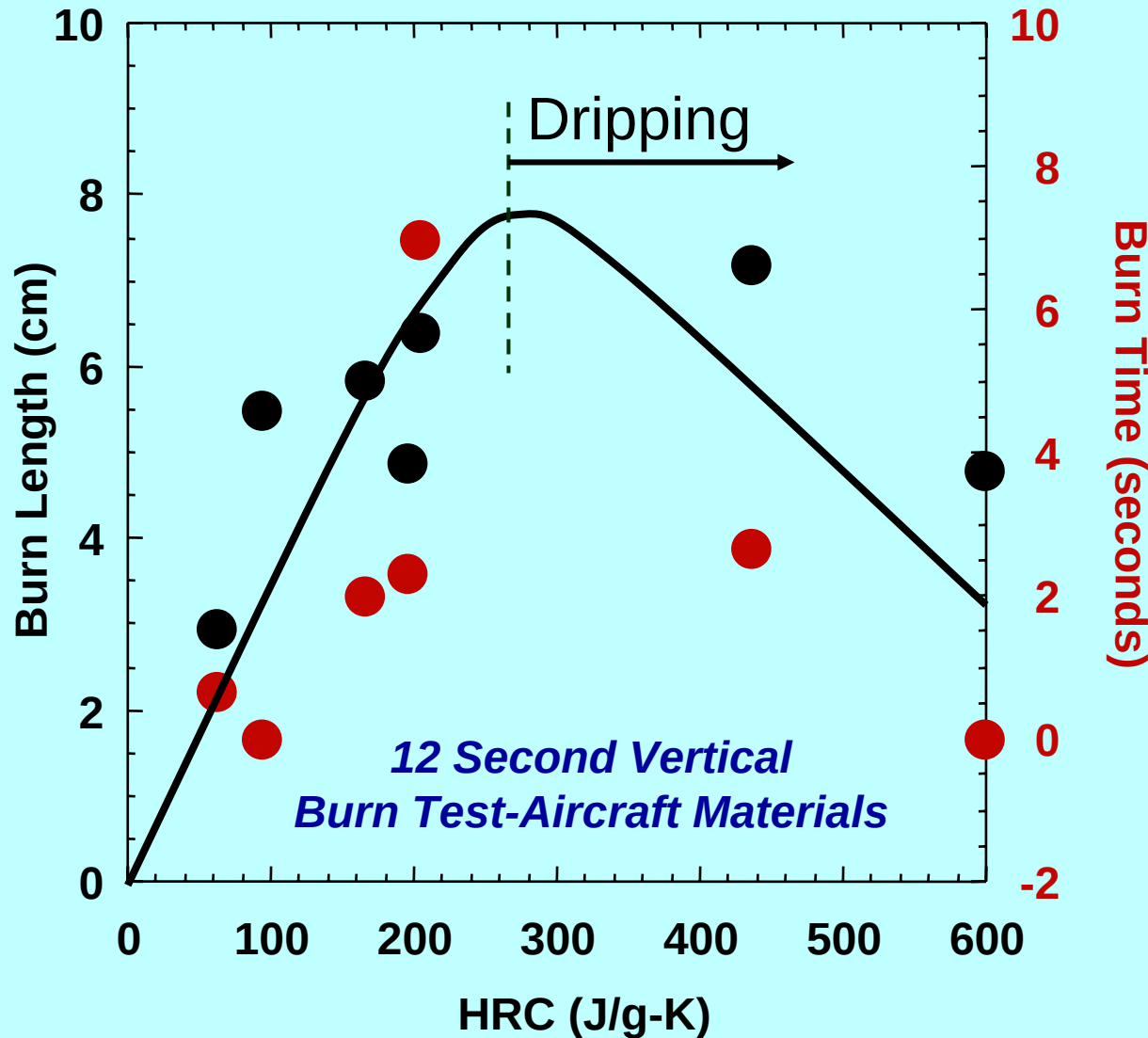


$$200 \text{ J/g-K} \approx \eta_c^* \leq \frac{\text{HRR}^* \eta_g}{(q''_{\text{flame}} - \text{CHF})}$$



