Microscale Combustion Calorimetry to Demonstrate Similarity of Adhesives

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Natallia Safronava, TAMI

Richard E. Lyon, FAA

Objectives

Determine correlation between Microscale Combustion Calorimetry (MCC) properties and FAR flammability tests for adhesives, potting compounds, edge fillers, etc.

Approach

- FSTG members provide cured adhesive specimens and FAR vertical Bunsen burner (VBB) test results:
  - 12 Second VBB
  - 60 Second VBB
- Conduct microscale combustion calorimeter (MCC) testing ASTM D 7309 at FAA on adhesives submitted by FSTG members.
- Correlate MCC flammability properties with FAR flammability (12s and 60s VBB) of adhesives
  - Qualitative (Pass/Fail) Analysis
  - Quantitative (Probabilistic) Analysis

Materials

7 companies participated in the study:

- Magnolia Plastics, Inc
- Bombardier Aerospace
- 3M
- Gulfstream Aerospace Corporation
- Magee Plastics Company
- Zodiac Aerospace
- Sell GmbH

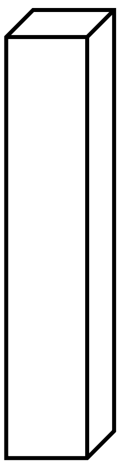
48 samples total were received for testing. 3 specimens with the same name were received from different suppliers.

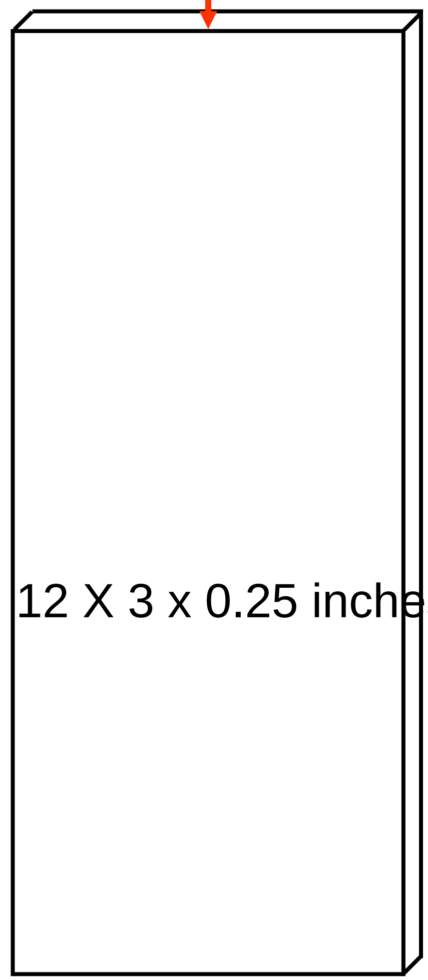
36 samples came with results for 12s and 33 samples for 60s Vertical burn test from the supplying companies.

Specimens for VBB Testing

Standard Specimen size,
Unless the actual size used in
Aircraft is smaller

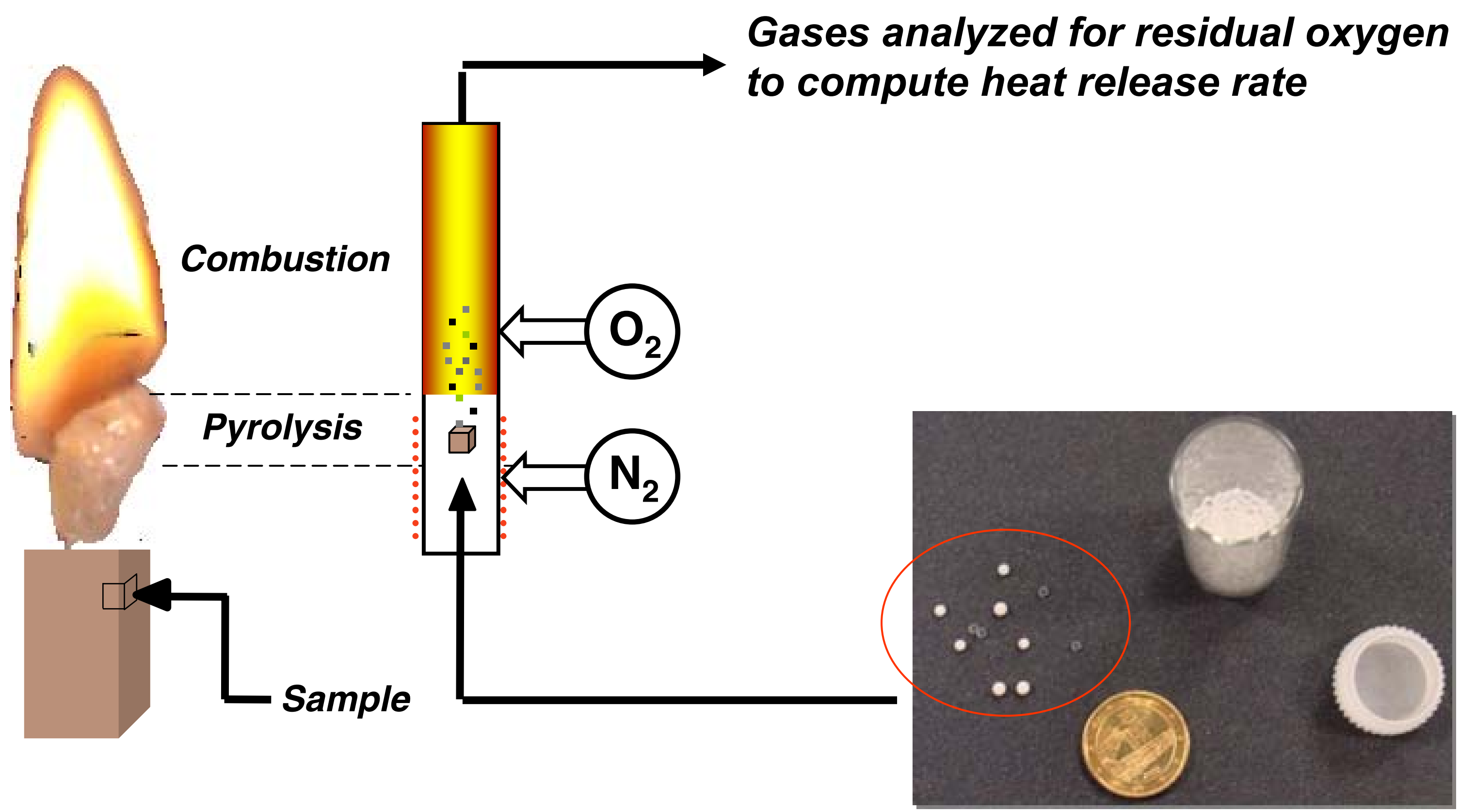
5 X 0.5 x 0.5 inches





…Other?

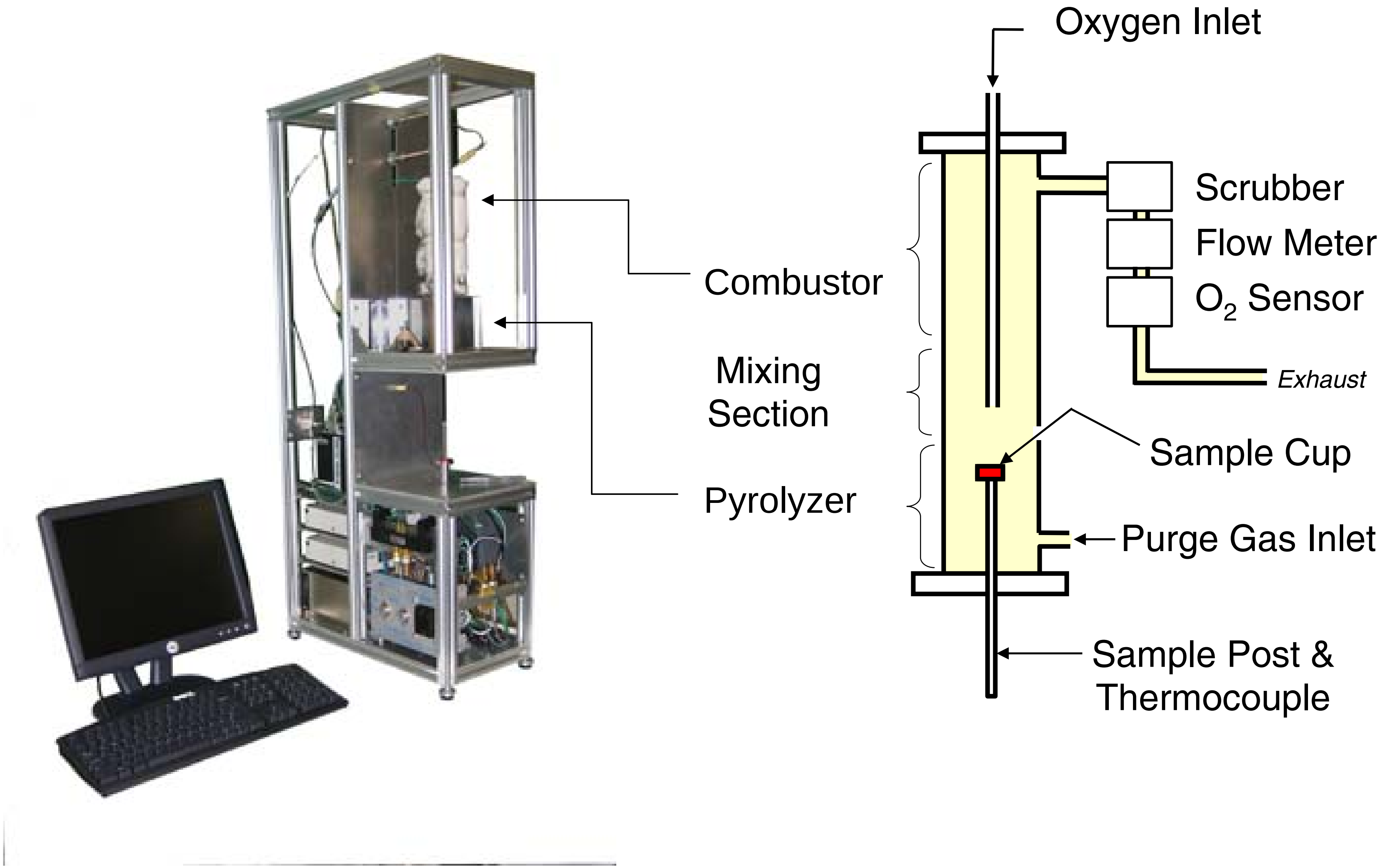
MCC Reproduces Elements of Flaming Combustion in Controlled Test



