

NOTE: MIL-STD-2031 has been redesignated as a Test Method Standard. The cover page has been changed for Administrative reasons. There are no other changes to this Document.

INCH-POUND

MIL-STD-2031

26 February 1991

DEPARTMENT OF DEFENSE  
TEST METHOD STANDARD

FIRE AND TOXICITY TEST METHODS AND QUALIFICATION  
PROCEDURE FOR COMPOSITE MATERIAL SYSTEMS USED IN HULL,  
MACHINERY, AND STRUCTURAL APPLICATIONS INSIDE NAVAL SUBMARINES



AMSC N/A

AREA CMPS

DISTRIBUTION STATEMENT A. Approved for public release; distribution is unlimited.

## MIL-STD-2031(SH)

### FOREWORD

1. This military standard is approved for use by the Naval Sea Systems Command, Department of the Navy, and is available for use by all Departments and Agencies of the Department of Defense.

2. Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Commander, Naval Sea Systems Command, SEA 55Z3, Department of the Navy, Washington, DC 20362-5101 by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

3. This standard establishes the fire and toxicity test methods, requirements, and the qualification procedure for composite materials and composite material systems to allow their use inside Naval submarines. This standard is needed to evaluate composite material systems not previously used for these applications. A "composite material system" is considered to be the basic composite material and any supplemental coating applied for enhancing fire resistance, abrasion resistance, or decorative purposes, etc. Composite material systems can offer significant weight and signature reduction advantages as well as desirable electromagnetic and chemical resistance characteristics. Fire limits for these materials will be invoked to make certain that these materials are not a primary fire source, are slow to ignite, can be extinguished using normal submarine fire fighting response to subdue a fire under anticipated operating conditions and that toxicity effects can be tolerated. It is important that design engineers, in addition to selecting materials for their physical and mechanical properties, ensure that the potential fire and toxicity characteristics of the composite material system are fit for the intended use in the closed environment of the naval submarine.

4. No single test method is adequate to evaluate the fire hazard of a particular composite material system. The behavior of a given material system in a fire is dependent not only on the properties of the fuel, but also on the fire environment to which the material system may be exposed. These standardized test methods for flammability and toxicity characteristics cover the spectrum from small scale tests through intermediate scale to large scale tests in order to better understand the fire hazard posed by the composite material system. Results from all of the tests may be required by the Naval Sea Systems Command to ensure the fitness of composite material systems for use in submarines at sea. It is anticipated that the test methods and qualification procedure standardized in this document will prove more appropriate to end item application contractors than to composite material manufacturers.

5. In addition to the tests cited in this standard, there are limitations for the components which are off-gassed during normal operations. Currently, all requests for material certification for this requirement must be sent by letter to the Department of the Navy, Naval Sea Systems Command, Gas Processing and Cryogenics Branch, Washington, D.C. 20362-5101.

## MIL-STD-2031(SH)

## CONTENTS

Paragraph	Page
1. SCOPE .....	1
1.1 Purpose .....	1
1.2 Application .....	1
1.3 Limitations .....	1
2. APPLICABLE DOCUMENTS .....	1
2.1 Non-Government publications .....	1
2.2 Order of precedence .....	2
3. DEFINITIONS .....	2
3.1 Application .....	2
3.2 Burn-through fire test .....	2
3.3 Combustion .....	2
3.4 Combustion gas generation .....	2
3.5 Composite .....	2
3.6 Composite material system .....	2
3.7 Certification .....	3
3.8 Flame spread index .....	3
3.9 Heat release rate .....	3
3.10 Ignitability .....	3
3.11 Ignition .....	3
3.12 Large scale open environment fire test .....	3
3.13 Large scale pressurizable fire test .....	3
3.14 Oxygen index .....	3
3.15 Oxygen-temperature index profile .....	3
3.16 Qualification .....	3
3.17 Quarter-scale fire test .....	4
3.18 Smoke .....	4
3.19 Smoke obscuration .....	4
3.20 Toxicity performance .....	4
4. GENERAL REQUIREMENTS .....	4
4.1 Fire characteristics and test methods .....	4
4.2 Qualification .....	4
4.2.1 Requirement .....	4
4.2.2 Procedures .....	6
4.2.3 Data package .....	6
4.2.4 Certification for use .....	6

## MIL-STD-2031(SH)

## CONTENTS

Paragraph	Page
5. DETAILED REQUIREMENTS .....	6
5.1 Test responsibility .....	6
5.2 Specimen requirements .....	6
5.3 Retest requirements .....	7
5.4 Test facilities .....	7
6. NOTES .....	7
6.1 Intended use .....	7
6.2 Implementation guidance .....	7
6.3 Consideration of data requirements .....	7
6.4 Subject term (key word) listing .....	8
 <b>Appendix</b>	
A. Standard operating procedure for oxygen-temperature index profile .....	9
B. (DTRC) burn-through fire test .....	14
C. Determination of the flashover potential of a lining material using a quarter-scale room fire test .....	20
D. Large scale open environment fire test .....	22
E. Large scale pressurizable fire test .....	26
F. N-Gas model smoke toxicity screening test .....	31
G. Test report technical content requirements .....	48
 <b>Figures</b>	
1. Test stand assembly .....	15
2. Sample mounting .....	17
3. Thermocouple locations .....	18
4. Pressurizable vessel dimensions .....	27
5. Combustion furnace .....	33
6. Furnace mounted on chamber .....	34
7. Exposure chamber design .....	35
8. Gas sampling flow .....	37
9. Animal restrainer design .....	38
10. Animal Restrainer .....	39
 <b>Tables</b>	
I. Fire characteristics and tests .....	4
II. Interlaboratory evaluation of douglas fir .....	42

## MIL-STD-2031(SH)

**1. SCOPE**

**1.1 Purpose.** This standard provides the fire and toxicity test methods, requirements and the qualification procedure to supply NAVSEA with the information needed to certify composite material systems, proposed for use in hull, machinery and structural applications, as fit for use in the closed environment of Naval submarines. This standard is applicable to all composites, such as, metallic, ceramic, carbon, or organic matrix materials used as alternatives to conventional metals.

**1.2 Application.** This standard applies to composite materials and composite material systems used in applications such as materials and composite gearbox covers, turbine covers, fan housings, vent ducting, isolation mounts, pipe hangars, piping, pumps, valves, decking, gratings, stanchions, ladders, hatches, stowage racks, weapons stowage and loading racks, logistic monorails, crew berthing, foundations, bedding plates, non-structural tanks, accumulators, air flasks, launch tubes, propeller shafts, and bearing housings. These are generic hull, machinery, and structural applications that have been identified as candidate systems for composite materials. It is not the intent to list each possible equipment/system component application or apply this standard to the submarine basic pressure hull structure or to items installed external to the pressure hull. Inquiries regarding approved or proposed applications shall be referred to the Naval Sea Systems Command (NAVSEA), Attention: Non-metallic Materials Branch.

**1.3 Limitations.** The preparing activity recognizes that there are definitions, test methods, specifications, and qualification procedures in use that are not contained in this standard. However, those that are contained herein have undergone coordination through an industry consensus process or through the DOD standardization process involved in the preparation of this standard. As a consequence, a certain degree of standardization has been achieved, and it is intended that this MIL-STD will further enhance the understanding of composite fire performance and the standardization of test methods and qualification procedure for composite use in a closed environment.

**2. APPLICABLE DOCUMENTS**

**2.1 Non-Government publications.** The following document(s) form a part of this document to the extent specified herein. Unless otherwise specified, the issues of the documents which are DOD adopted are those listed in the issue of the DODISS cited in the solicitation. Unless otherwise specified, the issues of documents not listed in the DODISS are the issues of the documents cited in the solicitation (see 6.2).

**AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)**

E 162                      **Standard Test Method for Surface Flammability of Materials Using a Radiant Heat Energy Source; (DOD adopted)**

MIL-STD-2031(SH)

- E 662 Test Method for Specific Optical Density of Smoke Generated by Solid Materials
- E 1354 Standard Test Method for Heat and Visible Smoke Release Rates for Materials and Products Using an Oxygen Consumption Calorimeter

(Applications for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.)

(Non-Government standards and other publications are normally available from the organizations that prepare or distribute the documents. These documents also may be available in or through libraries or other informational services.)

**2.2 Order of precedence.** In the event of a conflict between the text of this document and the references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

### 3. DEFINITIONS

**3.1 Application.** The application is specific end use hardware fabricated by utilizing a composite material system.

**3.2 Burn-through fire test.** The burn-through fire test is the test method to determine the time for a flame to burn-through a composite material system under controlled fire exposure conditions.

**3.3 Combustion.** Combustion is a chemical process of oxidation that occurs at a rate fast enough to produce heat and usually light as a glow or flame.

**3.4 Combustion gas generation.** The combustion gas generation is the rate of production of combustion gases (such as CO, CO<sub>2</sub>, HCl, HCN, NO<sub>x</sub>, SO<sub>x</sub>, halogen acid gases, and total hydrocarbons).

**3.5 Composite.** A composite is a material resulting from the controlled combination of two or more different materials at a macroscopic level; typically, a matrix that is reinforced with fibers, whiskers, or dispersions of another material.

**3.6 Composite material system.** Composite material system are the material characteristics which define the material and are relevant to fire and toxicity performance and are preserved in test specimens, these include as a minimum:

## MIL-STD-2031(SH)

- a. Chemical composition of constituents
- b. Geometric relationships between constituent materials in a composite layer (i.e., fiber volume fraction, ply construction, etc.)
- c. Stacking sequence for layers within the bulk structure
- d. Supplemental adhesives, additives or coatings applied for enhancing fire resistance, abrasion resistance, decorative purposes, etc.

**3.7 Certification.** Certification is the determination made by Naval Sea Systems Command as to the acceptability for use of the composite material system for the intended application.

**3.8 Flame spread index.** The flame spread index is a number or classification indicating a comparative measure derived from observations made during the progress of the boundary of a zone of flame under defined test conditions.

**3.9 Heat release rate.** The heat release rate is the heat evolved from a material, expressed per unit of exposed area, per unit of time.

**3.10 Ignitability.** Ignitability is the ease of ignition, as measured by the time to ignite in seconds, at a specified heating flux with a pilot flame.

**3.11 Ignition.** Ignition is the initiation of combustion as evidence by glow, flame, detonation or explosion. The combustion may be sustained or transient.

**3.12 Large scale open environment fire test.** A large scale open environment fire test is a method to test materials at full size of their intended application under controlled fire exposure to determine fire tolerance or ease of extinguishment.

**3.13 Large scale pressurizable fire test.** A large scale pressurizable fire test is a method to test materials using an enclosed compartment in a simulated environment under a controlled fire exposure.

**3.14 Oxygen Index.** Oxygen index is the minimum concentration of oxygen in a flowing oxygen nitrogen mixture capable of supporting flaming combustion of a material.

**3.15 Oxygen-temperature index profile.** A oxygen-temperature index profile is a plot of data points that define the temperature dependence of the oxygen index as the thermal environment of a material is changed.

**3.16 Qualification.** The qualification is the process of determining the acceptability of a composite material for use aboard a Naval submarine.

## MIL-STD-2031(SH)

**3.17 Quarter-scale fire test.** A quarter-scale fire test is a test method to determine the flashover potential of materials in a room when subjected to a fire exposure.

**3.18 Smoke.** Smoke is the airborne solid and liquid particulates and gases evolved when a material undergoes pyrolysis or combustion.

**3.19 Smoke obscuration.** Smoke obscuration is the reduction of light transmission by smoke as measured by light attenuation.

**3.20 Toxicity performance.** Toxicity performance is the potential toxic effects of combustion products (smoke and fire gases).

#### 4. GENERAL REQUIREMENTS

**4.1 Fire characteristics and test methods.** The fire characteristics to be investigated and the test methods and requirements for qualifying a composite material system for use aboard a naval submarine are defined in table I (see 6.3 and appendix G). Any exceptions to these requirements will be identified in the acquisition documents for the specified application (see 6.2) and must be approved by NAVSEA.

#### 4.2 Qualification.

**4.2.1 Requirement.** Use of composite material systems for interior submarine applications requires Naval Sea Systems Command approval of the fire and toxicity performance for the composite material system. Compliance with this requirement is the responsibility of all Naval activities and contractors that provide products, equipments, or services in support of Naval submarine construction or operation. The Non-metallic Materials Branch of the Naval Sea Systems Command has approval responsibility for use of composite material systems on Naval vessels.

TABLE I. *Fire characteristics and tests.*

Fire test/characteristics	Requirement	Test method
Oxygen-temperature index	Minimum	Appendix A
Percent oxygen at 25 °C	35	
Percent oxygen at 75 °C	30	
Percent oxygen at 300 °C	21	
Flame spread (index)	Maximum 20	ASTM E 162

## MIL-STD-2031(SH)

TABLE I. *Fire characteristics and tests – Continued.*

Fire test/characteristics	Requirement	Test method
Ignitability (seconds)	Minimum	ASTM E 1354
100 kW/m <sup>2</sup> irradiance	60	
75 kW/m <sup>2</sup> irradiance	90	
50 kW/m <sup>2</sup> irradiance	150	
25 kW/m <sup>2</sup> irradiance	300	
Heat release (kW/m <sup>2</sup> )	Maximum	ASTM E 1354
100 kW/m <sup>2</sup> irradiance		
Peak	150	
Average 300 seconds	120	
75 kW/m <sup>2</sup> irradiance		
Peak	100	
Average 300 seconds	100	
50 kW/m <sup>2</sup> irradiance		
Peak	65	
Average 300 seconds	50	
25 kW/m <sup>2</sup> irradiance		
Peak	50	
Average 300 seconds	50	
Smoke Obscuration	Maximum	ASTM E 662
D <sub>max</sub> occurrence	200 seconds	
Combustion gas generation	Maximum	ASTM E 1354
At 25 kW/m <sup>2</sup> irradiance		
CO	200 p/m <sup>2</sup>	
CO <sub>2</sub>	4 percent by volume	
HCN	30 p/m	
HCL	100 p/m	
Burn-through fire test	No burn-through in 30 minutes	Appendix B
Quarter-scale fire test	No flashover in 10 minutes	Appendix C
Large scale open Environment test	Pass	Appendix D
Large scale pressurizable Fire test	Pass	Appendix E
N-Gas Model smoke Toxicity screening test	No deaths Pass	Appendix F

<sup>1</sup>Kilowatt per square meter (kW/m<sup>2</sup>).<sup>2</sup>Parts per million (p/m).

