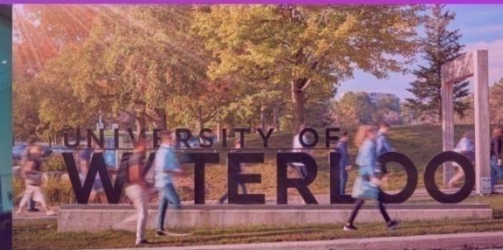
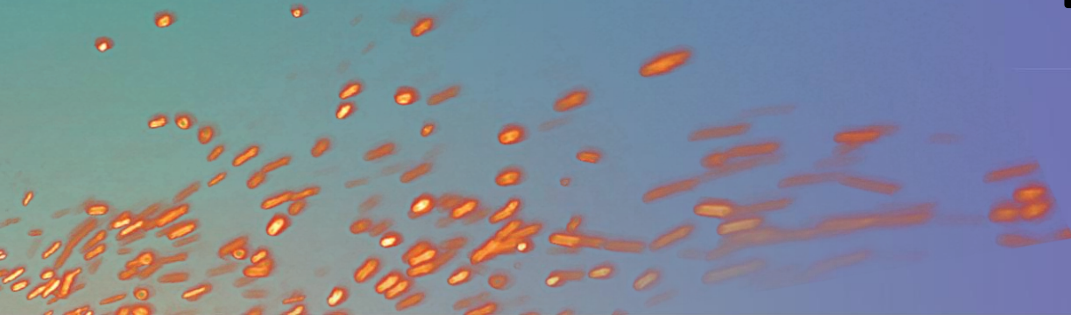




Measurement and Computation of Fire Phenomena

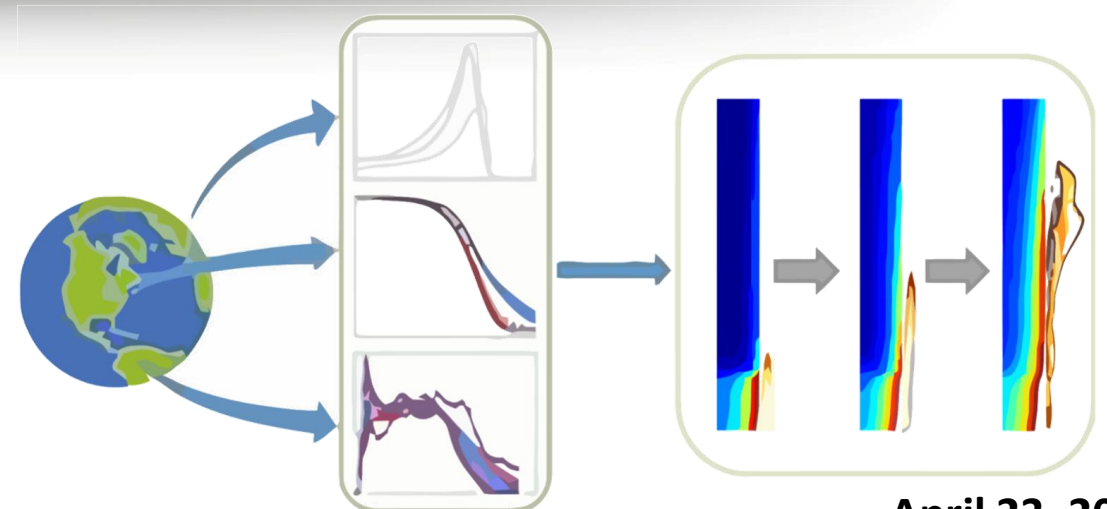
The MaCFP Condensed Phase Working Group

Experimental

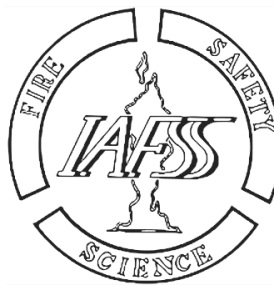


Organizing Committee:

- | | |
|---------------------|---|
| Benjamin Batiot | (University of Poitiers, France) |
| Morgan Bruns | (Virginia Military Institute, USA) |
| Simo Hostikka | (Aalto University, Finland) |
| Isaac Leventon | (National Institute of Standards and Technology, USA) |
| Yuji Nakamura | (Toyohashi University of Technology, Japan) |
| Pedro Reszka | (Universidad Adolfo Ibáñez, Chile) |
| Thomas Rogaume | (University of Poitiers, France) |
| Stanislav Stoliarov | (University of Maryland, USA) |



- Introduction
 - Motivation
 - Material Selection
 - The Github Repository & Virtual Discussion Forum
- Experimental Results
 - Milligram-scale (TGA, DSC, MCC)
 - Bench-scale (Cone Calorimeter, gasification apparatus, heat flow)
- Discussion
 - Focused series of questions based on workshop objectives



To make significant & systematic progress in fire modeling, based on a fundamental understanding of fire phenomena

Condensed Phase

Degradation Reaction

Mechanisms

Kinetics (A , E , ν)

Thermodynamics (h_i , c_p)

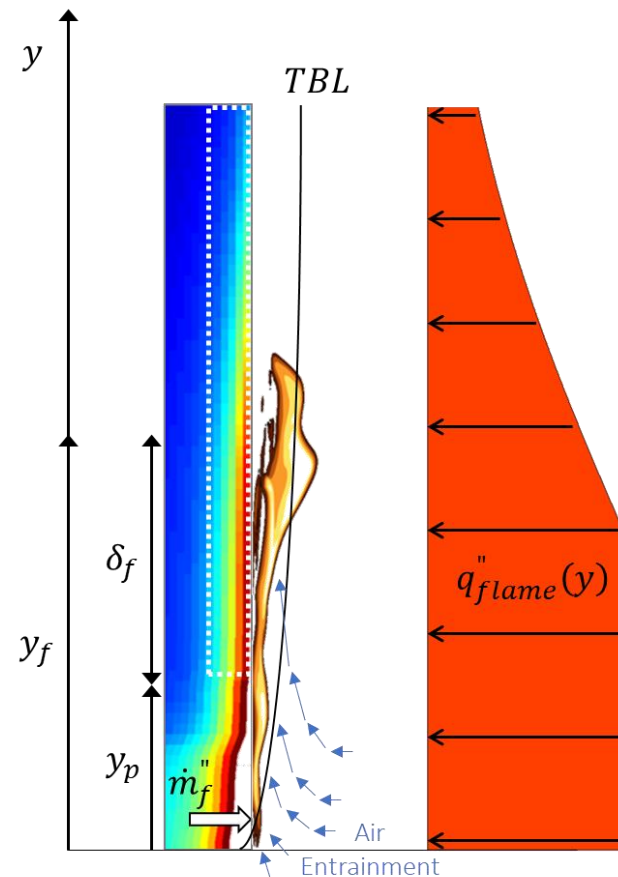
Heats of combustion (ΔH_c)

Heat & Mass Transport

Conductivity (k)

Interaction with radiation (α , ε)

Gas transfer, melt flow



Gas Phase

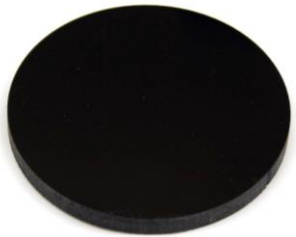
Buoyant flow

Turbulence

Flame radiation

Wall flame interaction

Flame extinction



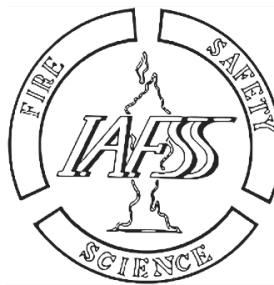
- Cast Black Poly(methyl methacrylate) (PMMA)

- Evonik ACRYLITE® cast black 9H01 GT
- Distributed in summer 2019
 - 100 mm by 100 mm by 6 mm slabs
 - 300 mg vials of powdered PMMA

- Suitable first reference material

- Maintains density/shape while burning
- Simple decomposition kinetics
- Low transparency to infrared radiation

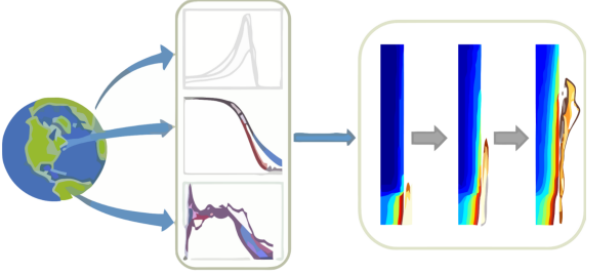
The identification of any commercial product or trade name does not imply endorsement or recommendation by NIST (or any other contributing institution).



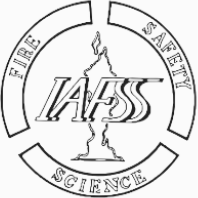
Preliminary Summary of Experimental Results

Preliminary Summary of Experimental Measurements

Predecisional Draft Report
Submitted to the 2021 MaCFP Condensed Phase Workshop
September 21, 2020

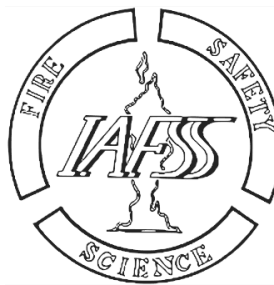


The diagram illustrates the flow of experimental data. It starts with a globe representing the source of data, which is then processed into three distinct line graphs. These graphs are then analyzed through a series of three heatmaps, showing the progression of data from raw measurements to more complex, multi-dimensional representations.

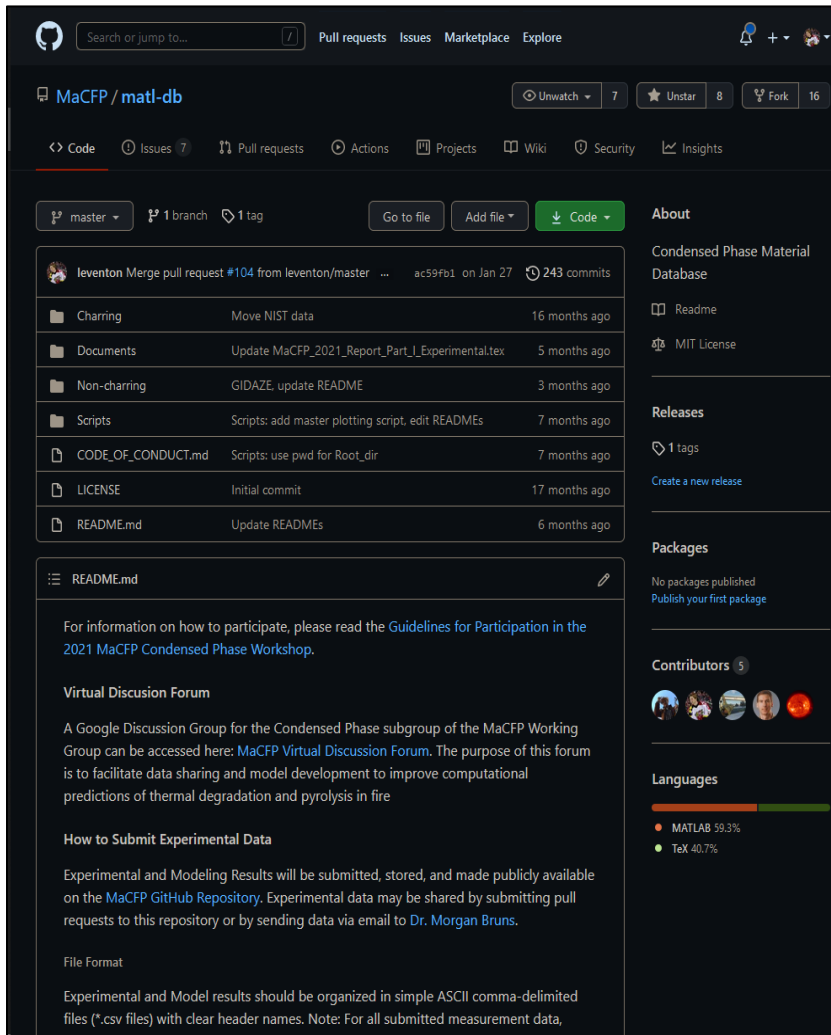


The MaCFP Condensed Phase Working Group Organizing Committee:
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Thomas Rogauze (University of Poitiers, France)
Stanislav Stolarov (University of Maryland, USA)

- Developing standard data set formats for experimental data on pyrolysis
- Developing requirements for data set quality and establishing a data review committee
- Quantifying the interlaboratory variability for comparable experimental datasets

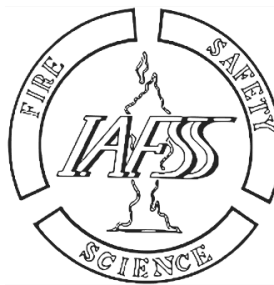


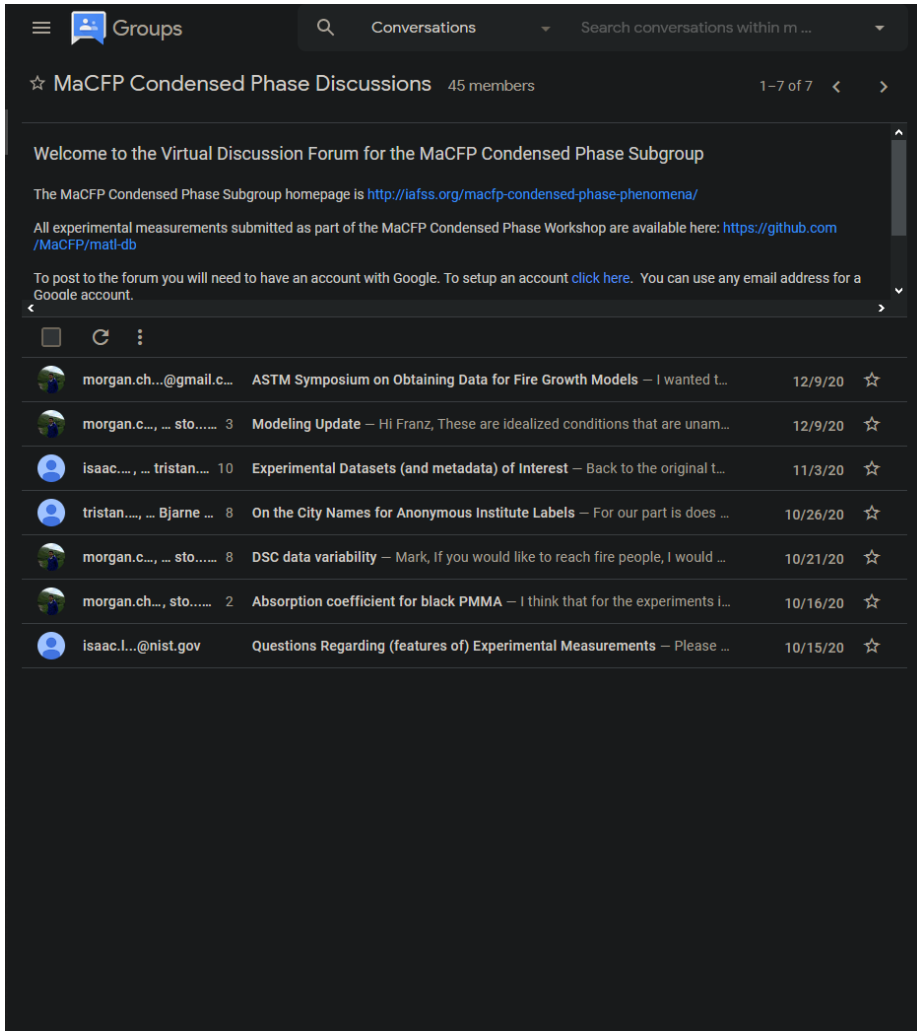
The MaCFP Repository (Github)



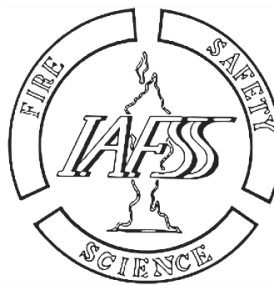
- <https://github.com/MaCFP/matl-db>
- Encourage participants to navigate Github to:
 - Access & compile most current datasets, reports
 - Review README files (descriptions of the test setup, conditions, and procedure)
- Some lessons from previous databases
 - Metadata is critical
 - Maintenance is necessary but not cheap
 - Must connect to applications

This summary is prepared for experts in the pyrolysis modeling community to provide critical review. Not all of the measurement data presented here have been through a formal review process and they should therefore be considered as pre-decisional draft results



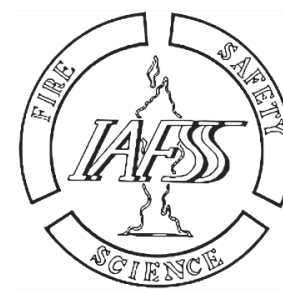


- <https://groups.google.com/g/macfp-condensed-phase-discussions>
- Encourage participants to visit Forum to:
 - Continue discussions started during workshop
 - Ask questions regarding measurements on Github Repository, related metadata, analysis of those results
 - Review measurement data/modeling approaches
 - Propose current/future measurement data of interest
 - What's needed (different scales, more detail at same scale)
 - What can you/your lab offer (measurement data, analysis, scripting, database management)

