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LABORATORY TEST METHODS FOR EVALUATING THE FIRE RESPONSE OF AEROSPACE MATERIALS

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for

National Aeronautics and Space Administration Ames Research Center Moffett Field, California 94035

Carlos J. Hilado, ⊉rincipal Investigator Fire Safety Center University of San Francisco 2130 Fulton Street San Francisco, California 94117



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LABORATORY TEST METHODS FOR EVALUATING THE FIRE RESPONSE OF AEROSPACE MATERIALS

Carlos J. Hilado Fire Safety Center University of San Francisco San Francisco, California 94117

#### INTRODUCTION

The introduction of new materials, and the design of new applications for existing materials, have together increased the number of possible interactions between materials and their environments. Fire safety requires that such combinations be evaluated for possible hazards to life and property, so that only materials which do not present unreasonable risk are permitted into specific applications.

Because no two fires are exactly alike, no single test method, or limited number of test methods, can reasonably be expected to simulate all possible fire situations. It should therefore be remembered that a particular test result is strictly valid only for those particular test conditions, and it becomes a matter of judgement as to what types of fire situations that particular test can be expected to simulate.

All fires develop in three stages: the pre-ignition conditions of initial exposure to heat, the pre-flashover conditions of the developing fire, and the post-flashover conditions of the fully developed fire. Reaching the pre-flashover stage indicates a failure of fire safety measures in the pre-ignition stage, and reaching the post-flashover stage indicates a failure of fire safety measures in the two preceding stages. It is both costeffective and common sense to deal with a fire in the pre-ignition stage, before it actually happens. The public in general, and fire protection personnel in particular, are instinctively aware of this fact, perhaps because they usually have more sense than money. The emphasis of much scientific work on the later stages of a fire, to the neglect of the pre-ignition stage, may indicate the presence of more money than sense in science, and presume the possession of more money than sense by the general public.

This dichotomy has extended to the development of laboratory test methods for the evaluation of materials. Some of the more recent tests developed have become so expensive in investment, facilities, and personnel that they have been priced out of reach of the relatively small laboratories which not only have the greatest need for such tests, but also have perhaps the most impact on the day-to-day maintenance of fire safety in materials. The test methods which were developed or evaluated at the University of San Francisco were intended to serve as means for comparing materials on the basis of specific responses under specified sets of test conditions, using apparatus, facilities, and personnel that would be within the capabilities of perhaps the majority of laboratories. The guidelines followed in selecting such test methods included the following: the apparatus should fit inside a four-foot-wide laboratory hood, preferably in a bench hood; the apparatus should not require special supporting facilities and construction, such as special power lines and air supply; the apparatus should be operated by a technician, and not by a much more expensive scientist.

Priority was given to test methods which showed promise of addressing the pre-ignition stage of a potential fire. Apparatus which could address the later stages of a fire as well were, of course, desirable. This priority blended well with the guidelines described, because test methods for the pre-ignition stage tend to require less elaborate apparatus, and less scientist employment, than test methods addressing the later stages.

With the purpose of screening in mind, and within the limitations described, these test methods are intended to indicate which materials may present more hazard than others under specific test conditions, and not necessarily explain why they differ.

These test methods will be discussed in this paper, arranged according to the stage of a fire to which they are most relevant. Some observations of material performance which resulted from this work will also be discussed.

# MATERIALS USED IN TEST METHOD DEVELOPMENT

Various aerospace materials were supplied by the Ames Research Center of the National Aeronautics and Space Administration. Many other materials were supplied by other sources, including the Johnson Space Center of the National Aeronautics and Space Administration, the Civil Aeromedical Institute of the Federal Aviation Administration, the Bureau of Home Furnishings of the State of California, Factory Mutual Research Corporation, SRI International, University of Pittsburgh, Merck and Company, CPR Division of the Upjohn Company, Jim Walter Research Corporation, Lockheed Palo Alto Research Laboratories, Stauffer Chemical Company, General Electric Company, Dow Corning Corporation, Phillips Petroleum Company, U. S. Borax Research Corporation, the Aeronautical Systems Division of the Air Force Systems Command, and the Eastern Forest Products Laboratory of the Canadian Forestry Service.

