Combustion Characteristics of Adhesive Compounds Used in the Construction of Aircraft Cabin Materials

July 2012

DOT/FAA/TC-TN12/12

U.S. Department of Transportation
Federal Aviation Administration

NOTICE

Technical Report Documentation Page

1. Report No. DOT/FAA/TC-TN12/12	2. Government Accession No.	3. Recipient's Catalog No.
4. Title and Subtitle COMBUSTION CHARACTERISTICS OF ADHESIVE COMPOUNDS USED IN THE CONSTRUCTION OF AIRCRAFT CABIN MATERIALS		5. Report Date July 2012
		6. Performing Organization Code
7. Author(s) Natallia Safronava[1] and Richard Lyon[2]		8. Performing Organization Report No.
9. Performing Organization Name and Address [1] Technology & Management International, LLC 1433 Hooper Avenue Toms River, NJ 08753 [2] Federal Aviation Administration William J. Hughes Technical Center Aviation Research Division Fire Safety Branch Atlantic City International Airport, NJ 08405		10. Work Unit No. (TRAIS)
		11. Contract or Grant No.
12. Sponsoring Agency Name and Address U.S. Department of Transportation Federal Aviation Administration Northwest Mountain Region - Transport Airplane Directorate 1601 Lind Avenue, SW Renton, WA 98057		13. Type of Report and Period Covered Technical Note
		14. Sponsoring Agency Code ANM-115

15. Supplementary Notes

The Federal Aviation Administration Aviation Research Division COR was Larry Fitzgerald.

16. Abstract

Adhesives are widely used in the aviation industry to construct lightweight and fatigue resistant aircraft cabin materials. Presently, there is no separate requirement for the flammability of adhesives, potting compounds, and fillers used in construction of cabin materials. The Flammability Standardization Task Group is an aircraft industry group that has proposed testing adhesives using the 12- and 60-second Vertical Bunsen Burner (VBB) Fire Tests requirements for cabin materials in Title 14 Code of Federal Regulations 25.853 to demonstrate that new adhesives have similar flammability to those used in certified cabin materials. Cabin materials pass or fail the VBB tests based on criteria for burn length, after-flame time, and time required for flaming drips to extinguish. The present study was conducted to determine whether the microscale combustion calorimeter (MCC) could also be used to establish similarity of aircraft adhesives, potting compounds, and fillers. To this end, thermal combustion properties were measured by MCC for 37 adhesives, edge fillers, and potting compounds, and the results were compared to VBB ratings to determine whether the former could be used to predict the latter.

17. Key Words Microscale combustion calorimetry, Heat of combustion, Adhesives, Vertical Bunsen Burner	18. Distribution Statement This document is available to the U.S. public through the National Technical Information Service (NTIS), Springfield, Virginia 22161. This document is also available from the Federal Aviation Administration William J. Hughes Technical Center at actlibrary.tc.faa.gov.

19. Security Classif. (of this report) Unclassified	20. Security Classif. (of this page) Unclassified	21. No. of Pages 25	22. Price

Form DOT F 1700.7 (8-72) Reproduction of completed page authorized

TABLE OF CONTENTS

	Page
EXECUTIVE SUMMARY	vii
INTRODUCTION	1
APPROACH	1
MATERIALS	3
METHODS	4
Thermal Combustion Properties	4
The VBB Test	5
Statistical Analysis	5
Qualitative Analysis	5
Quantitative Analysis	5
RESULTS	8
Thermal Combustion Properties	8
The VBB Test Results	10
Statistical Analysis	11
Qualitative Analysis	11
Quantitative Analysis	13
DISCUSSION	15
CONCLUSIONS	17
REFERENCES	17

LIST OF FIGURES

Figure		Page
1	The Probability of Passing a Fire Test p_i for a Sample Having a Fire Response Parameter $x = X/X^*$ for Various Exponents *b* According to Equation 3	3
2	Samples Received From FSTG Members	3
3	Probability Calculation for Bin Sizes of $n = 5$ and $n = 7$ Adhesive Samples	7
4	The *HRR* vs Temperature for Samples 1, 7, 18, and 21	10
5	Data for the 12- and 60-s VBB Tests Classification vs *HRC*	11
6	Data for the 12- and 60-s VBB Tests Classification vs *HR*	11
7	Data for the 12- and 60-s VBB Tests Classification vs HR_{gas}	12
8	Data for the 12- and 60-s VBB Tests Classification vs $1000/T_{\max}$	12
9	Data for the 12- and 60-s VBB Tests vs Volatile Fraction φ	13
10	Plots of p_i vs *HRC* for the 12- and 60-s VBB Tests	14
11	Plots of p_i vs Volatile Fraction φ for the 12- and 60-s VBB Tests	14
12	Plots of p_i vs HR_{gas} for the 12- and 60-s VBB Tests	14
13	Plots for p_i vs *HR* for the 12- and 60-s VBB Tests	15
14	Binary and Continuous Probability Distributions for the 60-s VBB Test Using *HR* as the Explanatory Variable	16

LIST OF TABLES

Table		Page
1	Pass/Fail Probability Distribution	2
2	Acceptance (Passing) Criteria for 14 CFR 25.853 VBB Tests	5
3	Tabular Representation of the Binning Process for the 60-s VBB Test	6
4	The MCC and VBB Test Results	8
5	Qualitative Results From 12- and 60-s VBB Tests	13
6	Values of X^*, b, MD, and Pearson's R for the 12-s VBB Test	15
7	Values of X^*, b, MD, and Pearson's R for the 60-s VBB Test	15

LIST OF SYMBOLS AND ACRONYMS

β	Heating rate
μ	Pyrolysis residue
φ	Volatile fraction
HR	Specific heat of combustion of the sample
HR_{gas}	Specific heat of combustion of the sample gases
HRC	Heat release capacity
HRR	Heat release rate
R	Pearson's correlation coefficient
T_{max}	Pyrolysis temperature
Q_{max}	Maximum value of HRR during experiment
CFR	Title 14 Code of Federal Regulations
FSTG	Flammability Standardization Task Group
MCC	Microscale combustion calorimeter
MD	Mean deviation
VBB	Vertical Bunsen Burner

EXECUTIVE SUMMARY

Adhesives are widely used in commercial aircraft and they play an important role in the aircraft industry because they provide a lightweight, fatigue resistant, and aerodynamically sound method of assembly. Presently, there is no separate requirement for the flammability of adhesives, potting compounds, and fillers used in the construction of cabin materials. This makes substitution or replacement of adhesives for various reasons (e.g., performance, supplier issues, and environmental regulations) costly because all cabin materials and parts must be fabricated and tested with the new adhesive, according to approved Federal Aviation Administration procedures (certificate). The Flammability Standardization Task Group (FSTG) is an aircraft industry working group that is interested in establishing a procedure to determine the similarity between different adhesives with regard to flammability by performing comparative tests of the adhesive separately from the cabin material/part/construction in which it is used and for which it was originally certified. To this end, the FSTG proposed testing adhesives by the 12- and 60-second Vertical Bunsen Burner (VBB) Fire Test requirement for cabin materials in Title 14 Code of Federal Regulations 25.853, a test that adhesives currently do not have to pass as separate components. Cabin materials pass or fail the VBB Test based on criteria for burn length, after-flame time, and the time required for flaming drips to extinguish. The present study was conducted to determine whether the microscale combustion calorimeter (MCC), a quantitative laboratory test for flammability, could be used to establish similarity of adhesives, potting compounds, and fillers used in the construction of aircraft cabin materials. The MCC thermal combustion properties were analyzed to determine whether there is a correlation between the MCC results and the VBB Test ratings and to what extent the former could be used to predict the latter. Probabilistic analyses demonstrated that qualitative (pass/fail) VBB Test results are predicted by quantitative thermal combustion properties of adhesives and edge-fill compounds.