○ Halogen containing

FLAME RESISTANCE: FAR 25.853 AND 25.855



Vertical Flame Test



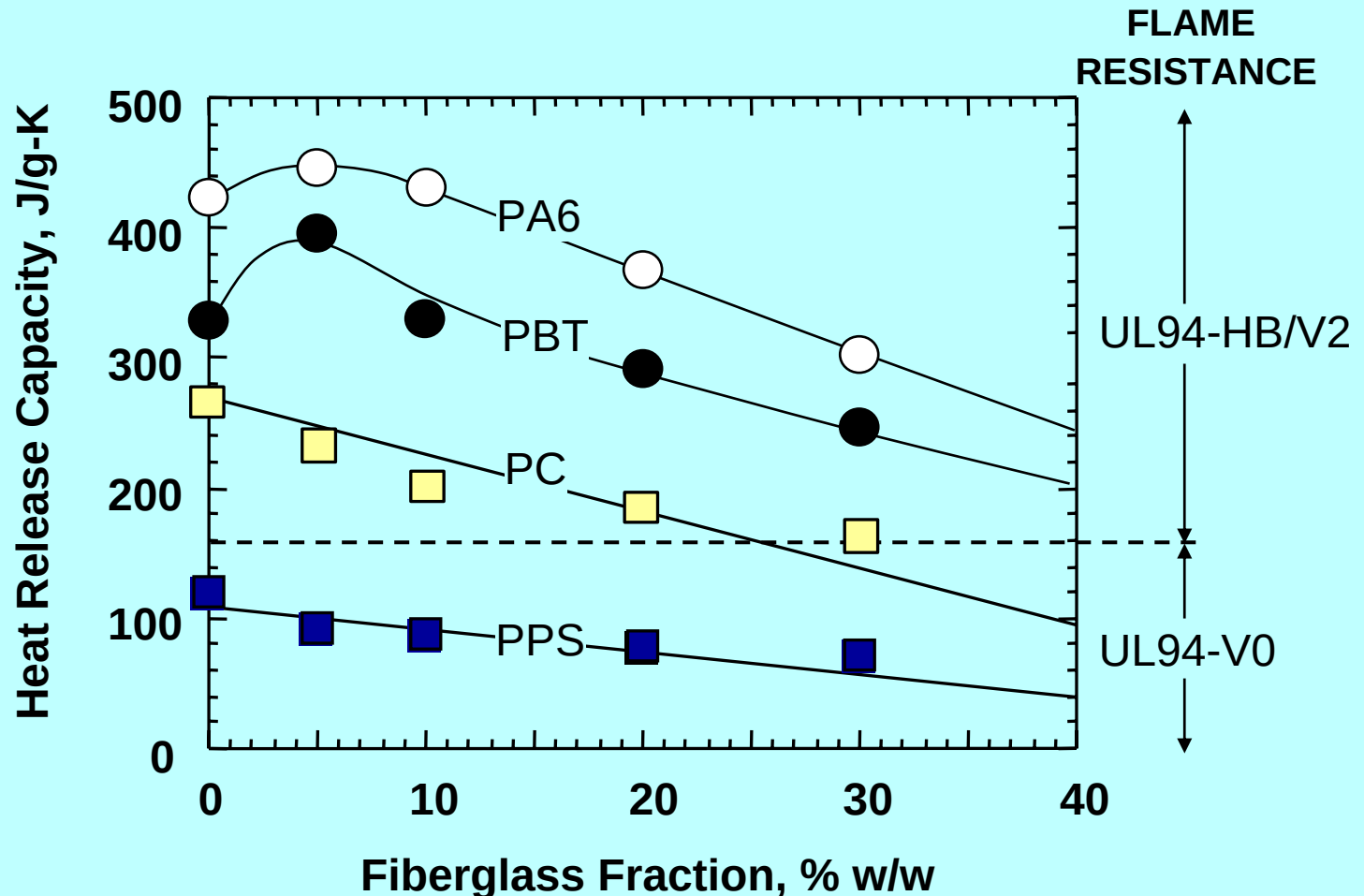
RESEARCH

FLAME RESISTANCE OF GFRP