## MIL-STD-2031(SH)

**4.2.2 Procedures.** Organizations having a composite material system application not previously approved by the Naval Sea Systems Command shall initiate the qualification procedure through direct contact or via a letter request to the Non-metallic Materials Branch. The request should include information on the planned composite material system application, chemical composition information, and benefits of the composite material system application. The application will be reviewed by the cognizant engineering group in NAVSEA. If the proposed request has merit, written qualification procedure guidance for fire testing will then be provided to the initiating organization. The results obtained from the tests conducted according to the methods of paragraph 4.1 will be used in performing a risk/benefit analysis inherent in the development of any new equipment and system. When the analysis is complete the requesting official will be notified of the acceptance or use category for the proposed application.

**4.2.3 Data package.** All test data and observations resulting from tests conducted by methods given in paragraph 4.1, shall be submitted to the Naval Sea Systems Command (see 6.3).

**4.2.4 Certification for use.** Test data and observations from these test methods and other information submitted to the Naval Sea Systems Command in support of the qualification procedure will be used in certifying a composite material system for the proposed application. The Naval Sea Systems Command will certify a composite material system prohibited and not permitted for any submarine application when warranted by the test data or analyses. The Naval Sea Systems Command will certify the composite material system is fit for use in specific limited applications based on test results and safety analysis. Composite material systems currently used in naval submarines are exempt from this qualification procedure for that specific application.

## 5. DETAILED REQUIREMENTS

**5.1 Test responsibility.** Unless otherwise specified (see 6.2), the supplier of the application item to the Navy shall be responsible for submitting the required data and information resulting from the tests required and requesting certification of the composite system for that application. The government reserves the right to perform independent tests where deemed necessary to ensure conformance with the prescribed requirements.

**5.2 Specimen requirements.** Unless otherwise specified by NAVSEA the specimen thickness shall be the minimum thickness intended for use of the composite material or as a composite material system. The minimum thickness of a composite material system permitted by this standard is 1/8 inch (3.18 mm). Material system characteristics shall be preserved when shape must be changed to fit into test apparatus. As a minimum, material characteristics to be preserved are chemical composition, geometric relationships between constituent materials in a composite layer, and stacking sequence for layers within the bulk structure, and coatings, cladding or additives, if appropriate. Edges which are not exposed in service shall be protected during testing when possible. To avoid the need for additional testing, test specimens shall be representative of any expected variations in the manufacturing process.

## MIL-STD-2031(SH)

**5.3 Retest requirements.** Once a composite material system has been certified by NAVSEA any change to the minimum material characteristics, as defined by the specimen requirements, shall be retested by the supplier for recertification by NAVSEA. The extent to which incremental tests are required will be determined by NAVSEA.

**5.4 Test facilities.** The contractor shall notify NAVSEA where the tests specified will be conducted. Except as otherwise directed, contractors may use their laboratories or other laboratories to perform the tests.

## 6. NOTES

(This section contains information of a general or explanatory nature that may be helpful, but is not mandatory.)

**6.1 Intended use.** The test methods and qualification procedures provided in this standard are intended to ensure that all information needed by Naval Sea Systems Command to certify a composite material system to be used aboard Naval submarines is developed.

**6.2 Implementation guidance.** When this standard is invoked, the following should be specified:

- a. Title, number, and date of this standard
- b. Issue of DODISS to be cited in the solicitation, and if required, the specific issue of individual documents referenced (see 2.1)
- c. When fire characteristics and test methods are other than as specified (see 4.1)
- d. When test responsibility is other than as specified (see 5.1).

**6.3 Consideration of data requirements.** The following data requirements should be considered when this standard is applied on a contract. The applicable Data Item Descriptions (DID's) should be reviewed in conjunction with the specific acquisition to ensure that only essential data are requested/provided and that the DID's are tailored to reflect the requirements of the specific acquisition. To ensure correct contractual application of the data requirements, a Contract Data Requirements List (DD Form 1423) must be prepared to obtain the data, except where DOD FAR Supplement 27.475-1 exempts the requirement for DD Form 1423.

Reference Paragraph	DID Number	DID Title	Suggested Tailoring
4.1 and Appendix G	D1-MISC-80653	Test reports	—

The above DID's were those cleared as of the date of this specification. The current issue of DOD 5010.12-L, Acquisition Management Systems and Data Requirements Control List (AMSDL), must be researched to ensure that only current, cleared DID's are cited on the DD Form 1423.

## MIL-STD-2031(SH)

### 6.4 Subject term (key word) listing.

Combustion gases  
Fire induced gases  
Fire limits  
Flammability  
Ignitability  
Pyrolysis  
Toxicity

## MIL-STD-2031(SH)

## APPENDIX A

STANDARD OPERATING PROCEDURE FOR OXYGEN-  
TEMPERATURE INDEX PROFILE

## 10. SCOPE

**10.1 Scope.** This method describes a procedure to measure the minimum oxygen concentration at temperatures from ambient, 25 degrees Celsius ( $^{\circ}\text{C}$ ) (75 degrees Fahrenheit ( $^{\circ}\text{F}$ )), to an elevated temperature, 300  $^{\circ}\text{C}$  (570  $^{\circ}\text{F}$ ), in a flowing mixture of oxygen and nitrogen that will just support flaming combustion. This procedure modifies ASTM D 2863 to measure the oxygen index at elevated temperatures. This appendix is a mandatory part of the standard. The information contained herein is intended for compliance.

## 20. APPLICABLE DOCUMENTS

**20.1 Non-Government publications.** The following document(s) form a part of this document to the extent specified herein. Unless otherwise specified, the issues of the documents which are DOD adopted are those listed in the issue of the DODISS cited in the solicitation. Unless otherwise specified, the issues of documents not listed in the DODISS are the issues of the documents cited in the solicitation (see 6.2).

## AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

D 2863      Standard Test Method For Measuring The Minimum Oxygen Concentration to Support Candle - Like Combustion of Plastics (Oxygen Index)

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.)

(Non-Government standards and other publications are normally available from the organizations that prepare or distribute the documents. These documents also may be available in or through libraries or other informational services.)

## 30. EQUIPMENT

**30.1 Equipment.** The Stanton-Redcroft FTA/HFTA apparatus or its equivalent shall be used for this test. The procedure described is for the Stanton-Redcroft device.

## MIL-STD-2031(SH)

**40. PROCEDURE**

**40.1 Preparation.** In accordance with the manufacturer's operating instructions, the apparatus shall be prepared for normal operation. Necessary connections to compressed gas supplies shall be made, checking for gas-tight connections, and electrical power shall be connected. Checking for sufficient fume hood draft to remove smoke and combustion product resulting from the test shall be performed.

**40.2 Specimens.** The specimens for testing shall be cut to size in advance. The normal size is 1/4 to 1/2 inch wide, 1/8 inch thick, and 6 inches long for self-supporting polymers and composite materials. Unless some special characteristic of the material that would invalidate the test results dictates special preparation (for example, a high moisture content), the samples are stored at room atmosphere and tested as received.

**40.2.1 Ignition.** If the material to be tested can be ignited readily by a 1 inch, normally adjusted Bunsen burner flame, the initial oxygen concentration should be set at 18 percent. If the material is difficult to ignite under these conditions, the initial oxygen concentration should be set at 25 percent. Make the necessary flow valve adjustments to maintain a total flow of 18 liters/minute.

**40.3 Calibration of oxygen analyzer.** Calibration shall include the following:

- a. *Position.* With the meter range set to the 100 percent scale, select position B1 and the meter should read 90-100 percent. This position has no adjustment.
- b. *Position B2.* Select position B2; the meter should read 90-100 percent; adjust as necessary.
- c. *Zero position.* Select the amplifier zero (AMP ZERO) position, and should read 0-10 percent. Adjust as necessary, N.B. This adjustment is extremely sensitive, proceed with caution.
- d. *Adjustment.* If adjustment was required, all settings should be rechecked to ensure that the required conditions have been established.
- e. *Pressure regulators.* Set the pressure regulators on the oxygen and nitrogen supply to 25-30 pounds per square inch (lb/in<sup>2</sup>).
- f. *Nitrogen.* Commence nitrogen flow, adjust for an overall flow rate of 18 liters/minute on the FTA module. Check to see that the oxygen analyzer flow rate registers approximately 100 milliliters per minute (mL/m); adjust as appropriate. The oxygen analyzer panel meter should give a steady reading of ZERO percent on both the 25 and 100 percent scales. The DVM should also register ZERO; adjust as appropriate. Cease nitrogen flow.

## MIL-STD-2031(SH)

- g. *Oxygen.* Commence oxygen flow, adjust for an overall flow rate of 18 liters/minute on the FTA module. Select the 100 percent range on the oxygen analyzer panel meter. After a short time, the steady reading should be 100 percent; the DVM should also register 100 percent. Adjust as required to ensure agreement on the panel meter and DVM. Cease oxygen flow. The calibration of the oxygen analyzer and adjustment of the DVM readout are now complete. The DVM will register oxygen concentrations between 0-100 percent.

**40.4 Determination of oxygen index at ambient temperature.** Determination of oxygen index at ambient temperature shall include the following:

- a. *Atmosphere.* Remove the chimney and place the specimen in the specimen holder. Replace the chimney and allow 30 seconds for the system to purge and establish the atmosphere surrounding the specimen.
- b. *Ignition.* Ignite the top of the specimen with the flame of an appropriate burner. Usually, a standard 14 ounce (oz) propane cylinder with narrow flame tip is used as the ignition source. When the specimen is well lit, remove the burner and start the timer to record burning time.
- c. *Criteria.* Observe the burning of the specimen. If the specimen burns longer than 3 minutes or burns beyond a length of 50 millimeters (mm), the oxygen concentration is too great and must be reduced. Select either criterion; time or burn length, depending upon the behavior of the material.
- d. *Calculations.* Once the proper mixture of oxygen and nitrogen has been obtained, 5 to 10 specimens shall be tested, to permit calculation of an average value of the oxygen index for the material under examination.

**40.5 Determination of oxygen index at elevated temperature.** Determinations at elevated temperatures are carried out in the same manner as the ambient temperature measurements except that the combination of the FTA and HFTA modules shall be used and the HFTA overall gas mixture flow rates may be varied with operating temperature to achieve constant flow velocity in the column surrounding the specimen.

**40.5.1 Gas flow rate.** The gas flow rate shall be adjusted to correspond to the desired initial oxygen index and test temperature. For submarine composite materials, three temperatures shall be of interest: 25, 75 and 300 °C (75, 165 and 570 °F). The corresponding flow rates shall be 9.7, 8.3, and 5.1 liters/minute for a 4 centimeter per second (cm/s) flow velocity. The flow of the oxygen-nitrogen mixture shall be halted and the air pump shall be started, with appropriate flow rate adjustment. The preheater and column heater controls shall be adjusted in accordance with the manufacturers instructions to attain the desired test temperature. When the test temperature has been reached, the air pump shall be shut off and oxygen-nitrogen flow shall be re-established.

## MIL-STD-2031(SH)

The following data, supplied by Stanton-Redcroft, shall give the temperature-flow rate relationship required to maintain a mean gas velocity of 4.0 cm/s about the test specimen.

Temperature		Flowmeter (L/min)
K	°C	
273	0	10.6
298	25	9.7
323	50	9.0
348	75	8.3
373	100	7.8
398	125	7.3
423	150	6.8
473	200	6.2
523	250	5.5
573	300	5.1
623	350	4.6
673	400	4.3

If the flow rate is held at 10.6 L/min throughout this temperature range, the mean gas velocity will vary between 4.0 cm/s at 0 °C (32 °F) and 9.9 cm/s at 400 °C (750 °F).

**40.5.2 Equilibrium.** The system shall be allowed to reach flow rate/column equilibrium. The test specimen, pre-mounted in its holder, shall be placed in the column with the aid of a long transfer handle or other appropriate device. The sample thermocouple level shall be set with the base of the test specimen and about 30 seconds shall be allowed for the sample to reach equilibrium with the column environment.

**40.5.3. Test initiation.** The specimen shall be ignited and the testing is conducted in a manner identical to the ambient temperature oxygen index determination.

**40.5.4 Average oxygen-index.** Once the proper oxygen-nitrogen mixture has been determined (equilibrium burning is sustained for 3 minutes or a 50 mm length) 5 to 10 specimens should be tested to permit the calculation of a average value of the oxygen index at the temperature level of interest.

**40.5.5 Required measurements.** Measurements of oxygen index shall be repeated until values have been obtained at 25 (ambient), 75, and 300 °C (75, 165 and 570 °F).

**40.5.6 Temperature index profile.** The average oxygen index values shall be graphed against temperature, and the best representative curve shall be plotted through the data points to construct the temperature index profile.

## MIL-STD-2031(SH)

**40.5.7 Additional data.** Should the measured oxygen index at 300 °C be lower than 21 percent, the oxygen index shall also be measured at 200 °C (390 °F). This additional data will help to define the temperature index profile more completely.

### 50. REPORT

**50.1 Precision of measurement.** Precision of measurement may be determined statistically, in accordance with ASTM D 2863, if the material is sufficiently well behaved under the experimental conditions of the test. From a practical standpoint, for specimens from the same sample, agreement to within 0.5 percent oxygen is possible; for specimens from different samples, agreement within 1.0 percent oxygen is possible. By the very nature of the materials under examination, it is unwise to expect too great a precision of measurement unless the material is of the nature of a "secondary standard" where behavior of the material is predictable. Poly(methylmethacrylate), PMMA, is such a secondary standard for oxygen index measurements. The expected oxygen index range for PMMA, under ambient conditions, 17.0-17.5 percent. PMMA is useful for determination of instrument performance and for initial training of technical personnel.

**50.2 Report contracts.** Test reports shall include the following:

- a. Nomenclature and code markings to identify the material evaluated and the manufacturer
- b. Dimensions of the test specimens
- c. Testing conditions of the oxygen index measurements
- d. Individual oxygen index values, corresponding temperatures, and the average oxygen index value for the particular set of measurements
- e. Tabular and graphic representations of the temperature index profile
- f. A description of any unusual specimen behavior such as prolonged glowing combustion after flameout, charring, dripping, bending, erratic burning, or excessive smoke evolution.

## MIL-STD-2031(SH)

## APPENDIX B

## DAVID TAYLOR RESEARCH CENTER BURN-THROUGH FIRE TEST

## 10. SCOPE

**10.1 Scope.** This test method describes a procedure to determine the time to burn through materials subjected to a temperature of approximately 1095 °C (2000 °F) for 1 hour under a controlled laboratory fire exposure. This is a temperature which may be experienced by direct flame impingement or from high heat fluxes associated with fluid-hydrocarbon fueled pool fires. The results from this test can be used to compare material performance in containing and preventing the propagation of fire, smoke, and fire gases between compartments or spaces. This appendix is a mandatory part of the standard.