INTRODUCTION

Adhesives are widely used in commercial aircraft and play an important role in the aircraft industry by providing a lightweight, fatigue resistant, and aerodynamically sound method of assembly. Epoxy-based film adhesives are used for metal-to-metal applications and honeycomb bonding, and liquid/paste adhesives are used for a variety of assembly operations including aircraft repair and maintenance. Presently, there is no separate requirement for the flammability of potting compounds or foams used to fill and reinforce the edges of fabricated sections of honeycomb composite panels or the epoxy adhesives used in bonded joints of cabin materials. This makes substitution or replacement of adhesives and edge-fill compounds (adhesive compounds) costly for reasons such as performance, supplier issues, and environmental regulations because all cabin materials and parts must be fabricated and tested with the new adhesive, according to approved Federal Aviation Administration procedures (certificate). The Flammability Standardization Task Group (FSTG) is an aircraft industry working group interested in methods of compliance based on establishing the similarity of different adhesive compounds. With regard to flammability, comparative testing of the adhesive compound was performed separately from the cabin material/part/construction in which it is used and for which it was originally certified [1]. To this end, the FSTG has proposed testing adhesive compounds using the 12- and 60-second Vertical Bunsen Burner (VBB) Fire Tests requirements for cabin materials in Title 14 Code of Federal Regulations (CFR) 25.853 [1]—tests that adhesive compounds currently do not have to pass as separate components. Cabin materials pass or fail the VBB Test based on criteria for burn length, after-flame time, and the time required for flaming drips to extinguish. The present study was conducted to determine whether the microscale combustion calorimeter (MCC), a quantitative laboratory test for flammability [2 and 3], supports the FSTG position that similarity of adhesive compounds can be established using VBB Tests. To this end, thermal combustion properties (i.e., heat release capacity, specific heat of combustion of the sample (HR), specific heat of combustion of the sample gases (HR_{gas}), pyrolysis residue (μ), and pyrolysis temperature ($T_{\max}$) were measured for 36 adhesives, potting compounds, and edge fillers used in the aircraft industry and the results correlated with the VBB Test results. Probabilistic analyses demonstrated that qualitative (pass/fail) VBB Test results are predicted by quantitative thermal combustion properties of adhesives and edge-fill compounds.

APPROACH

The objective of this study was to determine whether the pass/fail VBB Test ratings of adhesives can be predicted by the quantitative thermal combustion properties measured by the MCC. The VBB Test produces a qualitative result, Y_i, based on prescribed pass/fail criteria. Y_i results are presumed to be a function of the independent thermal combustion properties X_i. The response variable, Y_i, can be one of two possible outcomes, pass or fail, so it may be treated as a Bernoulli random variable with probability distribution. Table 1 shows the pass/fail probability distribution.

Table 1. Pass/Fail Probability Distribution

Test Result	Y_i	Probability
Pass	1	$P(Y_i = 1) \mid (X_i) \equiv p_i$
Fail	0	$P(Y_i = 0) \mid (X_i) \equiv 1\text{-}p_i$

$P(Y_i = 1 \mid X_i)$ is the probability that $Y_i = 1$ given X_i, and $P(Y_i = 0 \mid X_i)$ is the probability that $Y_i = 0$ given X_i. If N_P and N_F are the number of passing and failing results, respectively, in $N = N_P + N_F$ trials (tests) at a particular X_i, then $p_i = N_p/N$, $1\text{-}p_i = N_F/N$ and $N_P/N_F = p_i/(1\text{-}p_i)$ are the odds of passing the test [3 and 4]. An analog of the logistic model widely used in medical and social sciences [4 and 5] has been proposed for fire behavior [6] based on the assumption

$$\frac{N_F}{N_P} = \frac{1-p_i}{p_i} \propto X_i^n \tag{1}$$

Equation 1 becomes an equality by defining a parameter X^* with the units of X, such that $X_i = X^*$ when $N_F = N_P$ or when $p_i = 1/2$

$$\frac{p_i}{1-p_i} = \left(\frac{X^*}{X_i}\right)^b \tag{2}$$

The cumulative probability distribution for a single explanatory variable (thermal combustion property) X_i is obtained from equation 2

$$p_i = \frac{(X^*/X_i)^b}{1+(X^*/X_i)^b} = \frac{1}{1+(X_i/X^*)^b} \tag{3}$$

Equation 3 is plotted in figure 1 for b = 0, 1, 3, 10, and 30 over the range $x = 0$ to $x = 2$ of the normalized (dimensionless) combustion property $x = X/X^*$.

The adjustable parameter b in equation 3 indicates how wide the transition region is between the pass and fail results with respect to the thermal combustion property. A small value of b corresponds to a broad range of the thermal combustion property over which the transition from passing to failing results in the VBB Test is observed. Conversely, a large value of b corresponds to an abrupt transition between passing and failing results with respect to the thermal combustion property. Parameter X^* is the value of the independent/explanatory variable at which the probability of passing the test is 50%, i.e., when $p(X) = p(X^*) = 1/2$.

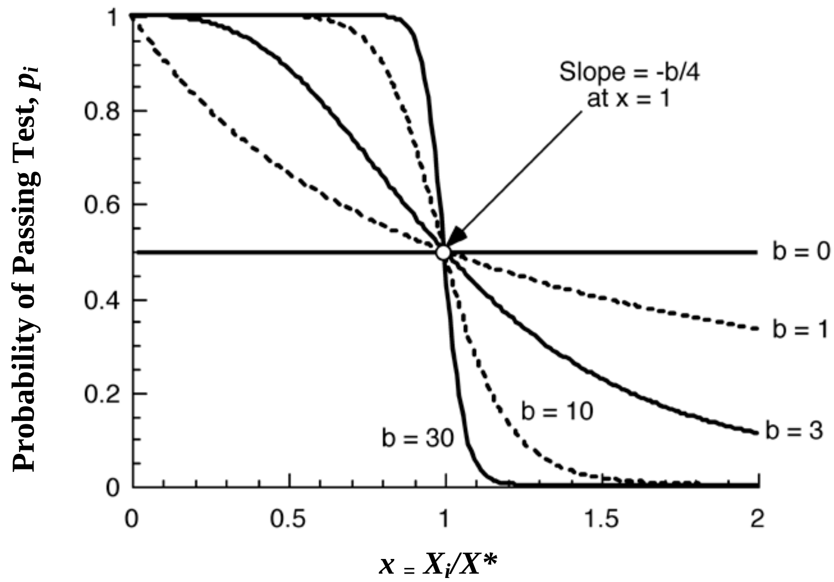

Figure 1. The Probability of Passing a Fire Test p_i for a Sample Having a Fire Response Parameter $x = X/X^*$ for Various Exponents b According to Equation 3