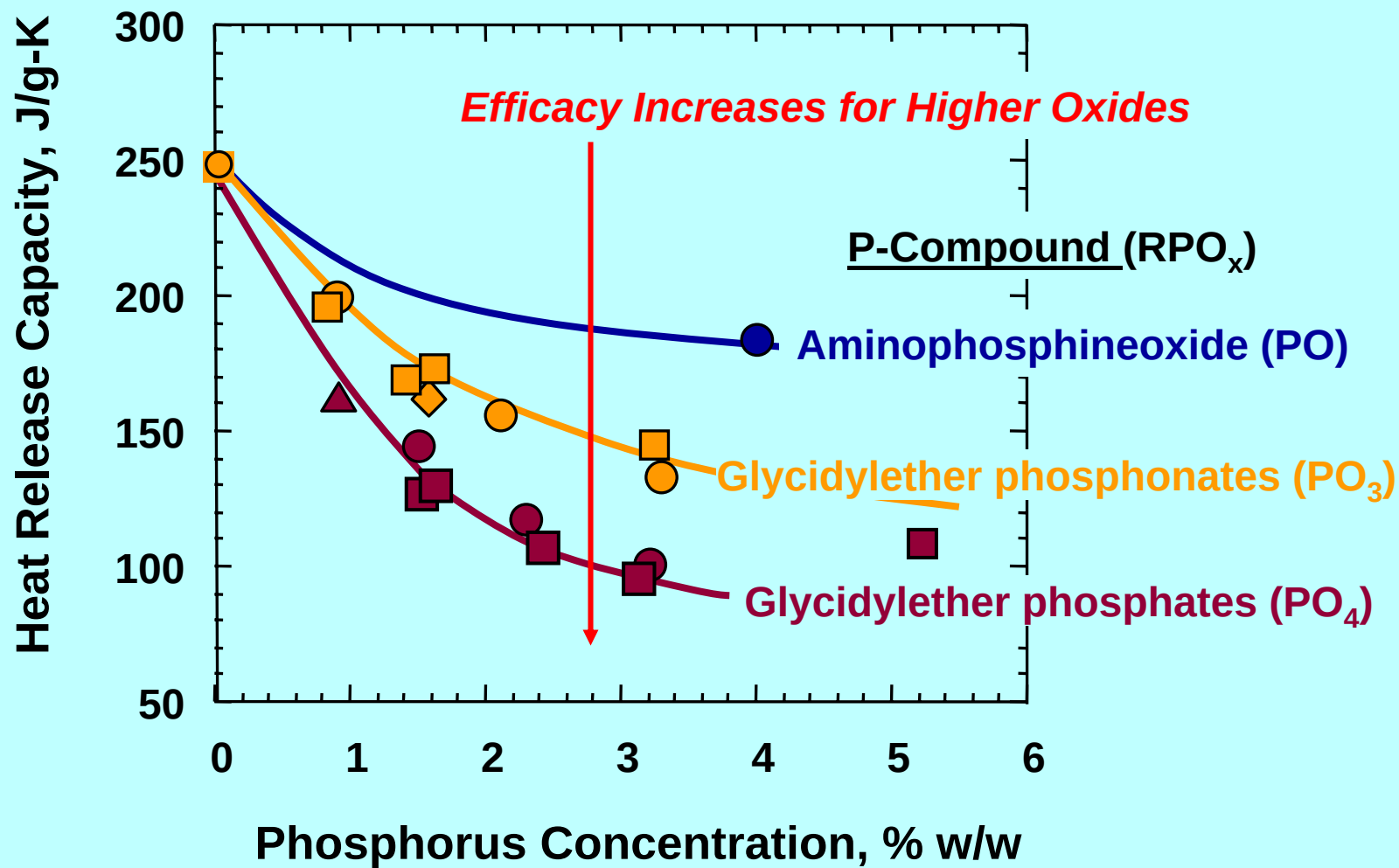


η_c has maximum for non-charring plastics (PA6, PBT).

η_c decreases monotonically for charring plastics (PC, PPS)