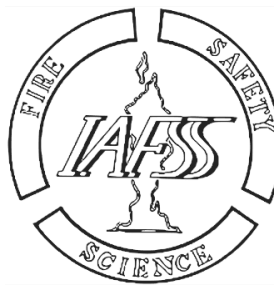


Thermogravimetric Analysis (TGA) Differential Scanning Calorimetry (DSC) Microscale Combustion Calorimetry (MCC)	Cone Calorimeter Fire Propagation Apparatus (FPA) Controlled Atmosphere Pyrolysis Apparatus (CAPA)
Test Conditions	
<ul style="list-style-type: none"> • Heating Rate [K min⁻¹] • Temperature Program: <ul style="list-style-type: none"> - Initial temperature - Conditioning isotherm (if used) - Maximum temperature • Sample mass [mg] • Sample geometry (e.g., powdered) • Calibration type, materials used, and frequency • Carrier gas and associated flow rate • Crucible type and volume 	<ul style="list-style-type: none"> • Radiant heat flux (kW m⁻²) • Heater Temperature • Extracting flow rate of the gas • Initial and Final Sample Mass • Sample holder geometry and characteristics • Thermal properties of backing insulation, if used
Test Outputs	
<ul style="list-style-type: none"> • Initial and Final Sample Mass [mg] • Time-resolved Sample Mass [mg] • Time-resolved Sample Temperature [K] 	<ul style="list-style-type: none"> • Sample Surface Area [m²] • Initial and Final Sample Mass [mg] • Time-resolved Sample Mass [mg] • Time-resolved Sample Back-Surface Temperature [K]

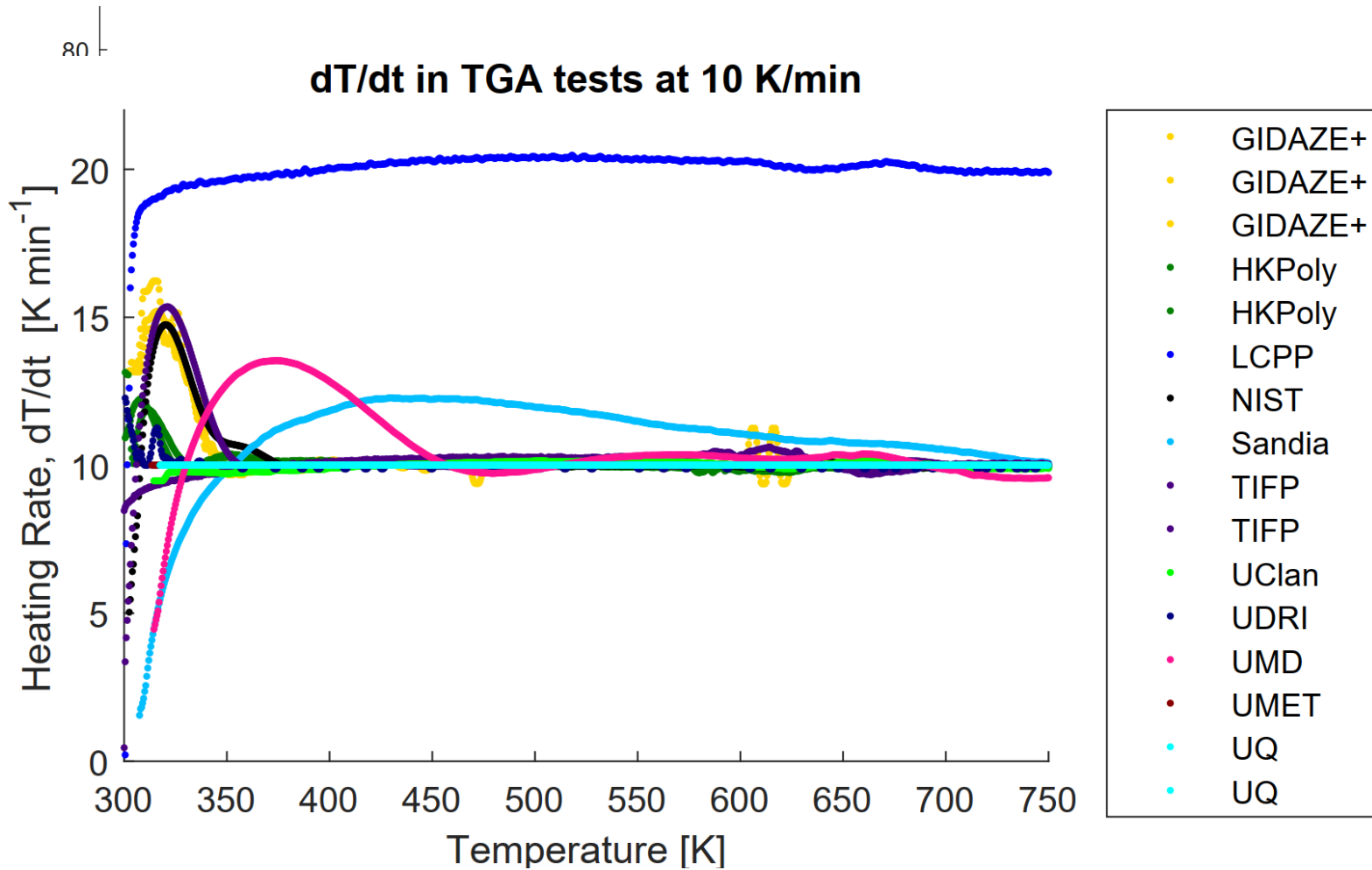
220 Experiments
16 Institutions
10 countries



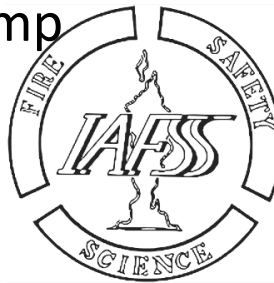
- 12 institutions
 - Up to 7 replicates from one lab under same conditions
- 4 gaseous environments
 - (N₂, 10 & 21 % O₂ in N₂, Ar)
- 9 heating rates
 - $1 \leq \frac{dT}{dt} \leq 100 \text{ K min}^{-1}$



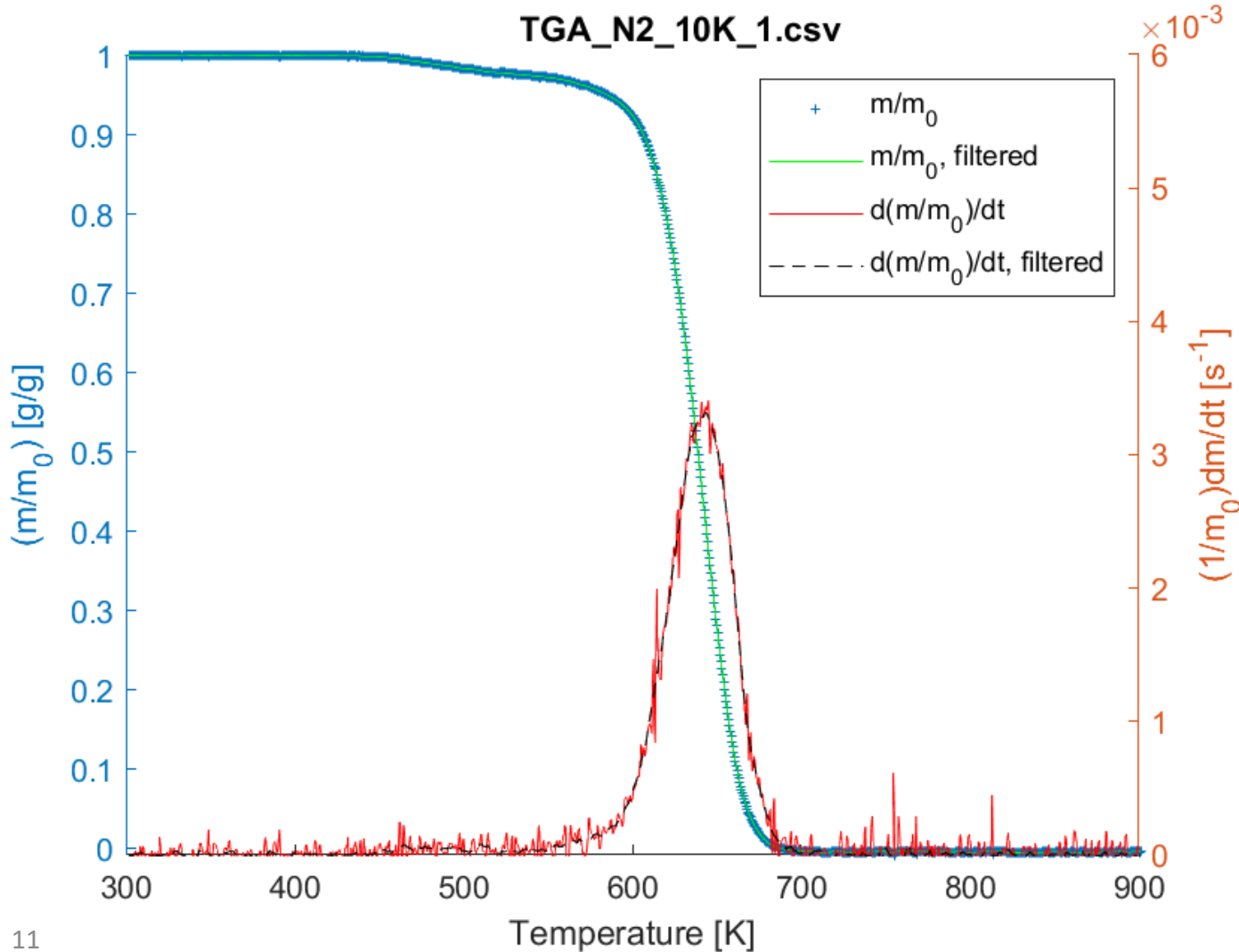
Thermogravimetric Analysis (TGA)



- Instantaneous heating rate observed during experiments may not match prescribed value
- Impact on analysis of:
 - Decomposition (rates) of materials with low thermal stability
 - Determination of temperature-resolved heat capacity at low Temp



Thermogravimetric Analysis (TGA)

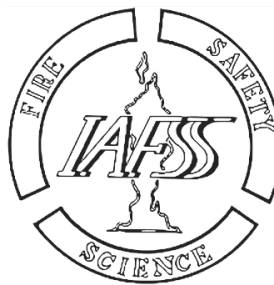


• Data Formatting

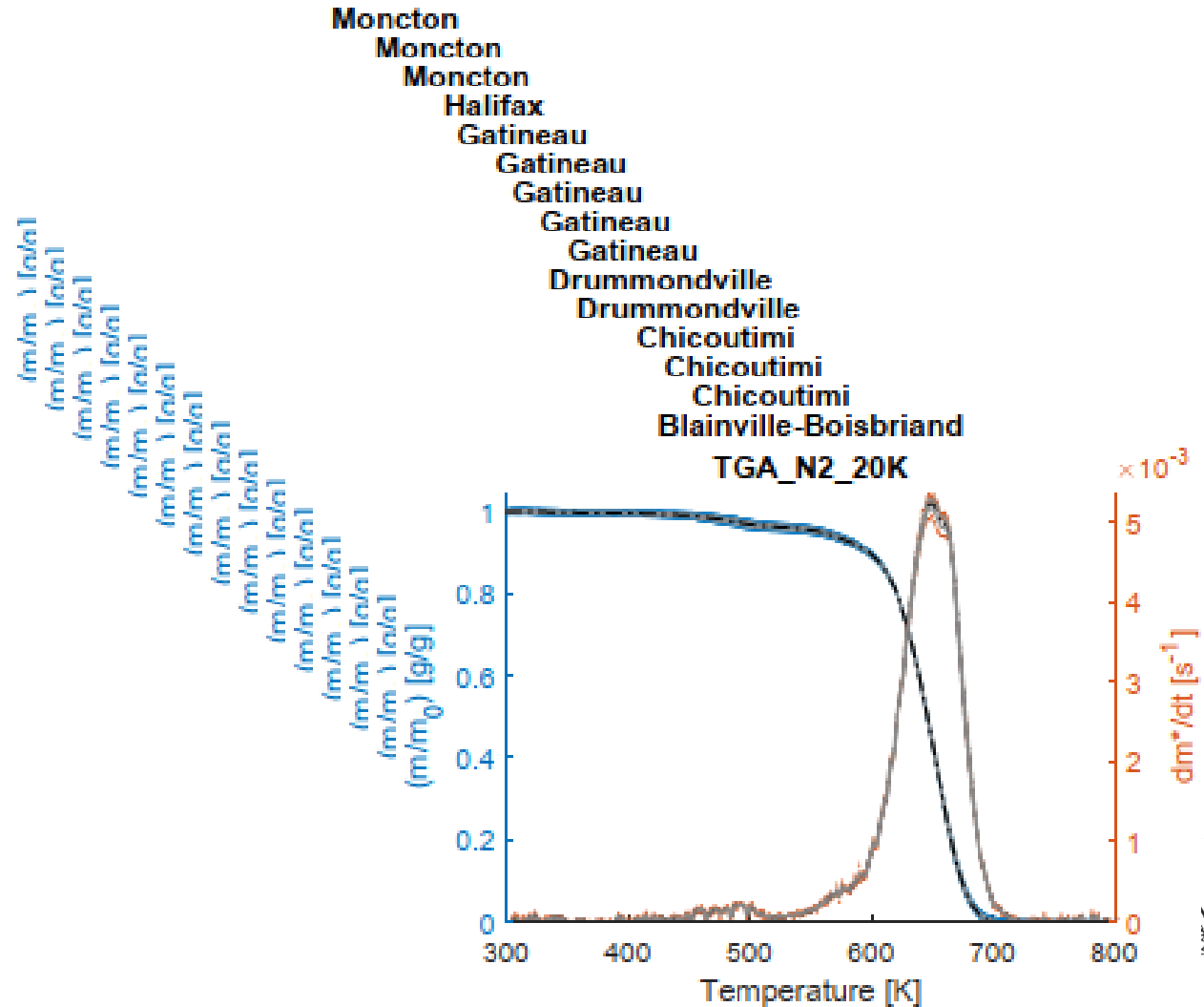
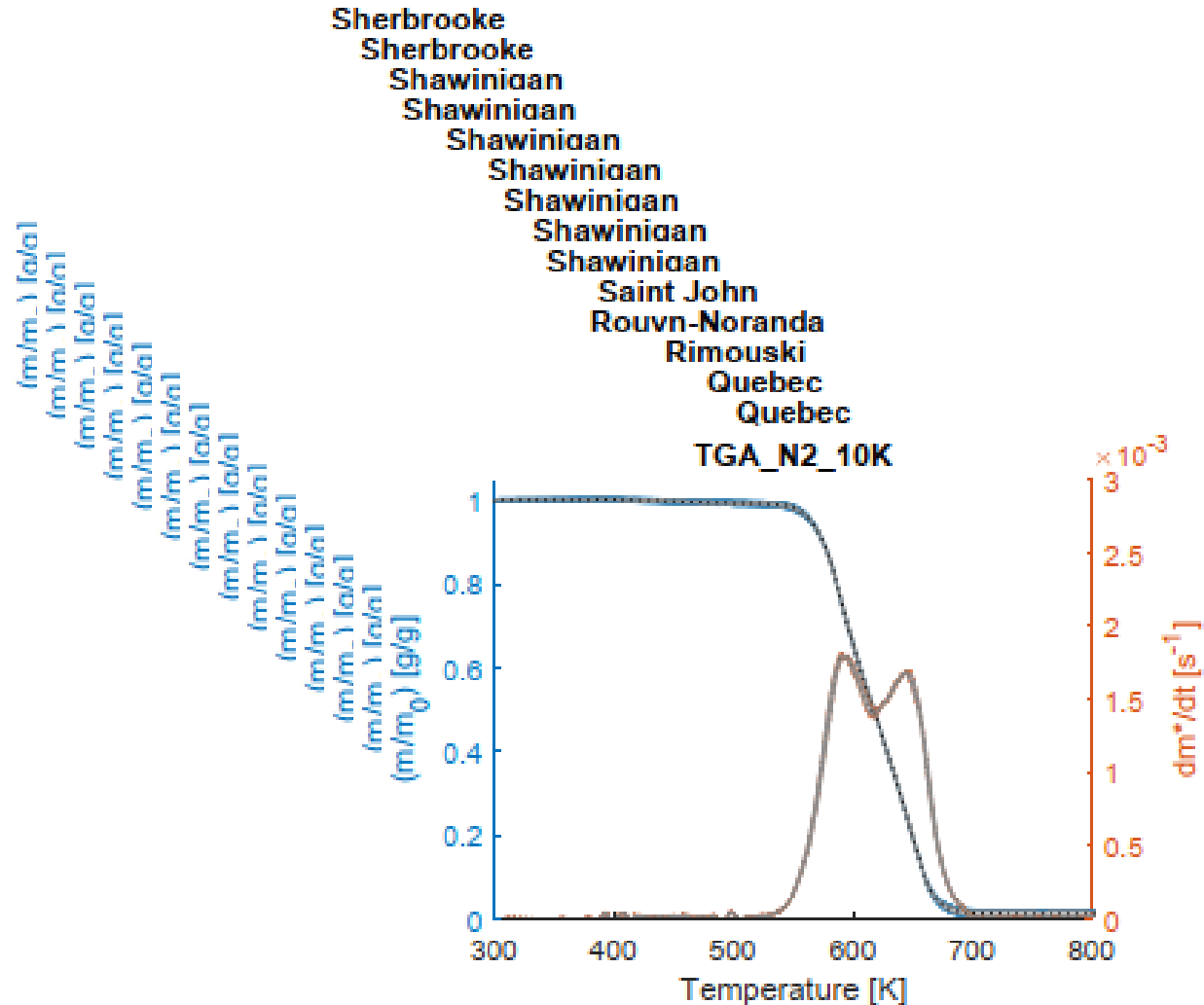
- [time (s) | Temp (K) | Mass (mg)]
- Tare measurements
- Reporting frequency [$\Delta T = 0.5$ K]
- README files \rightarrow metadata
 - Calibration (type, frequency)
 - Heating Program
 - Instrument, crucible description