MATERIALS

A total of 48 adhesives, potting compounds, and fillers used by the aircraft industry were received from FSTG members, as shown in figure 2. A few identical samples were received from different suppliers (two of them had questionable VBB Test ratings). Of these 48 samples, 36 had pass/fail ratings for the 12-s second VBB Test (12-s VBB Test) and 33 had pass/fail ratings for the 60-second VBB Test (60-s VBB Test). Details of burn length, after-flame time, and extinction time for flaming drips were not provided with the samples.

Figure 2. Samples Received From FSTG Members

METHODS

THERMAL COMBUSTION PROPERTIES.

A standard method was used [7] in which the sample was heated at a constant rate of temperature rise β = 1 K/s from ambient temperature to 900°C. The pyrolysis gases generated during the heating program were purged from the sample chamber with nitrogen, mixed with excess oxygen, and combusted at 900°C for 10 seconds. The rate of heat released by combustion of the pyrolysis gases was calculated from the flow rate of the gas stream and the oxygen consumed and divided by the initial sample mass to obtain the specific heat release rate (*HRR*) in units of Watts per gram of sample. The maximum value of *HRR* during the test was $Q'_{\max}$. Five thermal combustion properties were obtained during the test that are independent of sample mass and heating rate.

- Heat Release Capacity (*HRC*): The *HRC* is a derived quantity that represents the maximum capability (capacity) of a material to release combustion heat per degree of temperature rise during pyrolysis.

$$HRC = \frac{1}{\beta}\sum_{1}^{n} Q'_{\max,i} \tag{4}$$

  For a single component n = 1 and $HRC = Q'_{\max}/\beta$. For a multicomponent material exhibiting n separate from $Q'_{\max}$, the *HRC* is calculated as the sum of the individual $Q'_{\max}$ after deconvolution by peak fitting to remove overlap as per equation 4. The units of *HRC* are Joules per gram per degree Kelvin (J/g-K).

- Pyrolysis Temperature ($T_{\max}$): The temperature at which $Q'_{\max}$ occurs during the MCC test.

- Pyrolysis Residue (μ): The fraction of the original mass remaining at 900°C after pyrolysis in nitrogen during the MCC test.

- Specific Heat of Combustion of the Sample (*HR*): The integrated *HRR*, i.e., the area under the curve of *HRR* versus time, in units of Joules per initial mass of sample.

- Specific Heat of Combustion of the Sample Gases (HR_{gas}): The heat of combustion per unit mass of volatiles generated during the MCC heating program.

$$HR_{gas} = \frac{HR}{1-\mu} \tag{5}$$

THE VBB TEST.

The test method used to determine the ignition resistance of cabin materials was the 12- and 60-s VBB Test, respectively, as specified in 14 CFR 25.853 and 25.855 [8 and 9]. In the VBB Test, cabin materials were suspended vertically in a draft-free cabinet and ignited at the lower edge for 12 or 60 seconds, depending on the intended use of the cabin material, using a 38-mm (1.5-inch)-long flame from a Bunsen burner. The burner may be of the Bunsen or Tirrill type having a 10-mm outer diameter and the flame may be either a diffusion or pre-mixed flame of methane or natural gas. The outcome of this test was a pass or fail rating based on criteria for flame extinguish time, burn length, and drip extension time, as shown in table 2.

Table 2. Acceptance (Passing) Criteria for 14 CFR 25.853 VBB Tests

Test	Flame Extinguish Time (Seconds)	Burn Length (Inches)	Drip Extinguish Time (Seconds)
60-s VBB	≤15	≤6	≤3
12-s VBB	≤15	≤8	≤5

According to the standard, the sample should be a prismatic bar with the following minimum dimensions, unless the actual size used in the aircraft is smaller: 75-mm (3-inches) wide, 305-mm (12-inches) long, and 6-mm (1/4-inch) thick.

STATISTICAL ANALYSIS.

Qualitative Analysis. A qualitative analysis was performed on the 12- and 60-s VBB Test pass/fail results using each of the five thermal combustion properties as an explanatory variable. The objective was to determine if there was a distinct threshold value for each thermal combustion property that would guarantee a passing classification in the 14 CFR VBB Test for flammability. The analysis was conducted by ranking the VBB Test data by each thermal combustion property, recording the value of the thermal combustion property, and determining the upper limit for passing results, below which all the adhesives pass the VBB flammability tests. This approach is warranted if all possible adhesives are tested and analyzed to determine the value of the critical property. The critical value is specific to the data set, which, in this case, is 36 for the 12-s VBB Test and 33 for the 60-s VBB Test.

Quantitative Analysis. The construction of cabin materials for a certified airplane extends over many years as each new airplane is built. Over this time period, new adhesives are introduced and old ones are replaced as circumstances (e.g., supplier issues and environmental regulations) require. A more flexible analysis was attempted that accounted for the uncertainty of the pass/fail results over the range of the explanatory variable (thermal combustion property) for which both passing and failing VBB Test classifications were observed. Equation 3 was fit to the VBB Test results by nonlinear least square regression using the thermal combustion properties as explanatory variables. The VBB Test pass/fail results were coded as binary outcomes (pass = 1 and fail = 0) and b and X^* were treated as adjustable parameters. The analysis was conducted using the commercial spreadsheet program, KaleidaGraph (Synergy

Software), running on a personal computer. Identical results for b and X^* were obtained with other computational programs, Excel® (Microsoft®) and MATLAB® (The MathWorks, Inc.), on personal computers. To estimate the error of the cumulative probability distribution $p(X)$ obtained, the binary VBB Test results for the N = 33 or N = 36 adhesives were ranked by the predictor variables (thermal combustion properties) in ascending order and divided into $\sqrt{N} \approx 7$ groups/bins [3 and 4] having n = 5 samples in each group. The selection of bin size is important to provide a statistically valid sample for computing p_i while affording sufficient resolution to reveal trends in the test results. Table 3 demonstrates the bin-averaging procedure for the 60s VBB Test for which Y_i = 0 or 1, $p_i = \sum Y_i/n$, $X_i = HR_i$ and $X_{avg} = \sum X_i/n = \sum HR_i/n = HR_{avg}$. The results of this exercise for bin sizes n = 5 and n = 7 are shown in table 3 and compared in figure 3. Based on the closeness of these results, a bin size of n = 5 was chosen for quantitative analysis of the VBB Test versus the MCC data.