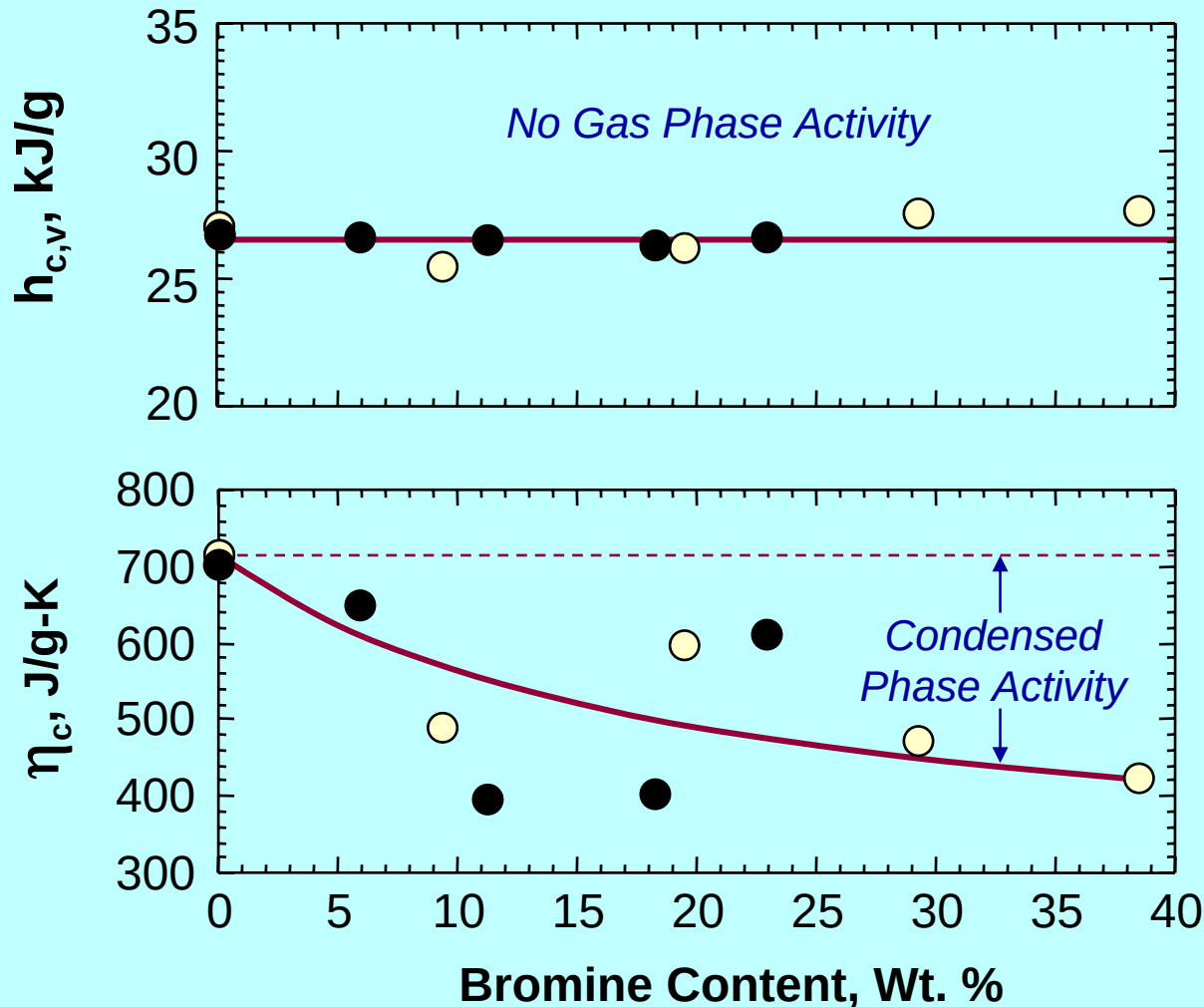
PHOSPHORUS-MODIFIED EPOXIES



BROMINATED EPOXIES (Adjusted for Fuel Fraction)



● Tetrabromobisphenol-A / DGEBA } + EMI-24 (2 wt.%)
● TetrabromoDGEBA / DGEBA



Heat of complete combustion of fuel gases is independent of [Br] in epoxy

Heat Release Capacity decreases with [Br] in epoxy



SUMMARY

CURRENT LIMITATIONS OF METHOD



- Small (mg) Specimens = No Fabricated Products, Assemblies, Laminates.
- Physical Effects Not Captured in Test Results:

Dripping
Swelling
Shrinking
Curling
Intumescence
Barrier (mass/heat) effects
etc.

Not So Important for FAA Tests
(Which Tend to Be More Severe)

- Complete Combustion Under Normal Operation
= Limited Sensitivity to Gas Phase/Halogen FR.