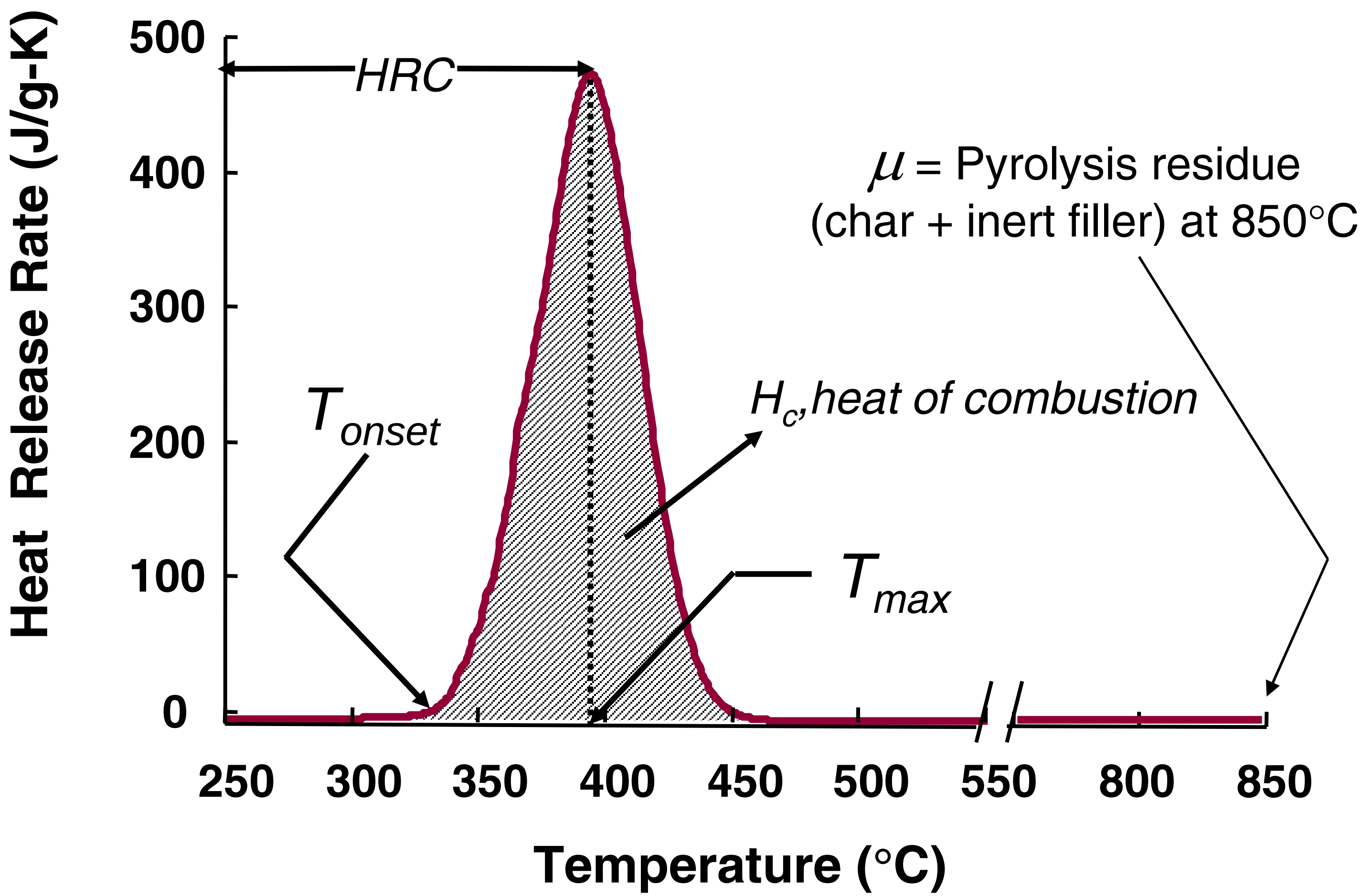
Flaming Combustion

Non-Flaming (Microscale) Combustion

FAA Microscale Combustion Calorimeter
ASTM D 7309



5 Thermal Combustion Properties Measured in MCC Test



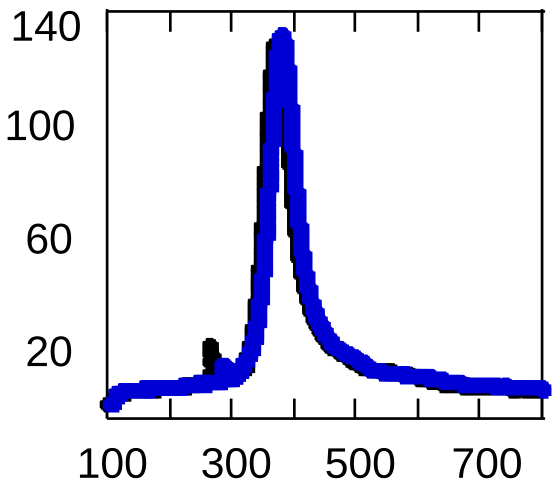
Results of MCC and VBB testing

#	Sample	12s VBB	60s VBB	T_{onset} (°C)	T_{max} (°C)	HR (kJ/g)	HR_{vol} (kJ/g)	HRC (J/g-K)	Char (%)
1	Epoxy Adhesive	Pass	Fail	325	345	19.8	26.1	335	24
2	Epoxy Adhesive	P	P	300	336	12.5	18.9	235	34
3	Epoxy Core Fill	P		311	375	17.6	23.5	220	25
4	Epoxy Core Fill	P		285	340	13.3	21.1	285	37
5	Epoxy Edge-Fill	P		235	311	13.8	20.6	200	33
6	Epoxy Adhesive	P	F	300	345	16.4	23.8	245	31
7	Adhesive	P	P	300	337	14.2	19.8	490	29
8	5 min Epoxy	P	P	307	348	10.3	14.7	335	30
9	10 min Epoxy	P	P	303	347	10.5	14.8	263	29
10	Filling compound		F	295	373	14.4	22.5	201	36
11	Filling compound	P	P	205	380	15	25	206	40
12	Potting compound	P	P	235	337	14.7	21.6	167	32
13	Phenolic resin	P	P	445	564	2.6	16.2	24	84
14	Epoxy	F	F	345	398	25.2	28.6	401	12
15	Epoxy	F	F	355	473	28.4	29.5	457	4
16	Liquid shim	F	F	350	412	16.3	25.6	206	36
17	Epoxy	P	P	315	356	10.3	14.7	279	30
18	Epoxy	P	P	315	346	13.5	18.2	249	26

