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An Approach to Evaluate Precision and Inter-Laboratory Variability of Flammability Test Methods for Aerospace Materials

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ABSTRACT

Materials selection for spacecraft is based on conventional flammability or ignition sensitivity acceptance tests. Current procedures for determining the inter-laboratory repeatability and reproducibility of aerospace materials flammability tests are not considering the dependence of data variability on test conditions and consequently attempts to characterize the precision of these methods were not successful. The inter-laboratory data variability is determined with tests conducted under arbitrary conditions, which consequently may not provide sufficient information to enable adequate determination of a method's precision. For evaluating the precision of NASA's flammability test methods, the protocol recommended includes selecting critical parameters and determining the 50% failure point by considering the specific failure criteria of each method using the critical parameter as a variable. Upon performing inter-laboratory round robin testing using this approach, the laboratories' performance could be evaluated by comparing the repeatability of the 50% failure point and/or the repeatability of critical conditions where the probabilities of passing and failing are unity, i.e., the transition zone repeatability. When a sufficient amount of data has been acquired with this method, an adequate estimation of precision of aerospace materials flammability test methods will be possible.

INTRODUCTION

Spacecraft fire safety emphasizes fire prevention, which is achieved primarily through the use of fire-resistant materials. Materials selection for spacecraft is based on conventional flammability or ignition sensitivity acceptance tests along with prescribed quantity limitations and configuration control for items that are nonpass or questionable. The NASA STD 6001 [1] Test 1 is the major method used to evaluate flammability of materials intended for use in the habitable environments of U.S. spacecraft; Test 4 is used to evaluate flammability of wire insulations. Although round robin tests involving up to four aerospace materials test laboratories were conducted during the last 10 years, attempts to characterize the precision of these methods were not successful. Current procedures for determining the inter-laboratory repeatability and reproducibility of aerospace materials flammability tests are not considering the dependence of data variability on test conditions. The inter-laboratory data variability is determined with tests conducted under arbitrary conditions, which consequently may not provide sufficient information to enable adequate determination of a method's precision.

The precision of a given test method is interlinked with the repeatability of the phenomena being evaluated. For example [2], normally the flammability behavior of materials follows an S-shape curve when the response is plotted versus the magnitude of a critical parameter, such as oxygen concentration (Figure 1). Below and above certain stimulus levels, the results would be more repeatable, and the flammability test evaluation methods would provide results that are insensitive to even a large change in variables. Tests conducted under transition zone conditions should be expected to provide less repeatable results, since in this zone smaller changes in test variables would have larger effects on results. It appears that the test conditions loci, relative to the flammability distribution curve (Figure 2, [3]), will dictate the repeatability and consequently should be carefully considered to prevent drawing erroneous conclusions on the statistical confidence of results.

A study was initiated on a methodology for characterizing precision of test methods used to evaluate aerospace materials when the test conditions strongly affect the repeatability of the phenomena evaluated. Normally, obtaining a relatively high level of confidence on results can be obtained only with a large number of samples if the test is conducted with sets of conditions that fall within the transition zone for a given material. Testing in zones removed from the transition zone would yield abnormally high precision for the methods, since the results would be insensitive to even large fluctuations of variables. This paper evaluates the feasibility of a method used to evaluate the repeatability and reproducibility of a test method by conducting a limited number of tests following a rigorous statistical method, such as the staircase (or 50%) method [4]. It is believed that the approach proposed would allow meaningful comparisons of laboratories' performance when the test conditions are likely to strongly affect the repeatability of the phenomena being investigated.

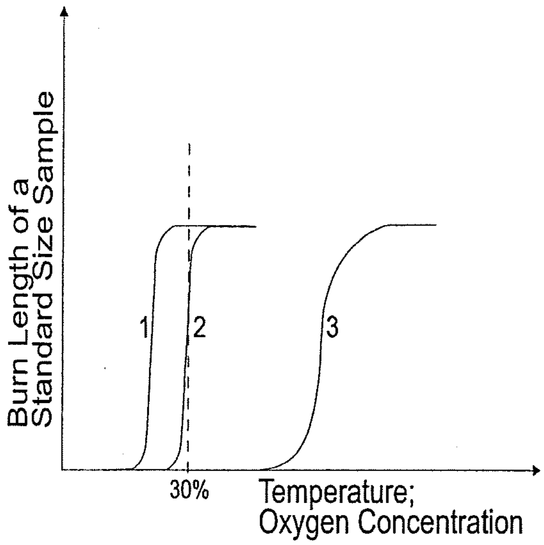

Figure 1. Flammability Distribution Curve: Generalized Dependency of a Flammability Response (i.e., Burn Length of a Standard-size sample) on the Magnitude of the Stimulus (i.e. Temperature or Oxygen Concentration)

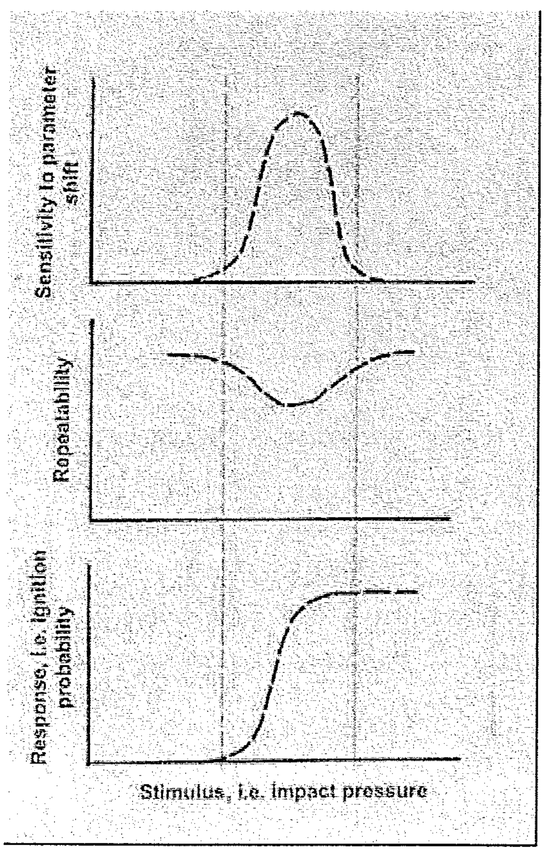

Figure 2. Expected Trends for Repeatability and Sensitivity to Parameter Shift in Relation with the Magnitude of the Stimulus and its Effects on Response.