## 20. APPLICABLE DOCUMENTS.

This section is not applicable to this appendix.

## 30. EQUIPMENT

**30.1 Test stand assembly.** A test stand assembly containing a test stand and a fire chamber shall be constructed to the dimensions shown in figure 1. A test stand assembly shall consist of the following:

**30.1.2 Mounting.** The test stand has a steel mounting plate approximately 4 by 4 feet which simulates a bulkhead and will allow a material specimen to be bolted to the plate for test and allow the fire chamber to be attached to control the fire exposure temperature.

**30.1.2 Fire chamber.** The fire chamber is a steel plate box approximately 3 feet wide, three feet deep, and 3 feet long. A 12 by 12 inch opening on one side serves as an air inlet and viewing port. The rear of the box is open for maintaining and mounting a propane burner. The burner shall be mounted 18 inches from the sample to be tested. A sliding plate door on top of the fire chamber is used to adjust air flow and temperature on the specimen.

**30.2 Propane burner.** A propane burner capable of providing 500,000 British Thermal Units per hour with a fuel source of at least 1-hour duration shall be used. The temperature at the face of the specimen shall be 1095 °C ± 110 °C (2000 °F ± 200 °F).

**30.3 Thermocouples.** The temperature of the flame and non-flame faces of the material specimen shall be measured using inconel-sheathed, Type K, 18-gauge chromel-alumel thermocouples (TCs). The time constant of the TC assemblies shall be less than 60 seconds and the TC accuracy shall be ±0.75%. At least eight TCs shall be used, two on the fire side and six on the non-fire side of the test specimen.



## MIL-STD-2031(SH)

**30.4 Filler insert.** A noncombustible material filler insert 1/2 inch thick shall be inserted between the steel mounting plate and specimen to provide a seal from fire gas flow to the edges and back face of the material specimen.

**40. PROCEDURE**

**40.1 Mounting.** Mount and bolt the 2- by 2-foot material specimen and filler insert to the steel mounting plate so that the 16-1/2 inch opening is entirely covered and is airtight. See figure 2.

**40.2 TC Installation.** Install eight TCs in the following locations (figure 3):

- a. One TC 2 inches from the center of the burner tip
- b. One TC against the center of the material specimen on the fire side
- c. Three TCs placed vertically on the non-fire side of the material specimen along its vertical centerline. The upper and lower thermocouples should be approximately 1 inch below and above, respectively, the edge of the 16-1/2 inch opening
- d. One TC placed 1-1/2 inches directly behind the center thermocouple of the three vertically mounted thermocouples on the non-fire side of the material specimen
- e. One TC placed 4 inches to the right of the bottom TC of the three vertically mounted thermocouples on the non-fire side of the material specimen
- f. One against the steel mounting plate 2 inches above the material specimen on the non-fire side.

**40.3 Fire chamber.** The fire chamber shall be attached to the test stand and tighten nuts on the test stand bolts. The propane burner tip shall be 18 inches from the material specimen.

**40.4 Burner temperature.** The burner and monitor shall be lit such that the temperature, as measured by the TC on the material specimen fire side, reads approximately 1095 °C (2000 °F) throughout the test. The airflow in the fire chamber shall be preset using a blank plate insert for the material specimen to obtain this temperature with the burner. The flame shall be applied to the material specimen for not less than 1 hour or until burn-through of the material occurs as demonstrated by flame penetration to the non-fire side of the material specimen.

MIL-STD-2031(SH)

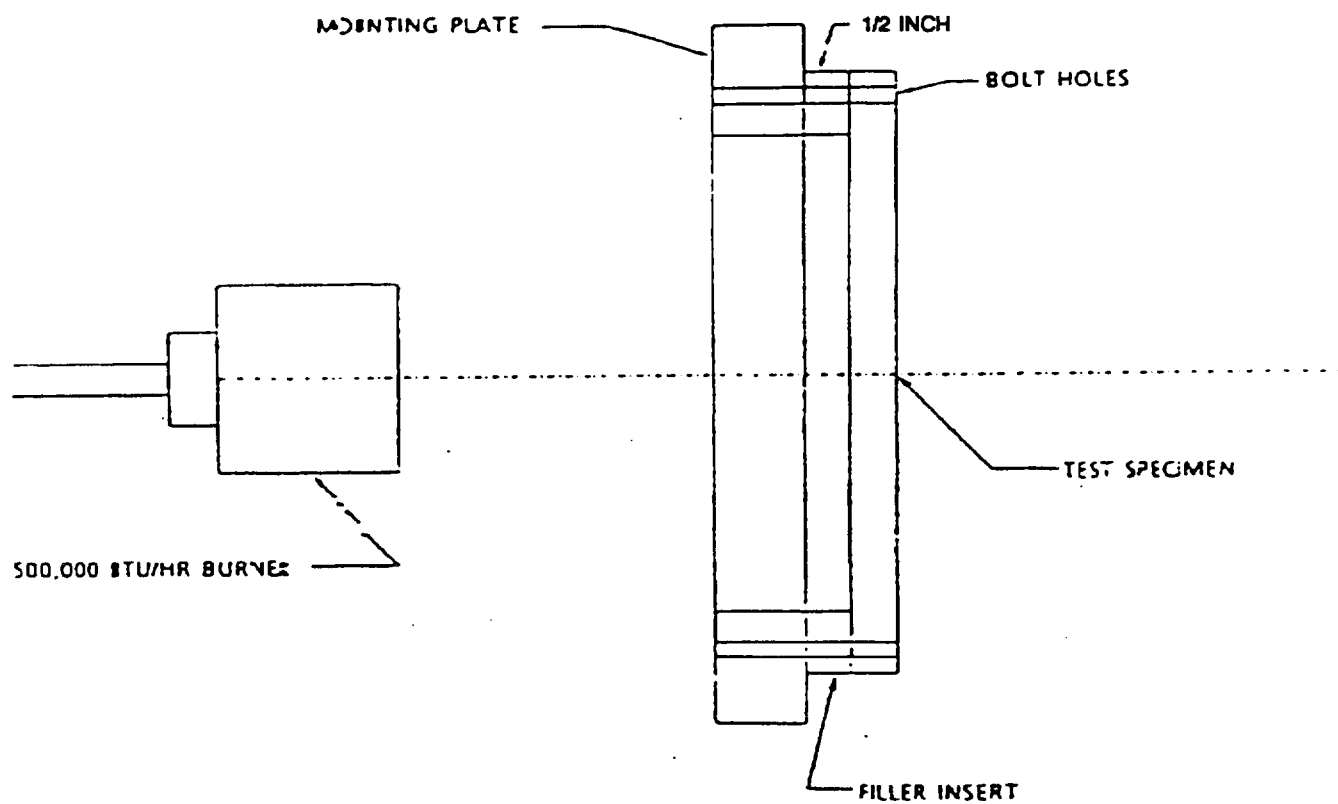
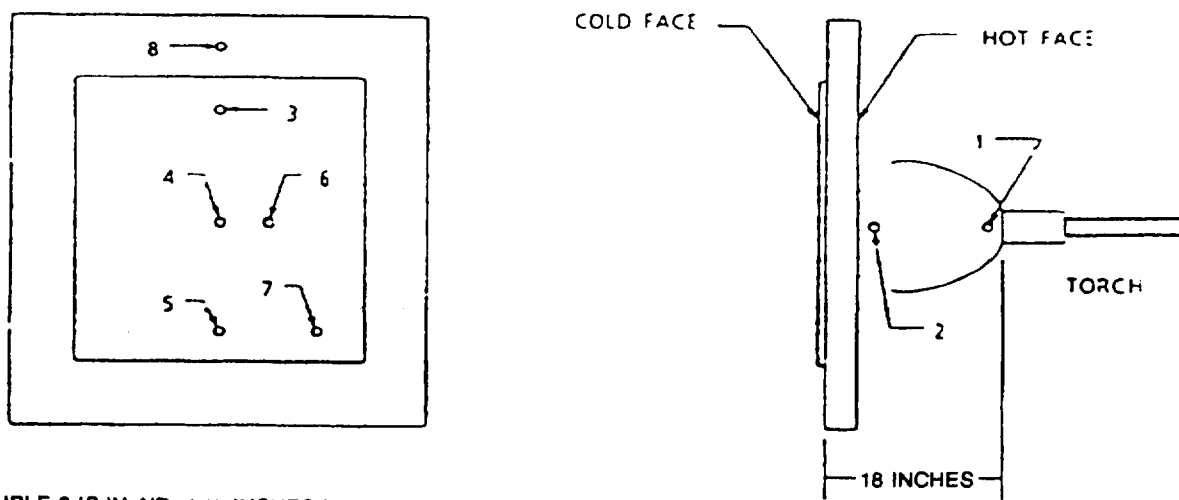


FIGURE 2. *Sample mounting.*

MIL-STD-2031(SH)



THERMOCOUPLE 6 IS IN AIR, 1-1/2 INCHES DIRECTLY BEHIND TC 4

FIGURE 3. Thermocouple locations.

## MIL-STD-2031(SH)

**40.5 Test data.** The test data from the eight TCs shall be recorded as a continuous function of time.

## 50. REPORT

**50.1 Report contents.** Test reports shall include the following:

- a. Sufficient nomenclature and code markings to identify the material evaluated and the manufacturer
- b. Time to smoke or fire gases (if they occur) are evolved from the non-fire side of the material specimen
- c. Time to burn-through material test specimen as evident by flame on the non-fire side. If burn-through does not occur within the 60 minutes, the time shall be reported as ">60 minutes".
- d. The time that the material specimen continues to burn on either side following removal of the propane burner source
- e. Tabular and graphic representations of the temperature profiles in 5 minute intervals for the three vertically mounted TCs and the TC mounted behind the center mounted TC. The temperature profile shall be recorded continuously and include temperatures during the test and for a period of 1 hour after the burner is removed
- f. Smoke generation (initial occurrence, amount, color, etc.)
- g. Visual inspection of fire damage to include any swelling, contraction, cracking, bulging, melting or dripping, separation of plies and effective material thickness remaining
- h. Visual record shall be obtained, for example video camera, to record the test set-up before starting the test, light-off of the propane burner with flame impingement on the material specimen, significant material destruction or fire gases on the fire side, and initial burn-through of the material specimen.

## MIL-STD-2031(SH)

## APPENDIX C

DETERMINATION OF THE FLASHOVER POTENTIAL OF A  
LINING MATERIAL USING A QUARTER-SCALE ROOM FIRE TEST

## 10. SCOPE

**10.1 Scope.** This method describes a procedure to determine the flashover potential of materials in a room when subjected to a fire exposure. The method described will yield a time from the introduction of the fire exposure until the moment of flashover. This appendix is a mandatory part of the standard. The information contained herein is intended for compliance.

**10.2 Justification.** In the interest of reducing cost and time associated with fire testing in a full size room (defined as a 10 by 10 by 8 feet high room having a 30 by 80 inch high doorway), a quarter-scale room fire test was devised to predict flashover potential of lining materials exposed to fire.

## 20. APPLICABLE DOCUMENTS

This section is not applicable to this appendix.

## 30. EQUIPMENT

**30.1 Quarter-scale room.** The quarter-scale room shall be constructed from a suitable ceramic insulation board and shall form an airtight box having a ceiling and four sides. The box shall be placed on a floor fabricated with the same material. The interior dimensions of the fully lined quarter-scale room shall be 30 by 30 by 24 inches high. The doorway is located at the center of one wall and shall be 19.5 inches wide and 17 inches high to secure the proper ventilation and fire development. The height between the finished ceiling and the top of the doorway shall be 7 inches. The floor of the model room shall extend at least 12 inches outside of the doorway. The box shall be removable to allow for application of ceiling and wall covering. The entire base of the box in contact with the floor shall be made airtight.

**30.2 Fire source.** A porous plate diffusion flame burner shall be used as the fire source. The burner shall be 3.5 by 3.5 by 3 inches high, consisting of a horizontal porous plate area of 3 by 3 inches with a .25 inch wide steel plate perimeter and steel plate sides and bottom.

**30.3 Thermocouples.** Four 10 mil chromel-alumel thermocouples shall be used, 1 inch and 3 inches below the center of the overhead and 1 inch and 2 inches below the top of the doorway.

## 40. PROCEDURE

**40.1 Material.** The test material shall fully line the walls and ceiling.

## MIL-STD-2031(SH)

**40.2 Temperature and humidity.** Prior to testing, the full-lined test room shall be conditioned for at least 24 hours at a relative humidity between 20 and 60 percent, and a temperature of  $23 \pm 5$  °C ( $73 \pm 10$  °F).

**40.3 Fire conditions.** The fire source shall be positioned on the floor snugly against one near corner of the test room. A flow rate of 0.64 cubic feet per minute ( $\text{ft}^3/\text{m}$ ) methane shall be used to produce a constant heat input to approximately 640 BTU per minute for the duration of the test.

**40.4 Thermocouple data.** The test data from the four thermocouples shall be recorded as a continuous function of time.

**50. REPORT**

**50.1 Report contracts.** Test reports shall include the following:

- a. Sufficient nomenclature and code markings to identify the material evaluated and the manufacturer
- b. Time to flashover, if it occurs. Flashover is characterized by thermal flux levels equal to or greater than 2 watts per square centimeter at the floor level. This corresponds to interior temperatures of 600 °C (1110 °F) or one of the doorway measurements reaching 500 °C (930 °F), whichever occurs first. Flashover shall not occur within 10 minutes.
- c. ~~The maximum temperature recorded and the time to reach that temperature if flashover is not achieved~~
- d. A video record shall be taken showing the test set-up, the point of maximum involvement and after the fire has been extinguished.

## MIL-STD-2031(SH)

## APPENDIX D

## LARGE SCALE OPEN ENVIRONMENT FIRE TEST

## 10. SCOPE

**10.1 Scope.** This method describes a procedure to test materials at full size in their intended application under a controlled laboratory fire exposure to determine fire tolerance or ease of extinguishment. The objective is to conduct tests to allow comparison of a new material application against the existing material application under the perceived fire threat. The intended application shall be tested under all the operating scenarios for the application; for example, in piping these conditions would include dry piping, stagnant fluid in the piping, and flowing fluid in the piping. Data shall be collected for fire growth, smoke generation, and combustion product toxicity in a large scale environment. The test data can be used to confirm small scale test results. This appendix is a mandatory part of the standard. The information contained herein is intended for compliance.

## 20. APPLICABLE DOCUMENTS

This section is not applicable to this appendix.