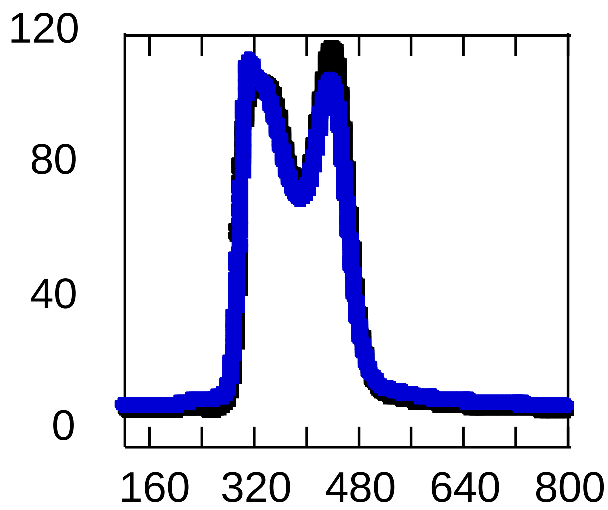
#	Sample	12s VBB	60s VBB	T_{onset} (°C)	T_{max} (°C)	HR (kJ/g)	HR_{vol} (kJ/g)	HRC (J/g-K)	Char (%)
19	Low density Filler	P	P	313	375	9	12.8	135	30
20	Low density Filler	P	P	285	364	16.2	23.1	205	30
21	Low density Filler	F	F	280	313	16.2	23.1	181	30
22	Adhesive	F	F	325	378	25.9	27.8	425	7
23	Adhesive	F	F	315	379	26	28.2	370	8
24	Adhesive	P	P	355	400	13.6	22.7	195	40
25	Adhesive	P		280	335	14.1	20.1	365	30
26	Edge Filling	P	P	280	445	16.4	23.4	185	30
27	Panel Adhesive	P	F	285	342	20	24.1	280	17
28	Adhesive	P	P	280	327	13.8	20	295	31
29	Edge Filling	P	P	313	382	7	10	80	30
30	Edge Filling	P	P	270	300	15.8	20.8	228	24
31	Panel insert	F	F	340	390	25	28.1	395	11
32	Panel&Insert adhesive	F	F	300	394	22	28.9	200	24
33	Fill product	F	F	275	311	18	23.1	310	22
34	Composite insert	F	F	320	379	21.2	26.2	320	19
35	Potting adhesive	P	F		353	15.6	22.3	328	30
36	Potting adhesive	P	P		366	15.2	23.8	196	36
37	Potting adhesive	P	P	190	353	10.2	19.4	121	47

Reproducibility of HRR for Samples from Different Sources Is Excellent

Tests of Filler from 2 Sources



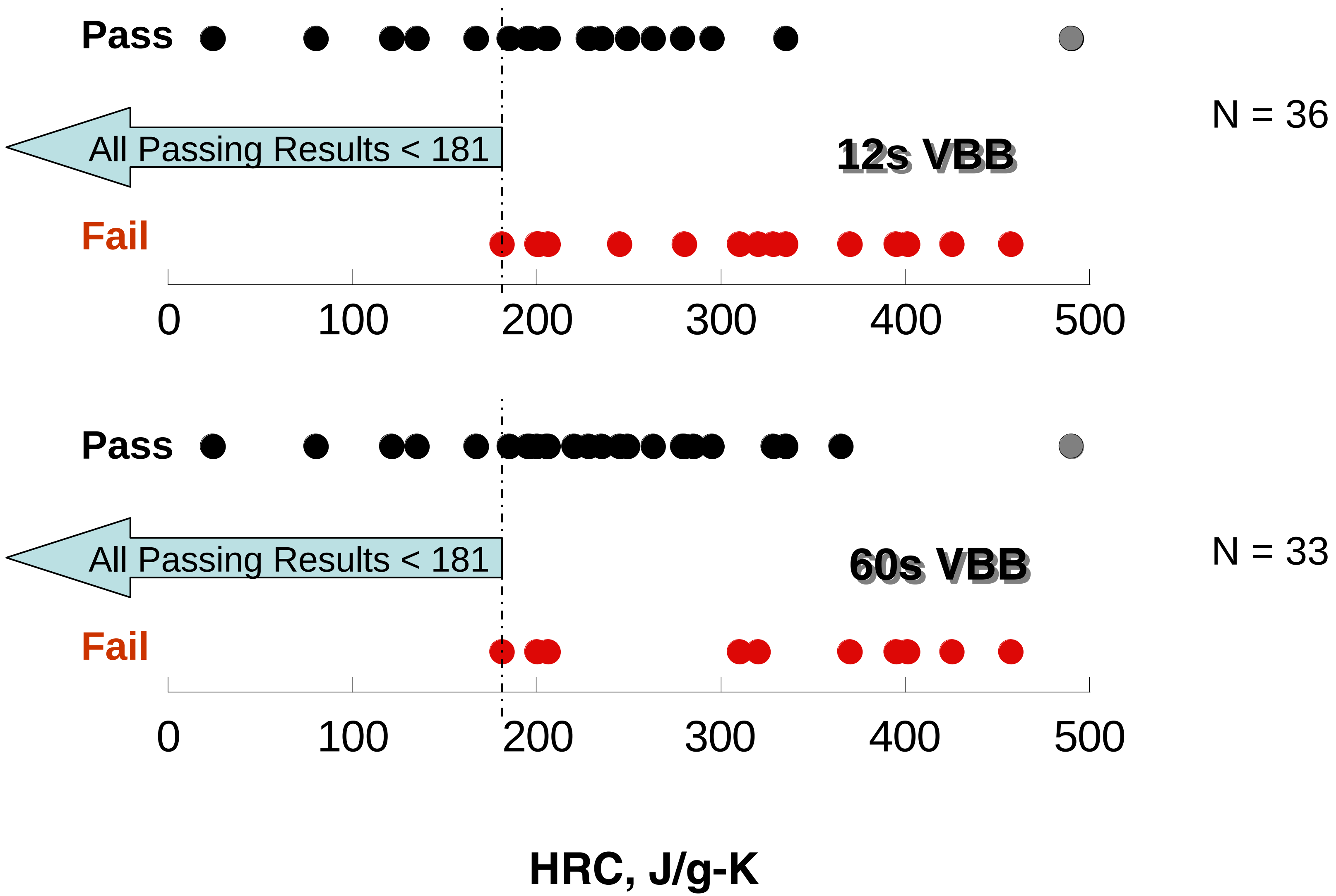
Tests of Low Density Filler from 2 Sources



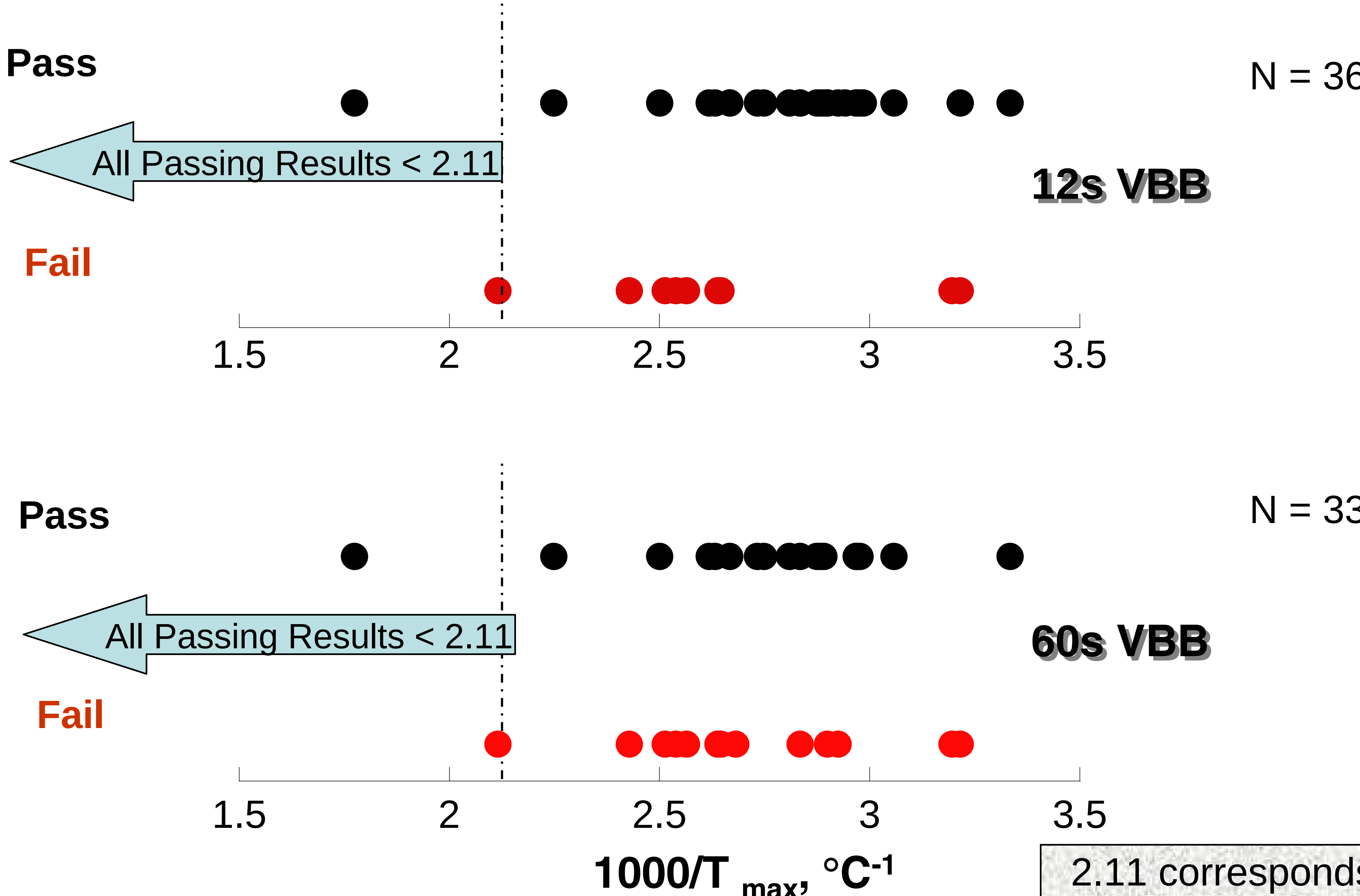
HRR (W/g) vs. Temperature (°C)