These materials were evaluated to obtain experience with the test methods being developed for a wide range of materials, to permit laboratory personnel to accumulate experience, and to keep personnel and methodology at peak efficiency during slack periods. They also permitted early application of these test methods to meeting human needs, and early demonstration of the relevance of NASA-supported technology to other national needs.

The relevance of some of the materials used to the development of test methods for aerospace materials is not immediately obvious. A substantial number of seat cushion materials were supplied by the Bureau of Home Furnishings of the State of California. Many of these materials were similar to those used in aircraft, and some were the same materials used in aircraft, because of the similarity between California and Federal Aviation Administration flame resistance requirements.

Wood is a relevant material for aircraft because certain passenger jet aircraft which had not been completely refurbished still contained wood in sidewall panels as late as 1977. Its major value, however, is as a reference material to provide a basis for comparison when this test methodology finds more general use.

# LABORATORY TEST METHODS DEVELOPED UNDER THIS PROGRAM

The following test methods, which will be discussed in perspective in later sections, were developed under this program. The methods are arranged on the basis of the stage of a fire to which they are relevant, and the fire response characteristic which they address.

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Pre-Ignition Stage
     Ignitability (Unpiloted)
          USF ignitability test method
     Flash Fire Propensity
          USF flash-fire screening test method
     Pyrolysis Gas Toxicity
          USF toxicity screening test method
     Smolder Susceptibility
          USF smoldering test method
     Smoldering Combustion Gas Toxicity
          USF smoldering toxicity test method
     Smoke (Unpiloted)
          none; NBS-Aminco smoke chamber used
Pre-Flashover Stage
     Ignitability (Piloted)
          USF ignitability test method
     Smoke (Piloted)
          none; NBS-Aminco and Arapahoe smoke chambers used
    Heat Release
          none; OSU release rate apparatus used
     Flame Spread
          none; method developed under separate Navy program
     Pyrolysis Gas Toxicity
          USF toxicity screening test method
     Flaming Combustion Gas Toxicity
          none; NBS-Aminco smoke chamber used
Post-Flashover Stage
     Ignitability (High Flux)
USF ignitability test method
     Smoke (High Flux)
          none; NBS-Aminco smoke chamber used
     Flame Spread (High Flux)
          none; method developed under separate Navy program
     Pyrolysis Gas Toxicity (High Flux)
          USF toxicity screening test method
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## PRE-IGNITION STAGE

## <u>Ignitability (Unpiloted)</u>

An ignitability test defines the range of conditions within which a material can be expected to remain in the pre-ignition stage. Ignition is the result of the combination of intensity of heat flux and time of exposure. The classic ignitability test requires exposure of the material to a known level of heat flux to determine the time needed for ignition. The pre-ignition stage is simulated by the absence of a pilot flame.

It should be observed that the traditional ignitability tests employing a burner flame have over the years served reasonably well as acceptance tests for certain applications, and will probably continue to play an important role. They have deficiencies in the inherent variability of the combination of a small heat source and a small area of specimen actually impacted, and the inability to discriminate between pre-ignition and pre-flashover conditions.

The USF ignitability test method (1-3) employs a Mellen high-flux heater and an Aminco specimen holder kept properly positioned with respect to reach other by a mounting frame. The heater is adjusted to the desired level of heat flux, and the specimens are then exposed in succession. The times at which smoking, charring, and ignition occur are recorded.

Ignitability test data have been published on aircraft interior composites (4), aircraft seat fabrics (5), insulation and building materials (6), rigid foam insulation (7), wood and cellulosic board (8), and furniture upholstery fabrics (9-12).

As anticipated, time to ignition decreased with increasing heat flux. In some cases, physical parameters such as density and construction were as important as the chemical composition of the material. The time to ignition of wood and cellulosic board appeared to be primarily a function of heat flux and material density, rather than of type of wood or cellulosic board (8).

Materials with thermal insulation characteristics can be expected to reach surface ignition more rapidly than less effective insulators that are therefore better heat sinks. With the increasing importance of energy conservation, materials possessing thermal insulation characteristics have become more desirable. This economic demand for a potentially hazardous characteristic emphasizes the need for careful design and engineering when dealing with these types of materials.

#### Flash Fire Propensity

Flash fires are a special form of fire hazard which combine aspects of ignitability, flammability, and heat release; they seem most likely to occur when substantial amounts of combustible vapors escape unburned from the vicinity of a fire and accumulate in an enclosure where they encounter an ignition source, perhaps even the original fire that produced them.