Table 3. Tabular Representation of the Binning Process for the 60-s VBB Test

HR_{avg} (J/g-K)	HR_i (J/g-K)	P/F	Y_i	$Y_{avg} = P_i$
7.8	2.6	*P*	1	1
	7	*P*	1	
	9	*P*	1	
	10.2	*P*	1	
	10.3	*P*	1	
12.1	10.3	*P*	1	1
	10.5	*P*	1	
	12.5	*P*	1	
	13.5	*P*	1	
	13.6	*P*	1	
14.4	13.8	*P*	1	0.8
	14.2	*P*	1	
	14.4	*F*	0	
	14.7	*P*	1	
	15.0	*P*	1	
15.8	15.2	*P*	1	0.6
	15.6	*F*	0	
	15.8	*P*	1	
	16.2	*P*	1	
	16.2	*F*	0	

Table 3. Tabular Representation of the Binning Process for the 60-s VBB Test (Continued)

HR_{avg} (J/g-K)	HR_i (J/g-K)	*P*/*F*	Y_i	$Y_{avg} = P_i$
17.3	16.3	*F*	0	0.2
	16.4	*P*	1	
	16.4	*F*	0	
	18.0	*F*	0	
	19.8	*F*	0	
22.6	20.0	*F*	0	0
	21.2	*F*	0	
	22.0	*F*	0	
	25.0	*F*	0	
	25.2	*F*	0	
26.7	25.9	*F*	0	0
	26.0	*F*	0	
	28.4	*F*	0	

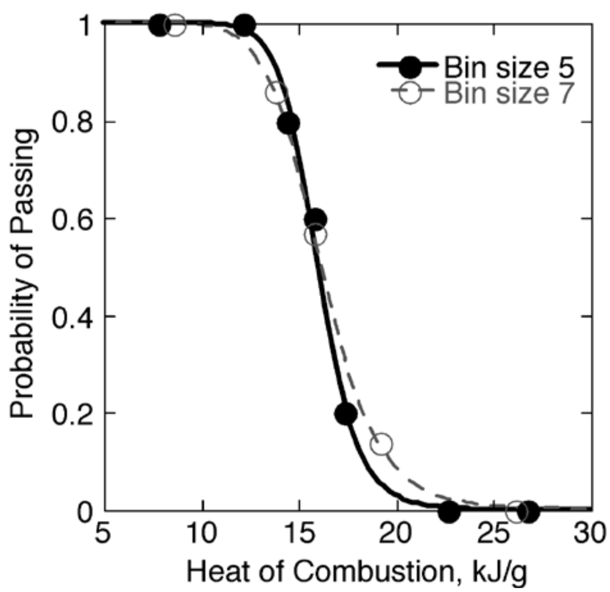

Figure 3. Probability Calculation for Bin Sizes of $n = 5$ and $n = 7$ Adhesive Samples

The appropriateness of the probability function for the VBB Test data was determined for each of the candidate explanatory variables (thermal combustion properties) by inspection of grouped (binned) experimental data compared to the model predictions and by calculating the correlation coefficient between the measured (bin-average) probability p_{meas} and the calculated probability, p_{calc}, using equation 3 with b and X^* obtained from the fit of the binary data. The correlation was estimated using Pearson's R.

$$R = \sqrt{1 - \frac{\sum_B (p_{meas} - p_{calc})^2}{\sum_B (p_{meas} - \bar{p}_{meas})^2}} \tag{6}$$

The mean deviation (MD) of predicted and grouped probabilities was also computed for each of the explanatory variables.

$$MD = \frac{\sum_B |p_{calc} - p_{meas}| / B}{\sum_B p_{meas} / B} = \frac{\sum_B |p_{calc} - p_{meas}|}{\sum_B p_{meas}} \tag{7}$$

RESULTS

THERMAL COMBUSTION PROPERTIES.

Average values for each thermal combustion property were calculated from triplicate MCC experiments and are shown in table 4 along with the VBB Test ratings provided by the FSTG members. Generic names for materials and applications were used to identify the samples. Figure 4 shows *HRR* versus temperature data for materials 1, 7, 18, and 21, respectively, from which the thermal combustion properties for these materials are shown with the complex *HRR* histories typical of multicomponent adhesives, potting compounds, and fillers.

Table 4. The MCC and VBB Test Results

Sample No.	Material	12-s VBB Test Result	60-s VBB Test Result	T_{max} (°C)	*HR* (kJ/g)	HR_{gas} (kJ/g-gas)	*HRC* (J/g-K)	μ (%)
1	Epoxy adhesive	*P*	*F*	345	19.8	26.1	335	24
2	Epoxy adhesive	*P*	*P*	336	12.5	18.9	235	34
3	Epoxy core fill	*P*		375	17.6	23.5	220	25
4	Epoxy core fill	*P*		340	13.3	21.1	285	37
5	Epoxy edge fill	*P*		311	13.8	20.6	200	33
6	Epoxy adhesive	*P*	*F*	345	16.4	23.8	245	31
7	Adhesive	*P*	*P*	337	14.2	19.8	490	29
8	5-minute epoxy	*P*	*P*	348	10.3	14.7	335	30
9	10-minute epoxy	*P*	*P*	347	10.5	14.8	263	29
10	Filling compound		*F*	373	14.4	22.5	201	36

Table 4. The MCC and VBB Test Results (Continued)

Sample No.	Material	12-s VBB Test Result	60-s VBB Test Result	T_{max} (°C)	*HR* (kJ/g)	HR_{gas} (kJ/g-gas)	*HRC* (J/g-K)	μ (%)
11	Filling compound	*P*	*P*	380	15	25	206	40
12	Potting compound	*P*	*P*	337	14.7	21.6	167	32
13	Phenolic resin	*P*	*P*	564	2.6	16.2	24	84
14	Epoxy	*F*	*F*	398	25.2	28.6	401	12
15	Epoxy	*F*	*F*	473	28.4	29.5	457	4
16	Liquid shim	*F*	*F*	412	16.3	25.6	206	36
17	Epoxy	*P*	*P*	356	10.3	14.7	279	30
18	Epoxy	*P*	*P*	346	13.5	18.2	249	26
19	Low-density filler	*P*	*P*	375	9	12.8	135	30
20	Low-density filler	*P*	*P*	364	16.2	23.1	205	30
21	Low-density filler	*F*	*F*	313	16.2	23.1	181	30
22	Adhesive	*F*	*F*	378	25.9	27.8	425	7
23	Adhesive	*F*	*F*	379	26	28.2	370	8
24	Adhesive	*P*	*P*	400	13.6	22.7	195	40
25	Adhesive	*P*		335	14.1	20.1	365	30
26	Edge filling	*P*	*P*	445	16.4	23.4	185	30
27	Panel adhesive	*P*	*F*	342	20	24.1	280	17
28	Adhesive	*P*	*P*	327	13.8	20	295	31
29	Edge filling	*P*	*P*	382	7	10	80	30
30	Edge filling	*P*	*P*	300	15.8	20.8	228	24
31	Panel insert	*F*	*F*	390	25	28.1	395	11
32	Panel and insert adhesive	*F*	*F*	394	22	28.9	200	24
33	Fill product	*F*	*F*	311	18	23.1	310	22
34	Composite insert	*F*	*F*	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*	353	10.2	19.4	121	47