Same Sample has Both Pass & Fail VBB ratings

VBB versus Heat Release Capacity

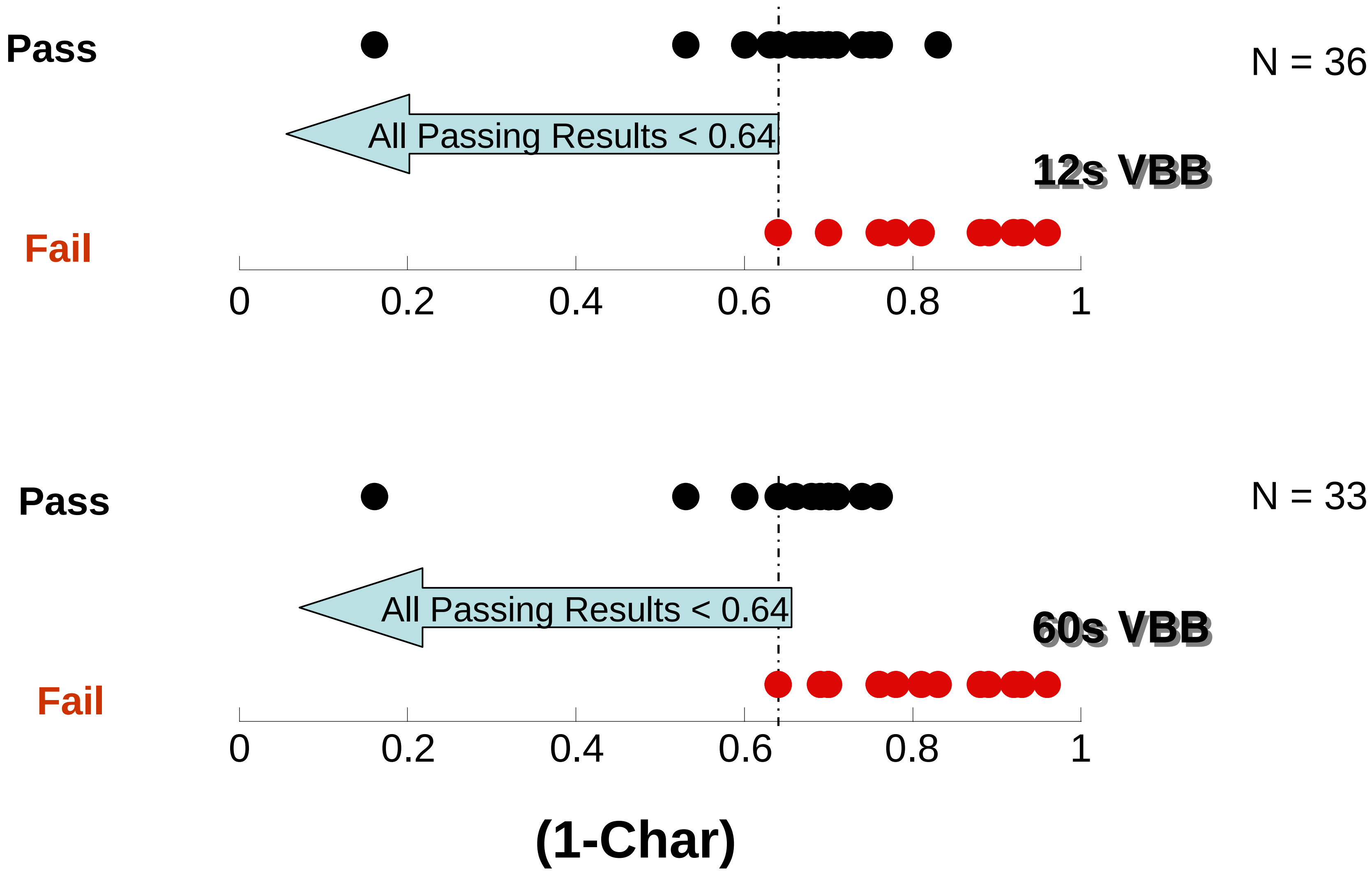


VBB versus 1000/Temperature at Max HRR (T_{max})