## 30. EQUIPMENT

**30.1 Fire test cell.** As a minimum, a 20- by 20-foot enclosure of at least 3600 cubic feet shall simulate a shipboard compartment. The fire compartment enclosure, preferably steel, must be able to withstand temperatures up to 1095 °C (2000 °F) for the 30 minutes duration of the test. The base of the test cell shall be a noncombustible material such as concrete. The fire test cell shall have the following capabilities:

- a. A method to measure fuel consumption by the volume of fuel being added to the fire pan or a weight load cell if no fuel is to be added.
- b. An exhaust system which allows collection of all products of combustion for oxygen consumption measurement/analysis. The exhaust system is also necessary for environmental considerations. The David Taylor Research Center (DTRC) fire test cell or equal shall use a water spray in the lower exhaust duct to create air flow and to settle out smoke particulate.
- c. A drainage system that shall drain the water and fire products released from the exhaust duct and to drain the base of the test cell of extinguishment and ancillary fluids is needed. Holding tanks for the drained fluids shall be used.
- d. A gas sampling arrangement to sample the fire gas products for at least CO, CO<sub>2</sub>, O<sub>2</sub>, HCN and HCl

## MIL-STD-2031(SII)

- e. A smoke obscuration measuring system is recommended but optional.
- f. The test cell should have available connections and ancillary equipment to provide fluids and air flow to enable the material application to be operated in normal modes. These capabilities do not have to be made available in all cases, but the design of the test cell shall include the requirements. A steel sided test cell is easier to alter when adding additional ancillary services.

**30.2 Fuel supply.** The fuel supply shall include the following:

- a. *Fuel.* For structural tests the use of a clean burning fuel such as heptane or hexane is recommended. For fire extinguishment tests, fuels found in the submarine such as diesel fuel or 2190 TEP hydraulic fluid should be utilized.
- b. *Fire pan.* A stainless steel all welded construction pan sized for the fire shall be used. It should have connections to add fuel and to maintain the water base layer during burning operations.
- c. *Fuel supply system.* A fuel pump (manual or automatic) with a flow meter shall provide a continuous flow of fuel from the supply containers to the fire pan.
- d. *Water base layer.* A continuous supply of water to the fuel pan shall maintain the water base layer.

**30.3 Extinguishment.** For safety purposes a charged fire hose shall be available during all fire tests. Standard submarine extinguishing systems shall be used for ease of extinguishment tests. The following existing extinguishment systems shall be used: 2 1/2 gallon AFFF extinguisher, 18 pound PKP extinguisher, 30 pound CO<sub>2</sub> extinguisher, and the Navy all purpose nozzle with 30 lb/in<sup>2</sup> water pressure.

**30.4 Thermocouples.** Temperatures of the flame and material application shall be measured using inconel-sheathed, type K, 18-gauge chromel-alumel thermocouples. The time constant of the thermocouple assemblies shall be less than 60 seconds. Thermocouple accuracy shall be plus or minus 0.75 percent. At least four thermocouples shall be utilized: one above the fire pan to measure the flame temperature, one in the overhead, one at the level of the material application in the flame, and one at the level of the material application outside the flame. Additional thermocouples shall be used as appropriate for the material application being tested such as internal to piping or ducting.

**30.5 Video system capability.** Video camera and equipment shall be used to record the test setup before test start, light off of the fuel pan, duration of the test and post fire damage shall be used. At least one of the cameras shall have a test time generator.

## MIL-STD-2031(SH)

**30.6 Digital data collection system capability.** A digital data collection system to continuously record thermocouple, flux meter, and radiometer data shall be used when available.

### 40. PROCEDURE

#### 40.1 Preparation.

**40.1.1 Cleaning.** The fire test cell shall be completely cleaned between fire tests.

**40.1.2 Test plan.** A detailed test plan for the fire test to include time sequences and data collection requirements shall be developed and rehearsed prior to initiation of the actual test.

**40.1.3 Calibration.** Calibration of the fuel supply system shall be confirmed and the burning rate of the fuel supply shall be calculated. When analysis of the combustion gases from the composite is required, the base level of combustion products formed from the fuel system shall be determined as a function of the time the fuel is burning.

**40.1.4 Ancillary services.** The material application and the ancillary services for the operating mode and failure sensing system shall be installed.

**40.1.5 Data and monitoring systems.** The data collection sensors and monitoring systems shall be installed and calibrated.

**40.1.6 Fire fighting equipment.** The fire fighting equipment shall be laid out and the fire hose component shall be tested.

#### 40.2 Procedure. Test procedure shall be as follows:

- a. Turn on test instrumentation
- b. Turn on fuel supply and water supply to the fire pan and establish flow and proper pressure
- c. Turn on the water spray system to the exhaust system
- d. With all data collection sensors operational and personnel at their stations, initiate the test by igniting the fire pan
- e. Maintain the established fuel flow and observe temperatures, fire plume, and ancillary services performance
- f. Record the test data continuously throughout the test

## MIL-STD-2031(SH)

- g. The test shall be conducted for 30 minutes or until the application fails. The fire may be extinguished by stopping the fuel pump and allowing the fire to subside or with the fire hose. When the material application fails either visually or by the sensing system, secure the fuel pump.

**50. REPORT****50.1 Report contents. Test reports shall contain the following:**

- a. Sufficient nomenclature and code markings to identify the material evaluated and the manufacturer
- b. The time-temperature curve on the fire from the overhead thermocouple and from the thermocouple outside the plume by the application
- c. Failure time of the material application being tested. If failure does not occur, the time is reported as " >30 minutes ".
- d. The fuel consumption during the fire
- e. Continued burning of the material after the fire pan is extinguished
- f. Visual inspection of fire damage to include any swelling, contraction, cracking, bulging, melting or dripping, separation of plies and effective material thickness remaining
- g. Extinguishment test data (if run)
  - (1) Pre-burn time
  - (2) Time to extinguish
  - (3) Reflash (time)
  - (4) Time to extinguish reflash
  - (5) Submarine extinguishing system used.
- h. When required, gas sampling (if collected) data for CO, CO<sub>2</sub>, O<sub>2</sub>, HCN, and HCl
- i. When required, smoke obscuration data (if collected)
- j. Total heat release by the material application if an oxygen consumption capability exists
- k. Additional observations, if any.

## MIL-STD-2031(SH)

## APPENDIX E

## LARGE SCALE PRESSURIZABLE FIRE TEST

## 10. SCOPE

**10.1 Scope.** This method describes a procedure to test materials in their intended application in a simulated submarine environment under a controlled laboratory fire exposure. The results from the test method shall be used to verify material performance under the effects of an enclosed environment and relative location within the submarine. The test shall provide data for fire growth, smoke generation, and combustion product toxicity in a large-scale pressurized environment. The test data shall also be used to confirm small scale test results. This appendix is a mandatory part of the standard. The information contained herein is intended for compliance.

## 20. APPLICABLE DOCUMENT

## 20.1 Government documents.

**20.1.1 Other Government documents, drawings, and publications.** The following other Government documents, drawings, and publications form a part of this document to the extent specified herein. Unless otherwise specified, the issues are those cited in the solicitation.

## DOCUMENTS

## NAVAL RESEARCH LABORATORY (NRL)

NRG 8643

Large-Scale Pressurizable Fire Test Facility – Fire I (DTIC  
No. ADA 124288)

(Application for copies should be addressed to the Naval Publications and Forms Center, (Attn: NPODS), 5801 Tabor Avenue, Philadelphia, PA 19120-5099.)

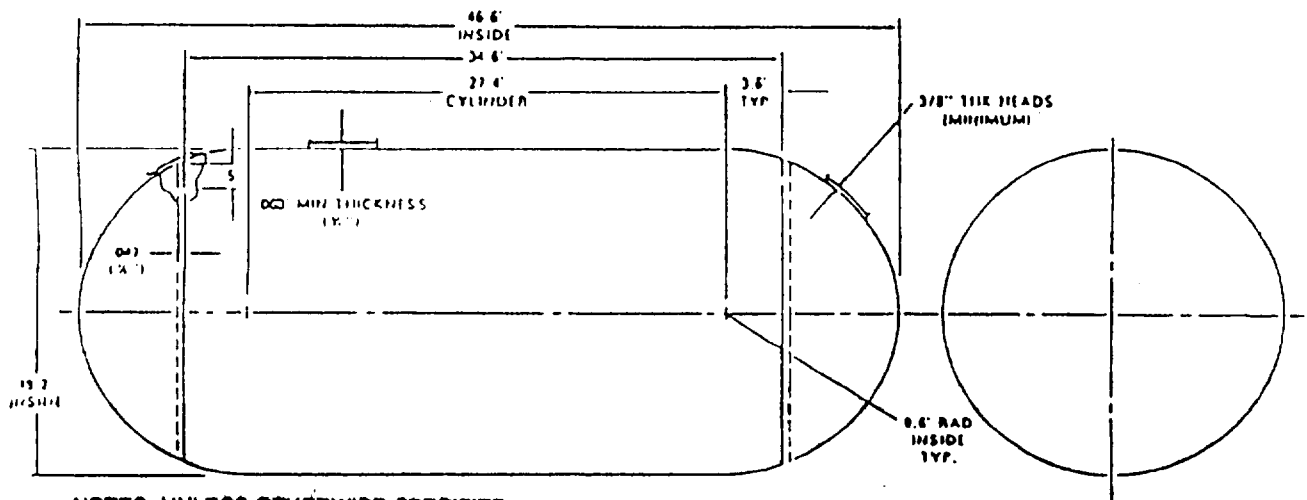
## 30. EQUIPMENT

**30.1 Chamber.** A large (10,000 cubic feet or greater) cylindrical chamber with hemispherical sections on the ends, capable of being pressurized to 618 kilopascal (kPa) (89.7 lb/in<sup>2</sup>) and withstanding 230 °C (450 °F) shall be used. The chamber shall be of sufficient diameter to allow mock-up of at least two separate levels of a submarine. Figure 4 provides configuration and dimensions for the Naval Research Laboratory's pressurizable chamber – Fire I. The chamber should have the following capabilities:

- a. A pressure rated access door at least 3 feet wide by 6 feet high to provide entry for reconfiguration materials and test materials

## MIL-STD-2031(SH)

## APPENDIX E



## NOTES: UNLESS OTHERWISE SPECIFIED

1. ALL DIMENSIONS IN FEET
2. MATERIAL: ASTM 285 GRADE C STEEL
3. FINISH: PRIME COAT OUTSIDE SURFACE & PAINT
4. CONTRACTOR TO PRESSURE TEST PER ASME. CODE FOR 78 LB/IN<sup>2</sup> WORKING PRESSURE INTERNAL AT NRL
5. INSTALLATION AT NRL
6. TANK VOLUME:  
     SPHERE 3.706 CU FT  
     CYLINDER 7.933 CU FT  $\pm$  3%  
     TOTAL 11.639 CU FT  $\pm$  3%
7. EXTERIOR LIFTING LUGS OPTIONAL
8. WELDED CONSTRUCTION

FIGURE 4. Pressurizable vessel dimensions.

## MIL-STD-2031(SH)

- b. A venting system at the top of the chamber to enable reduction in pressure within the chamber
- c. A drain system at the bottom of the chamber to enable the removal of water and other fire extinguishing fluids
- d. An inert gas (nitrogen) pressurization system to enable fire suppression when required
- e. Visual observation view ports to enable videotaping and test monitoring
- f. Instrumentation ports to provide feed-throughs for electric sensing and pressure measurements within the vessel
- g. A rupture disk assembly for the chamber is recommended
- h. A chamber electronic control system, a gas sampling system, and closed-circuit television.

**30.2 Liquid fuel supply system.** A specialized liquid fuel supply system shall be used when available to provide for steady burning rates and measured fuel use rates. Such a system shall include an array of tapered-edge fire pans with various cross-sectional areas and a constant-level, liquid fuel supply system which allows the measure of fuel-loss (burning-rate) history. For test of materials a single measured supply such as a 5-gallon pan of n-heptane shall be used.

**30.3 Thermocouples.** Temperatures in the chamber shall be monitored with thermocouple arrays in each end of the chamber. These thermocouples shall have chromel-alumel wires with diameters of 0.2 mm and have ceramic insulation enclosed in 304 stainless steel jackets 1 mm in diameter. Thermocouple arrays for sensing temperatures in the vicinity of the test material application shall be included as required to properly record test results.

**30.4 Radiometers.** Wide and narrow angle radiometers installed on a tree stand shall be used to obtain radiation measurements in the vicinity of the test material application.

**30.5 Laser/detector system.** A laser/detector system or systems shall be used to measure optical smoke density in various locations and to correlate with the smoke particle analyzers.

**30.6 Particle analyzer system.** A filtration system shall be used to measure smoke particle mass concentration at 2 minute intervals.

**30.7 Gas analysis system.** A continuous type gas analyzer system shall be used to monitor the concentrations for O<sub>2</sub>, CO, and CO<sub>2</sub>. A grab sampling system is used to collect and store for future analysis the concentrations for total hydrocarbons, O<sub>2</sub>, CO, CO<sub>2</sub>, HCl, and HCN.

## MIL-STD-2031(SH)

**40. PROCEDURE**

**40.1 Cleaning.** The large-scale pressurizable fire test chamber shall be completely cleaned between fire tests.

**40.2 Test Plan.** A detailed script for the fire test to include time sequences and data collection requirements shall be developed and rehearsed prior to the initiation of the actual test.

**40.3 Calibration.** Calibration of the fuel supply system shall be confirmed.

**40.4 Burning rate.** The burning rate of the fuel shall be calculated.

**40.5 Mock-up.** Submarine configuration mock-up structures and equipments shall be installed.

**40.6 Test material installation.** The test material application shall be installed.

**40.7 Data collection installation.** Data collection sensors, monitoring systems, and the atmospheric analyzers shall be installed and calibrated.

**40.8 Test initiation.** With data collection sensors operational and personnel at their station, the test shall be initiated by igniting the fuel supply.

**40.9 Test data.** The test data shall be recorded continuously throughout the test.

**40.10 Extinguishment.** The test shall be secured by fuel supply burnout, chamber nitrogen extinguishment system, or by a planned extinguishment test procedure.

**50. REPORT**

**50.1 Report contents.** Test reports shall contain the following information:

- a. Sufficient nomenclature and code markings to identify the material evaluated and the manufacturer
- b. Temperatures in both °C and °F
- c. Radiometer response in watts per square centimeter( $W/cm^2$ )
- d. Pressure response of the test chamber in  $lb/in^2$  absolute
- e. Gas concentrations of total hydrocarbons as grams per cubic centimeter ( $g/m^3$ );  $O_2$ ,  $CO$ , and  $CO_2$  as percentage by volume and  $HCl$  and  $HCN$  as p/m

MIL-STD-2031(SH)

- f. Smoke production shall incorporate the following: average particle diameter micro meters (  $\mu$ m ), aerosol density in grams per cubic meter ( $\text{g}/\text{m}^3$ ), calculated number density ( $\text{particles}/\text{cm}^3$ ), visual range in meters (m), smoke particulate mass generation rate in grams per second ( $\text{g}/\text{s}$ ), and total mass of particulates in grams (g).
- g. Fire damage estimate shall be reported as percent of the material area damaged or destroyed. Include visual inspection of the fire damage for swelling, contraction, cracking, bulging, melting or dripping, separation of plies and effective material thickness remaining.
- h. Fuel consumption during the fire
- i. The time of any continued burning of the material after the fire pan has been extinguished
- j. Additional observations, if any.