• Processing Data

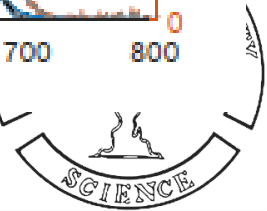
- Savitzky Golay filter
 - $\Delta T = 15$ K window
 - Third order (cubic fit)



Thermogravimetric Analysis (TGA)

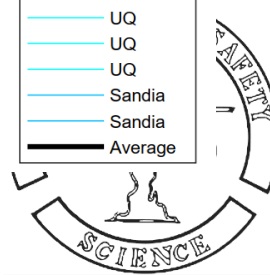
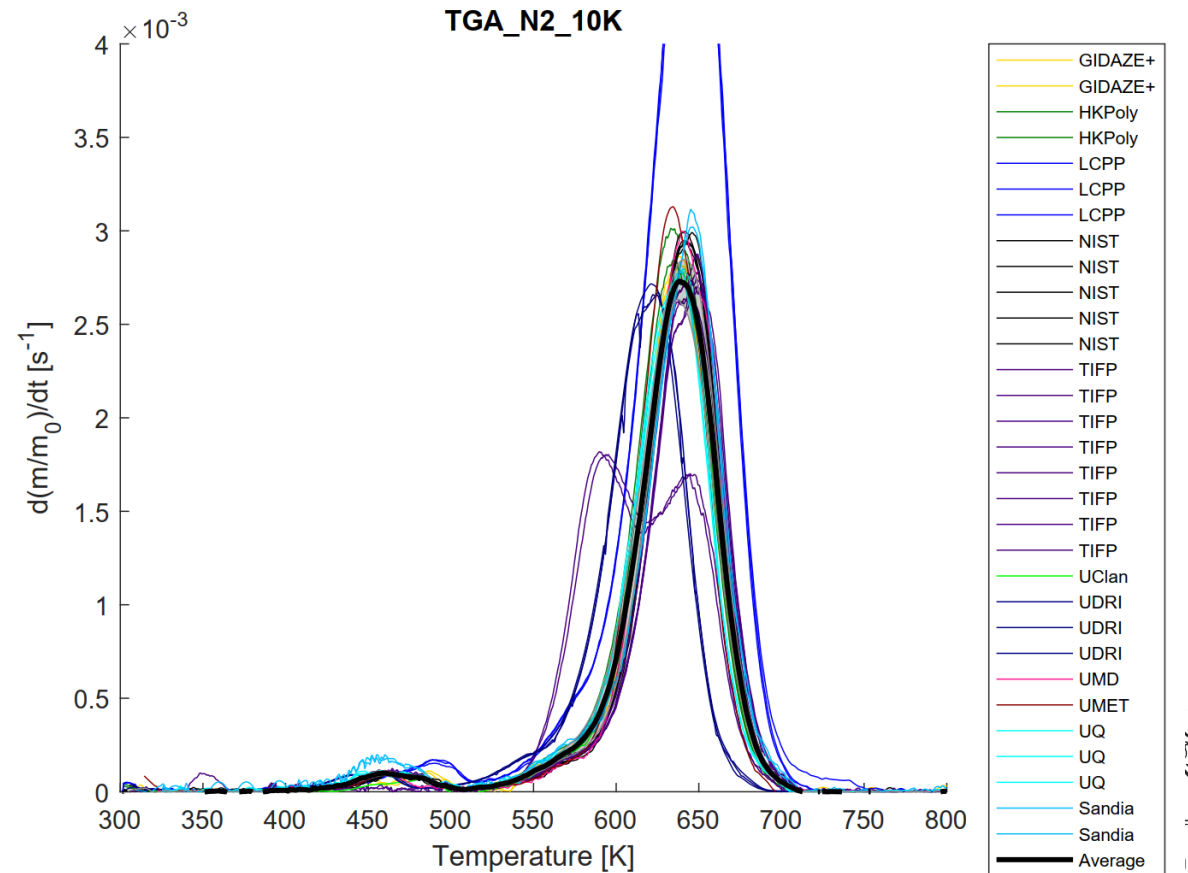
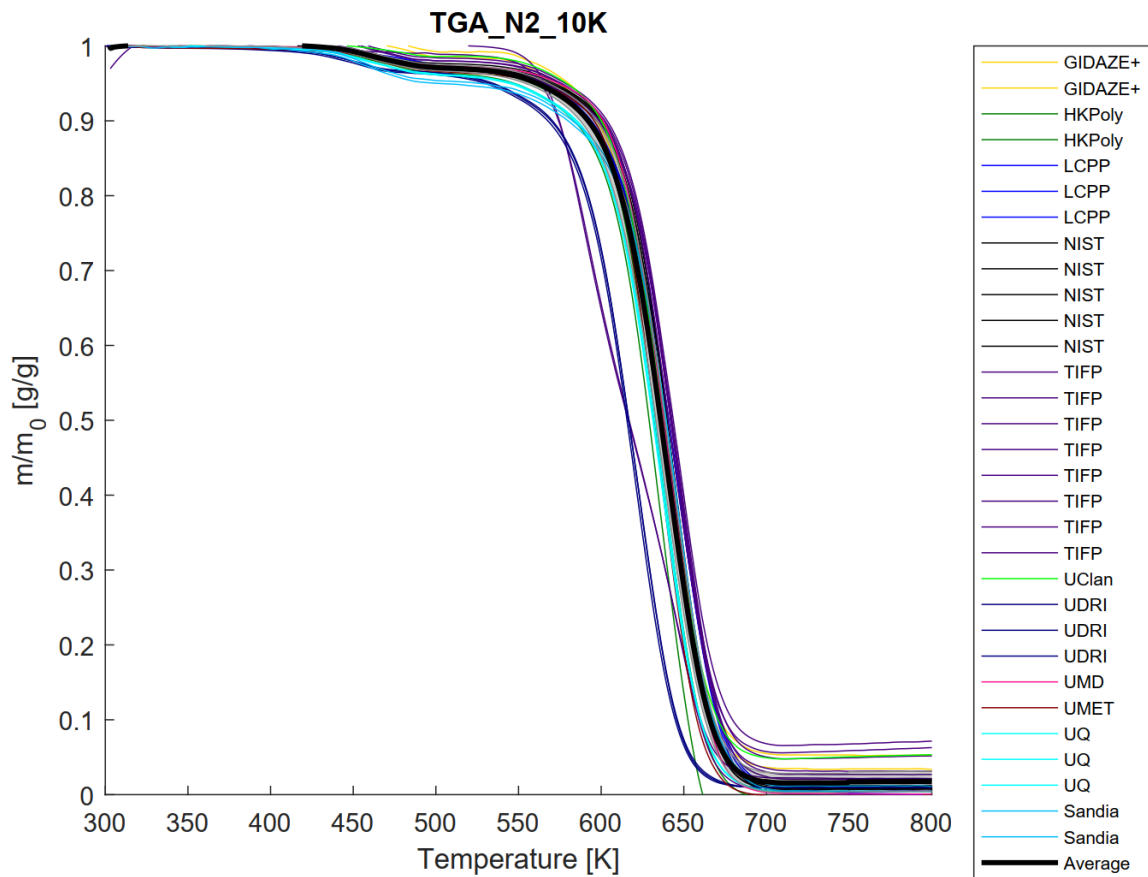


Analyze **all** datasets (multiple heating rates, environments)

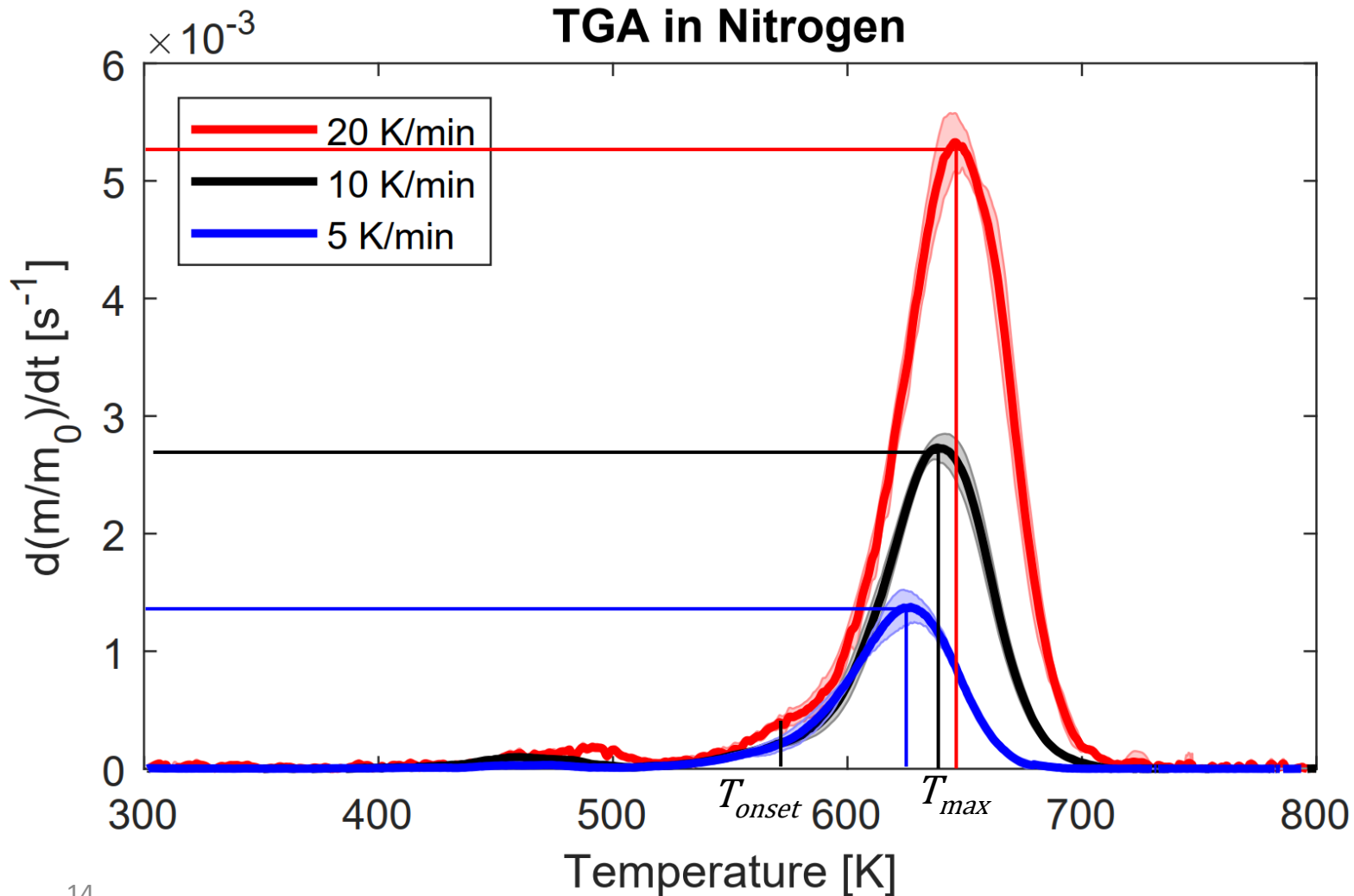


Thermogravimetric Analysis (TGA)

Nitrogen Environment, $\frac{dT}{dt} = 10 \text{ K/min}$

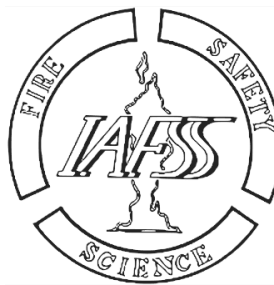


Thermogravimetric Analysis (TGA)



Tabulated Values

- Onset temperature of decomposition, T_{onset} (K)
- Peak normalized mass loss rate
- The Temperature at which it occurs, T_{max} (K)



Thermogravimetric Analysis (TGA)

	5 K/min		10 K/min		20 K/min	
	Mean	Std Dev	Mean	Std Dev	Mean	Std Dev
Aalto	-	-	-	-	-	-
DBI_Lund	-	-	-	-	589	5
Edinburgh	-	-	-	-	-	-
FM	-	-	-	-	-	-
GIDAZE+	-	-	580 ^A	A	-	-
HKPoly	-	-	581	3	-	-
LCPP	555	1	577	2	577	2
NIST	-	-	589	1	-	-
Sandia	-	-	580 ^{AC}	1 ^A	-	-
TIFP	-	-	579	1	-	-
UClan	-	-	584	B	-	-
UDRI	-	-	561	2	-	-
UMD	-	-	591	B	-	-
UMET	579	B	588	B	595	B
UQ	-	-	578	B	-	-
Average	561	12	585^D	5	584	8

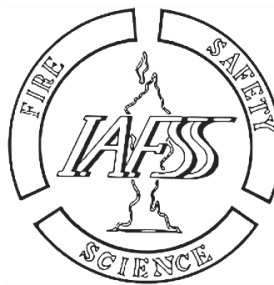
- Onset temperature of decomposition, T_{onset} (K)
 - Defined as the lowest temperature at which normalized mass loss rate exceeds 10 % of its peak value
 - Tabulated values shown here: tests conducted in Nitrogen

^A Calculated based on two values

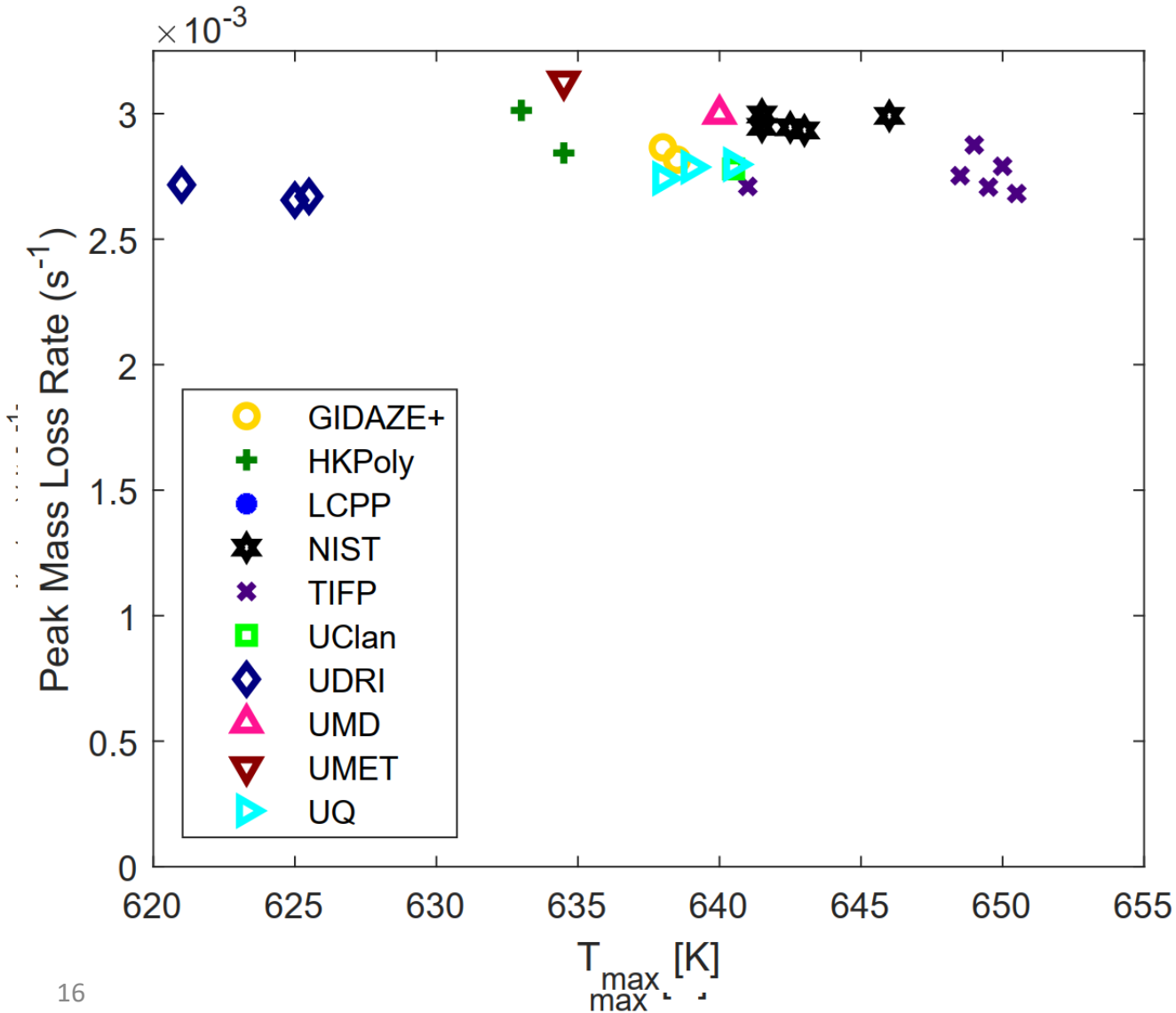
^B Standard deviation not calculated, single datapoint