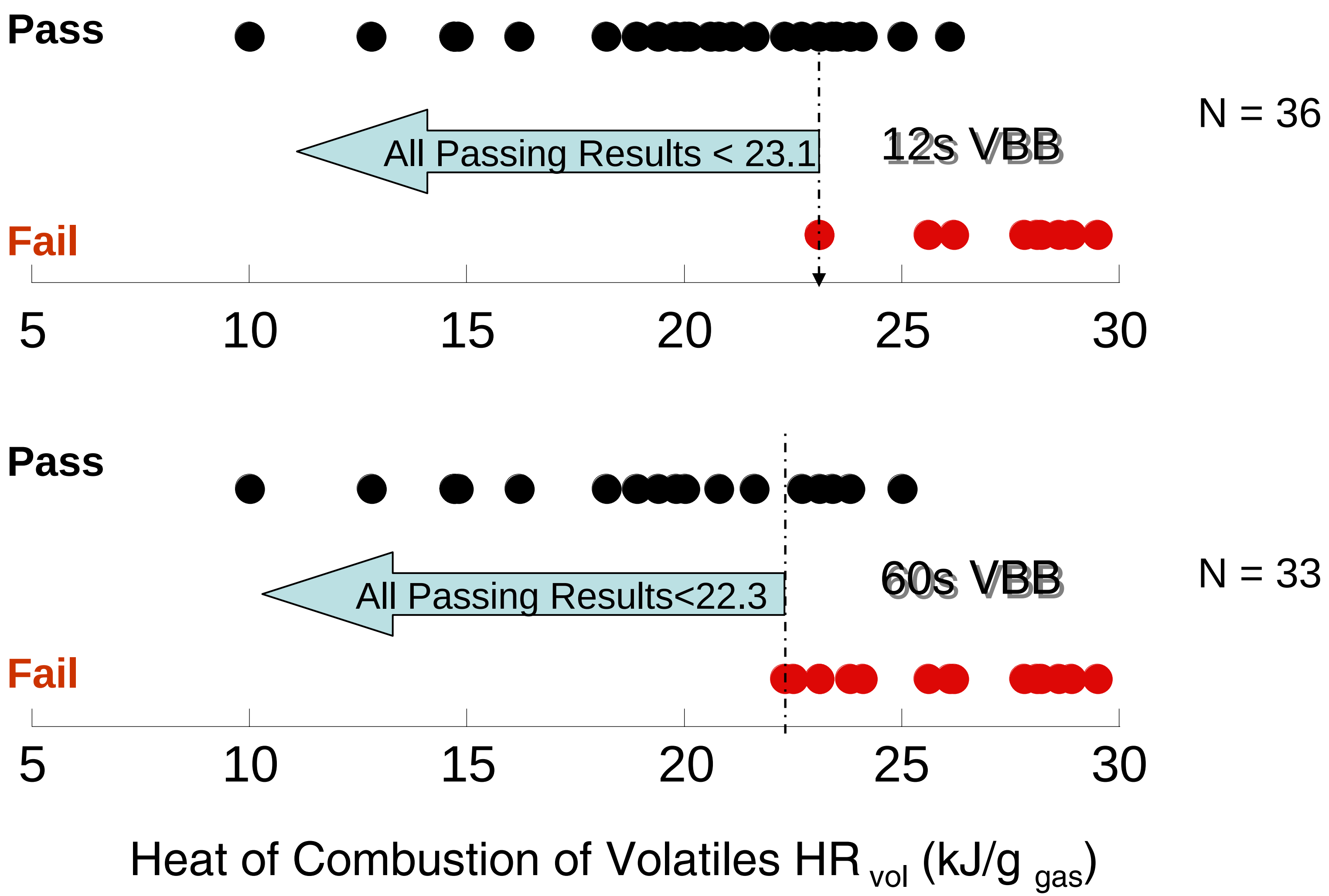


2.11 corresponds to T max= 473°C

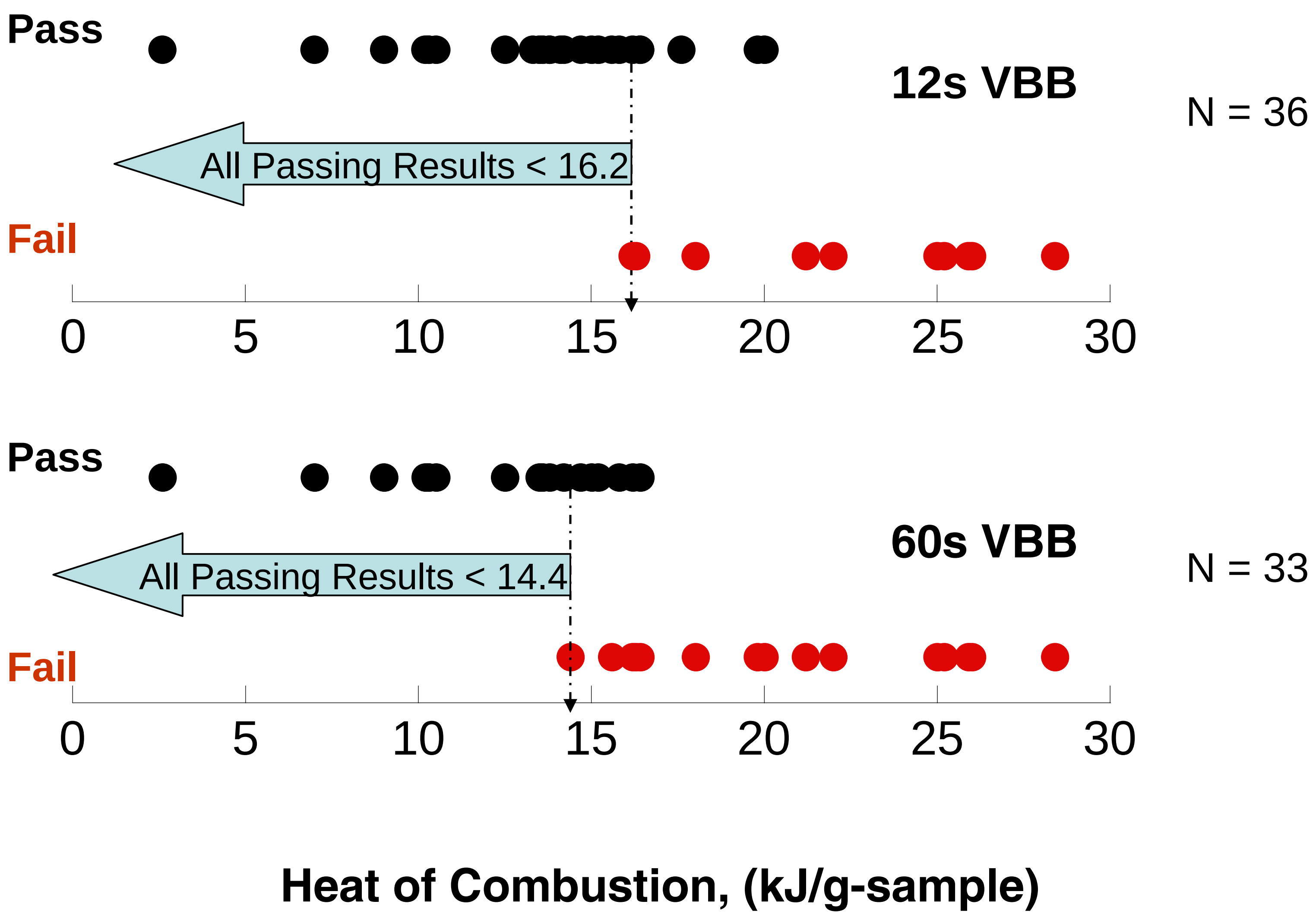
VBB versus Volatile Fraction (1-Char)



VBB versus Heat of Combustion of Volatiles



VBB versus Heat of Combustion



Aircraft Cabin Materials

$\langle\eta_c\rangle$	η_c	pHRR	y	p_B
36	1	8	0	1/10 = 0.10
	9	25	0	
	10	59	0	
	13	13	0	
	15	65	0	
	22	31	0	
	29	55	0	
	50	30	0	
	105	54	0	
	108	92	1	
127	115	61	0	4/10 = 0.40
	120	49	0	
	120	123	1	
	121	60	0	
	121	52	0	
	127	109	1	
	129	64	0	
	136	65	0	
	140	69	1	
	141	110	1	
174	152	90	1	5/10 = 0.50
	153	30	0	
	155	85	1	
	156	45	0	
	159	72	1	
	173	106	1	
	192	36	0	
	196	32	0	
	201	74	1	
	203	65	0	
254	215	213	1	8/10 = 0.80
	215	150	1	
	230	97	1	
	235	258	1	
	248	55	0	
	261	44	0	
	265	188	1	
	276	199	1	
	295	93	1	
	298	211	1	
355	301	232	1	10/10 = 1.0
	316	166	1	
	345	115	1	
	349	207	1	
	359	90		

Etc.

10 points consecutive average

Transform Quantitative Data into Qualitative Data Using Pass/Fail Criterion

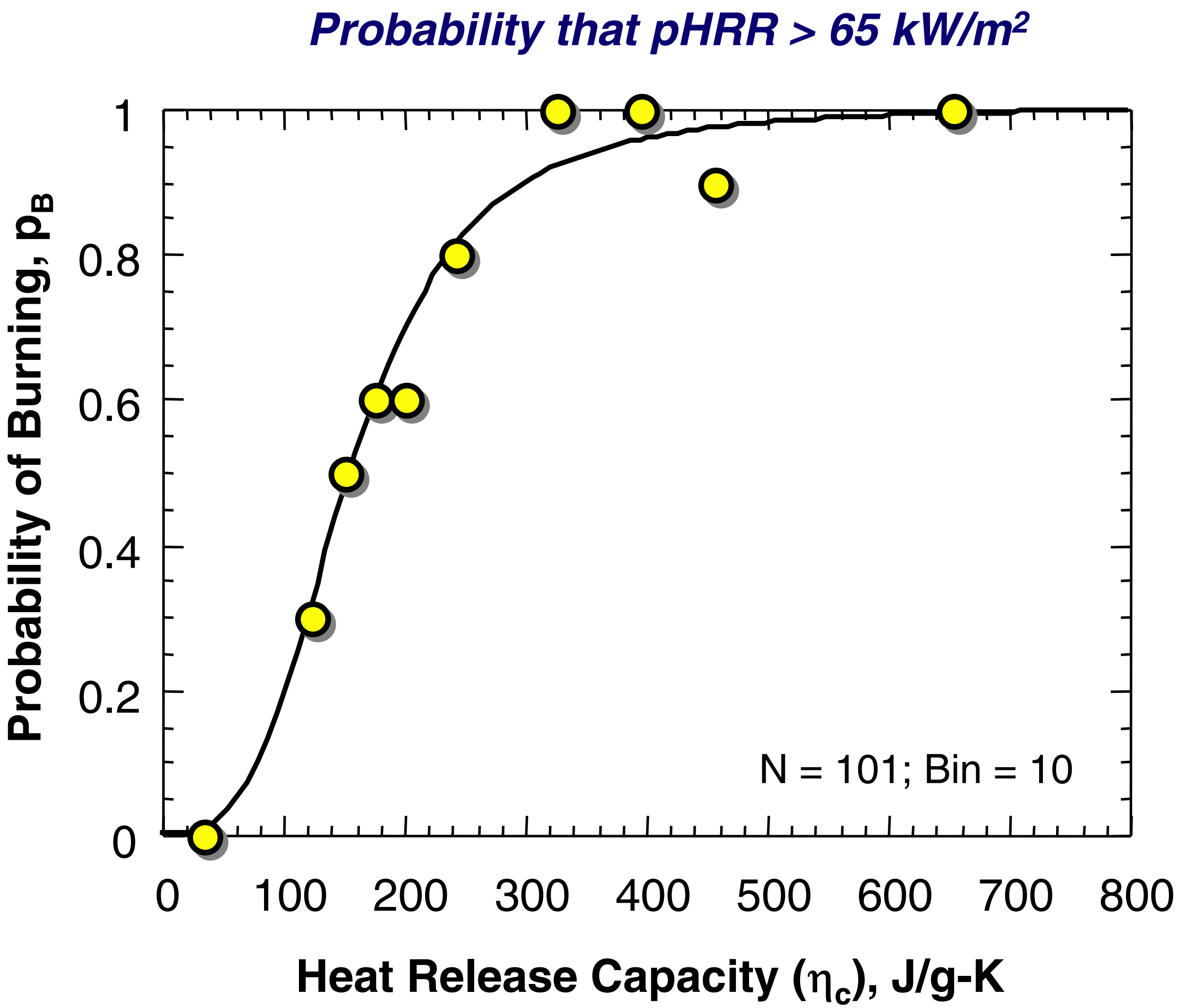
<u>14 CFR 25.853</u>

- HRR $\leq$ 65 kW/m^2 = Pass; **y = 0**
- HRR > 65 kW/m^2 = Fail; **y = 1**

$$p_B = \bar{y} = \frac{\sum_{n} y_i}{n}$$

*A Statistical Model For the Results of Flammability Tests, Richard E. Lyon, Fire & Materials 2009, San Francisco, CA