P = Pass
F = Fail

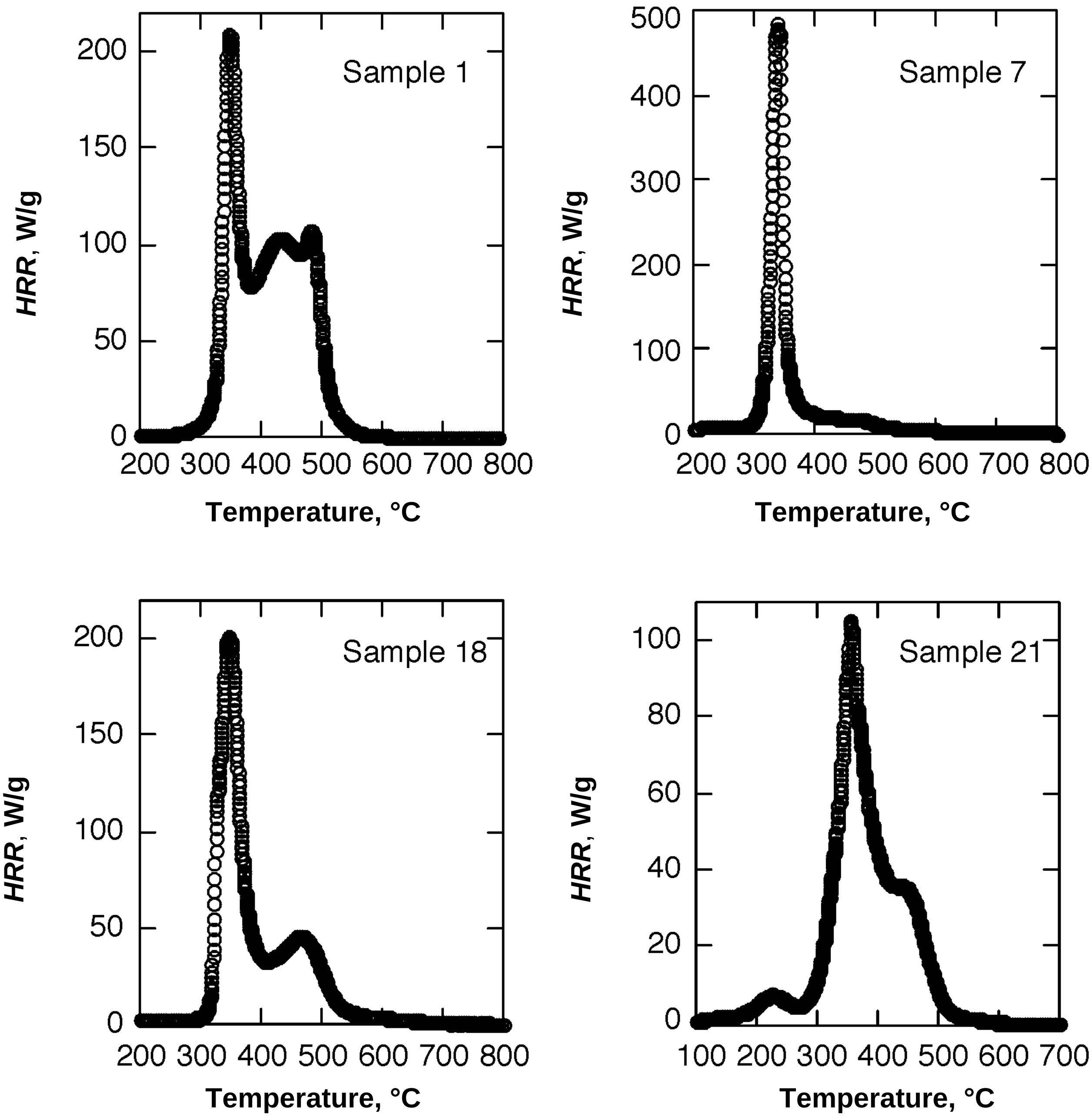

Figure 4. The *HRR* vs Temperature for Samples 1, 7, 18, and 21

THE VBB TEST RESULTS.

Pass and fail results for the 12-s VBB Tests of 36 adhesives and the 60-s VBB Tests of 33 adhesives are listed in table 4 with the thermal combustion properties of each material.

STATISTICAL ANALYSIS.

Qualitative Analysis. The qualitative pass/fail results in table 4 are plotted versus the value of each thermal property—*HRC*, *HR*, HR_{gas}, $1000/T_{\max}$ and the volatile fraction $\varphi = (1-\mu)$ for both the 12- and 60-s VBB Tests in figures 5 through 9. Passing results from the VBB Test are shown as filled circles, and failing results are shown as open circles. The threshold for failing the VBB Test is shown as a dotted vertical line located at the lowest value of the thermal combustion property for which a failing result was recorded. The intercept of the dotted vertical line with the *x* axis is the critical/threshold value $X_{critical}$, below which none of the samples failed the VBB Test in this group. The total number of specimens in the 12-s VBB Test plots of figures 5 through 9 is $N = 36$, while $N = 33$ for the 60-s VBB Test data in these figures. The values of $X_{critical}$ for the thermal combustion properties for the 12- and 60-s VBB Tests are listed in table 5.