^C Tests conducted in Argon

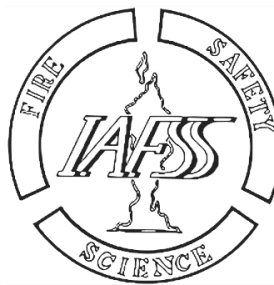
^D Average excludes outliers



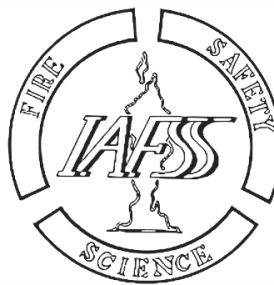
Thermogravimetric Analysis (TGA)



- Peak normalized mass loss rate and the Temperature at which it occurs, T_{max} (K)
 - Tabulated values shown here: tests conducted in Nitrogen at $10 K min^{-1}$

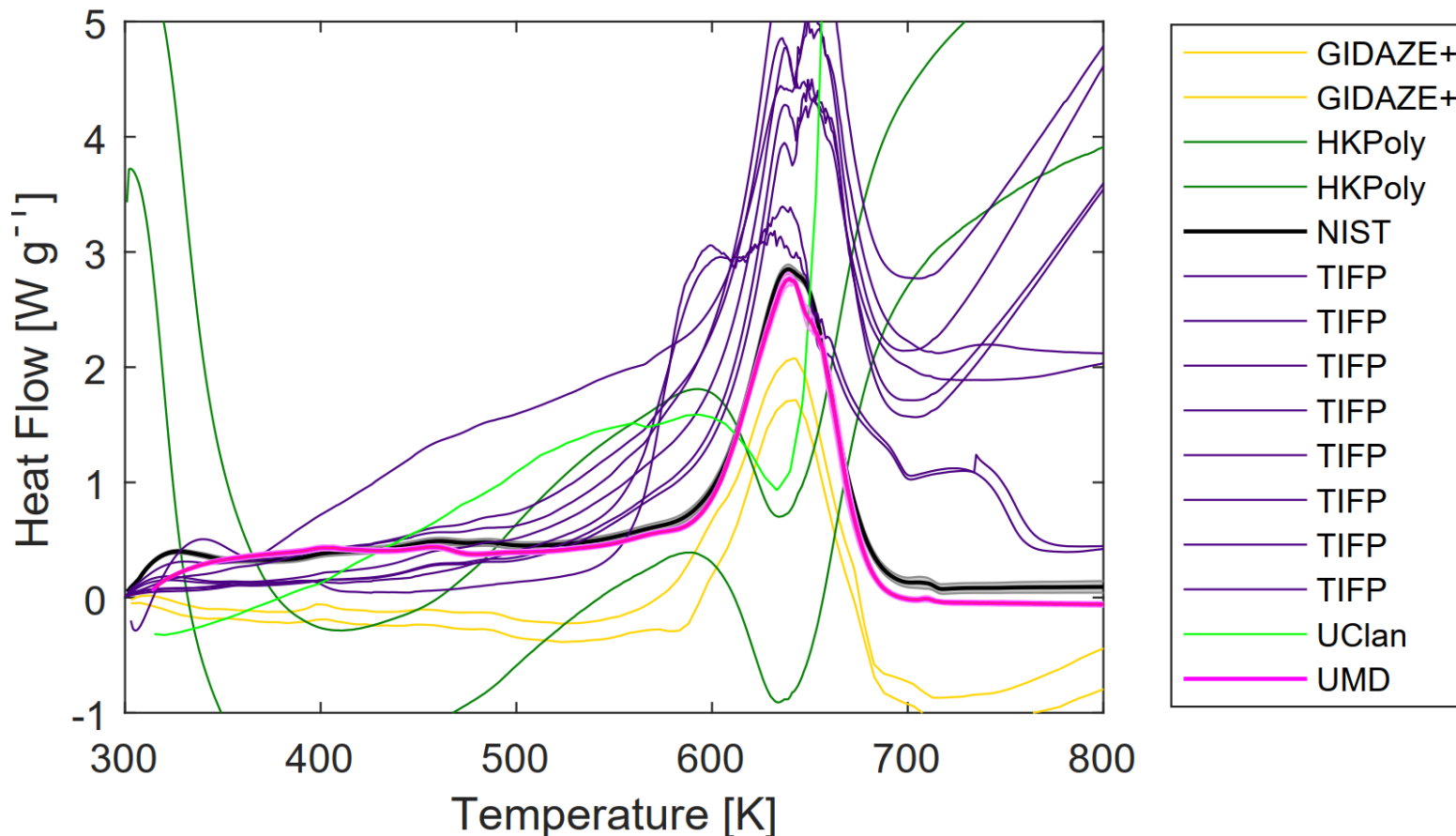


- 9 institutions
 - Up to 7 replicates from one lab under same conditions
 - Typically only 2-3 replicates
- 4 gaseous environments
 - (N₂, 10 & 21 % O₂ in N₂, Ar)
- 5 heating rates
 - $3 \leq \frac{dT}{dt} \leq 50 \text{ K min}^{-1}$



Differential Scanning Calorimetry (DSC)

Nitrogen Environment, $\frac{dT}{dt} = 10 \text{ K/min}$



• Data Formatting

Time	Temperature	Heat Flow
[s]	[K]	[W/g]

• Consistency

- Endothermic \leftrightarrow positive (UP)

• Reporting frequency [$\Delta T = 0.5 \text{ K}$]

• README files \rightarrow metadata

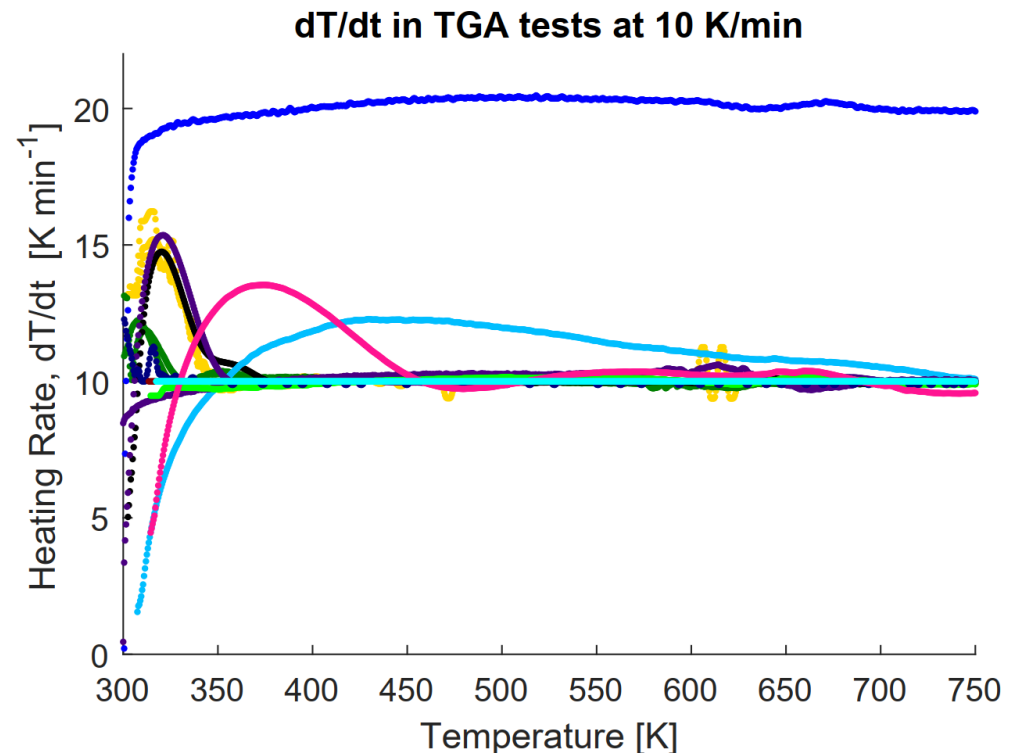
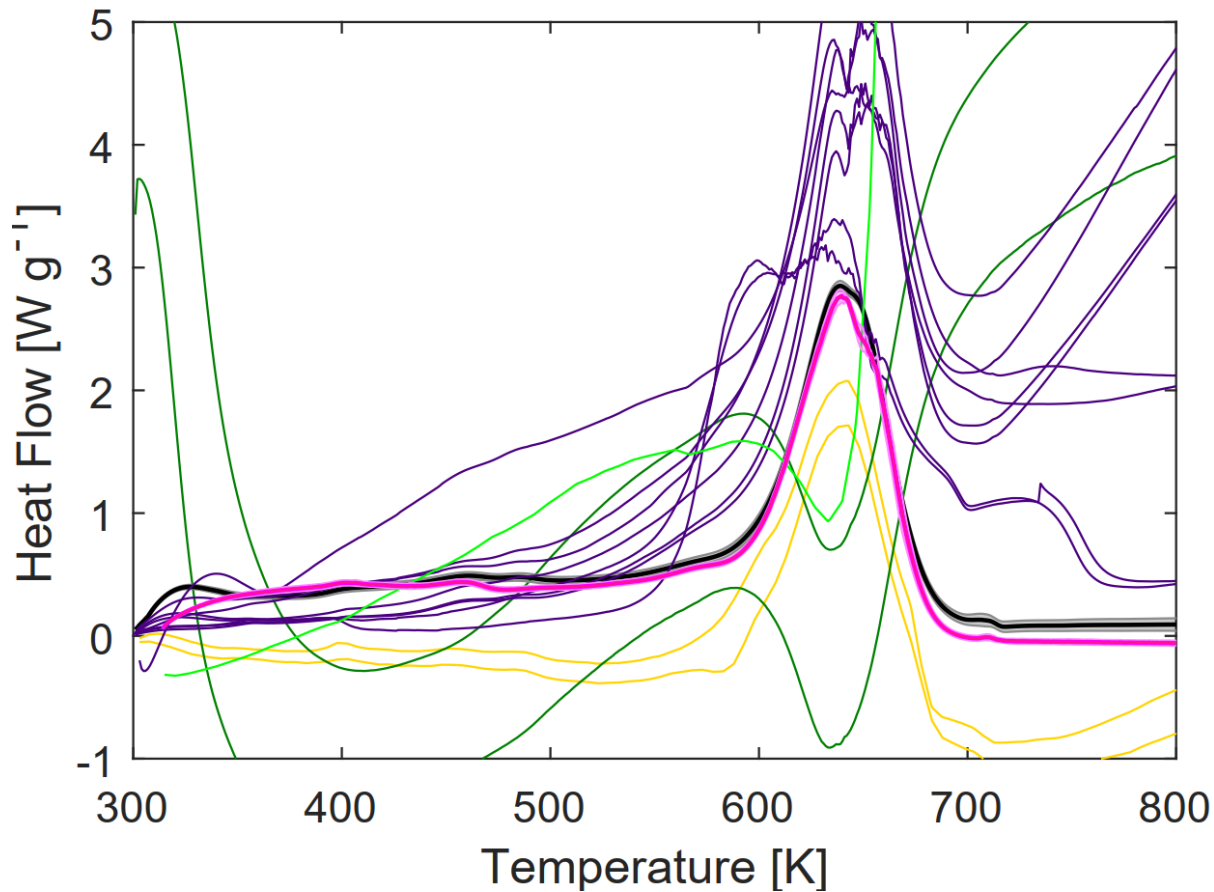
- Calibration (type, freq)
- Heating program
- Instrument, crucible description

• Calibration & Baseline

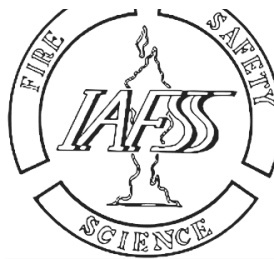
Differential Scanning Calorimetry (DSC)

Determination of heat capacity, c_p

Nitrogen Environment, $\frac{dT}{dt} = 10 \text{ K/min}$



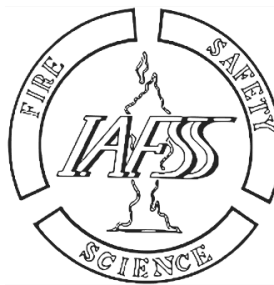
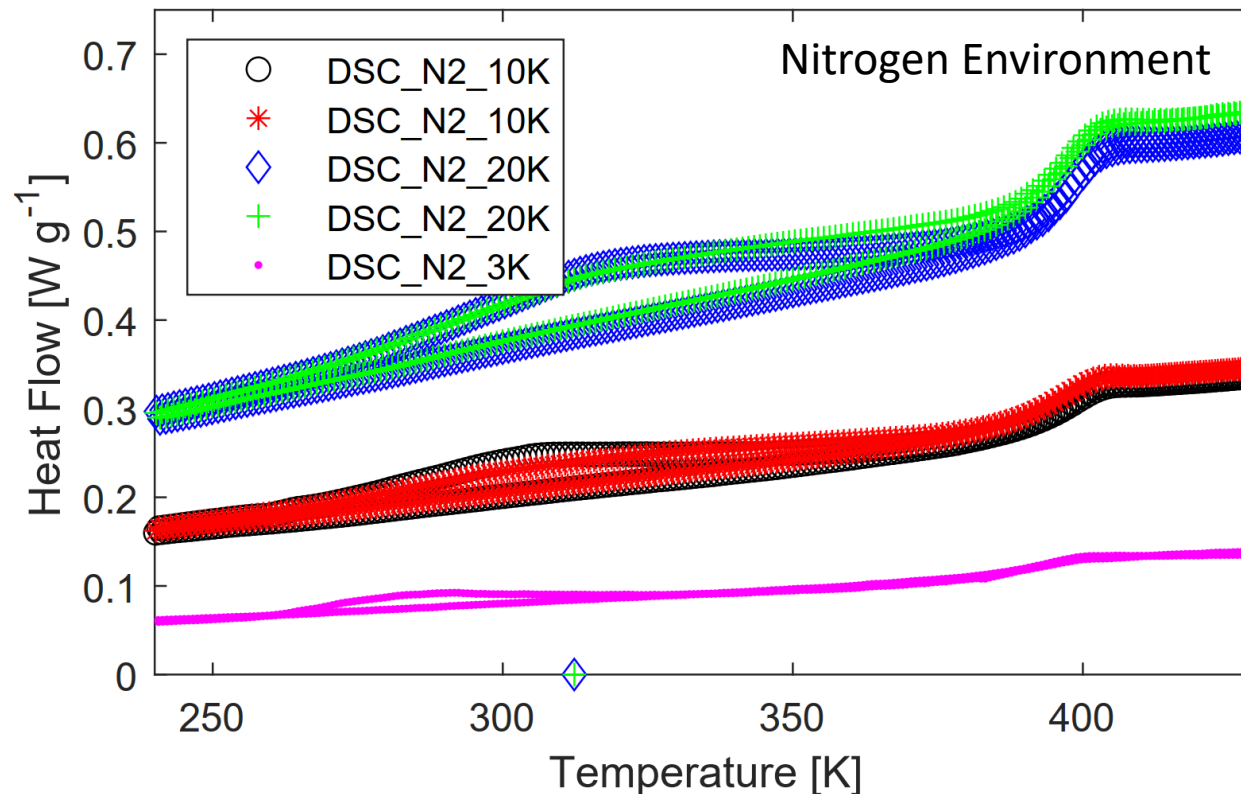
$$\text{Heat flow} = \sum_{j=1}^{N_c} \left(\xi_j c_{p,j} \frac{\partial T}{\partial t} + \sum_{i=1}^{N_r} r_i h_{r,i} \right)$$



Differential Scanning Calorimetry (DSC)

Determination of heat capacity, c_p

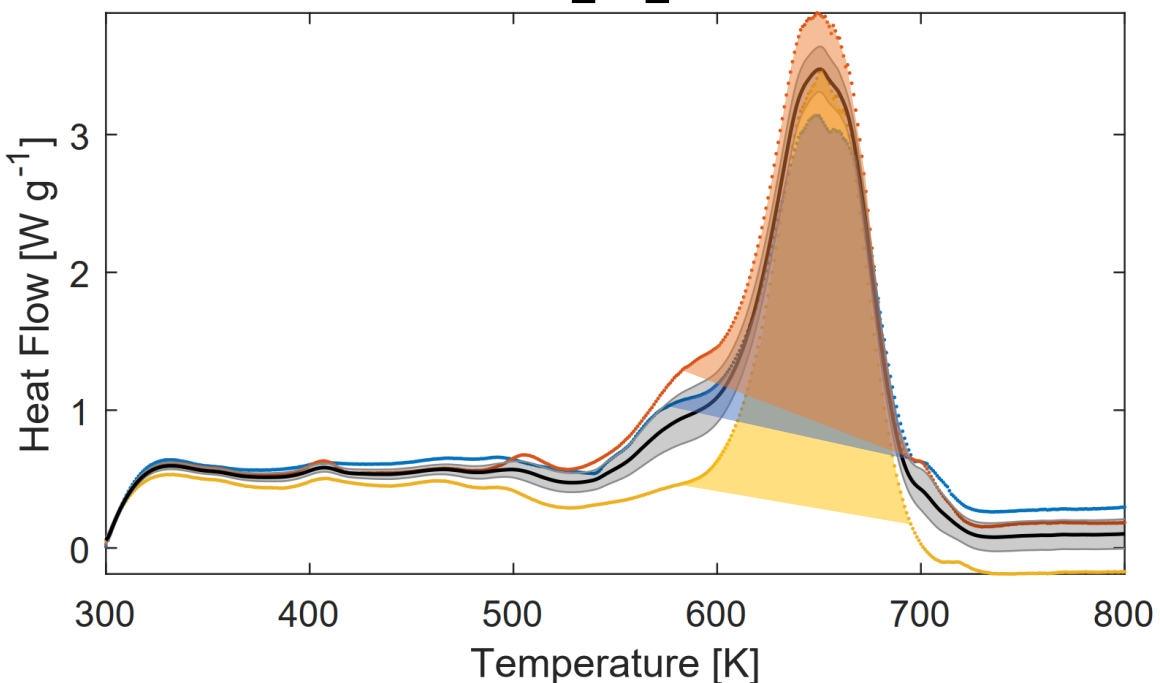
Low temperature measurements (UMET) for determination of heat capacity, c_p