Aircraft Cabin Materials



14 CFR 25

Probability of Burning

The ***Odds of Self Sustained Burning*** in a flame test are assumed to be related to the thermal combustion properties: x_c = HR, HRC, T_p, $(1-\mu)$ and their critical values, x^*

$$\text{Odds of Burning} = \frac{p_B}{1 - p_B} \equiv \left(\frac{x_c}{x^*}\right)^n$$

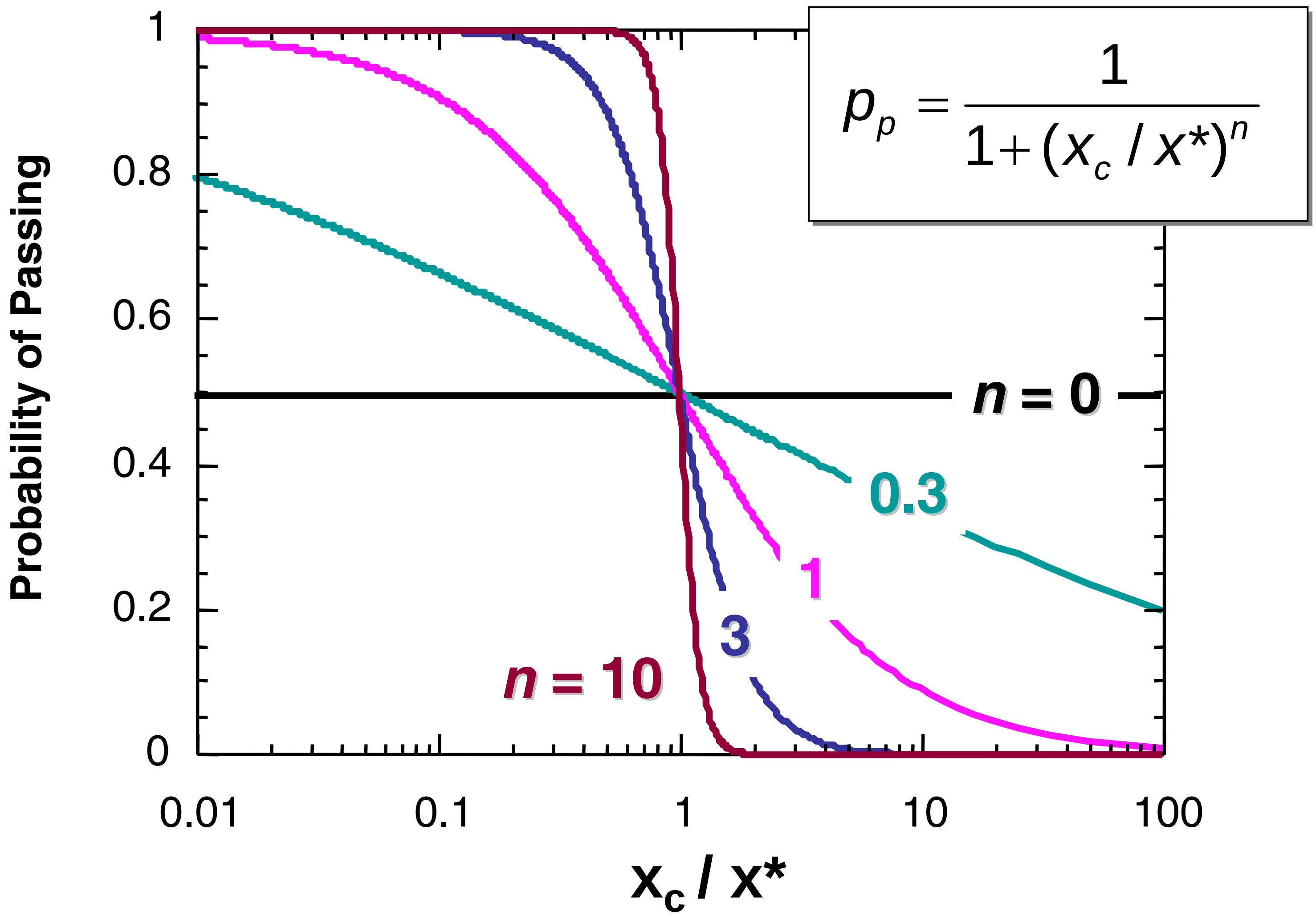
The ***Probability of Burning (Failing)*** is:

$$p_B = \frac{(x_c/x^*)^n}{1 + (x_c/x^*)^n}$$

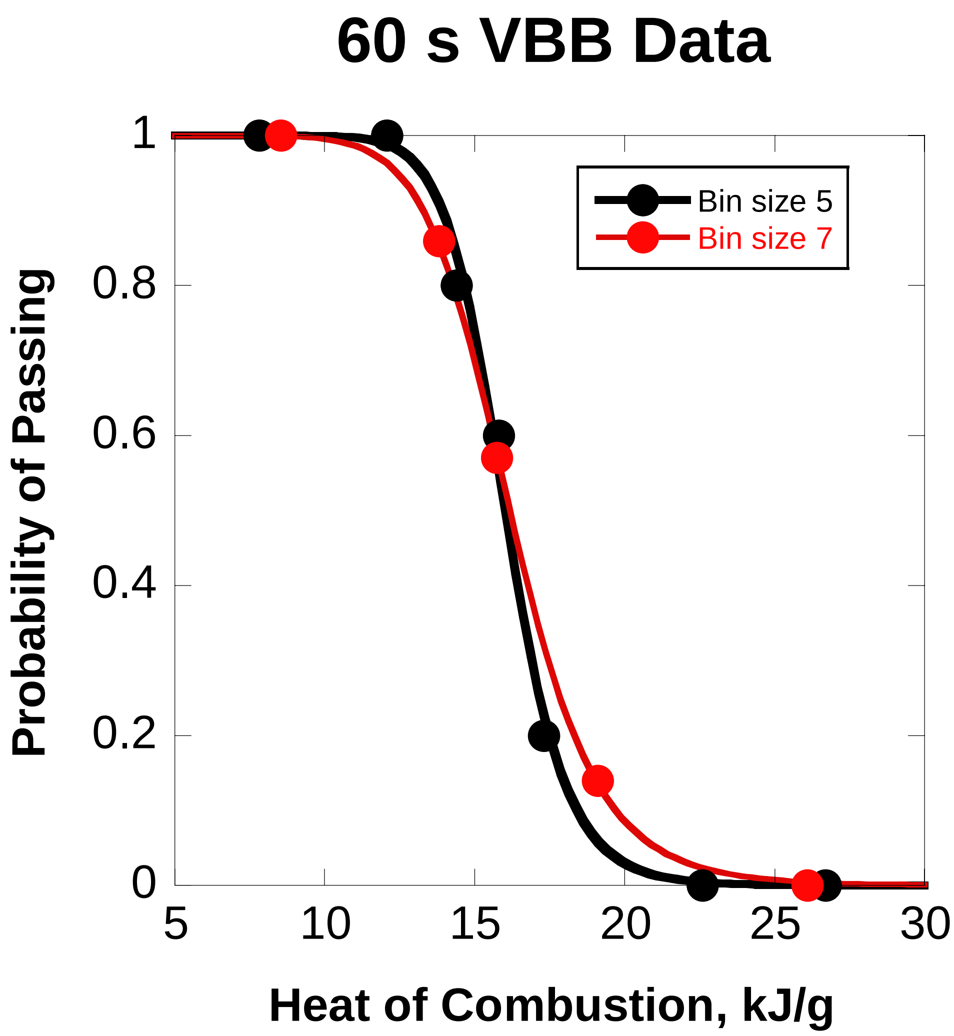
So the ***Probability of Not Burning (Passing)*** is:

$$\boxed{P_p = 1 - P_B = \frac{1}{1 + \left(x_c/x^*\right)^n}}$$

Dependence of P_p on Model Parameters



Bin Size in Statistical Analysis

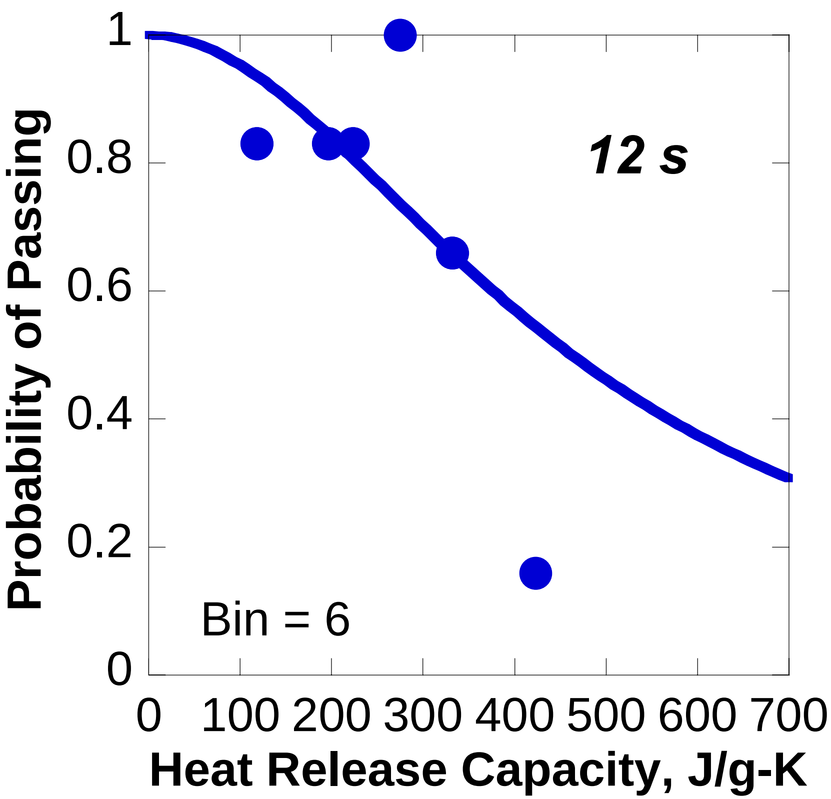


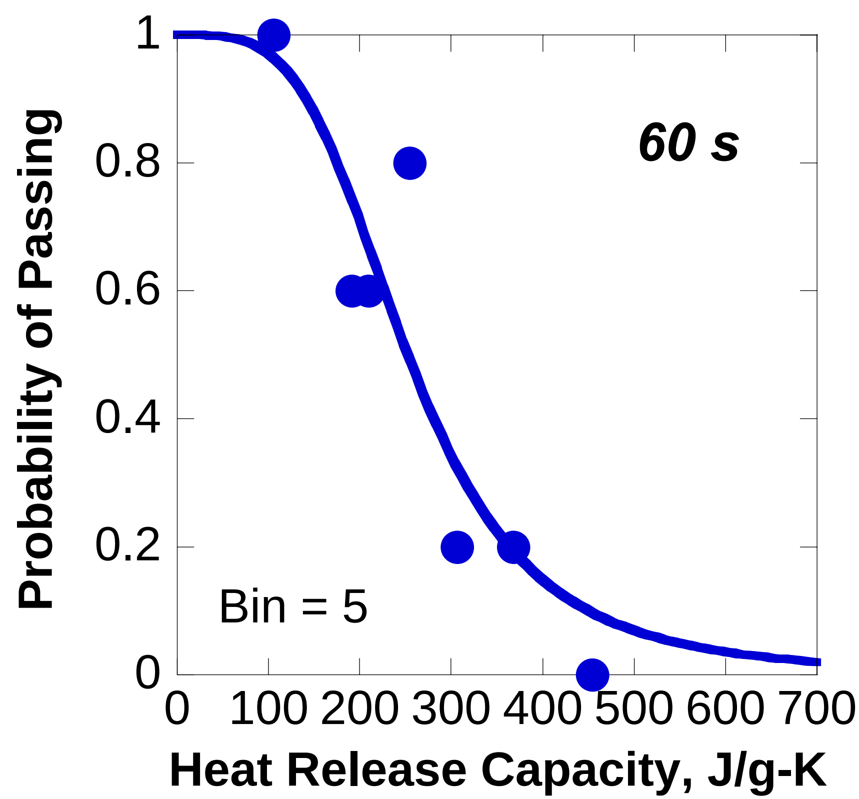
Important:

-representative bin size
(number of elements in the bin)