## MIL-STD-2031(SH)

## APPENDIX F

## N-GAS MODEL SMOKE TOXICITY SCREENING TEST

## 10. SCOPE

**10.1 Scope.** This laboratory test method shall determine whether the combustion products from a material are extremely or unusually toxic. It shall be based on a method prepared by the National Institute of Standards and Technology (NIST) from their experiments conducted with rats on the combined lethal concentration of the major fire gases. The test shall be for preliminary screening purposes to rapidly estimate toxicity and shall provide information on the gases responsible for toxicity. The test method shall assume that the toxicity of combustion products is determined by a small number of major fire gases. The procedure shall entail burning samples of test materials in one of three modes: flaming, non-flaming, and non-flaming/flaming transition. Analytical tests are first conducted without animals. The concentrations of a small set of gases are measured – CO, CO<sub>2</sub>, HCN, and low O<sub>2</sub>. Based on the N-Gas Model equations for this combination of gases, an LC<sub>50</sub> value is predicted for the material. Very low predicted LC<sub>50</sub> values indicate that a material has extreme toxicity. Tests are then conducted with laboratory rats at 80 percent of the estimated LC<sub>50</sub> value. If none of the animals die from a 30 minute exposure or within a 24 hour post-exposure period to these gases, the predicted LC<sub>50</sub> is considered usable for hazard assessment calculations and indicates that the combustion products do not contain an unusually toxic material. If any animals die, then other gases are present that interact with the initial set of four gases to affect the toxicity of the combustion products. This result means that further toxicity testing shall be necessary to identify any unknown toxicant and to determine a more precise LC<sub>50</sub> value for the material. The advantages of this screening test over current test methods that assess smoke toxicity are that it is rapid, economical, minimizes the use of animals as compared to "classical LC<sub>50</sub>" determinations, and provides information on the gases responsible for toxicity in addition to the potency of the combustion gases. This appendix is a mandatory part of the standard. The information contained herein is intended for compliance.

## 20. APPLICABLE DOCUMENTS

## 20.1 Government documents.

**20.1.1 Other Government documents, drawings, and publications.** The following other documents, drawings, and publications form a part of this document to the extent specified herein. Unless otherwise specified, the issues are those cited in the the solicitation.

## NATIONAL INSTITUTE OF HEALTH (NIH)

## Guide for the Care and Use of Laboratory Animals

(Application for copies should be addressed to the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.)

## MIL-STD-2031(SH)

**20.2 Non-Government publications.** The following document(s) form a part of this document to the extent specified herein. Unless otherwise specified, the issues of the documents which are DOD adopted are those listed in the issue of the DODISS cited in the solicitation. Unless otherwise specified, the issues of documents not listed in the DODISS are the issues of the documents cited in the solicitation.

## AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

D 1929 Standard Test Method for Ignition Properties of Plastics; (DOD adopted)

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.)

(Non-Government standards and other publications are normally available from the organizations that prepare or distribute the documents. These documents also may be available in or through libraries or other informational services.)

**30. EQUIPMENT**

**30.1 Combustion system.** A furnace with a controller capable of operating up to 800 °C (1470 °F) and controlled to plus or minus 10 °C ( $\pm 20$  °F) is required as shown in figure 5. The furnace shall be connected to the bottom of the exposure chamber under a stainless steel plate that contains a cooling coil through which cold water shall continuously run during the experiment (figure 6). Figure 7 shows the exposure chamber design with furnace attached. A quartz 1000-ml beaker, 9 cm inside diameter (id) by 15 cm high, is used to hold the material sample. The beaker with material to be tested shall be inserted into the furnace after the furnace has been connected to the exposure chamber.

**30.2 Chemical analysis.** Instruments capable of obtaining continuous measurements for O<sub>2</sub>, CO<sub>2</sub>, and CO shall be used. A non-dispersive infrared (IR) technique is suggested for CO and CO<sub>2</sub>.

**30.2.1 Range of measurement.** For carbon monoxide the specific instrument shall measure a range of 0–1 percent (1 percent equals 10,000 p/m) with an accuracy of 0.01 percent (100 p/m). For carbon dioxide the specific instrument shall measure a range of 0–10 percent with an accuracy of 0.15 percent (1,500 p/m). For the oxygen measurements, an instrument operating on the paramagnetic principle shall be used. The instrument shall measure a range of 0–25 percent with an accuracy of 0.1 percent. A non-continuous gas chromatographic sampling technique shall be used as alternative equipment as long as the same sampling accuracy for each gas is maintained. The measurements shall be made every two to three minutes.

MIL-STD-2031(SH)

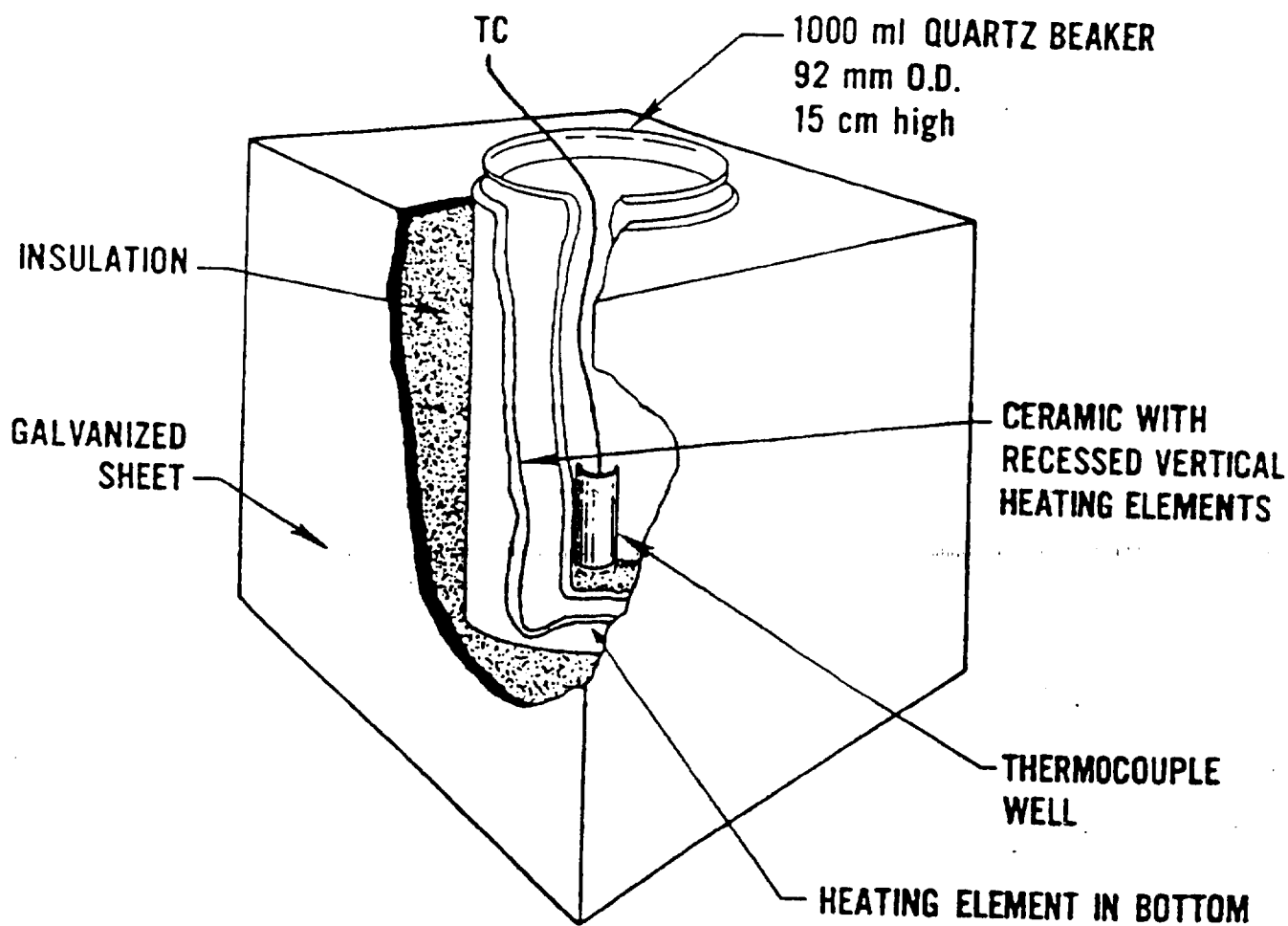


FIGURE 5. *Combustion furnace.*

MIL-STD-2031(SH)

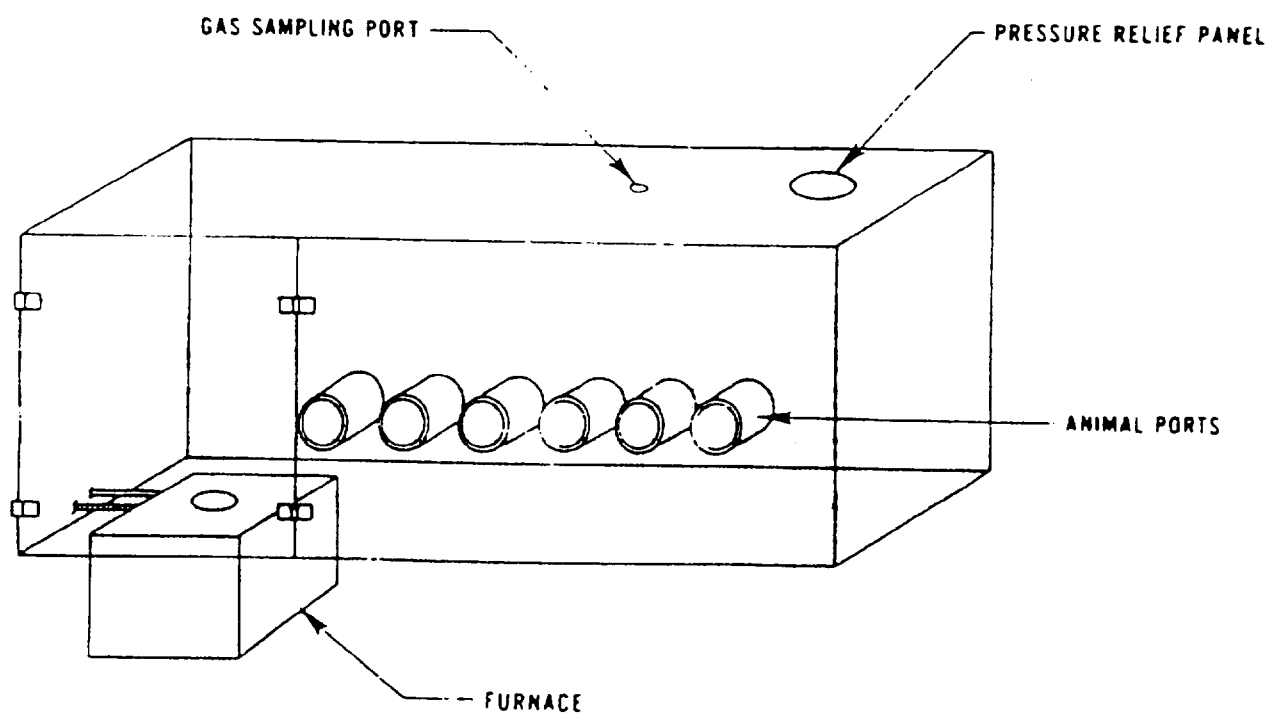


FIGURE 6. *Furnace mounted on chamber.*

MIL-STD-2031(SH)

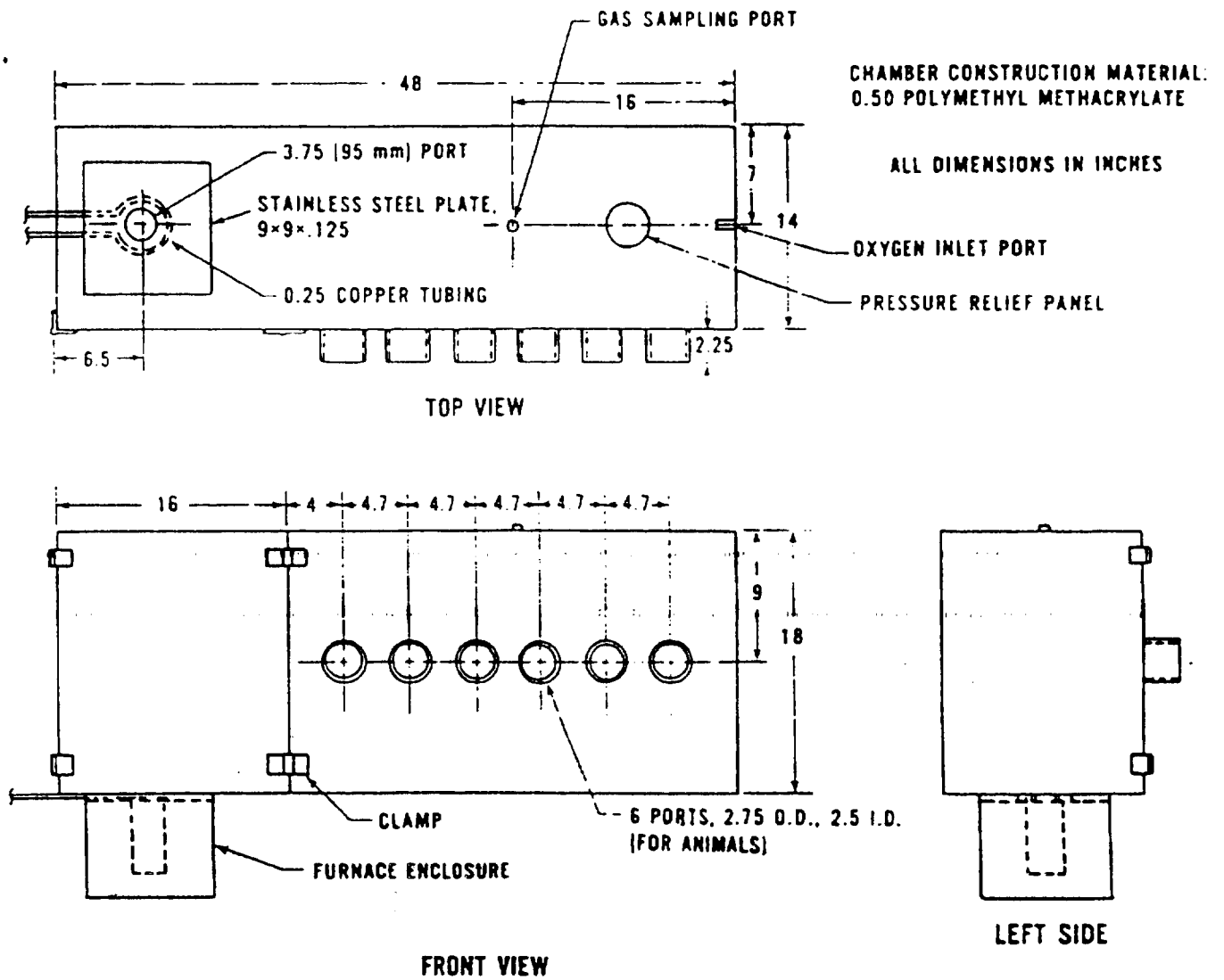


FIGURE 7. Exposure chamber design.