STATUS OF MICROCALORIMETRY: Commercial



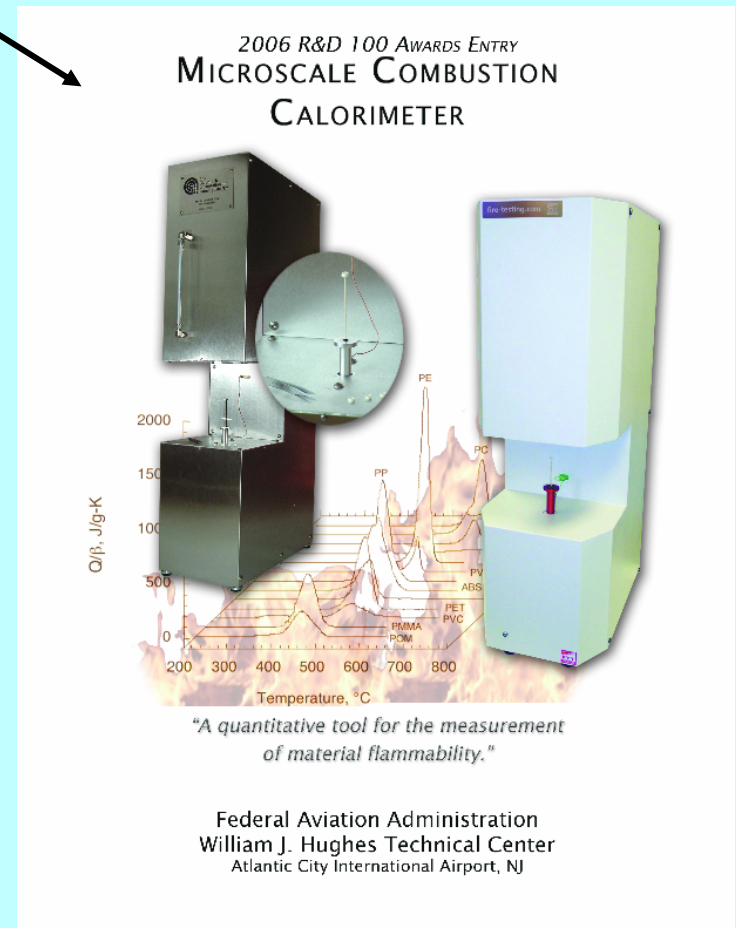
- R&D 100 Award Submitted by FAA

- Two licensed manufacturers

The Govmark Organization,
Farmingdale, NY.

Fire Testing Technology,
East Grinstead, England.

- Over ten (>10) commercial units in operation in industry, government, academia.



STATUS OF MICROCALORIMETRY: Regulatory



Draft Document November 3, 2005

Standard Test Method for Determining Flammability Parameters of Materials Using Controlled Heating and Oxygen Consumption Calorimetry

INTRODUCTION

A method for determining flammability parameters of materials under laboratory conditions is useful as a screening test for materials flammability and for research purposes. The method uses controlled heating to thermally decompose the sample, thermal oxidation of the evolved gases in excess oxygen, and oxygen consumption calorimetry to measure the heat release characteristics of materials under conditions that simulate a fire environment in a laboratory test. Measured heat release characteristics are the maximum amount, rate, and temperature of heat release by combustion of sample gases evolved during controlled heating. Tests can be conducted in which the sample is thermally decomposed in an oxygen-free (anaerobic) or in an oxygen-containing (aerobic) environment.

1. Scope

- 1.1 This test method establishes a quantitative procedure for determining flammability parameters of materials in a laboratory environment using controlled heating of a minimum quantity of sample and thermal oxidation of the sample gases released during heating.
- 1.2 Tests can be performed in which the sample is thermally decomposed in an oxygen-free (anaerobic) or oxidizing (aerobic) environment.
- 1.3 The net heat of combustion of the sample gases is determined from the mass of oxygen consumed to completely oxidize the gases generated during controlled thermal decomposition of the sample.
- 1.4 The net heat of combustion of the sample is determined from the mass of oxygen consumed to completely oxidize the gases and solid residue generated during controlled thermal oxidative decomposition.
- 1.5 The heat release rate is determined from the rate of oxygen consumption.
- 1.6 The sample can be a material or composite material of any condensed phase (solid, non-volatile liquid, slurry) in any form (fiber, film, powder, pellet, droplet).
- 1.7 This test method has been developed to facilitate material development and research.
- 1.8 *This standard may involve hazardous materials. This standard does not purport to address all of the safety concerns associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

- 2.1 ASTM Standards
 - D 635 Standard Test Method for Rate of Burning and/or Extent and Time of Burning of Plastics in a Horizontal Position.
 - D 1929 Standard Test Method for Determining Ignition Temperat

Work Items Submitted for Standard Test Method in:

ASTM Committee D20 (Plastics)

ISO Technical Committee TC 61
(Plastics)

FAA (?)

• “Screening Method” in
Fire Test Handbook?

• Adapt to AC Materials