-sufficient number of points
to fit the curve

Heat Release Capacity as a Predictor Passing VBB Test

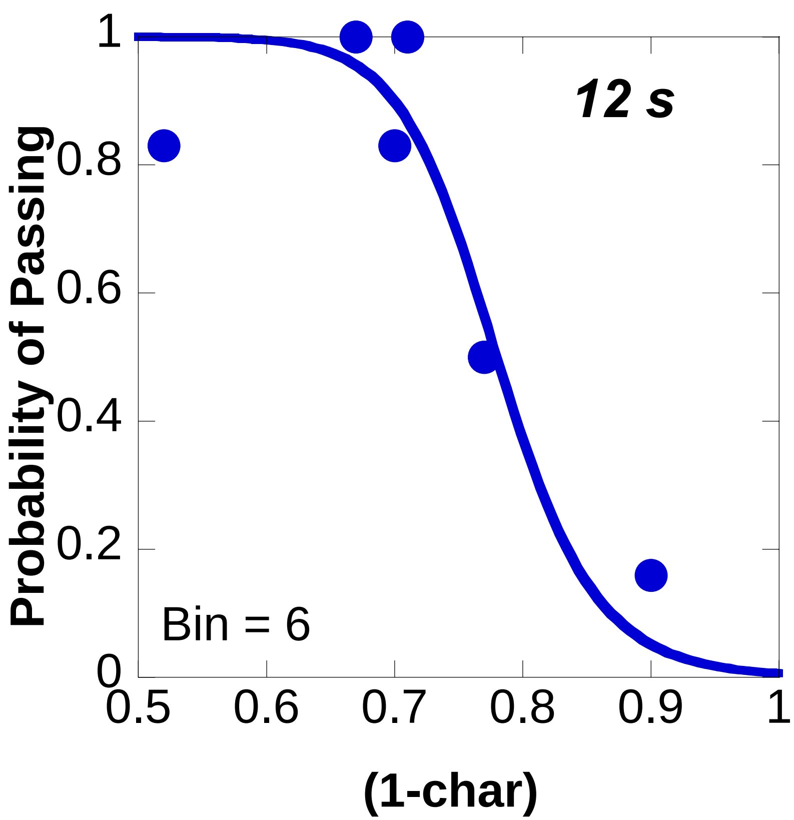




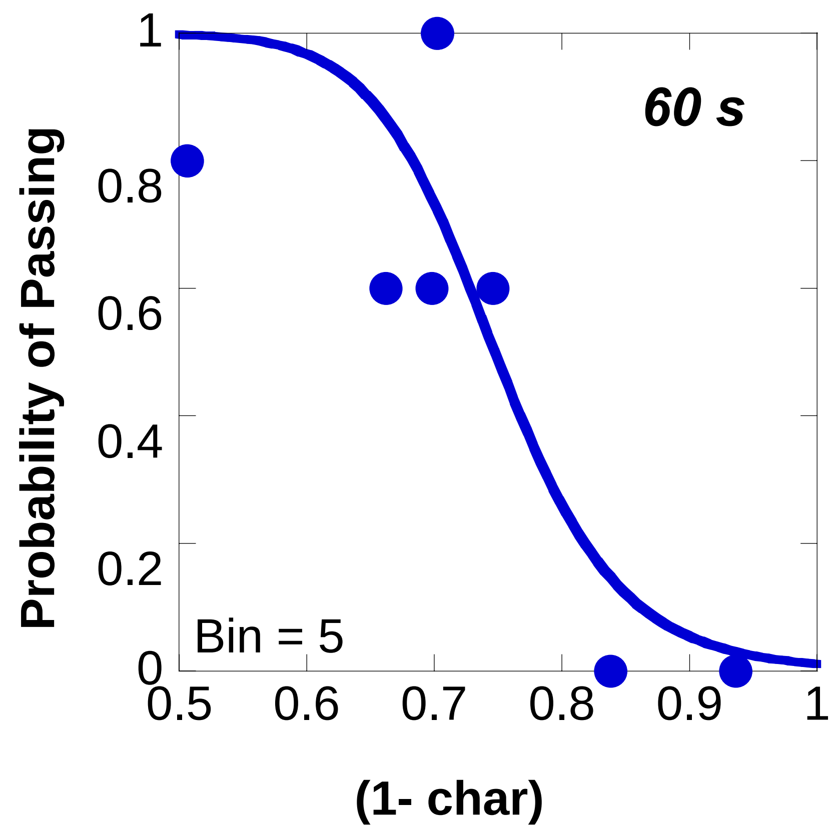
η* = 463 J/g-K
n = 1.9
R = 0.68

η* = 252 J/g-K
n = 3.8
R = 0.90

Volatile fraction (1- Char yield) as a Predictor Passing VBB Test

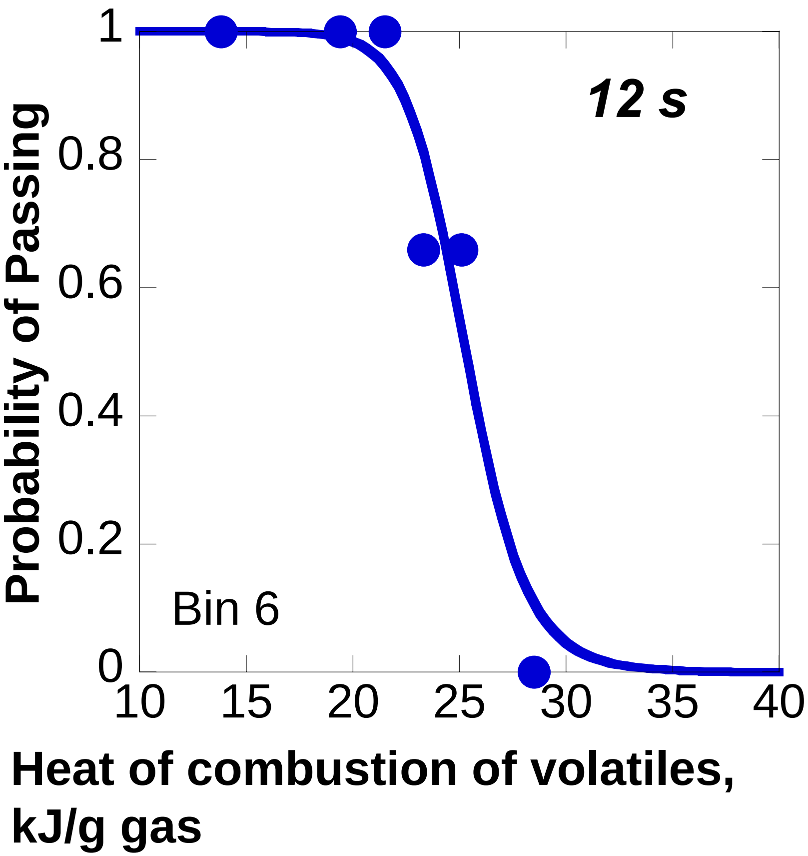


(1-μ) = 0.77*
n = 20
R = 0.93

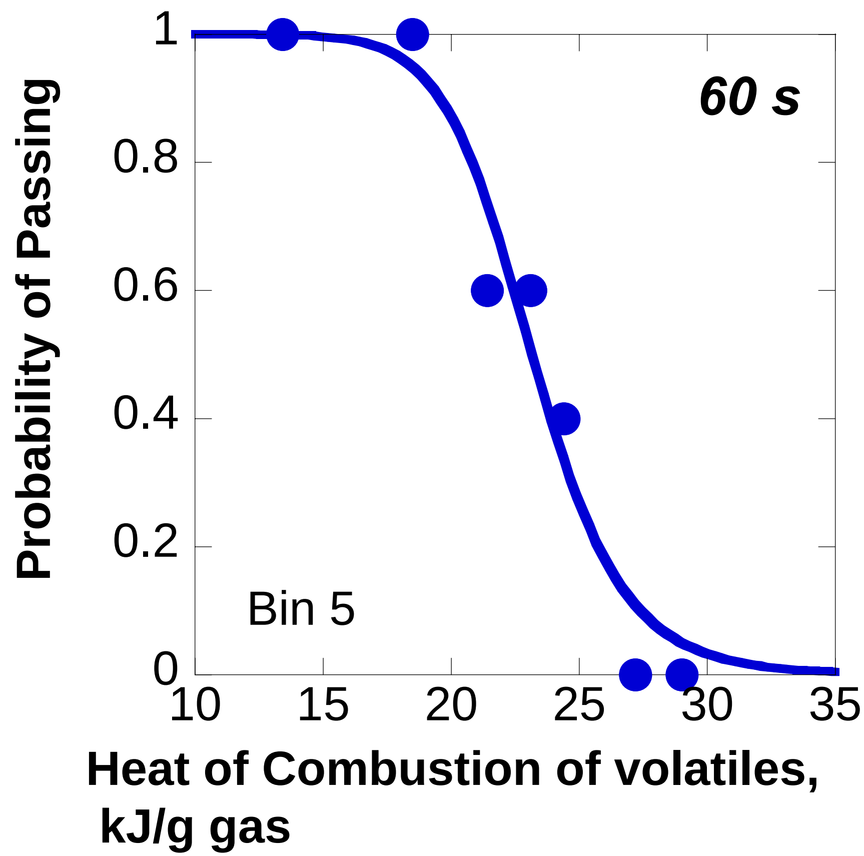


(1-μ) = 0.74*
n = 15.4
R = 0.85

Heat of Combustion of volatiles as a Predictor Passing VBB Test

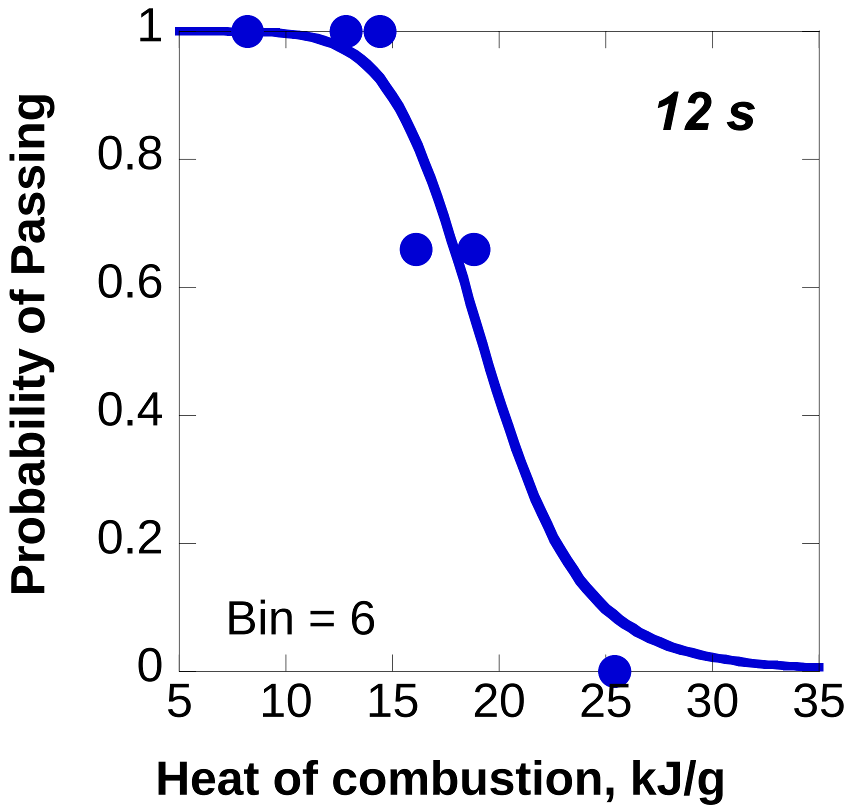


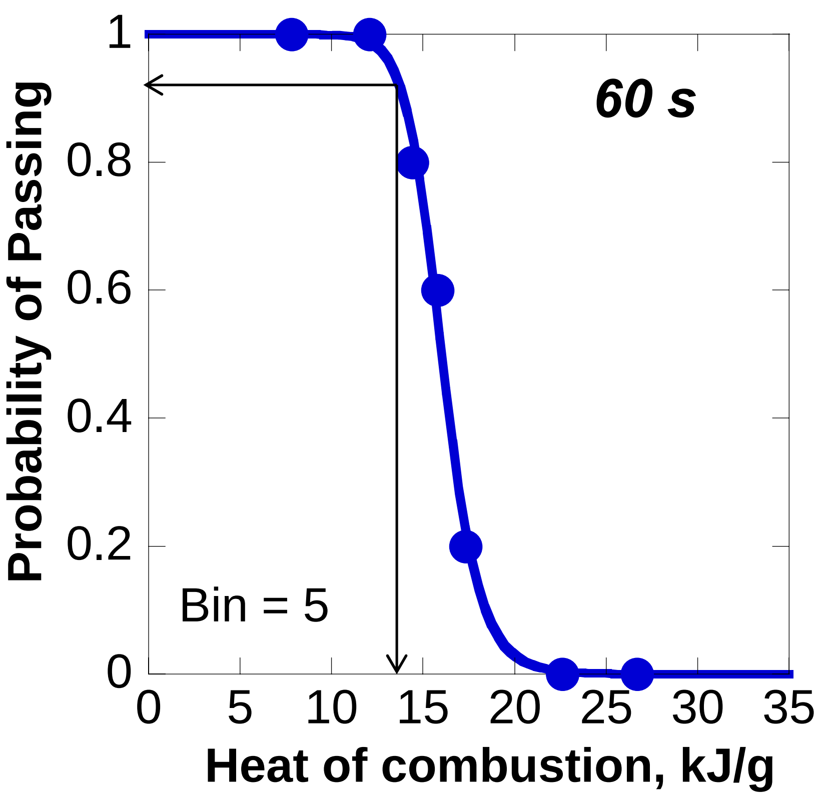
HR_{vol} = 25.3 kJ/g_{gas}*
n = 17.7
R = 0.96



HR = 23.1 kJ/g_{gas}*
n = 13.0
R = 0.98

Heat of combustion as a Predictor Passing VBB Test





HR* = 19.3 kJ/g
n = 8.5
R = 0.96

HR* = 16.0 kJ/g
n = 15.7
R = 0.99

Conclusions

- Microscale Combustion Calorimeter properties that are highly correlated with 12s and 60s VBB results are:
  - Total Heat Release of Sample (HR)
  - Heat of Combustion of Fuel Gases (HR_{vol})
- A limited number of samples were tested in this study.
- Sample size was not controlled in the VBB test and is a source of variability in the P/F results.