## MIL-STD-2031(SH)

**30.2.2 HCN Measurements.** For the HCN measurements a gas-tight syringe shall be used to obtain a sample every 3 minutes to be analyzed with a gas chromatograph equipped with a thermionic detector.

**30.2.3 Continuous monitoring.** Continuous monitoring of O<sub>2</sub>, CO<sub>2</sub>, and CO shall be accomplished by the removal of some of the products from the chamber. To provide concurrent monitoring of these gases, a gas sampling port, sample tubing and pump system shall be used to provide a flow of approximately 0.5 liters/min/gas. Two liters per minute shall be removed via the gas sampling port located at the animal nose level in the geometric center of the exposure chamber, analyzed and pumped back into the chamber (figure 8) at the left side of the chamber above the furnace.

**30.2.4 Toxicity levels.** Under some circumstances assessing the toxicity of generated gases instead of oxygen deprivation may be important. Oxygen shall be supplied to the exposure chamber as needed to maintain the concentration between 16–21 percent. If this is done, the total volume shall be corrected for this addition when calculating the mass loading of combustion products (chamber volume plus added volume at room temperature).

**30.2.5 Calibration.** Instrumentation for the measurement of CO, CO<sub>2</sub>, and O<sub>2</sub> shall be calibrated before each test using standard gas mixtures of a combination of CO, CO<sub>2</sub>, and O<sub>2</sub> in nitrogen. The return tubes shall be disconnected during calibration of the analytical instruments to prevent the inadvertent accumulation of calibration gases in the animal chamber.

**30.3 Temperature measurements.** Suitable temperature sensors shall be placed in the air close to the animals' noses. These instruments shall be measure temperature to 60 °C (140 °F). The environmental temperature of the chamber shall be recorded continuously during the 30 minute animal exposure.

**30.4 Animal exposure system.** A nominal 200 liter rectangular animal exposure chamber including the combustion furnace shall be used. The exposure chamber shall be made of 1.2 cm (0.5 inch) clear polymethylmethacrylate with inside dimensions of 122 x 36 x 46 cm (48 x 14 x 18 inches). Six animal ports shall be positioned as shown on figure 7 and shall be constructed of polymethylmethacrylate tubing 6.3 cm (2.5 inch) id having a 0.3 cm (1/8 inch) wall thickness. A blow-out panel shall be provided in the top of the exposure chamber on the right side away from the furnace to provide pressure relief in case of an explosion.

**30.4.1 Animal restrainers.** Animal restrainers designed to permit exposure of only the heads of the rats shall be used. Figures 9 and 10 provide details for a typical animal restrainer system.

**30.5 Animals.** Adult male rats weighing 225–300 grams that are 3–4 months of age shall be used. Fischer-344 rats or equivalent shall be used. Healthy animals shall be used in testing.

## MIL-STD-2031(SH)

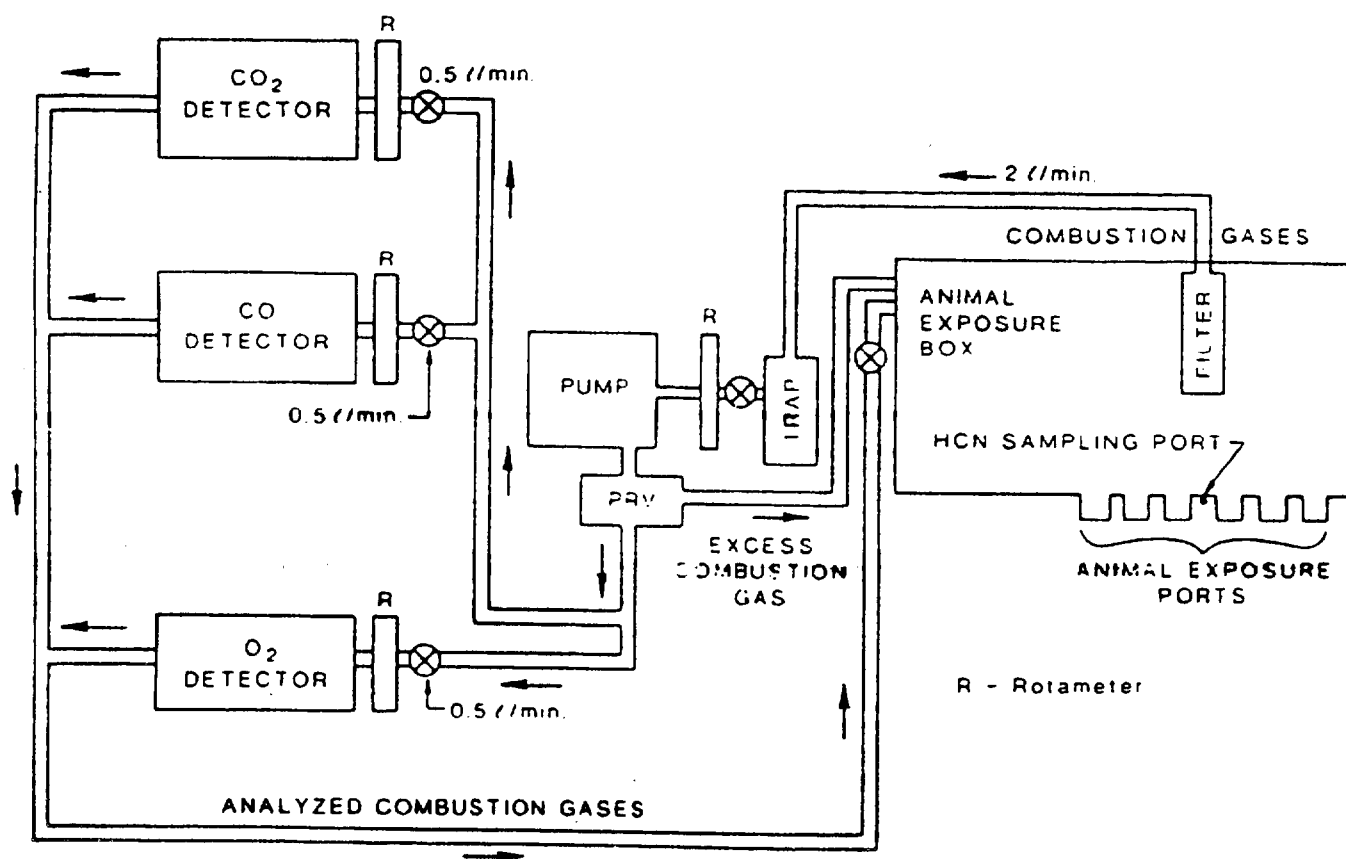


FIGURE 8. Gas sampling flow.

MIL-STD-2031(SH)

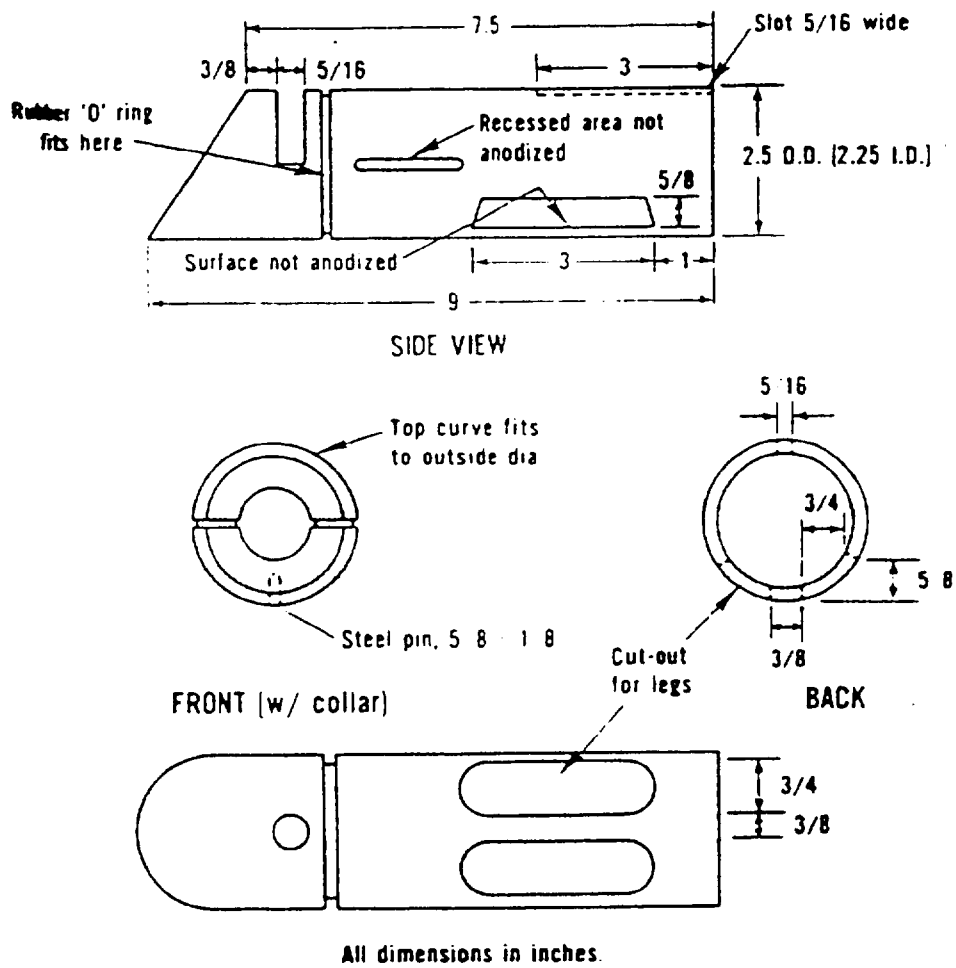


FIGURE 9. Animal restrainer design.

MIL-STD-2031(SH)

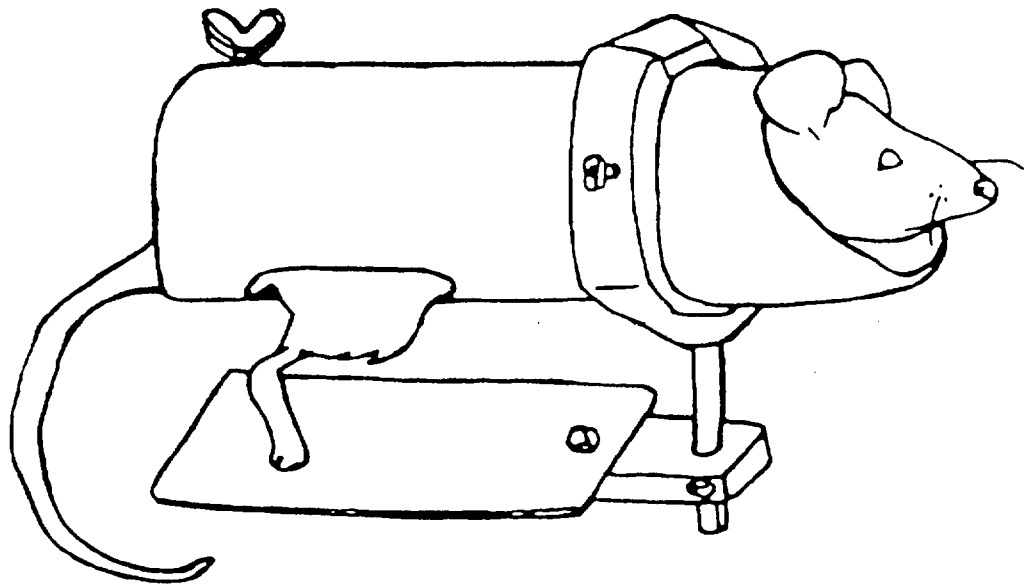


FIGURE 10. *Animal restrainer.*

## MIL-STD-2031(SH)

**30.5.1 Acclimation.** Animals received from a supplier shall be housed at the testing laboratory for a minimum of 7 days to allow them to become acclimated to the laboratory conditions before being used in testing.

**30.5.2 Care.** Animal care and maintenance shall be performed in accordance with the procedures outlined in the National Institute of Health's "Guide for the Care and Use of Laboratory Animals". The rats shall be housed individually in suspended stainless steel cages and provided with food and water ad libitum. Twelve hours of fluorescent lighting per day shall be provided using an automatic timer. All animals (including the controls) shall be weighed daily from the day of arrival until the end of the post-exposure observation period.

**30.6 Definitions specific to the procedure.** The following definitions are considered important to the performance and understanding of this smoke toxicity screening test:

<i>Acute toxicity:</i>	Harmful effects leading to lethality from a single short inhalation exposure to the combustion products of materials
<i>Mass loading:</i>	Amount of material placed in furnace (grams)
<i>Mass consumed:</i>	Amount of material (grams) depleted in the furnace during combustion. This is determined by weighing the charged quartz beaker before and after an experiment.
<i>Loaded concentration:</i>	Mass loading per unit of exposure chamber volume in milligrams per liter (mg/L)
<i>Consumed concentration:</i>	Mass consumed per unit of exposure chamber volume (mg/L)
<i>LC<sub>50</sub>:</i>	Concentration that is determined statistically to produce death in 50 percent of the test animal population exposed for 30 minutes and observed for a period of at least 14 days. This value may be calculated based on either mass loaded or mass consumed, as specified. The lethal effects of the individual and combined four gases for this procedure occurs within 24 hours.
<i>Extreme toxic potency:</i>	A property of smoke where very small amounts of the combusted or thermally decomposed material are lethal, i.e., the LC <sub>50</sub> value is very small. The value at which smoke exhibits extreme toxicity depends on the fire scenario and should be decided by the concerned individuals.