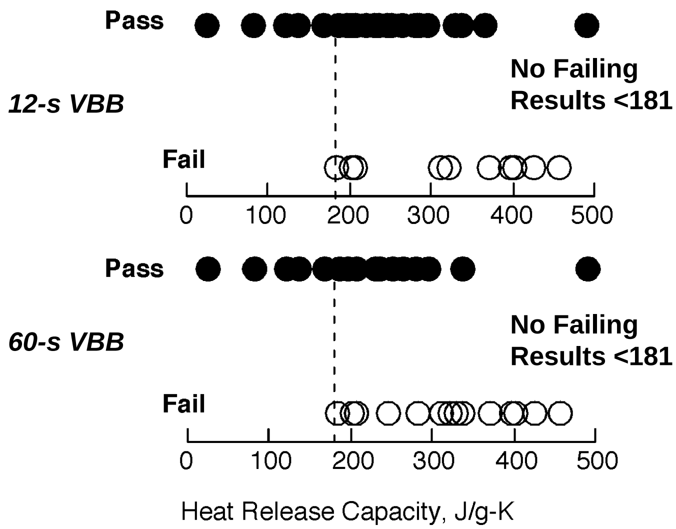

Figure 5. Data for the 12- and 60-s VBB Tests Classification vs *HRC*

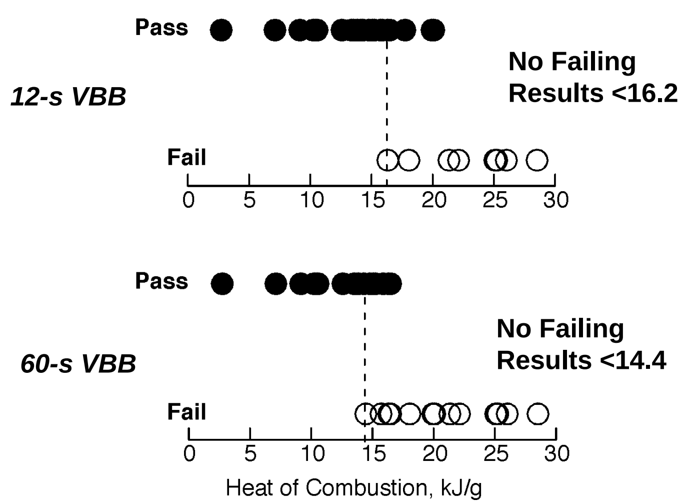

Figure 6. Data for the 12- and 60-s VBB Tests Classification vs *HR*

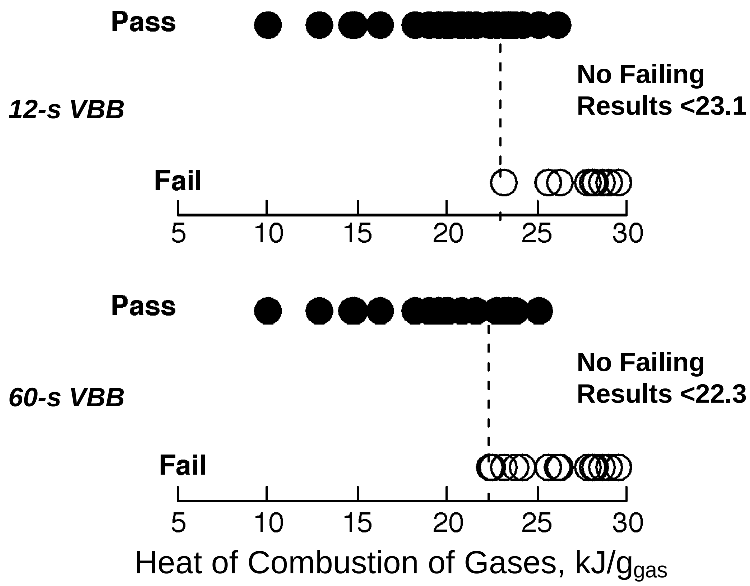

Figure 7. Data for the 12- and 60-s VBB Tests Classification vs HR_{gas}

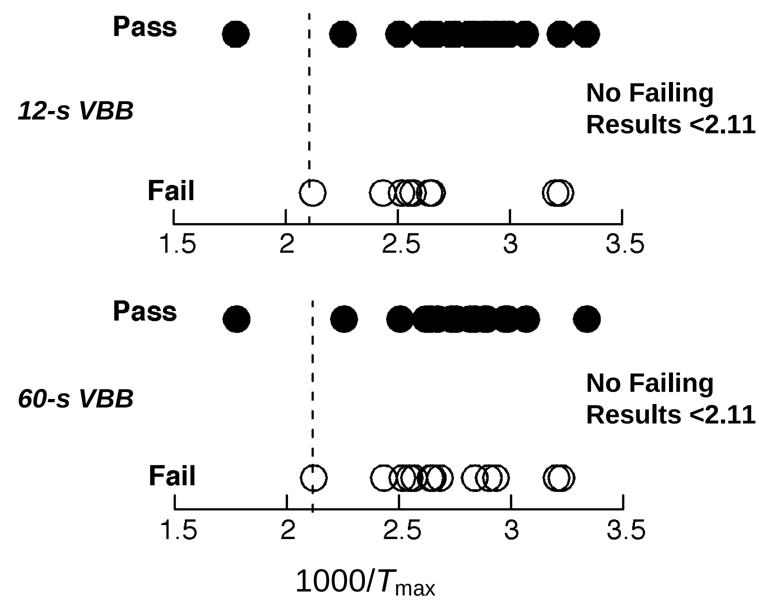

Figure 8. Data for the 12- and 60-s VBB Tests Classification vs $1000/T_{max}$

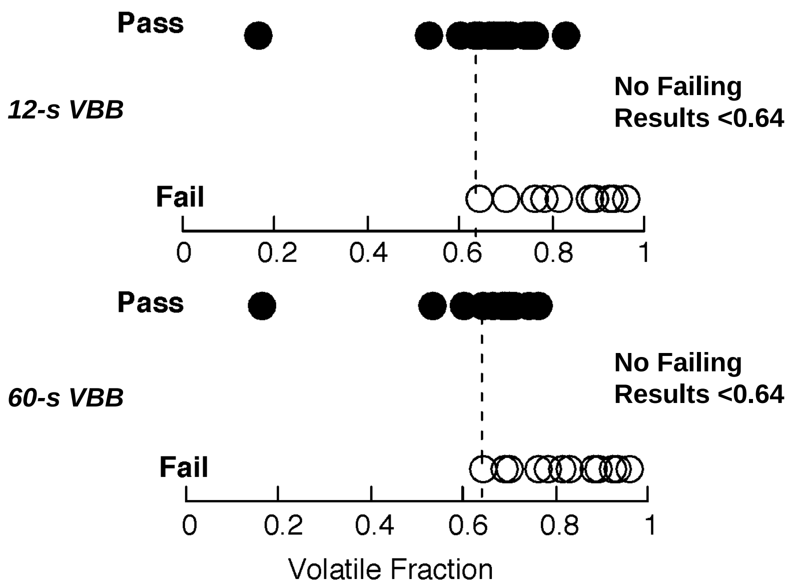

Figure 9. Data for the 12- and 60-s VBB Tests vs Volatile Fraction φ

Table 5. Qualitative Results From 12- and 60-s VBB Tests

Thermal Combustion Property, X_i	Critical Value of Thermal Combustion Property, $X_{critical}$	
	12-s VBB Test	60-s VBB Test
HRC (J/g-K)	181	181
φ (g-gas/g-sample)	0.64	0.64
HR (J/g-sample)	16.2	14.4
HR_{gas} (J/g-gas)	23.1	22.3
$T_{\max}$ (°C)	474	474

Quantitative Analysis. Four of the five thermal combustion properties in table 5 were used as explanatory variables in the quantitative analysis, as shown in figures 10 through 13. The pyrolysis temperature $T_{\max}$ was rejected as an explanatory variable because the probability function (equation 3) could not be fit to the binary data due to the low correlation.