Differential Scanning Calorimetry (DSC)

Determination of heat of reaction, h_r

DBI_Lund
DSC_N2_20K

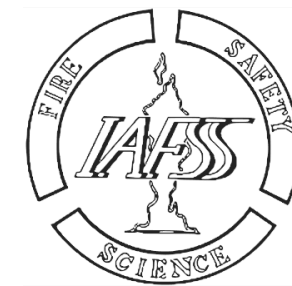


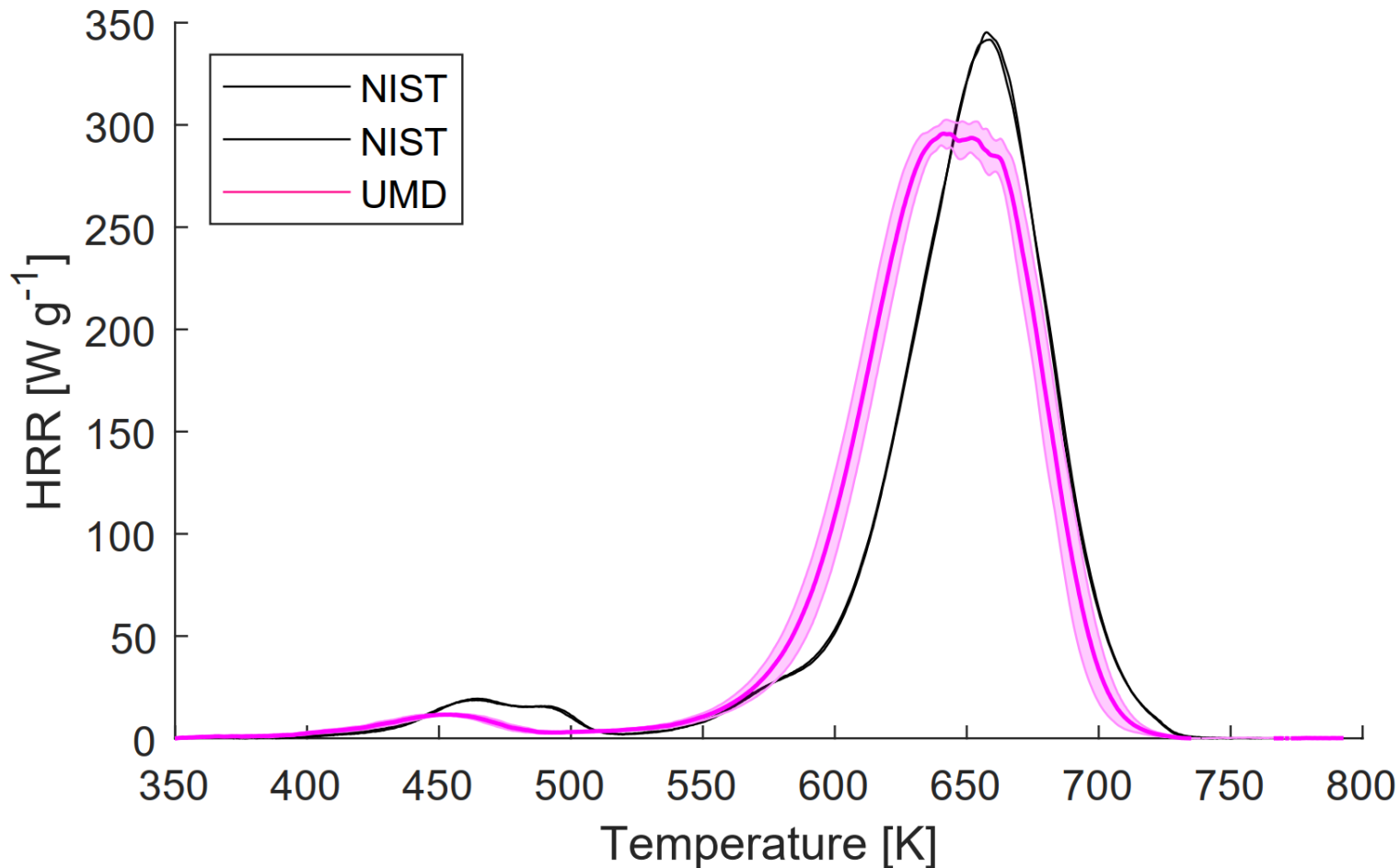
Estimated heats of reaction, h_r (J/g), in anaerobic DSC tests

Institution	Carrier Gas	Heating Rate (K/min)	Heat of Reaction (J/g)					
			Test 1	Test 2	Test 3	Test 4	Test 5	Test 6
Sandia	Argon	1	1183					
		10	461	452				
		50	231	255	245			
DBI_Lund	Nitrogen	20	407	491	509			
GIDAZE+	Nitrogen	10	773	835				
NIST	Nitrogen	10	716	718	718	705	710	
TIFP	Nitrogen	10	1104	980	770	985	976	906
UMD	Nitrogen	10	696					

Nitrogen Environment, $\frac{dT}{dt} = 20 \text{ K/min}$

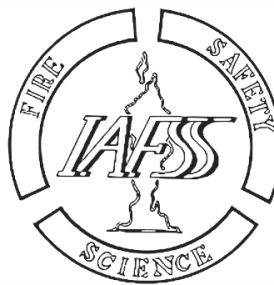
$$h_r = \frac{(q_{\text{tot}} - q_{\text{baseline}})}{\Delta m}$$





- Nitrogen environment
 - 100 cc min⁻¹ UHP N₂
- Single Heating rate
 - $\frac{dT}{dt} = 60 \text{ K min}^{-1}$
- Heat of Combustion
 - $\Delta H_c = 23.5 \text{ or } 24.5 \text{ kJ g}^{-1}$

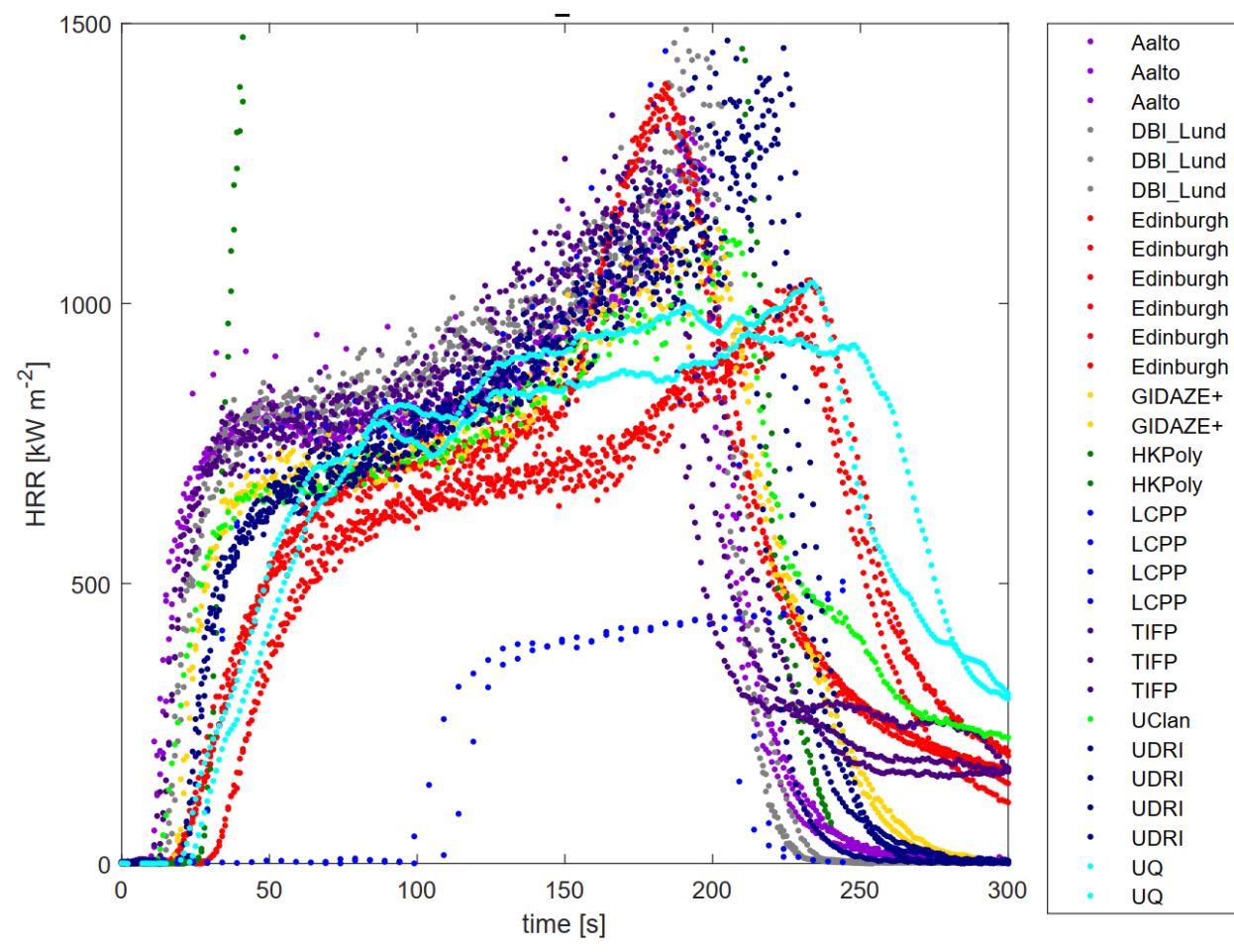
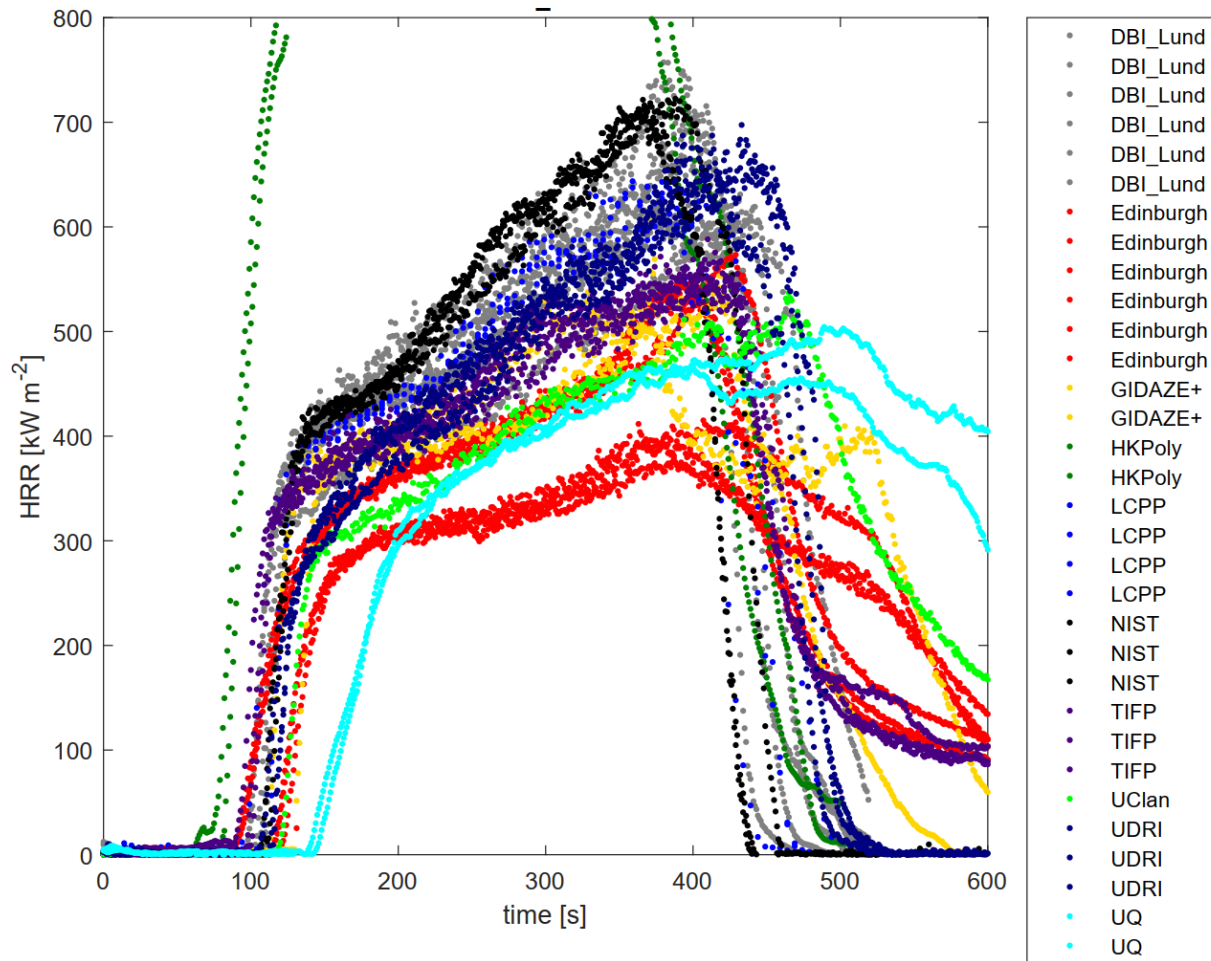
- 10 institutions
 - Up to 6 replicates from one lab under same conditions
- 3 incident heat fluxes
 - $25 \leq q_{ext}'' \leq 65 \text{ kW m}^{-2}$
- Measurement Data (1 Hz)
 - Sample Mass [g]
 - Heat Release Rate [kW m^{-2}]
 - Back Surface Temperature [K]



Cone Calorimeter: Impact of Backing Insulation

External Heat Flux, $q''_{ext} = 25 \text{ kW m}^{-2}$

External Heat Flux, $q''_{ext} = 65 \text{ kW m}^{-2}$

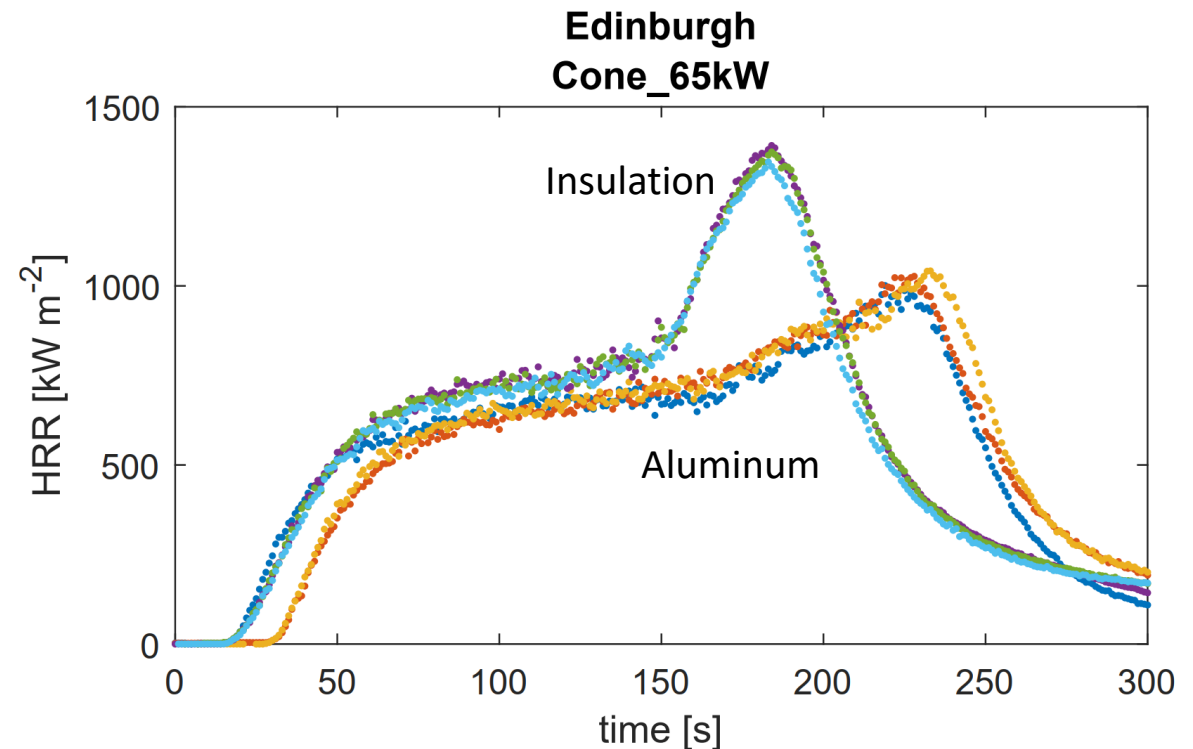
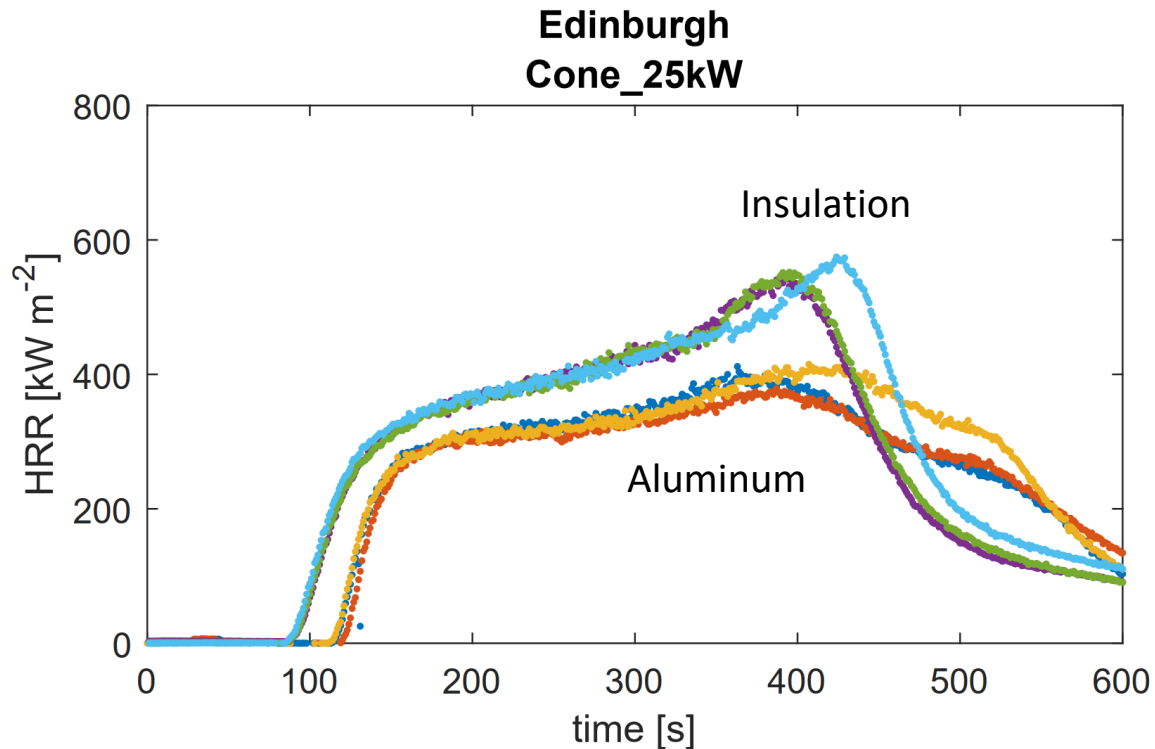