The basic approach to laboratory evaluation of flash-fire propensity involves propagation of fuel-air mixtures in a tube. The USF flash-fire screening test method (13-15) employs a Lindberg horizontal tube furnace to pyrolyze 0.10 g of material at 800°C and a vertical combustion tube to develop the flammable mixture and observe the flash fire. The height of the flash fire and the time to flash fire are recorded.

The materials exhibiting the greatest flash-fire propensity under these particular test conditions included polyurethane flexible foam, polyethylene plastics, and wood. It should be remembered that this screening test provides only an indication of the flash-fire propensity of the material, not a complete predictable level of performance. In a full-scale fire situation, such factors as thickness of the material, area of exposed surface, and tendency to form a surface char obviously will not be the same for all materials.

#### Pyrolysis Gas Toxicity

There are two basic approaches to evaluating toxicity: the analytical approach and the bioassay approach. The analytical approach consists of isolating, identifying, and measuring the various compounds in the mixture of gases evolved, and seeking to predict the overall or mean toxicity of the entire mixture on the basis of what is known about the toxicity of the individual compounds. The bioassay approach simply uses the laboratory animal as the integrator of all the information which the analytical approach seeks to obtain and synthesize.

The USF toxicity screening test method (16-20) uses the bioassay approach because it is more cost-effective and timeeffective for screening purposes. A 1.00 g sample of material is placed in a weighed quartz boat, centered in a quartz tube, and the tube is placed in a Lindberg horizontal tube furnace. The sample is heated at a rate of 40°C/min from 200°C to 800°C without forced air flow past the sample. The pyrolysis tube is connected to a 4.2 liter hemispherical chamber, which contains four Swiss Webster male mice. The animals are under continuous observation, and times to various responses are recorded. The test is terminated upon the death of the last surviving animal or after 30 minutes, whichever occurs first. A sample of the chamber atmosphere is taken for analysis before the chamber is opened. The quartz boat containing the sample is allowed to cool, and then weighed to determine the amount of char remaining. Over 300 materials have been evaluated under these particular test conditions, which are intended to simulate the pre-ignition stage as well as the non-flaming aspects of the pre-flashover stage of a fire. The most extensive single compilation includes 270 materials (18); less extensive compilations, some of which include more recent data, deal specifically with wood (21), silicone polymers (22), cellular polymers (23), synthetic polymers (24), furniture upholstery fabrics (25-27), thermal insulation materials (28), and plastics and elastomers (29).

Under these particular test conditions, of the 15.4 million tons of plastics sold in the United States each year, 85.7 per cent appeared to be less toxic than wood and only 0.3 per cent more toxic than wood, with about 14 per cent undetermined but probably equivalent. Of the major generic types of plastics, only polyoxymethylene, polyphenylene sulfide, and polyether, polyaryl, and polyphenyl sulfones appeared to be more toxic than wood under these particular test conditions.

Of the fibers and fabrics, wool and modacrylic appeared to be the most toxic under these particular test conditions; rayon, nylon, and polyolefin appeared to be less toxic than the natural fibers (cotton, silk, and wool).

To obtain an indication of the effects of test conditions on relative toxicity, a variety of materials were evaluated under different test conditions (18,23,25). The relative rankings of materials tended to remain the same in spite of changes in test conditions and in test methods. Some significant reversals in rankings were observed, however.

#### Smolder Susceptibility

Smoldering is a distinctive form of combustion which has been encountered in certain materials which tend to form a porous char. The most common example is charcoal. Materials which are known to be susceptible to smoldering include cellulosic fabrics, cotton batting, some formulations of polyurethane flexible foams, and cellulosic loose fiber fill insulation.

The USF smoldering test method for fabric/cushion combinations (30) employs an L-shaped wooden test stand which holds vertical (back) and horizontal (seat) panels of fabric over polyurethane flexible foam or cotton batting substrates. A king-size non-filter cigarette is placed at the abutment of the vertical and horizontal panels and covered with a layer of cotton bed sheeting material. The L-shaped wooden test stand is based on apparatus used by the Bureau of Home Furnishings of the State of California. The USF smoldering test method for cellulosic loose fiber fill insulation (31) employs a rectangular stainless steel opentop box which holds the sample material. A king-size non-filter cigarette is lighted and inserted vertically in the center of the sample. The stainless steel open-top box is based on apparatus used for Federal Specification HH-1-515D.