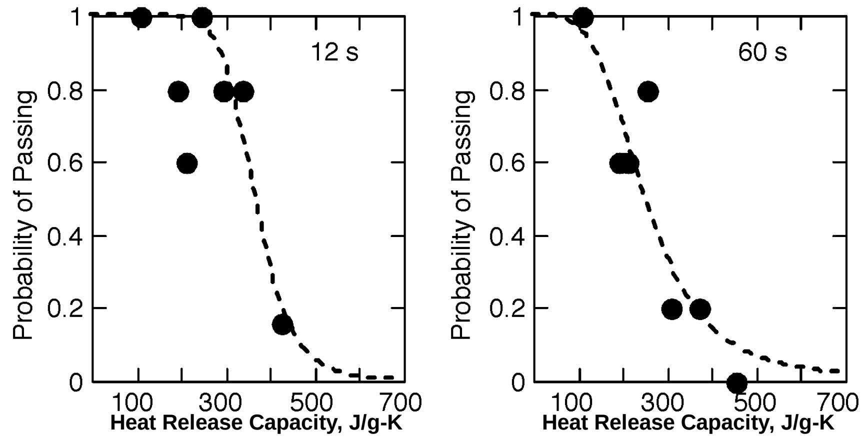

Figure 10. Plots of p_i vs HRC for the 12- and 60-s VBB Tests

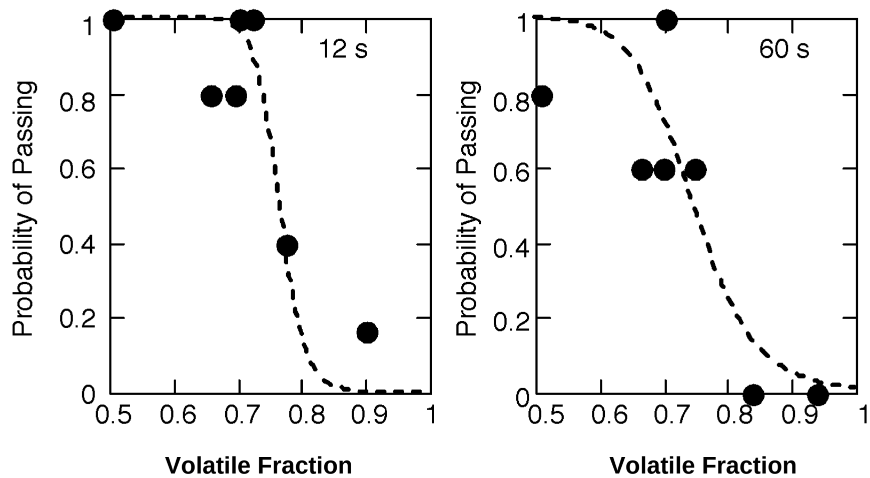

Figure 11. Plots of p_i vs Volatile Fraction φ for the 12- and 60-s VBB Tests

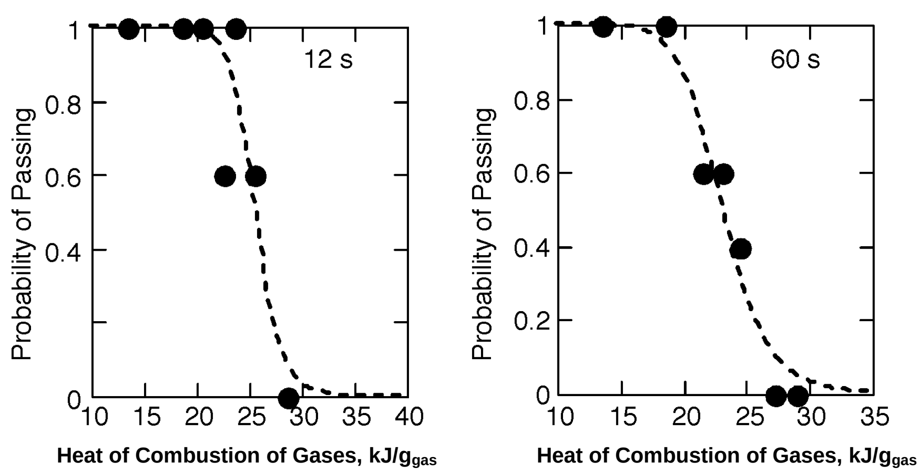

Figure 12. Plots of p_i vs HR_{gas} for the 12- and 60-s VBB Tests

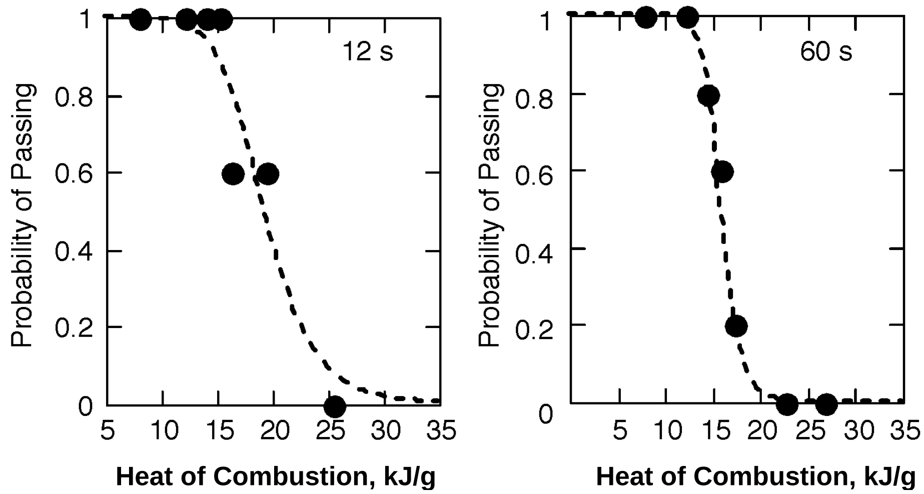

Figure 13. Plots for p_i vs *HR* for the 12- and 60-s VBB Tests

The regression coefficients for the four thermal combustion properties that were amenable to quantitative analysis are listed in tables 6 and 7 for the 12- and 60-s VBB Tests, respectively, along with the goodness-of-fit estimates (equations 5 and 6) for the probability function.

Table 6. Values of *X**, *b*, MD, and Pearson's *R* for the 12-s VBB Test

Predictor Variable	*X**	*b*	*R*	MD (%)
HRC	365 J/g-K	3.8	0.74	24
φ	0.78	19.9	0.74	16
HR_{gas}	25.4 kJ/g	21.6	0.91	13
HR	19.3 kJ/g	8.7	0.95	11

Table 7. Values of *X**, *b*, MD, and Pearson's *R* for the 60-s VBB Test

Predictor Variable	*X**	*b*	*R*	MD (%)
HRC	262 J/g-K	2.9	0.95	26
φ	0.74	15.4	0.85	30
HR_{gas}	23.0 kJ/g-gas	15.1	0.99	14
HR	16.0 kJ/g	17.6	1.0	4

DISCUSSION

This study was conducted to determine whether the MCC could be used to discriminate between adhesives, potting compounds, and fillers (i.e., adhesive compounds) with regard to their flammability in the 14 CFR Part 25 VBB Test. The VBB Test of adhesive compounds used in the construction of cabin materials has been proposed as a surrogate for full-scale fabrication and fire testing to certify parts made with substitute adhesives [1]. This study did not address whether the VBB Test result of an adhesive compound was a good predictor of 14 CFR Part 25 fire tests of cabin materials fabricated with the adhesive compound. However, this study did address whether, and to what extent, the MCC could be used to predict the VBB Test results for adhesive compounds tested separately from fabricated cabin materials.