Cone Calorimeter: Impact of Backing Insulation



Institution	Backing Material (Insulation or Metal Block)	[Frame: yes/no?]
Aalto	Two layers (each ~1 cm thick) of ceramic wool with a density of 65 kg/m ³ as specified in standard ISO 5660-1 [1]; its other thermal properties are not known.	
DBI & Lund	Wrapped (bottom and sides) in aluminum foil, and placed on top of two 13 mm thick layers of Morgan Thermal Ceramics Superwool SW Plus (Density 64 kg/m ³)	
Edinburgh	Wrapped with 1 layer of aluminum foil, backed by 12 sheets of 3 mm thick Superwool XTRA Paper (k, ρ in README)	
HK Poly	Backing Insulation: thermal conductivity= 0.1 W/m-K, density = 800 kg/m ³ , heat capacity = 0.5 [Thickness, material type?]	
LCPP	Monolith substrates (3x, each 12 mm thick) [material type/thermal conductivity / thermophysical properties?]	
NIST	One-inch thick (2.54 cm) layer of Kaowool Blanket. Density = 128 kg/m ³ . Thermal Conductivity 0.06, 0.012, 0.21, 0.3 W/m-K (at 260, 538, 816, 1093 C, respectively)	
TIFP	Earth-alkali silicate wool, [thickness?], thermal conductivity at 600K 0.16 kW/m/K	
UCLan	Glass wool [thickness / density / thermal properties?]	
UDRI	Ceramic wool thermal conductivity (measured via guarded hot plate: 0.04 W/m-K at 30 C) [thickness?]	
UQ	Vermiculite with Dow Corning Dowsil 340 paste [thickness, material properties?]	
Edinburgh	Wrapped with 1 layer of aluminum foil, in contact with an aluminum block 10 mm thick	
GIDAZE+	Ceramic fibre backing pad at the sides of the sample and the aluminum block and at the rear face of the aluminum block [thickness of both, thermal properties of insulation?]	

Cone Calorimeter: Impact of Backing Insulation



Back /sides of sample wrapped with aluminum foil,
wrapped sample then placed on either:

Insulation: 12 sheets of 3 mm thick Superwool XTRA Paper

Aluminum: 10 mm thick aluminum block

Density:	$\rho_{\text{insulation}} \approx 200 \text{ kg m}^{-3}$		$\rho_{\text{aluminum}} \approx 2700 \text{ kg m}^{-3}$	
Temperature:	200 °C	400 °C	600 °C	1000 °C
$k_{\text{insulation}} [\text{W m}^{-1} \text{K}^{-1}]$	0.05	0.08	0.13	0.3
$k_{\text{aluminum}} [\text{W m}^{-1} \text{K}^{-1}]$	215*	249*	-	-

Cone Calorimeter: Heat Release

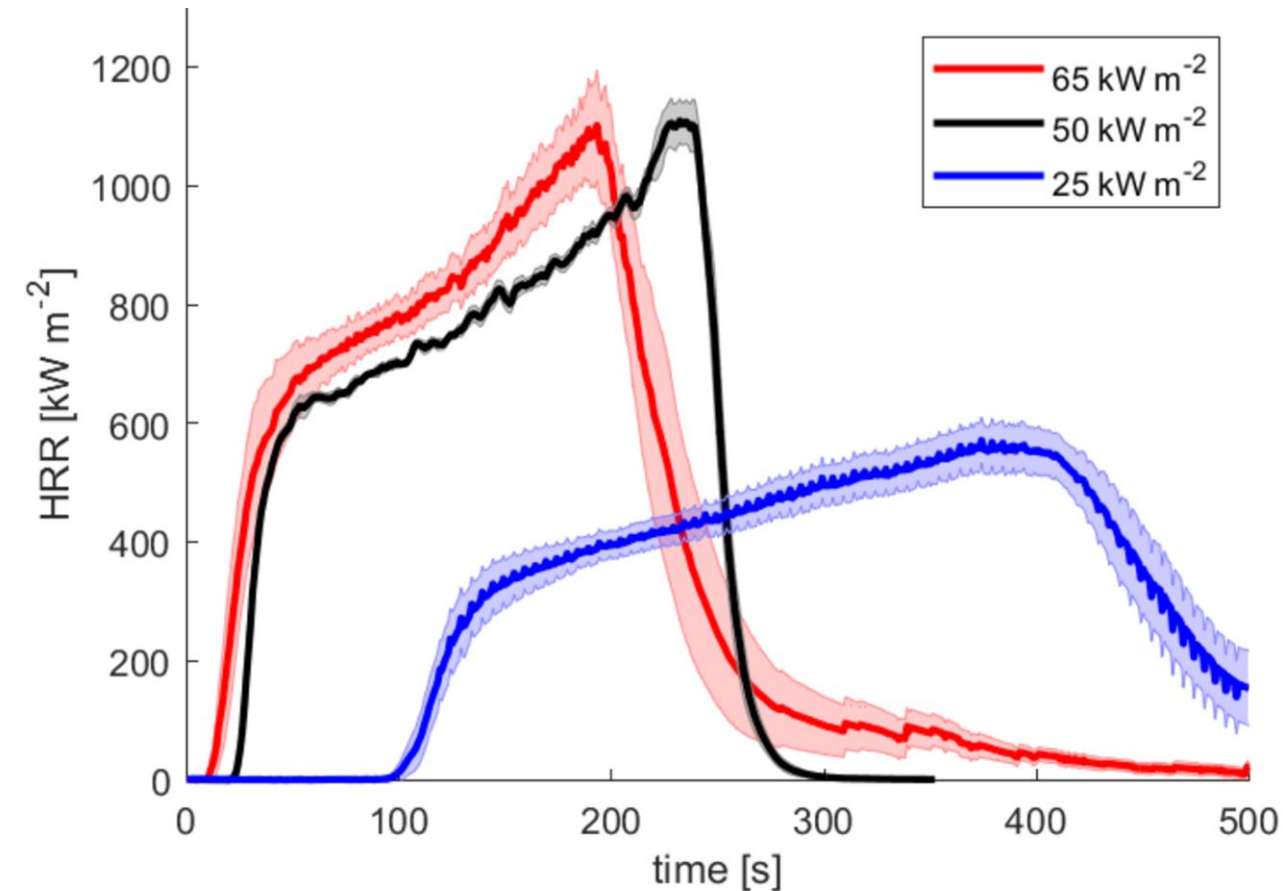
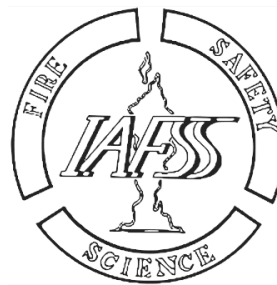
Lyon & Quintiere. *Comb. and Fl.*, 2007

Time to ignition, t_{ign} , in each cone calorimeter experiment is defined as the time at which $HRR \geq 24 \text{ kW/m}^2$

q_{ext}'' [kW m ⁻²]	Average (lab) t_{ign} [s]
25	99 to 148
50	26 +/- 2
65	12 to 30

Heat of combustion, ΔH_c : total energy released per gram of gaseous volatiles produced (kJ/g) when $HRR \geq 240 \text{ kW/m}^2$ (i.e., ten times the critical ignition HRR)

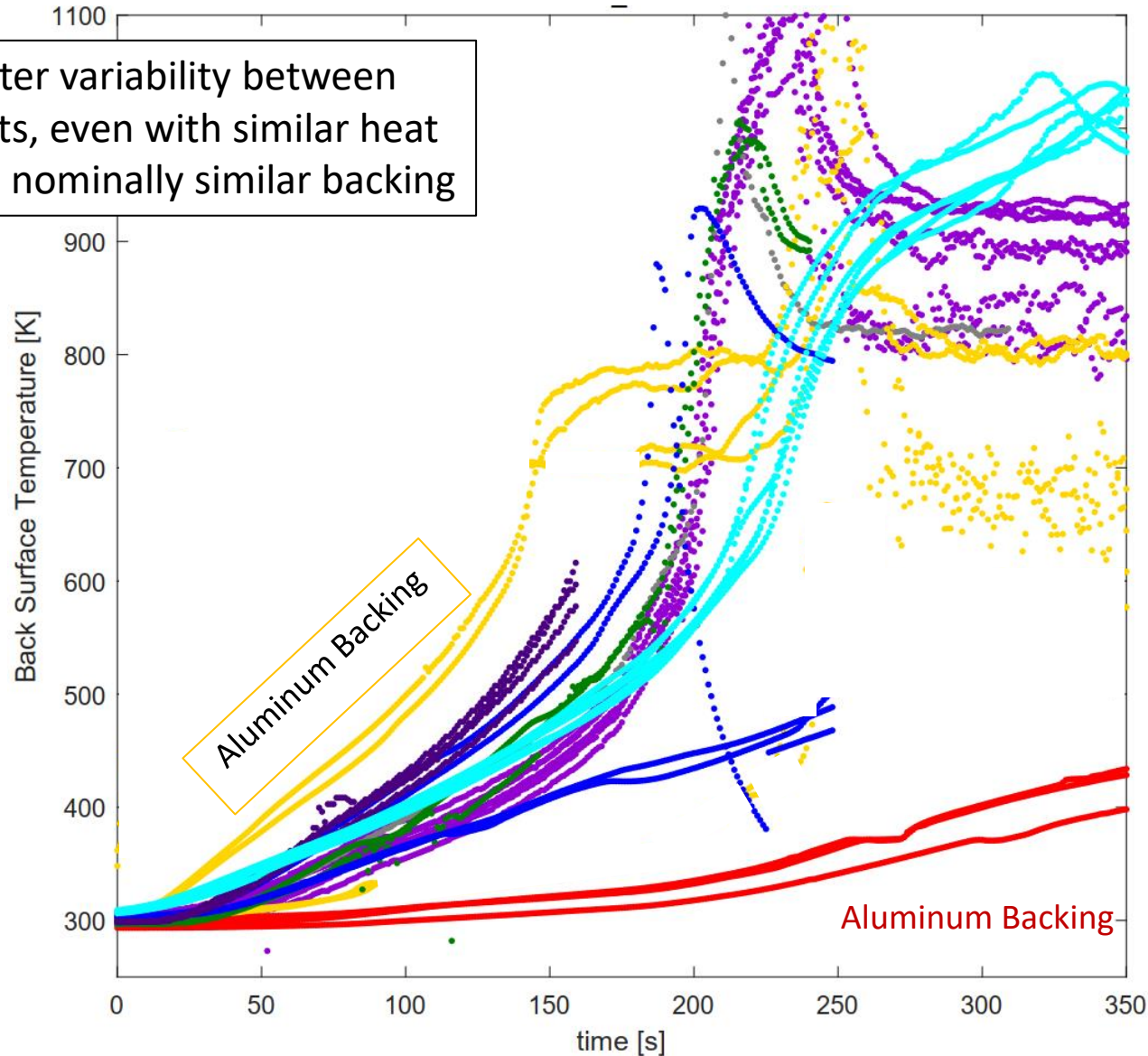
q_{ext}'' [kW m ⁻²]	Average (lab) ΔH_c [kJ g ⁻¹]
25	22.0 to 24.9
50	24.5 +/- 0.3
65	22.3 to 26.1



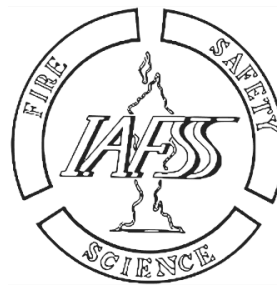
Average curves presented here represent the aggregate of data as received, some of which may require corrections by the original submitting institution.

Cone Calorimeter: Back Surface Temperatures

Significantly greater variability between institutional datasets, even with similar heat release rate profiles, nominally similar backing



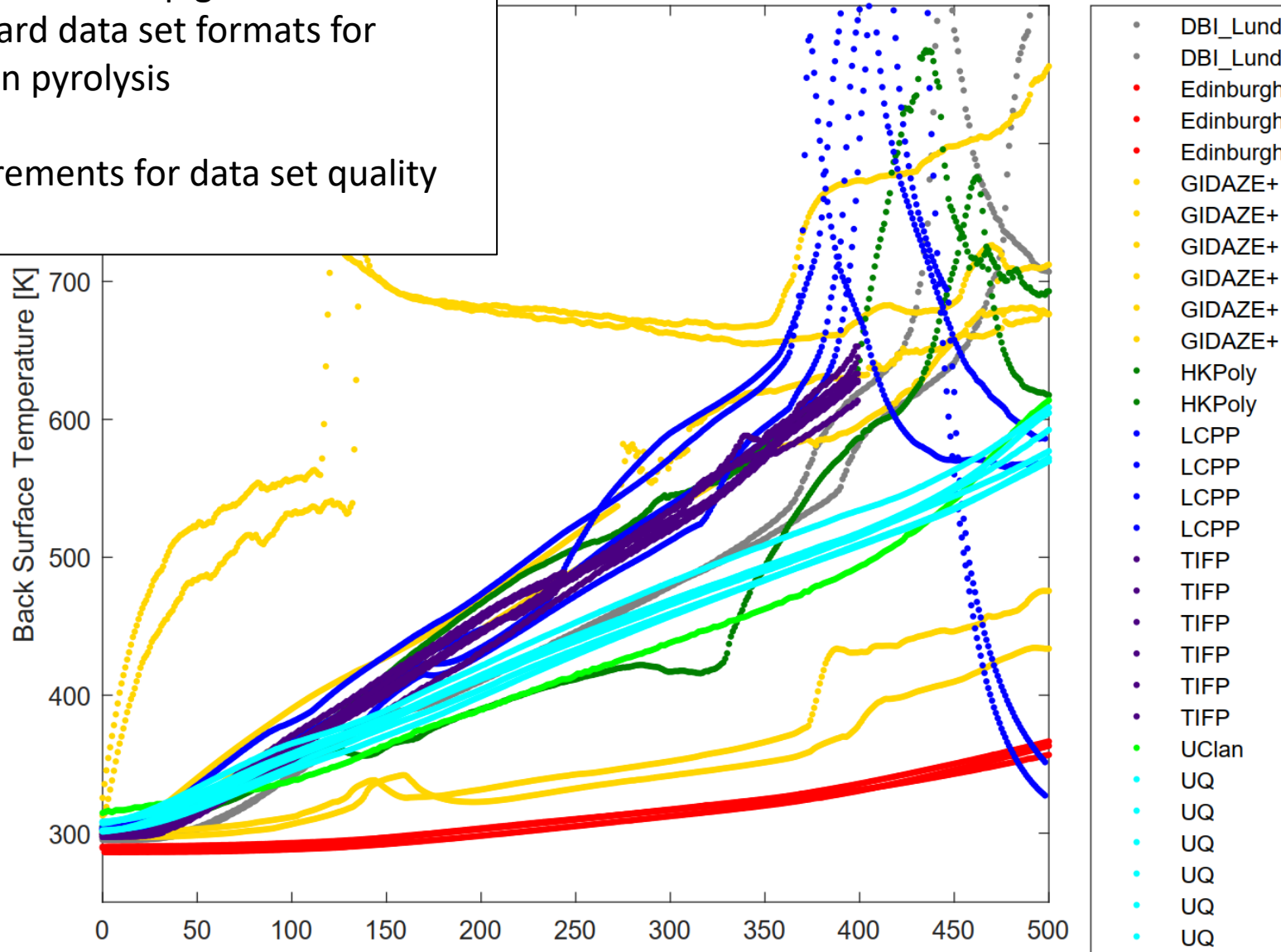
External Heat Flux:
 $q_{ext}'' = 65 \text{ kW m}^{-2}$