Experimental

Materials

Two materials were used for flammability tests, Kydex 100, which is a PVC/PMMA alloy, and Nomex, an aromatic polyamide. The flammability test samples were approximately 6.4 cm wide and 30.5 cm long, which are standard NASA STD 6001 Test sample dimensions. The material thicknesses were approximately 1.6 mm for Kydex and 0.32 mm for Nomex.

Test Systems and Procedures

Flammability tests were conducted in a 1400 L test chamber connected to a vacuum pump and oxygen and nitrogen supplies. The test system met the NASA STD 6001 Test 1 requirements [1]. All tests were conducted at 101.3-kPa total pressure and following NASA STD 6001 Test 1 procedures, with the exception that no K-10 paper was used. The test environment was established by evacuating the test chamber and back filling with oxygen and nitrogen. The test environment was mixed by forced convection inside the test chamber and its oxygen content was verified prior to testing.

Initially, the limiting oxygen indices were determined by conducting sequential tests, as recommended by ASTM D 2863 [5], and using a step size of one percent oxygen by volume. The limiting oxygen indices were calculated with the "up-and-down method for small samples" [4]. This method has been adopted by both ISO 4589 [6] and D 2863 for determining the "minimum oxygen concentration required to support combustion of plastics." The failure criteria consisted of burn length of at least 15.2 cm, the same as for NASA STD 6001 Test 1.

Upon determining the limiting oxygen indices, the tests focused on determining the flammability transition interval. Five tests were conducted under conditions of increasing and decreasing oxygen concentration stepwise by one percent until a probability of unity for both passing and failing the burning criteria was obtained.

Results and Discussion

The test results are shown in Table 1. The limiting oxygen indices following the ASTM D2863 logic are shown in Table 2. Data for both Kydex and Nomex appears to be repeatable, with relatively small standard deviations. The preliminary results indicate that both materials appear to be suitable for evaluating intra- and inter-laboratory repeatability. Additional repeatability and round robin testing involving several laboratories will be required to acquire sufficient information to determine with statistical confidence if flammability data from particular laboratories fall outside expected repeatability and reproducibility values. The suggested approach could be extended to test methods intended to determine ignition sensitivity of aerospace materials.

Table 1. Probability of Failing NASA STD 6001 Test 1 criteria in various oxygen concentrations

Test Material	*Oxygen Concentration Vol %*	*Samples Tested*	*Probability of Sample Failing 15.2-cm Burn Criteria*
Kydex 100	29	10	0.0
	30	5	0.4
	31	5	0.6
	32	5	0.8
	33	5	0.8
	34	5	1.0
Nomex	22	5	0.0
	23	2	0.4
	24	5	0.2
	25	5	0.6
	27	5	0.8
	28	5	1.0

Table 2. Upward Flame Propagation in Quiescent Environments; Limiting Oxygen Index, and Repeatability Statistics

Test Materials	*Limiting Oxygen Index Values*[1,2]					*Average*[1]	*Range*[1]	*Standard Deviation*[1]
Kydex 100	30.5	32.5	32.5	31.5	32.3	31.9	30.5-32.5	0.77
Nomex	25.8	26.0	24.7	25.9	25.0	25.5	24.7-26.0	0.53

CONCLUSION

Current procedures for determining the inter-laboratory repeatability and reproducibility of aerospace materials flammability tests are not considering the dependence of data variability on test conditions. The inter-laboratory data variability is determined with tests conducted under arbitrary conditions, which consequently may not provide sufficient information to enable adequate determination of a method's precision. For evaluating the precision of NASA's flammability and ignition sensitivity test methods, the following protocol is recommended. Select one or two critical parameters for each method. For example, oxygen concentration could be considered for all types of flammability tests. Upon selecting the critical parameter(s), determine the 50% failure point considering the specific failure criteria of each method and using the critical parameter as a test variable. One can also use ASTM D 2863 test logic to determine the limiting oxygen index of NASA STD 6001 Tests 1 or 4 (which in fact provides the 50% failure point). For flammability test methods, an alternative or additional test method would be to perform testing under critical conditions removed stepwise from the 50% point to determine the critical conditions where both probabilities of passing and failing become unity upon conducting a statistically adequate number of tests under each condition. Perform inter-laboratory round robin testing using this approach. The laboratories' performance could be evaluated by comparing the repeatability of the 50% failure point (or the limiting oxygen index values for flammability) and/or the repeatability of critical conditions where the probabilities of passing and failing are unity, i.e., the transition zone repeatability. When a sufficient amount of data has been acquired with this method, an adequate estimation of precision of aerospace materials flammability will be possible. Additional repeatability and round robin testing involving several laboratories will be required to acquire sufficient information to determine with statistical confidence if flammability data from particular laboratories fall outside expected repeatability and reproducibility values. The approach suggested will be extended to test methods intended to determine ignition sensitivity of aerospace materials.

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