In the case of upholstery fabric/cushion combinations, smolder susceptibility is not a function of the fabric alone, or of the cushion alone, but of the specific fabric/cushion combination. A fiberglass board substrate is not used because it appears to be a well-intentioned pseudo-scientific selection which provides neither relevance to real life nor impartial safety factors. Only the actual cushioning materials are used in the test method. For comparing fabrics, polyurethane flexible foam of the appropriate densities for back and seat cushions are used. In general, 100 per cent cellulosic fabrics exhibit the greatest susceptibility to smoldering.

Differences in physical behavior such as melting and charring can critically affect the ability of the fabric and cushion to support smoldering combustion. The confidence with which performance can be predicted on the basis of chemical composition alone has been weakened by the finding that some materials which were more resistant to flame ignition proved to be more susceptible to smoldering.

#### <u>Smoldering Combustion Gas Toxicity</u>

In cases where smoldering combustion continues for long periods of time without ever reaching ignition, the principal hazard is presented by the toxicity of the gases produced.

The USF smoldering toxicity test method (32,33) employs a 244 liter animal exposure chamber in which eight Swiss Webster male mice are exposed in two stainless steel cages. The source of the gases is the L-shaped wooden test stand for fabric/cushion combinations, or the stainless steel open-top box for cellulose insulation. After the igniting cigarette is lighted and placed in position, the stand or box is placed in the chamber which is then sealed. The animals are under continuous observation. The test is terminated upon the death of the last surviving animal or after 90 minutes, whichever occurs first. A sample of the chamber atmosphere is taken for analysis before the chamber is opened.

Cellulosic fabric/cotton batting combinations seemed to be significantly more hazardous than cellulosic fabric/polyurethane foam combinations with respect to toxicity of gases from smoldering combustion. With both fabric/cushion combinations and cellulose insulation, toxicity appeared to be a function of the amount of material consumed by smoldering.

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The ratio of available surface for smoldering to compartment volume in this system when fabric/cushion combinations are tested is  $1,795 \text{ cm}^2/\text{m}^3$ ; this represents a safety factor of 5:1 for a couch in a California living room, and 100:1 for a three-seat unit in a wide-body jet passenger aircraft.

### Smoke (Unpiloted)

Smoke is an important fire response characteristic because visibility is a factor in the ability of occupants to escape, and in the ability of firefighters to suppress a fire. Tests for smoke-producing characteristics employ one of two measurement techniques: gravimetric and optical. The USF Fire Safety Center uses the Arapahoe smoke chamber, which employs the gravimetric technique, and the NBS-Aminco smoke chamber, which employs the optical technique, but only the NBS-Aminco smoke chamber in the unpiloted mode is applicable to the pre-ignition stage.

Compilations of NBS-Aminco smoke data obtained under unpiloted conditions have been published (34-36). Of the 15.4 million tons of plastics sold in the United States each year, about 48 per cent appeared to produce more smoke than wood under these particular test conditions; these included polyethylene, polypropylene, ABS, and thermoset polyesters. The remaining 52 per cent appeared to either be comparable to wood or to produce less smoke than wood under these particular test conditions.

A more extensive discussion of smoke test data is contained in a later section.

### PRE-FLASHOVER STAGE

#### Ignitability (Piloted)

The use of a pilot flame in an ignitability test simulates the proximity of an open-flame ignition source. It should be remembered that during the pre-flashover stage some materials may be exposed to radiant heat at a sufficient distance from the flames to make unpiloted ignition more relevant.

A heat flux level of 5 W/cm<sup>2</sup> may be considered an arbitrary border between the pre-flashover and post-flashover stages. Test procedures involving higher levels of heat flux may not be always relevant to the pre-flashover stage.

The USF ignitability test method is adapted to piloted ignition by the addition of a small pilot flame approximately 25 mm (1 in) above the top of the specimen to ignite any flammable mixtures which may be formed. Ignitability test data have been published on furniture upholstery fabrics (11) and airfield pavement materials (37).