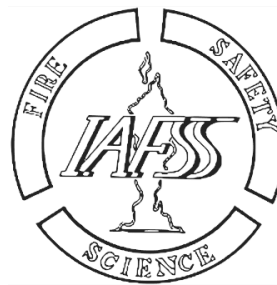
Cone Calorimeter: Back Surface Temperatures

Consider Workshop goals:

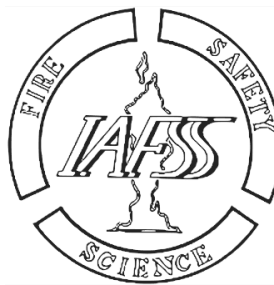
1. Developing standard data set formats for experimental data on pyrolysis
2. Developing requirements for data set quality



External Heat Flux:
 $q_{ext} = 25 \text{ kW m}^{-2}$



- 5 institutions; 3 experimental apparatus
 - Controlled Atmosphere Pyrolysis Apparatus (CAPA)
 - Controlled Atmosphere Cone Calorimetry
 - Fire Propagation Apparatus (FPA) → Tungsten Lamps: 2600KHeater Temperature: 800 to 1200 K
- Incident heat fluxes between
 - $25 \leq q_{ext}'' \leq 65 \text{ kW m}^{-2}$
- Measurement Data (1 Hz)
 - Sample Mass [g]
 - Back (or Front) Surface Temperature [K]
- Requests to experimentalists:
 - README files → metadata

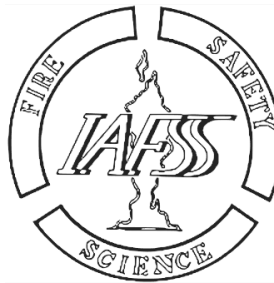


- Data Formatting
 - [time (s) | Mass (g) | Temp (K)]
 - Tare measurements
 - Initial mass = sample mass, m_0
 - Reporting frequency [1 Hz]
- Processing Data
 - Calculate mass flux as numerical derivative of sample mass
 - Apply Savitzky-Golay filter
 - Calculate average mass flux

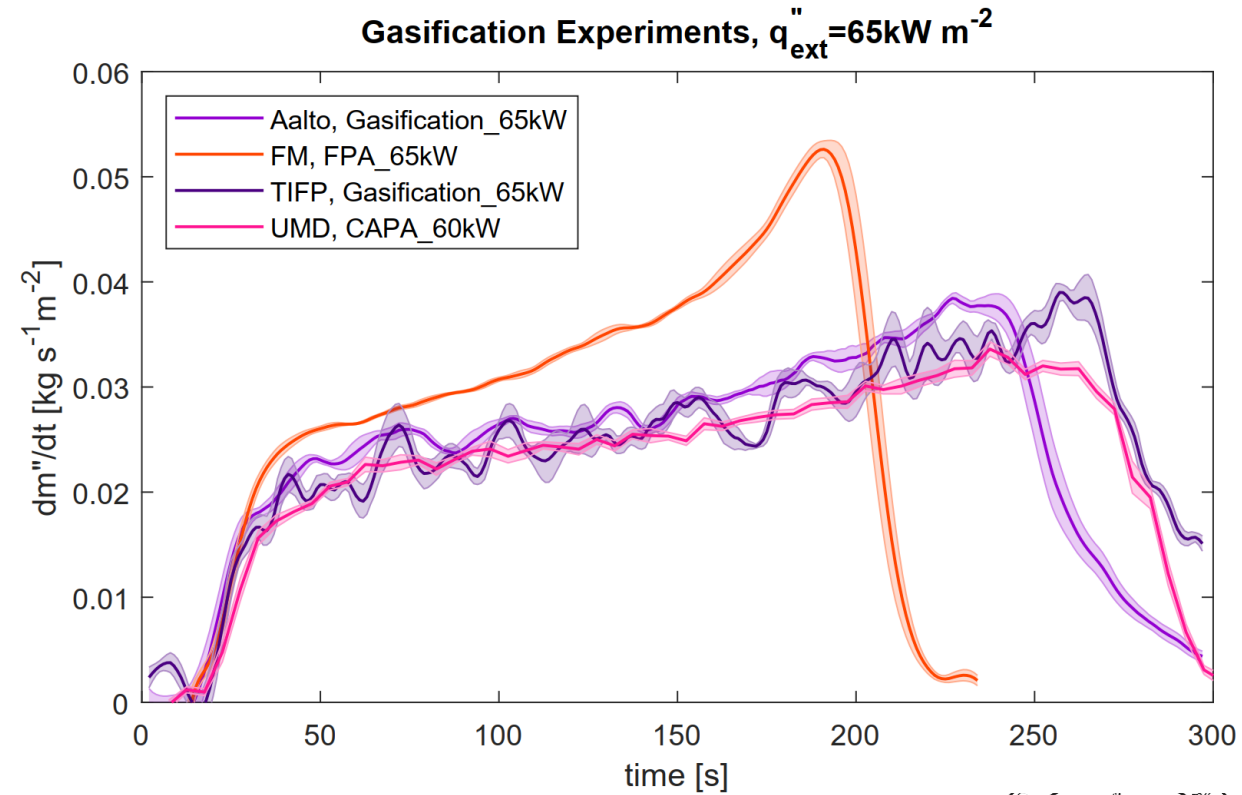
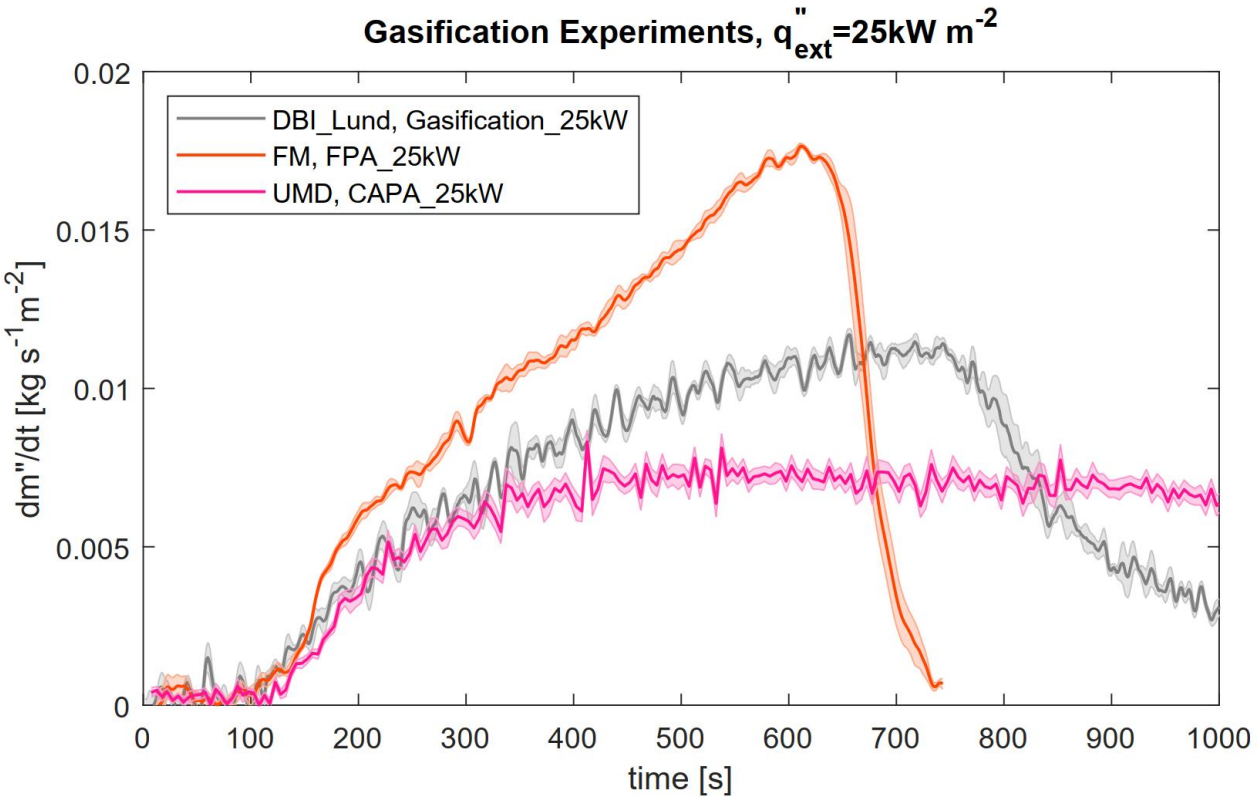
$$\dot{m}''_{i,j} = \frac{1}{A} \frac{\dot{m}_{i-2,j} - \dot{m}_{i+2,j}}{t_{i+2} - t_{i-2}}$$

filter

$$\overline{\dot{m}''_i} = \frac{1}{\sum_{j=1}^{N_j} N_i} \sum_{j=1}^{N_j} \sum_{i'=i-n}^{i+n} \dot{m}''_{i',j}$$

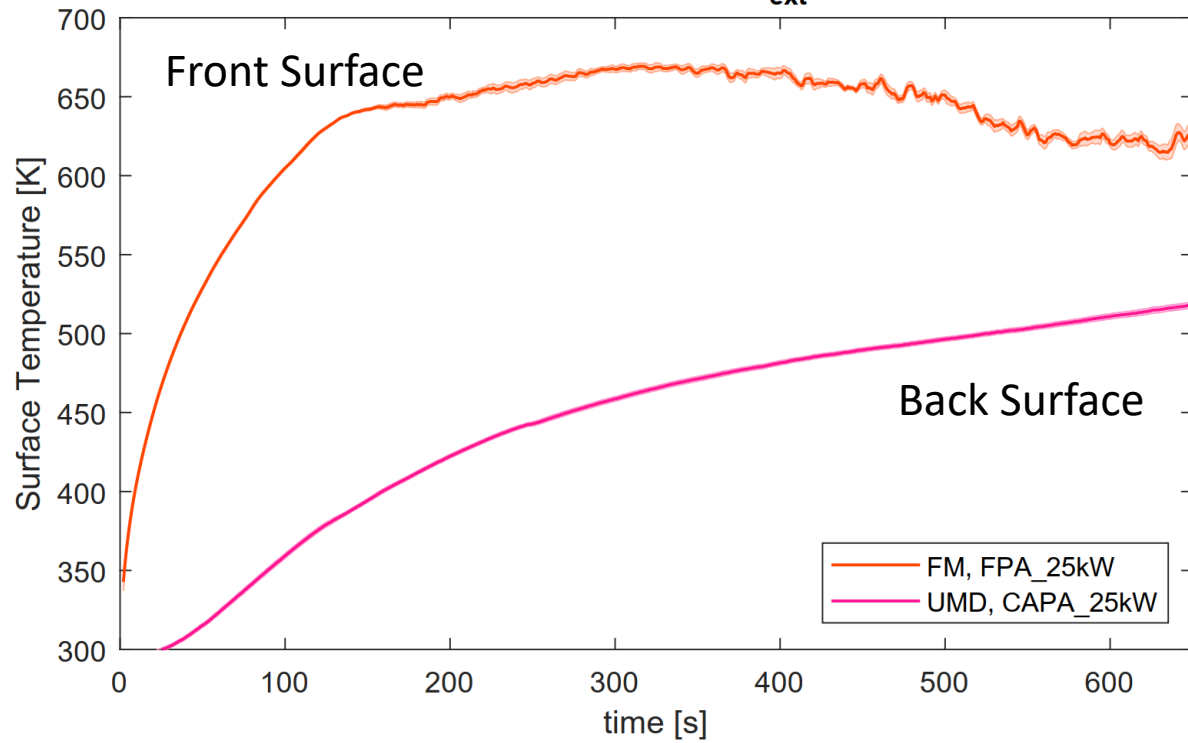


Anaerobic Gasification Experiments Mass Loss Rate

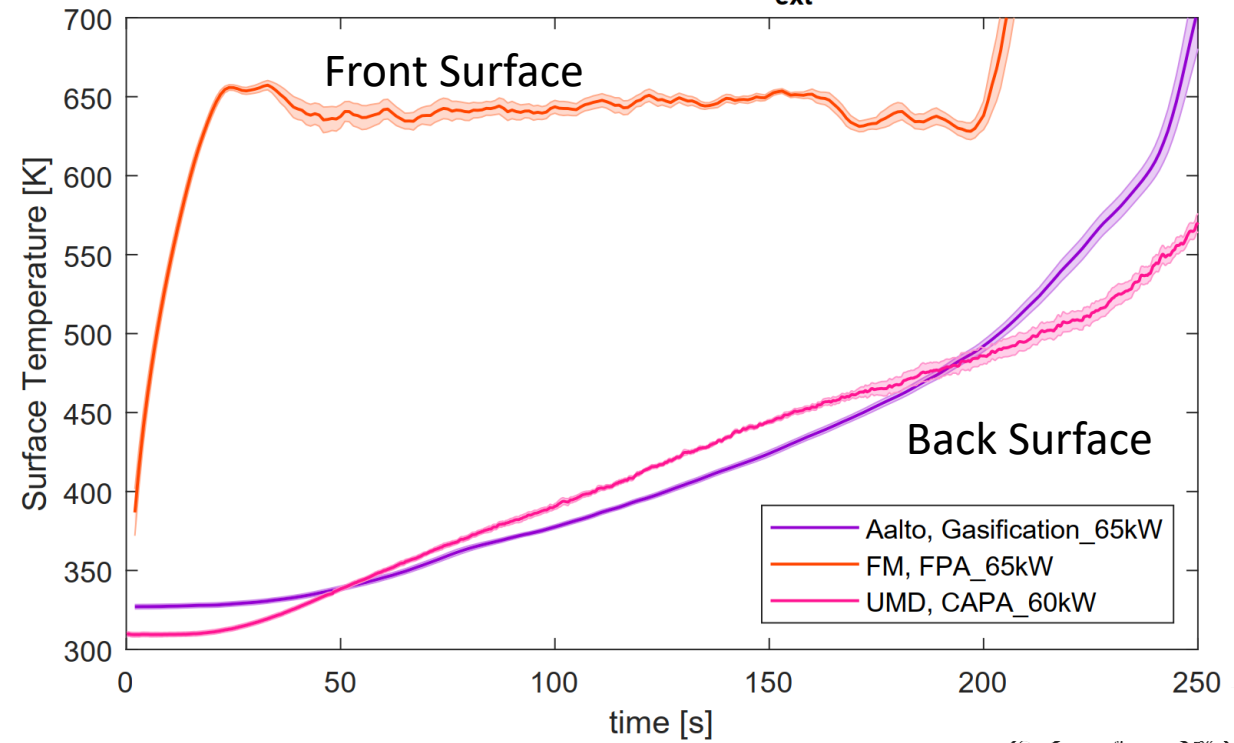


Anaerobic Gasification Experiments Surface Temperature

Gasification Experiments, $q''_{\text{ext}}=25\text{kW m}^{-2}$



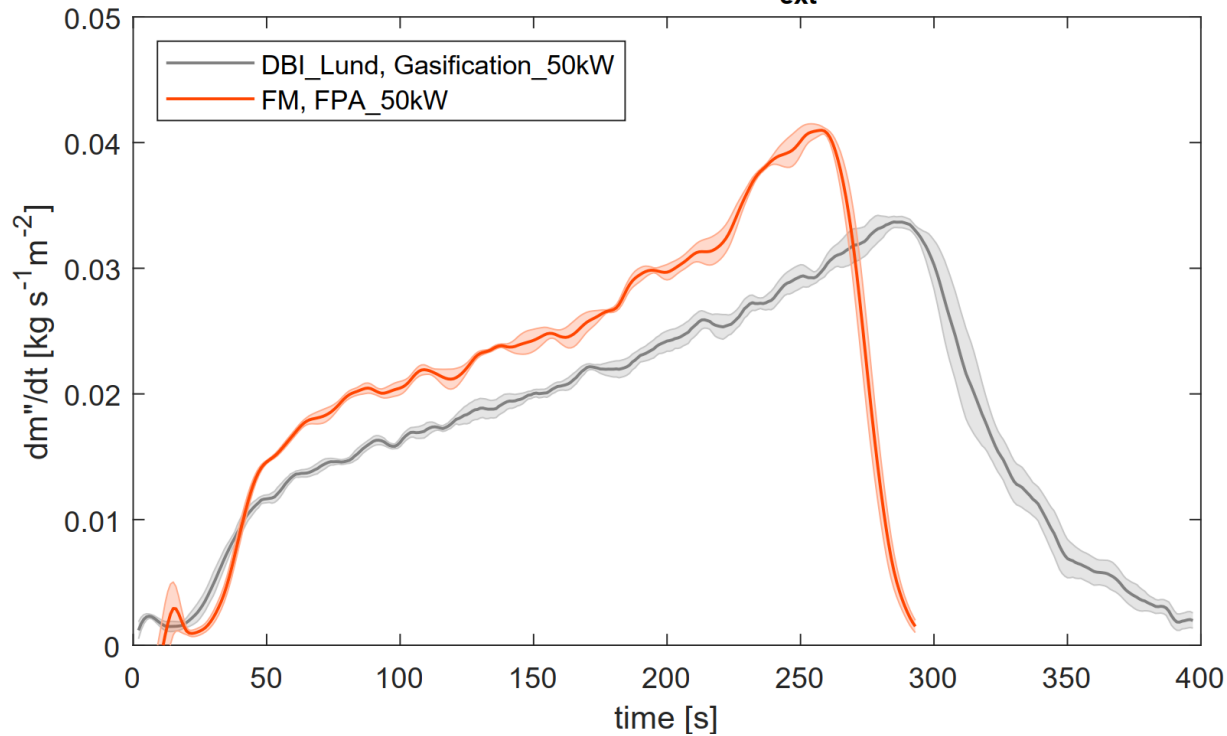
Gasification Experiments, $q''_{\text{ext}}=65\text{kW m}^{-2}$