It was found that there was not a clear demarcation between passing and failing results in the VBB Test with regard to most thermal combustion properties. This was expected given that flame extinction is a critical phenomenon and is sensitive to small variations in intrinsic (material) and extrinsic (size-dependent) properties, as well as the conditions of the test. For these reasons, the transition from passing to failing results occurred over a range of the thermal combustion property examined but was centered at a particular value, X^*. In fact, this transition occurs over the range $\Delta X \approx \pm 2X^*/b$ of the explanatory variable. This behavior is shown in figure 14, which is a plot of the binary (qualitative) probabilities for *HR* from table 4 as per figure 6, and the cumulative (quantitative) probability shown as a continuous solid line calculated using equation 3 and the parameters $X^* = 16.0$ kJ/g and $b = 17.6$ from table 7. The transition from passing to failing for the binary probabilities, $14 < HR < 17$ kJ/g in figure 14 is approximately $X^* \pm 2X^*/b = 16.0 \pm 2$ kJ/g, or $14 < HR < 18$ kJ/g. Thus, the probability of passing the 12- or 60-s VBB Test for a new adhesive compound not included in this study can be determined from its MCC thermal combustion properties, X_i using equation 3, and the parameters in tables 6 and 7.

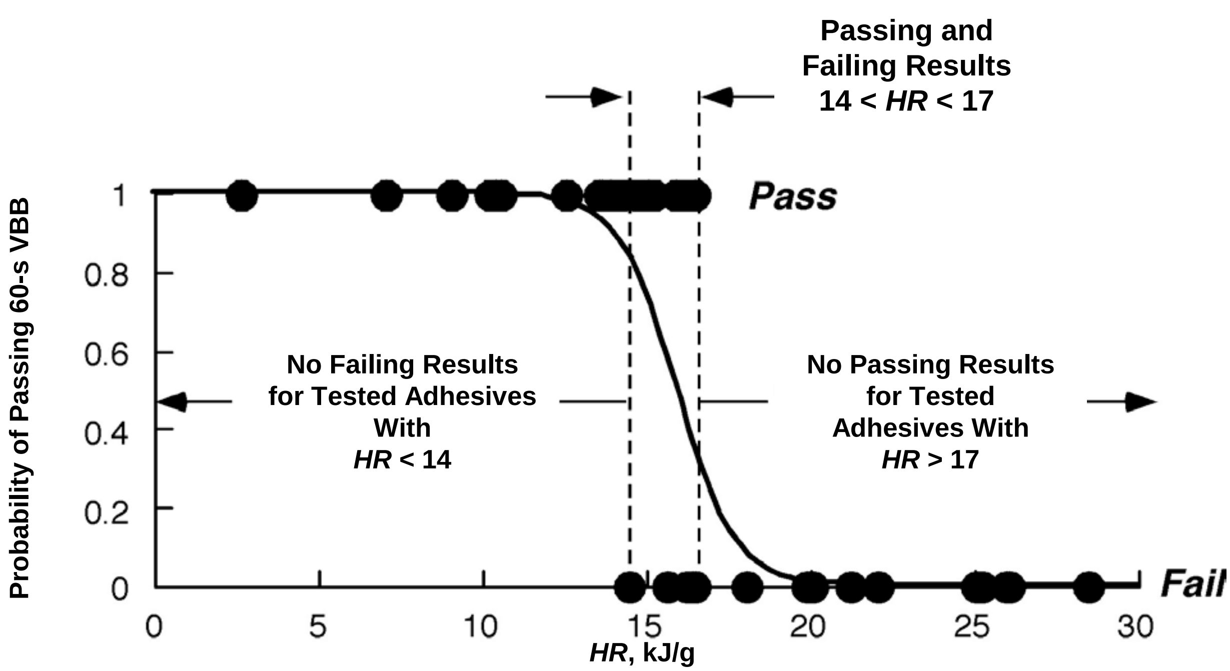

Figure 14. Binary and Continuous Probability Distributions for the 60-s VBB Test Using *HR* as the Explanatory Variable

CONCLUSIONS

The thermal combustion properties of epoxy adhesives, edge fillers, and potting compounds used in the construction of aircraft cabin materials were measured with a microscale combustion calorimeter. The thermal combustion properties were evaluated as predictors of Title 14 Code of Federal Regulations Part 25 Vertical Bunsen Burner (VBB) Test pass/fail results of standard rectangular bar specimens (75 x 300 x 6 mm) of the adhesive compounds. It was found that the heat released by combustion of the sample, *HR*, and the heat released by combustion of the sample gases, HR_{gas}, were excellent predictors of the pass/fail classification of the adhesives, potting compounds, and fillers. Moreover, the probability of passing the 12- and 60-second VBB Tests can be calculated for new adhesive compounds using the continuous probability distribution function and parameters determined in this study.

REFERENCES

1. "Flammability Standardization Group Update," *International Aircraft Materials Fire Test Working Group Meeting*, Bremen, Germany, June 23, 2011.

2. Lyon, R.E., Walters, R.N., and Stoliarov, S.I., "Thermal Analysis of Flammability," *Journal of Thermal Analysis and Calorimetry*, Vol. 89, No. 2, 2007, pp. 441-448.

3. Lyon, R.E., Walters, R.N., and Stoliarov, S.I., "A Thermal Analysis Method for Measuring Polymer Flammability," *Journal of ASTM International*, Vol. 3, No. 4, April 2006, pp. 1-18.

4. Neter, J., Wasserman, W., and Kutner, M.H., *Applied Linear Regression Models*, Richard D. Irwin, Inc., Homewood, Illinois, 1983.

5. Hosmer, D.W. and Lemeshow, S., *Applied Logistic Regression*, John Wiley & Sons, New York, 1989.

6. Lyon, R.E., Safronava, N., Walters, R.N., and Stoliarov, S.I., "A Statistical Model for the Results of Flammability Tests," *Fire and Materials 2009*, San Francisco, California, January 26-28, 2009.

7. "Standard Test Method for Determining Flammability Characteristics of Plastics and Other Solid Materials Using Microscale Combustion Calorimetry," ASTM D 7309-07, American Society for Testing and Materials (International), West Conshohocken, Pennsylvania, 2007.

8. U.S. Federal Register, Title 14 Code of Federal Regulations Parts 1-49, "Aeronautics and Space, Vol. 1, Chapter I: Federal Aviation Administration," 2007.

9. Hill, R., "Aircraft Material Fire Test Handbook," FAA report DOT/FAA/AR-00/12, April 2000.