## MIL-STD-2031(SH)

- Unusual toxicity:* A property of a material whose combustion product toxicity can not be explained by the quantity of known, major toxic gases present
- Autoignition temperature:* The lowest furnace temperature at which a material sample introduced into the test furnace will spontaneously ignite within 30 minutes.

**40. PROCEDURE**

**40.1 Pre-test and test conditions.** All tests shall be conducted in a room or enclosed space having an ambient temperature of  $22 \pm 3$  °C. ( $70 \pm 5$  °F) and relative humidity of  $50 \pm 10$  percent at the time of test.

**40.1.1 CAUTION:** Provision shall be made for removing combustion products from the exposure chamber without contaminating the work area and exposing the test operators. The exposure chamber shall be housed in a chemical hood or other ventilated enclosure.

**40.1.2 Inside exposure chamber.** Inside exposure chamber wall surfaces shall be cleaned when changing the test material; changing temperature of decomposition; following test runs where toxicologically significant combustion products are suspected of accumulating as particulates; or, as visual inspection may indicate.

**40.1.3 Pre-experiment check.** Prior to experiments involving the actual test material, the system shall be checked to determine that the analytical and combustion systems are operating correctly. To check the entire system, a standard material shall be run. Douglas fir (4.5 g for non-flaming and 7.1 g for flaming combustion) conditioned for 48 hours at  $22 \pm 3$  °C. ( $70 \pm 5$  °F) and relative humidity of  $50 \pm 10$  percent shall be used. The  $LC_{50}$  data from 7 laboratories for the 30 minute exposure and 14 day post-exposure observation period is shown in Table II. An NIST Standard Reference Material may be available for use in the future. If the  $LC_{50}$  results for the non-flaming and flaming decomposition of Douglas fir conditions fall within the 95 percent confidence limits of the mean of these laboratories, the performance of the system is considered acceptable.

## MIL-STD-2031(SH)

TABLE II. *Interlaboratory evaluation of douglas fir LC<sub>50</sub> (30 minutes + 14 days).*

Laboratory	Non-flaming	Flaming
1	16.7(14.5 - 19.3)	35.8(28.6 - 44.9)
2	27.6(22.9 - 33.3)	45.3(39.0 - 52.7)
3	26.8(21.3 - 33.7)	28.0 <sup>3</sup>
4	24.0(19.9 - 29.0)	29.6(22.7 - 38.6)
5	25.9(20.0 - 33.5)	38.4(35.2 - 41.9)
NIST <sup>1</sup>	20.4(16.4 - 25.3)	41.0(33.0 - 50.9)
NIST <sup>2</sup>	22.8(20.2 - 25.8)	39.8(38.2 - 41.4)
8	18.5(17.3 - 19.8)	29.8(23.9 - 37.1)
Mean ± 95 percent confidence limits	22.8(13.4 - 32.2)	36.0(21.1 - 50.8)

<sup>1</sup>NIST small furnace.

<sup>2</sup>NIST large furnace.

<sup>3</sup>Estimated.

In this table, the numbers quoted are as calculated from the data provided by various laboratories. The 95 percent confidence limits reflect only statistical variations.

**40.1.4 Check-out procedure.** The instruments for CO<sub>2</sub>, CO, and HCN measurements shall be zeroed and a base line established during the check-out procedure. Oxygen concentration shall be recorded prior to initiating the exposure.

**40.1.5 Warm-up.** The furnace shall be brought up to the desired temperature and the system allowed to reach equilibrium 10 minutes before the start of the experiment. During this warm-up period and the recording of pre-exposure data, the door of the exposure chamber shall be left open.

**40.2 Test materials.** Material samples to be evaluated for toxicity shall be conditioned in a constant humidity chamber maintained at 50 ± 10 percent relative humidity at a room temperature of 22 ± 3 °C (70 ± 5 °F) for a period of 48 hours prior to testing.

**40.2.1 Sizing of sample.** The size of the test specimen for the initial test shall vary depending on the expected toxicity of its combustion products. A reasonable initial loading for an unknown material is about 5 grams. For example, 5 grams of material thermally degraded leads to a concentration of combustion products of 25 mg/L (grams of material loaded/chamber volume). Based on the results of the initial test, sample sizes for subsequent tests shall be selected to provide sufficient information to predict the LC<sub>50</sub> concentration. Prior to the start of the experiment, the weight of the beaker and sample material shall be recorded.

## MIL-STD-2031(SH)

**40.2.2 CAUTION:** A maximum sample mass of 8 grams shall be specified to reduce the risk of creating an explosive mixture.

**40.2.3 Specimens.** Test specimens shall be representative of the materials from which they are taken. Samples of composite materials and non-homogeneous assemblies must contain proportional quantities of each component. Whenever possible, the test specimen shall be a single piece of the same thickness as the material being tested.

**40.3 Autoignition temperature.** The procedure for determining the autoignition temperature shall begin with the cup furnace stabilized at 500 °C (930 °F). One gram of the material shall be introduced into the furnace. If autoignition does not occur, the process shall be repeated by increasing the temperature at 50 °C intervals until autoignition is observed. If autoignition occurs at 500 °C (930 °F), the furnace temperature shall be decreased in 50 °C increments until autoignition is not observed. At either temperature, additional tests shall be performed until the autoignition temperature is bracketed to within 25 °C. If autoignition is not observed by 800 °C (1470 °F), then this shall be the maximum test temperature for the material.

**40.3.1 Note:** The electric spark or ethanol used to ensure immediate flaming of the material during toxicological testing in the flaming condition shall not be used while determining the autoignition temperature.

**40.3.2 Larger sample.** The observed autoignition temperature is dependent on apparatus, procedure, and sample size. A larger sample may ignite at a lower temperature, so it is recommended that before actual testing begins, a larger sample size (6 or 8 grams) be tested at the non-flaming temperature. Autoignition temperatures determined by this procedure may differ from those measured according to ASTM D 1929.

**40.4 Trial run.** For each new test material, a trial run without animals shall be performed using the non-flaming, flaming, or non-flaming/flaming transition modes as determined by the fire scenario of concern. This procedure shall determine:

- a. The degree of oxygen depletion during combustion of the sample
- b. That the average chamber temperature over the 30 minute exposure period measured at the nose position of the animal does not exceed 40 °C (105 °F)
- c. That the proper conditions have been established for conducting either non-flaming or flaming combustion exclusively.

**40.4.4 Flaming test.** For the flaming test exposure, two drops of ethanol shall be added to the sample and an electrically heated wire or electric spark used to ensure immediate ignition of the test material.

## MIL-STD-2031(SH)

**40.4.5 Alternate procedure.** If the trial run shows that the average temperature in the exposure chamber will exceed 40 °C (105 °F), the electrical power to the furnace may be cut when the sample is completely degraded. In this case, although the furnace is shut off before the 30 minute exposure is completed, the exposure chamber shall remain closed and the animals shall receive a full 30 minute exposure. The length of time required to degrade a sample that produces CO shall be determined by monitoring the increase of CO concentration. For those materials that do not produce CO, another degradation product shall be analytically monitored. When the concentration of the monitored gas reaches a steady state for 2 minutes, the heater shall be turned off.

**40.5 Analytical tests.** Two analytical experiments without animals shall be conducted first under separate non-flaming, flaming, or non-flaming/flaming transition conditions to determine the concentrations of CO, CO<sub>2</sub>, and HCN that would be generated and the O<sub>2</sub> depletion levels from different mass loadings of each material. If the trial run does not produce an excessive exposure temperature and, for non-flaming tests, the sample does not ignite, then the trial run may be considered one of the two analytical tests. These species concentrations shall be used to predict the LC<sub>50</sub> value for the selected combustion mode. The animals shall be exposed to the mass of material that is equivalent to 80 percent of this predicted value.

**40.5.1 Test initiation.** To initiate the analytical experiments, the weighed samples shall be placed in the preheated, preweighed cup in the furnace and the door of the exposure chamber immediately closed. Placement of the sample into the furnace shall designate the starting time of the exposure. The material shall be allowed to decompose for 30 minutes.

**40.6 N-Gas Model prediction for LC<sub>50</sub> mass of material.** Using the gas concentrations obtained during the analytical tests, calculate the N-Gas Model predictions with the listed equations. These equations have been empirically derived to predict when 50 percent of the animals should die either within the 30 minute exposure or within a 24 hour post-exposure period. This occurs when the equation is approximately equal to 1. These N-Gas Model equations are based on NIST studies of the toxicological interactions of two to four of these gases. The current model equations do not predict post-exposure deaths occurring after 24 hours. Based on anticipated material composition, use one of the three equations listed below to determine the material mass loading that produces the LC<sub>50</sub> value, that is when the equation is approximately equal to 1. To determine the amount of material that shall be used in the animal exposure tests, take 80 percent of the material mass calculated for the LC<sub>50</sub> value.

$$(1) \text{ For CO and HCN: } [\text{CO}]/\text{LC}_{50}\text{CO} + [\text{HCN}]/\text{LC}_{50}\text{HCN} \approx 1 \pm 0.2$$

where the brackets indicate the actual concentration of the specified gases; the LC<sub>50</sub> value of CO is based on 30 minute exposures and is 6600 p/m and the LC<sub>50</sub> value of HCN is 160 p/m for 30 minute exposures or 110 p/m for 30 minute exposures plus 24 hour post-exposure deaths.

$$(2) \text{ For CO, HCN, and CO}_2: m[\text{CO}]/([\text{CO}_2]-b) + [\text{HCN}]/\text{LC}_{50}\text{HCN} \approx 1 \pm 0.2$$

## MIL-STD-2031(SH)

where the term  $m$  equals minus 18 and  $b$  equals 122,000 when the  $\text{CO}_2$  concentration is 5 percent or less. If the  $\text{CO}_2$  concentration is above 5 percent,  $m$  and  $b$  equal 23 and minus 38,600, respectively. Other values are the same as equation (1).

$$(3) \text{ For CO, HCN, CO}_2 \text{ and low O}_2: \\ m[\text{CO}]/([\text{CO}_2]-b) + [\text{HCN}]/\text{LC}_{50}\text{HCN} + 21 - [\text{O}_2]/(21-5.4) \approx 1 \pm 0.2$$

where 5.4 is the percent  $\text{O}_2$  that causes 50 percent of the animals to die in 30 minutes. Other values are the same as for equations (1) and (2).

## NOTE

The values used in the above equations are dependent on the test protocol, the source of test animals, and the strain of rat selected. These equations represent the best data concerning the toxicological interaction of these gases as of the spring of 1989.

**40.7 Animal exposure tests.** For each decomposition mode, a single animal experiment shall be performed using 80 percent of the predicted  $\text{LC}_{50}$ . In each animal exposure experiment, six rats shall be exposed. The animals shall be placed in restrainers, then inserted into the six portholes located along the front of the exposure chamber such that only the heads of the animals are exposed. In the non-flaming or flaming experiments, the animals in their restrainers shall be placed in the portholes before the test sample is dropped into the cup furnace. In the non-flaming/flaming transition experiments, the portholes shall be blocked from the inside of the chamber with rubber stoppers during the first phase. Five minutes into the second phase of decomposition, the animals in their restrainers shall be inserted into the portholes so that the stoppers are pushed into the chamber while simultaneously exposing the animals' heads to the combustion atmospheres. The animals shall remain in place for 30 minutes. Because all of the combustion products shall be kept in the chamber, these exposures shall consist of both the initial generation of the test atmosphere, which usually lasts about 5 minutes, as well as the later steady-state conditions. The toxicological endpoint shall be death, which occurs during the 30 minute exposure or the post-exposure 24 hour observation period. In all experiments, the  $\text{CO}$ ,  $\text{CO}_2$ ,  $\text{O}_2$ , and  $\text{HCN}$  shall be measured throughout the exposures. The gas concentrations used in the N-Gas Model shall be the time-weighted average values which shall be calculated for the exposure period.

**40.8 Animal post-test procedure.** Although this protocol shall be limited to animal lethality up to 24 hours after the termination of an exposure, observation of the animals' condition post-exposure, and up to 14 days following the exposure shall be important to ensure that toxicological syndromes are consistent with the gases included in the equations specified in 40.6. Animals shall be removed while still in the restrainer and examined for the following responses: animal's eyes shall be examined for reflexes, redness, tearing, and corneal opacity; and, animal's nose and mouth shall be examined for any discharge and respiratory difficulties such as gasping, wheezing, and rapid or slow breathing. After removal of the animal from the restrainer, the investigator shall examine

## MIL-STD-2031(SH)

the animal's exploratory behavior (characterized by trying to escape and exploring his surroundings); righting reflex (animal shall be placed on his back and the ability to right himself shall be scored as rapid, slow or non-existent); or, posture (animal shall be lifted from table by his tail and placed back on table noting irregularities such as limp hind legs). After checking these responses, the animals shall be returned to their cages and weighed daily for 14 days. If still losing weight on day 14, they shall be kept until they show signs of recovery (three successive days of weight gain) or die. Animals shall not be used in more than one experiment.

**40.9 Post-test time-weighted average procedure.** Immediately following the animal test, the quartz beaker shall be removed from the furnace, allowed to cool, and then weighed to determine the mass of material consumed. (The beaker and material shall also be weighed before the experiment).

The average 30 minute (animal exposure time) concentrations of CO, CO<sub>2</sub>, O<sub>2</sub>, and HCN shall be calculated according to the following equation:

$$\bar{C}_i = \frac{\sum_{j=1}^n [C_j * \Delta t_j]_i}{30} \quad i = (\text{CO, CO}_2, \text{O}_2, \text{HCN})$$

where: C<sub>j</sub> = gas concentration of ith species during Δ t<sub>j</sub> time interval

t<sub>j</sub> = measurement time for the jth interval (minute)

n = number of readings, such that  $\sum_{j=1}^n t_j = 30$

If none of the exposed animals die either during the exposure or within 24 hours post-exposure period, then the predicted LC<sub>50</sub> value shall be estimated adequately. If one or more of the animals die at 80 percent of the predicted LC<sub>50</sub> value, then other gases may be acting in conjunction with the major gases measured. In this case or in the case animals die during the 14 day post-exposure monitoring period, further animal tests may be necessary.