As anticipated, time to ignition decreased with increasing heat flux. The presence of a pilot flame tended to result in shorter times to ignition. In general, the relative performance of materials appeared to be the same as in the case of unpiloted ignition.

# Smoke (Piloted)

The use of a pilot flame, or an open-flame heat source, tends to maintain flaming combustion conditions. This can drastically alter the nature of the smoke evolved from the material being evaluated. It should be remembered that during the pre-flashover stage some materials may te far enough removed from the flames to make unpiloted conditions more relevant.

The Arapahoe smoke chamber, which measures smoke by the gravimetric technique, uses a burner flame as the heat source. The NBS-Aminco smoke chamber, which measures smoke by the optical technique, uses radiant heat and a pilot burner to provide piloted ignition. Data have been published for the Arapahoe smoke chamber (34-36,38-40) and for the NBS-Aminco smoke chamber under piloted conditions (34-36).

Because both unpiloted and piloted conditions may be relevant at various times and locations in the pre-flashover stage, it should be emphasized that test conditions can markedly affect the smoke-producing characteristics of some materials. Wood, polyolefins, and polyurethanes gave higher smoke values under unpiloted conditions than under piloted conditions. PVC, polysulfone, and polycarbonate exhibited the opposite trend, with higher smoke values under piloted conditions than under unpiloted Some materials, in contrast, seem to be relatively unaffected by the test conditions. The styrene-based plastics (polystyrene, ABS, and SAN) and thermoset polyesters appeared to exhibit consistently high smoke values, and the phenolics seemed to exhibit consistently low smoke values.

Of the 15.4 million tons of plastics sold in the United States each year, about 60 per cent appear to produce more smoke than wood under piloted conditions; these include polystyrene, ABS, SAN, thermoset polyesters, polyethylene, and polyvinyl chloride. The remaining 34 per cent appear either to be comparable to wood or to produce less smoke than wood.

These discussions are based on specific optical density values which have not been adjusted for material deposited on the optical system, because the author considers such adjustments unrealistic and misleading. Smy ke deposited on the optical system was originally in the atmosphere obstructing visibility, and the use of the density adjustment gives manufacturers a reward for producing smoke that is heavier and more prone to deposit on surfaces, which could very well include the eyes and the respiratory system.

With certain materials such as softwoods, NBS smoke test data may be influenced by the type of pilot burner used (40). As a result, data obtained by different laboratories at different times using different pilot burners may not be comparable.

Airfield pavement materials were evaluated for smoke in the NBS-Aminco smoke chamber, using exposure to radiant heat with piloting, and exposure to burning JP-4 fuel (41). The standard laboratory procedure tended to give opposite results from behavior in contact with burning fuel, indicating that radiant exposure with piloting does not necessarily correlate with exposure to burning fuel.

Smoke data for materials evaluated in different thicknesses demonstrate that changing the amount of material present or available can affect smoke production to a greater extent than differences in chemical composition. In many situations, careful design and engineering can control smoke production more effectively than chemical modification of materials.

## Heat Release

The amount of heat released from a material and the rate at which that heat is released influence the temperature of the fire environment and the rate of fire spread, and consequently the severity of burn injuries and the extent of property damage.

Of the various methods of evaluating heat release which have been developed, only one, the Ohio State University (OSU) release rate apparatus, has developed adequate experience. This apparatus is used at the USF Fire Safety Center, but it cannot be considered as meeting the guidelines described earlier for suitable laboratory screening tests; it does not fit within a 4-foot wide space and remain easily operable, and the clearance required is about 8 feet, higher than the average canopy hood; the compressed air requirements are greater than wood be available from many building systems. The OSU apparatus, nevertheless, is closer to being a laboratory test in scale than some other massive devices.

#### Flame Spread

Flame spread has long been considered one of the most important fire response characteristics because it influences the rate of spread of a fire and the time available for escape.

The Underwriters Laboratories 25-foot tunnel has been, and will probably continue to be, the most important test for evaluating flame spread. Although some materials have been notable exceptions, this method has over the years served reasonably well as an acceptance test for building materials. In the absence of a credible replacement, most laboratory methods for evaluating flame spread have been aimed at predicting performance in the 25-foot tunnel.