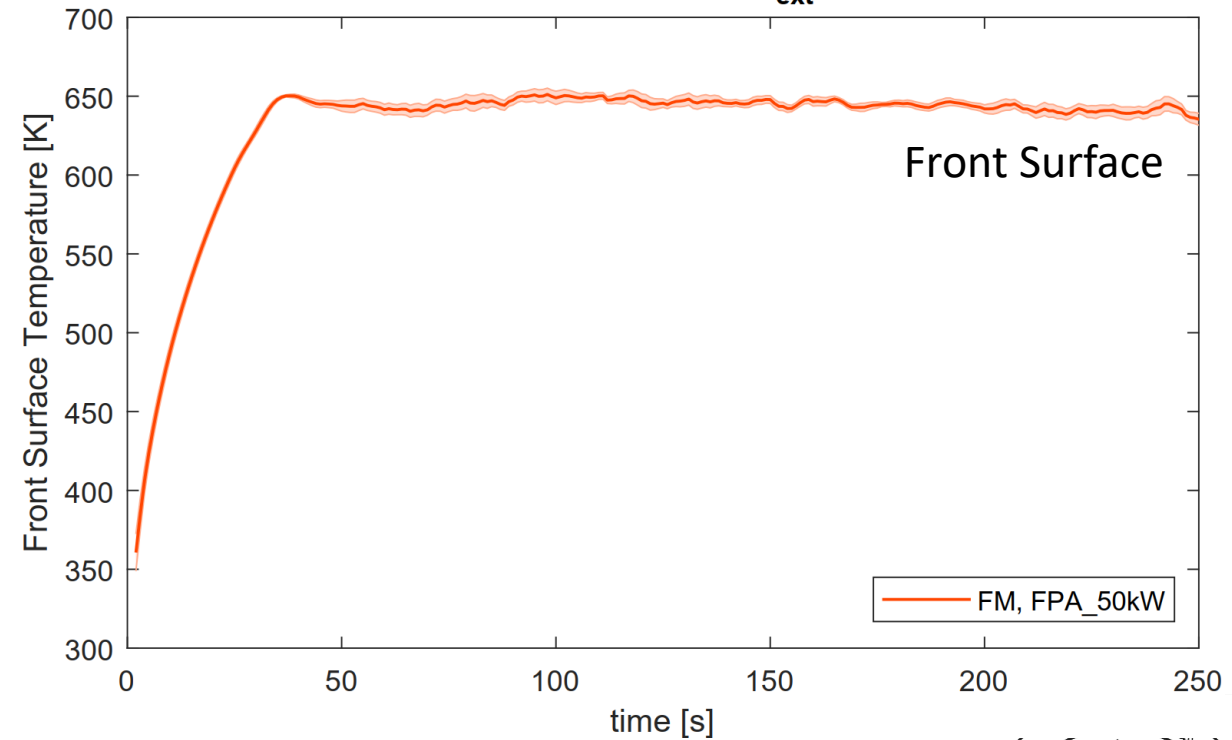
Anaerobic Gasification Experiments

Mass Loss Rate and Front Surface Temperature

Gasification Experiments, $q''_{\text{ext}} = 50 \text{ kW m}^{-2}$

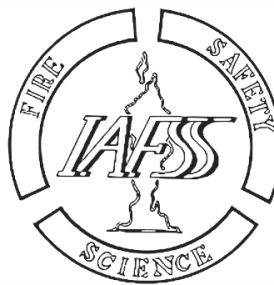


Gasification Experiments, $q''_{\text{ext}} = 50 \text{ kW m}^{-2}$



“Direct” Measurements of Thermal Conductivity and Diffusivity

- 2 institutions; 3 experimental apparatus
 - Thermal conductivity (2x)
 - Thermal diffusivity (1x)



“Direct” Measurements of Thermal Conductivity

“Direct” measurements match within 15-20%
between different apparatus

- UMET
 - Transient Plane Source Method
 - TPS 2500S-Hot Disk

Sample	Temperature (K)	Thermal Conductivity (W/m/K)	Mean Dev. (K)
PMMA 1	295	0.220	9.63×10^{-4}
	295	0.210	2.69×10^{-4}
	295	0.209	2.68×10^{-4}
PMMA 2	293	0.209	8.65×10^{-4}
	293	0.208	2.60×10^{-4}
	293	0.209	1.99×10^{-4}

- DBI & Lund
 - Netzsch HFM 446 Medium

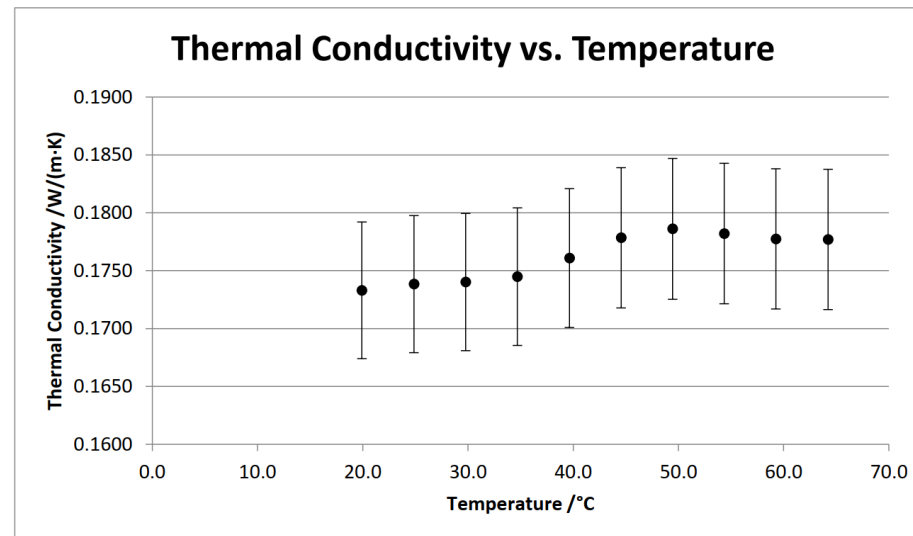
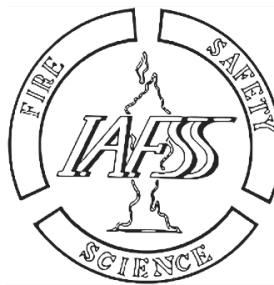


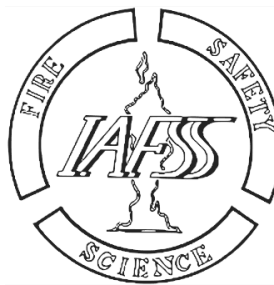
Figure 13 Measured thermal conductivity of PMMA



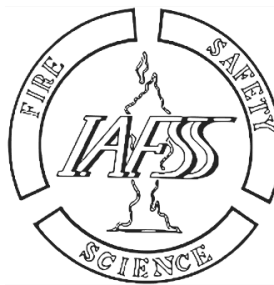
“Direct” Measurements of Thermal Diffusivity

- UMET
 - Laser Flash Diffusivity
 - Netzsch Light Flash Apparatus (LFA 467)

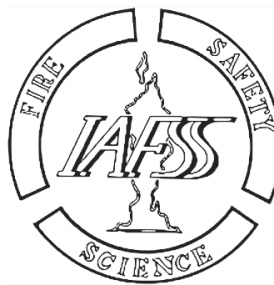
Sample	Temperature (K)								
	298	303	313	323	333	343	353	363	373
	Sample Thermal Diffusivity (mm ² /s)								
PMMA 1	0.117	0.115	0.111	0.110	0.109	0.106	0.105	0.103	0.102
PMMA 2	0.112	0.112	0.110	0.108	0.107	0.106	0.104	0.103	0.101



- Repository of Experimental Data: <https://github.com/MaCFP/matl-db>
 - A digital archive, version-controlled, of well-documented experiments that can be used as targets for pyrolysis model calibration and validation
 - Initial scripts written for data analysis
 - Progress towards developing standard data set formats for experimental data
- Preliminary, predecisional draft report of initial results prepared and shared for critical review: <https://github.com/MaCFP/matl-db/releases>
 - Data review: initial requirements for data set quality
 - <https://github.com/MaCFP/matl-db/tree/master/Non-charring/PMMA>
 - Initial quantification of inter-laboratory variability for comparable experimental datasets

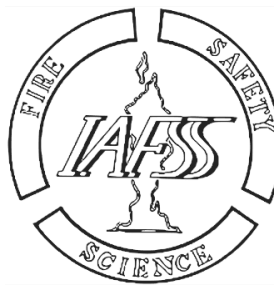


- Test conditions, procedure, and initial calibration may have a meaningful impact on measurement results
 - Calibration is critical, especially for DSC measurements
 - Variations in data sets
 - Stochastic vs. identifiable causes
 - Identify correlations, quantify sources of error
 - Further analysis to be provided in final report
- Use care when selecting measurements from various sources, *clearly* identify test setup and conditions



MaCFP Objectives as Discussion Topics

1. Developing standard data set formats for experimental data on pyrolysis
2. Developing requirements for data set quality
3. Next Steps (Experimental) for MaCFP 2024
Proposals & Commitments
4. Open Discussion



1. Developing standard data set formats for experimental data on pyrolysis

How to identify these datasets with missing information (regarded as containing errors)?

Information / Formatting of READMEs: sufficient?

Metadata needed/wanted

Test setup description

Resolution of measurement data

Calibration information

Structure of repository

Data Submission

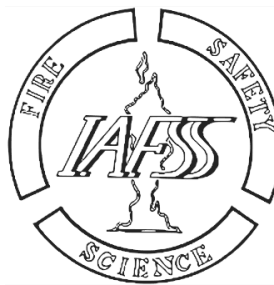
Tare sample mass; HRR and heat flow baselines (start and end)

Github PR; at a minimum must match .csv file / data format

Suggested Proposal:

Established tests: all data must be submitted with complete README/metadata

New test types: research submit all information deemed necessary, feedback to standardize



2. Developing requirements for data set quality

How to identify these datasets that need partial or complete edits?
(regarded as containing errors)

What constitutes 'good' data?

Requirements for calibration (number / type / frequency)

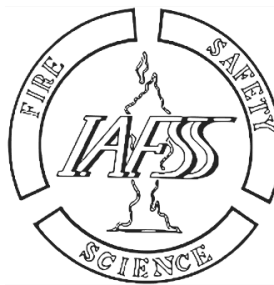
<https://github.com/MaCFP/matl-db/tree/master/Non-charring/PMMA>

Key factors influencing material response during tests

Outlier Criteria: Identification of clearly incorrect behavior in measurement data

Removal of extraneous measurements

(e.g., poor thermal contact of thermocouples later in test)



2. Developing requirements for data set quality

How to interpret and use measurement data in this repository for pyrolysis model calibration and validation

Key factors influencing material response during tests

In this directory, experimental measurements from both mg- and g-scale tests (conducted by 16 unique fire safety science institutions around the world) are available. Every effort was made to remove variability between results due to potential differences in material composition by sharing samples of the same exact PMMA, prepared for mg- and g-scale experiments; that is, all tests were performed on the exact same material, from the same source, and prepared in the same form (excluding minor variations, ~5%, in sample slab thickness, as produced by the manufacturer).

In many cases, data from the same test type under the same nominal test conditions that was provided by different institutions showed qualitative agreement; in others, variations are apparent. Some variations between datasets are simply stochastic (i.e., random, unavoidable noise in repeated tests). However, others may result from systematic causes such as calibration differences in mg-scale experiments or sample holder, insulation type, and/or heater temperature in g-scale experiments. When such measurement data is used as a reference for comparison with the results of numerical simulations, it is the responsibility of users of this data to be aware factors that can affect this smaterial flammability response. A short summary of these factors is provided below. For further detail, the user of this data is referred to the [Preliminary Summary of Experimental Measurements Document](#)

Milligram-scale experiments

- Heating Rate
- Gaseous Environment
- Crucible Type
- Apparatus Calibration (should be performed for identical test conditions: heating rate, gaseous environment, crucible type)
- Baseline Correction (especially for DSC measurements)
- Initial Sample Mass (and geometry)

Gram-scale experiments

- Incident Heat Flux (and uniformity across sample's surface)
- Gaseous Environment (and flow rate, which can affect convective heat transfer at the sample's surface prior to gasification or burning)
- Backing Material (e.g., the presence or absence of an insulating substrate, and its thermal properties)
- Heater Temperature
- Exposed Sample Surface Area
- Sample Holder Characteristics (including the use/non-use of a retainer frame)
- Baseline Correction (especially for HRR measurements)
- Temperature Measurement Instrumentation and Location (e.g., IR camera vs. thermocouple; thermal contact)

Outlier Criteria: Identification of clearly incorrect behavior in measurement data

With the help of the experimentalists who performed these experiments and shared their data, significant effort has been made to provide consistent formatting of measurement results and to identify and correct small errors that may have arisen either during testing, submission, or compilation and standardization of formatting. Despite these efforts, clear outliers can sometimes be identified. Criteria defining these clear outliers are defined below; it is suggested that, for further analysis, users omit data sets that do not meet these criteria.

Milligram-scale experiments

For all mg-scale tests, average steady state heating rate must match nominal conditions.

Thermogravimetric Analysis (TGA)

In anaerobic conditions, a single reaction peak should be observed at the following temperatures (T_{max}):

- $dT/dt = 5 \text{ K/min}$: $T_{max} = 625 \text{ K} \pm 7.5 \text{ K}$
- $dT/dt = 10 \text{ K/min}$: $T_{max} = 640 \text{ K} \pm 7.5 \text{ K}$
- $dT/dt = 20 \text{ K/min}$: $T_{max} = 650 \text{ K} \pm 7.5 \text{ K}$

In oxygenated environments (21 vol. % O₂) for tests conducted at $dT/dt = 10 \text{ K/min}$, two reaction peaks should be observed at the following temperatures (T_{max}):

- $T_{max1} = 580 \text{ K} \pm 7.5 \text{ K}$
- $T_{max2} = 605 \text{ K} \pm 7.5 \text{ K}$

Differential Scanning Calorimetry (DSC)

- At all heating rates, *integral* heat flow must be positive for all times/temperatures

Microscale Combustion Calorimetry (MCC)

- Heat of combustion, $H_c = 24 \text{ kJ/g} \pm 1.5 \text{ kJ/g}$

Gram-scale experiments

Cone Calorimetry

1. Heat Release Rate Measurements (HRR) Heat of combustion, $H_c = 23.5 \text{ kJ/g} \pm 2.5 \text{ kJ/g}$
2. Back Surface Temperature Measurements Prior to sample burnout (which occurs at approximately 400 s, 250s, and 200 s with incident heat fluxes of 25, 50, and 65 kW/m², respectively), temperature, T, should increase monotonically. That is, mean $dT/dt > 0$ (across any 20 s interval).

Gasification Experiments (CAPA, FPA, Controlled Atmosphere Cone)

1. Back Surface Temperature Measurements Prior to sample burnout, back surface temperature should increase monotonically. That is, mean $dT/dt > 0$ (across any 20 s interval).
2. Front Surface Temperature Measurements Prior to sample burnout, front surface temperature should increase rapidly before reaching a relatively constant value equal to the pyrolysis temperature of this PMMA ($\sim 650 \text{ K} \pm 10 \text{ K}$)



3. Next Steps (Experimental) for MaCFP 2024

New Materials (e.g., charring materials, natural fuels, composites, transparent to radiation)?

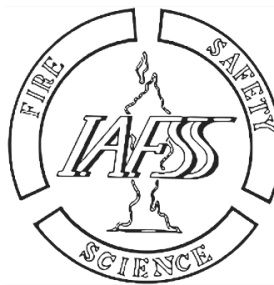
Research effort must connect to critical applications

Additional data needed at mg- or bench-scale?

(e.g., evolved gas analysis, spectrally resolved properties)
the actual voltages / Edinburgh requests...

Who will conduct experiments, procure & distribute material(s)?

If you want to be more involved, please contact us directly



4. Open Discussion (Experimental focus)

Discussion Forum: <https://groups.google.com/g/macfp-condensed-phase-discussions>