## 50. REPORT

**50.1 Report contents.** The following data shall be included in a report for each test performed on each material:

- a. Description of the material tested
- b. Autoignition temperature

MIL-STD-2031(SH)

- c. Type of test (i.e., analytical, animal)
- d. Combustion condition (i.e., non-flaming, flaming, or non-flaming/flaming transition)
- e. Amount of material loaded into the furnace
- f. Amount of material consumed (difference between amount loaded into the furnace and amount of residual material)
- g. Time-weighted average (during a 30 minute animal exposure) for O<sub>2</sub>, CO, CO<sub>2</sub>, HCN, animal exposure box temperature and any other gases
- h. For each animal test, report animal mortality during the 30 minute exposure; 30 minutes plus 24 hours after the exposure; and, 30 minutes plus post-exposure period (14 days) day deaths occur
- i. Estimated LC<sub>50</sub>
- j. Value of the N-Gas Model equation (paragraph 40.6) for experiments performed at the estimated LC<sub>50</sub> value
- k. Actual LC<sub>50</sub> value, if determined by "classical LC<sub>50</sub>" procedures
- l. Additional observations, if any.

MIL-STD-2031(SH)

APPENDIX G

TEST REPORTS TECHNICAL CONTENT REQUIREMENTS

10. SCOPE

**10.1 Scope.** This appendix covers information that shall be included in the test reports when specified in the contract or order. This appendix is mandatory only when data item description DI-MISC-80653 is cited on the DD Form 1423.

20. APPLICABLE DOCUMENTS

This section is not applicable to this appendix.

30. TEST REPORTS

**30.1 (Oxygen-temperature LOI) standard operating procedure for oxygen-temperature index profile.**

**30.1.1 Source.** Name and address of the testing laboratory and date of test.

**30.1.2 Nomenclature.** Noun name, chemical name, manufacturer, and code designations.

**30.1.3 Dimensions.** Length, width, and thickness in inches.

**30.1.4 Experimental conditions.** Humidity, room temperature, time period specimen at experimental conditions.

**30.1.5 Profile.** Tabular results and graphic representations of the temperature index profile at 25 (ambient), 75 and 300 °C (75, 165, and 570 °F) and other temperatures as appropriate. Include results from each specimen test.

**30.1.6 Behavior.** Descriptions of any unusual specimen behavior such as prolonged glowing combustion after flameout, charring, sparking or dripping, bending, erratic burning, or excessive smoke evolution.

**30.2 (Flame Spread) surface flammability of materials using a radiant heat energy source.**

**30.2.1 Source.** Name and address of the testing laboratory and date of test.

**30.2.2 Nomenclature.** Noun name, chemical name, manufacturer, and code designations.

**30.2.3 Dimensions.** Length, width, and thickness in inches.

## MIL-STD-2031(SH)

**30.2.4 Specimen type.** Type of test specimens (molded slab, core, skin, surface treated, etc.) and whether tested with or without backing or aluminum foil.

**30.2.5 Specimen quantity.** Number of specimens tested and their exposure time (15 minutes or completely destroyed).

**30.2.6 Flame spread index.** Average flame spread index for the set of specimens and range and any visual characteristics of the individual specimens.

**30.2.7 Behavior.** Description of any unusual specimen behavior such as sparking or dripping.

**30.3 (Ignitability) test method for heat and visible smoke release rates for materials and products using an oxygen consumption calorimeter.**

**30.3.1 Source.** Name and address of the testing laboratory and date of test.

**30.3.2 Nomenclature.** Noun name, chemical name, manufacturer, and code designation.

**30.3.3 Specimen quantity.** Number of replicate specimens (minimum of three) tested under the same heat flux level (25, 50, 75, and 100 kW/m<sup>2</sup>) with their thickness and mass. The exhaust system flow rate at each heating flux level is required.

**30.3.4 Mounting.** Test orientation (either vertical and horizontal), specimen mounting and whether optional retainer or wire grid were used.

**30.3.5 Ignition.** Time to ignition for each specimen in seconds.

**30.3.6 Behavior.** Description of any unusual specimen behavior such as sparking or dripping.

**30.4 (Heat release) test method for heat and visible smoke release rates for materials and products using an oxygen consumption calorimeter.**

**30.4.1 Source.** Name and address of testing laboratory and date of test.

**30.4.2 Nomenclature.** Noun name, chemical name, manufacturer, and code designations.

**30.4.3 Specimen quantity.** Number of replicate specimens (minimum of three) tested under the same heating flux levels of 25, 50, 75, and 100 kW/m<sup>2</sup> with their thickness and mass. The exhaust system flow rate of each heating flux level is required.

**30.4.4 Mounting.** Test orientation (either vertical or horizontal), specimen mounting and whether optional retainer or wire grid were used.

**30.4.5 Release rate curve.** Heat release rate (per unit area) curve.

## MIL-STD-2031(SH)

**30.4.6 Release rate average.** Average heat release rate for 60 seconds determined by 30 seconds each side of the maximum heat release rate value in the first 300 seconds after ignition.

**30.4.7 Total heat.** Total heat released by the specimen.

**30.4.8 Average heat.** Average heat of combustion for the entire test period.

**30.4.9 Mass.** Mass remaining after test and mass loss rate for 60 seconds determined by 30 seconds each side of the maximum heat release rate value in the first 300 seconds after ignition.

**30.5 (Smoke obscuration) specific optical density of smoke generated by solid materials.**

**30.5.1 Source.** Name and address of the testing laboratory and date of test.

**30.5.2 Nomenclature.** Noun name, chemical name, manufacturer, and code designations.

**30.5.3 Test conditions.** Number of specimens tested and the test conditions; type of exposure, the exposure period, and temperature of chamber wall.

**30.5.4 Behavior.** Descriptions of any unusual specimen behavior such as prolonged glowing combustion after flameout, charring, sparking or dripping, swelling, contraction, cracking, bulging, erratic burning, or excessive smoke evolution.

**30.5.5 Smoke.** Observations of the smoke-generating properties of the specimens during exposure, such as color of smoke, nature of the settled particulate matter, etc.

**30.5.6 Time versus  $D_s$ .** Tabulation of time versus  $D_s$  (rounded to two significant figures) for each run of the three test specimens.

**30.5.7 Test results.** Test results (rounded to two significant figures) including the average and range on each set of specimens for  $D_m$  with time of occurrence, and  $D_m$  (corr).

**30.6 (Combustion gas generation) test method for heat and visible smoke release rates for materials and products using an oxygen consumption calorimeter.**

**30.6.1 Source.** Name and address of the testing laboratory and date of test.

**30.6.2 Nomenclature.** Noun name, chemical name, manufacturer, and code designations.

**30.6.3 Specimen quantity.** Number of replicate specimens (minimum of three) tested under the heating flux level of  $25 \text{ kW/m}^2$  with their thickness and mass. The exhaust system flow rate at the heating flux level shall be stated.

## MIL-STD-2031(SH)

**30.6.4 Mounting.** Test orientation (either vertical or horizontal), specimen mounting and whether optional retainer or wire grid were used.

**30.6.5 Gases.** Combustion gases (as a minimum CO, CO<sub>2</sub>, HCl, O<sub>2</sub> reported) measured at each heating flux level. Report NO<sub>x</sub>, SO<sub>x</sub>, halogen acid gases and total hydrocarbon concentrations when collected.

**30.7 DTRC burn-through fire test.**

**30.7.1 Source.** Name and address of testing laboratory and date of test.

**30.7.2 Nomenclature.** Noun name, chemical name, manufacturer, and code designation.

**30.7.3 Dimensions.** Length, width, and thickness in inches.

**30.7.4 Time to smoke.** Time to smoke or fire gases visually start to evolve from non-fire side of material specimen.

**30.7.5 Time to burn-through.** Time to burn-through of the specimen as evident by flame on non-fire side of the material.

**30.7.6 Continued burn time.** The time that the material specimen continues to burn after removing the fire source.

**30.7.7 Profile.** Tabular and graphic representations of the temperature profiles in 5 minute intervals of the three vertically mounted thermocouples and the thermocouple mounted behind the center mounted thermocouple.

**30.7.8 Smoke.** Description of any smoke generation as to time of initial occurrence, color, and relative amount.

**30.7.9 Visual inspection.** Description of visual inspection of fire damage to the material specimen to include any swelling, contraction, cracking, bulging, melting or dripping, separation of plies and effective material thickness remaining.

**30.8 Determination of the flashover potential of lining material using a quarter-scale room fire test.**

**30.8.1 Source.** Name and address of testing laboratory and date of test.

**30.8.2 Nomenclature.** Noun name, chemical name, manufacturer, and code designations.

## MIL-STD-2031(SH)

**30.8.3 Flashover.** Time to flashover, if it occurs. Flashover is characterized by thermal flux levels equal to or greater than 2 watts per square centimeter at the floor level.

**30.8.4 Temperature.** Time and temperature to maximum temperature recorded if flashover does not occur.

**30.8.5 Observations.** Visual observations such as smoke, fire in overhead, etc.

**30.9 Large scale open environment fire test.**

**30.9.1 Source.** Name and address of testing laboratory and date of test.

**30.9.2 Nomenclature.** Noun name, chemical name, manufacturer, and code designations.

**30.9.3 Time-temperature.** Time-temperature curve on the fire from the overhead thermocouple and from the thermocouple by the application outside the fire plume.

**30.9.4 Failure time.** Failure time, if it occurs, of the material application.

**30.9.5 Fuel consumption.** Fuel consumption during the fire in gallons.

**30.9.6 Continued burn.** The time that the material continues to burn after extinguishing the fire pan.

**30.9.7 Visual inspection.** Description of visual inspection of fire damage to the material application to include any swelling, contraction, cracking, bulging, melting or dripping, separation of plies.

**30.9.8 Extinguishment.** Extinguishment test data when conducted: pre-burn time, time to extinguishment, reflash, and time to extinguish reflash.

**30.9.9 Gases.** Combustion gas results for CO in p/m, CO<sub>2</sub>, and O<sub>2</sub> in percentage by volume, HCl in p/m, and any other gases collected.

**30.9.10 Smoke obscuration.** Smoke obscuration data when collected.

**30.9.11 Heat release.** Total heat release by the material application when collected.

**30.9.12 Observations.** Additional observations, if any.

**30.10 Large scale pressurizable fire test.**

**30.10.1 Source.** Name and address of testing laboratory and date of test.

## MIL-STD-2031(SH)

**30.10.2 Nomenclature.** Noun name, chemical name, manufacturer, and code designations.

**30.10.3 Time-temperature.** Representative time-temperature curve on the fire.

**30.10.4 Responses.** Temperatures in both °C and °F, radiometer response in  $W/cm^2$  and pressure in  $lb/in^2$  absolute.

**30.10.5 Gases.** Gas concentrations of total hydrocarbons as  $g/m^3$ ;  $O_2$ , CO, and  $CO_2$  as percentage by volume; and, HCl and HCN as parts per million.

**30.10.6 Smoke.** Smoke production shall include the following: average particle diameter ( $\mu m$ ), aerosol density ( $g/m^3$ ), calculated number density (particles/ $cm^3$ ), visual range (m), smoke particulate mass generation rate ( $g/s$ ), and total mass particulates (g).

**30.10.7 Visual inspection.** Fire damage estimate shall be reported as percent of material area damaged or destroyed. Include visual inspection of the fire damage for swelling, contraction, cracking, bulging, melting or dripping, separation of plies and effective material thickness remaining.

**30.10.8 Fuel consumption.** Fuel consumption during the fire.

**30.10.9 Continued burn.** The time that the material continues to burn after extinguishing the fire pan.

### **30.11 N-Gas Model Smoke Toxicity Screening Test.**

**30.11.1 Source.** Name and address of the testing laboratory and date of test.

**30.11.2 Nomenclature.** Noun name, chemical name, manufacturer, and code designations.

**30.11.3 Autoignition.** Autoignition temperature.

**30.11.4 Test type.** Type of test (i.e. analytical, animal).

**30.11.5 Combustion.** Combustion condition (i.e. non-flaming, flaming, or non-flaming/ flaming transition).

**30.11.6 Material loaded.** Amount of material loaded into the furnace.

**30.11.7 Material consumed.** Amount of material consumed (difference between amount loaded into the furnace and the amount of residual material).

**30.11.8 Gases.** Time-weighted average (during a 30 minute exposure) for  $O_2$ , CO,  $CO_2$ , HCN (and any other gases), and animal exposure box temperature.

## MIL-STD-2031(SH)

**30.11.9 Mortalities.** Animal mortalities (number of deaths and time after initiating animal exposure) during the 30 minute exposure, 30 minutes plus 24 hours after exposure, and 30 minutes plus 14 days post-exposure period.

**30.11.10  $LC_{50}$**  Estimated  $LC_{50}$ .

**30.11.11 N-gas model equation.** Value of the N-gas model equation for experiments performed at the estimated  $LC_{50}$  value.

**30.11.12 Observations.** Additional observations, if any.

**Custodians:**

Army - MR  
Navy - SH

**Preparing activity:**

Navy-SH  
(Project CMPS-0068)

# STANDARDIZATION DOCUMENT IMPROVEMENT PROPOSAL

## INSTRUCTIONS

1. The preparing activity must complete blocks 1, 2, 3, and 8. In block 1, both the document number and revision letter should be given.
2. The submitter of this form must complete blocks 4, 5, 6, and 7.
3. The preparing activity must provide a reply within 30 days from receipt of the form.

NOTE: This form may not be used to request copies of documents, nor to request waivers, or clarification of requirements on current contracts. Comments submitted on this form do not constitute or imply authorization to waive any portion of the referenced document(s) or to amend contractual requirements.

### I RECOMMEND A CHANGE:

1. DOCUMENT NUMBER  
MIL-STD-2031(SH)

2. DOCUMENT DATE (YYMMDD)  
26 February 1991

3. DOCUMENT TITLE Fire and Toxicity Test Methods and Qualification Procedure for Composite Material Systems Used in Hull, Machinery, and Structural Applications Inside Naval Submarines

4. NATURE OF CHANGE (Identify paragraph number and include proposed rewrite, if possible. Attach extra sheets as needed.)

### 5. REASON FOR RECOMMENDATION

### 6. SUBMITTER

a. NAME (Last, First, Middle Initial)

b. ORGANIZATION

c. ADDRESS (Include Zip Code)

d. TELEPHONE (Include Area Code)

e. DATE SUBMITTED (YYMMDD)

(1) Commercial

(2) AUTOVON

(If applicable)

### 8. PREPARING ACTIVITY

a. NAME Technical Point of Contact (TPOC):  
Mr. William Dunham (SEA 51431)

b. TELEPHONE (Include Area Code)

(2) AUTOVON

PLEASE ADDRESS ALL CORRESPONDENCE AS FOLLOWS:

TPOC: 703 602-0146

8-332-0146

c. ADDRESS (Include Zip Code)

Commander, Naval Sea Systems Command

IF YOU DO NOT RECEIVE A REPLY WITHIN 45 DAYS, CONTACT:  
Defense Quality and Standardization Office