The Monsanto 2-foot tunnel is used at the USF Fire Safety Center, partly because it meets the guidelines by fitting into a 4-foot-wide laboratory hood. The work done with it has been limited to surface films for aircraft interior panels.

One USF method for evaluating flame spread employs an array of fuel pans surrounding one end of a 2 by 1 ft horizontal specimen to produce a fire plume which radiates as much a 7 W/cm<sup>2</sup> heat flux on the specimen surface. This method does not meet the guidelines because it requires air access on all sides and substantial smoke removal capabilities. Work with this method has been limited to airfield pavement materials (42).

# Pyrolysis Gas Toxicity

During the pre-flashover stage, materials which are not exposed to flame ignition sources may undergo pyrolysis upon heating, by radiation or other modes. The rising temperature program of the USF toxicity screening test method would be expected to be relevant to this aspect of the pre-flashover stage. The discussion in the preceding section would therefore be applicable here.

# Flaming Combustion Gas Toxicity

Under flaming conditions, the toxic threat may decrease in importance relative to the thermal threat to occupants of a compartment. The gases produced by flaming combustion, however, may be carried into other compartments where the thermal threat is not significant.

The NBS-Aminco smoke chamber is potentially useful for flaming combustion gas toxicity tests because of its large volume and steel construction. The poor visibility and limited access, however, make it more suitable for the analytical approach rather than the bicassay approach. The work which has been done has been limited to airfield pavement materials (41).

# POST-FLASHOVER STAGE

#### <u>Ignitability (High Flux)</u>

The USF ignitability test method can be considered relevant to the post-flashover stage when heat flux levels of 5 W/cm<sup>2</sup> and higher are used. At these levels of heat flux, it becomes more difficult to discriminate between materials; perhaps there may be relatively little difference between most materials in real fires after flashover occurs.

It does not appear necessary to evaluate materials at heat flux levels above 8 W/cm<sup>2</sup> in the light of the heat flux levels observed in full-scale fires. Evaluation of materials at higher levels, as high as 15 W/cm<sup>2</sup>, may merely constitute scientific one-upsmanship.

#### Smoke (High Flux)

The NBS-Aminco smoke chamber can be considered relevant to the post-flashiver stage if a high-flux heater is used to produce heat flux levels of 5 W/cm<sup>2</sup> and higher, but heat flux above 8 W/cm<sup>2</sup> does not appear useful as noted earlier.

#### Flame Spread (High Flux)

The USF method employing an array of fuel pans (42) can be considered relevant to the post-flashover stage if the fire plume is maintained to produce heat flux at the specimen surface consistently in the 5 to 8 W/cm<sup>2</sup> range.

## Pyrolysis Gas Toxicity (High Flux)

The USF toxicity screening test method provides for exposure of the sample material to constant fixed temperatures in some procedures (18,19). A furnace temperature of 800°C corresponds to a radiant heat flux of 7.5 W/cm<sup>2</sup>.

The data obtained using a fixed temperature of 800°C are less extensive than those obtained using the rising temperature program, with the most extensive single compilation including 53 materials (43). Under these particular test conditions, of the 15.4 million tons of plastics sold in the United States each year, 75.7 per cent appeared to be less toxic than wood, 10.0 per cent not significantly different, and only 0.3 per cent more toxic than wood, with about 14 per cent undetermined but probably equivalent.

### DISCUSSION

Much of the information obtained from laboratory test methods, or for that matter from more costly full-scale tests, will not come as news to firefighters and other fire-protection personnel. One of the motives for developing laboratory test methods of sensible proportions is to obtain information at less cost. With the increasing number of materials which need to be evaluated, our limited resources have to be used in a more cost-effective manner.

The data obtained in the course of evaluating a wide range of materials under various test conditions indicate that physical structure and test conditions are fully as important as chemical composition. The design and engineering of the application appear to have as much, if not more, effect as changes in chemical composition.

The aerospace materials evaluated generally appear to have fire response characteristics superior to those exhibited by materials in general use. The design of synthetic polymers for increasing char yield appears to reduce flammability in the majority of cases, and to reduce toxicity in some cases, with sulfur-containing polymers the most significant exception.

## CONCLUSION

Limited resources and common sense demand that laboratory test methods limit their requirements to those which are necessary to their mission. The laboratory test methods developed under this program appear to be cost-effective means of comparing materials.

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