

# Standard Test Method for Determining the Combustion Behavior of Metallic Materials in Oxygen-Enriched Atmospheres<sup>1</sup>

This standard is issued under the fixed designation G124; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon  $(\varepsilon)$  indicates an editorial change since the last revision or reapproval.

# 1. Scope

- 1.1 This test method covers test apparatus and techniques to determine the minimum test gas pressure and sample temperature that supports self-sustained burning and the regression rate of the melting surface of a standardized sample of a metallic material that has been ignited using a promoter.
- 1.2 The data obtained from this test method are dependent on the precise test sample configuration and provide a basis for comparing the burning characteristics of metallic materials. No criteria are implied for relating these data for the suitability of a material's use in any actual system.
- 1.3 Requirements for apparatus suitable for this test method are given, as well as an example. The example is not required to be used.
- 1.4 This test method is for gaseous oxygen or any mixture of oxygen with inert diluents that will support burning, at any pressure or temperature within the capabilities of the apparatus used.
- 1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.
- 1.6 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

#### 2. Referenced Documents

2.1 ASTM Standards:<sup>2</sup>

G63 Guide for Evaluating Nonmetallic Materials for Oxygen Service

- G88 Guide for Designing Systems for Oxygen Service
- G93 Practice for Cleaning Methods and Cleanliness Levels for Material and Equipment Used in Oxygen-Enriched Environments
- G94 Guide for Evaluating Metals for Oxygen Service

# 3. Terminology

- 3.1 Definitions:
- 3.1.1 *burn length*, *n*—the burn length is the length of the sample that has been consumed by combustion.
- 3.1.1.1 *Discussion*—The burn length is determined by subtracting the post-test sample length from the pretest sample length (which does not include the promoter length or region used by the test sample support.)
- 3.1.2 *flammable material*, n—a material is defined in this standard as flammable if a standard rod sample burns more than 3 cm (1.2 in.) above the promoter (1, 2).<sup>3</sup>
- 3.1.3 *highest no-burn pressure, n*—the maximum gas pressure (at a specified oxygen concentration and fixed sample temperature) at which a material does not burn more than 3 cm (1.2 in.) above the promoter in a minimum of five tests.
- 3.1.4 highest no-burn temperature, n—the maximum sample temperature (at a specified oxygen concentration and pressure) at which a material does not burn more than 3 cm (1.2 in.) above the promoter in a minimum of 5 tests.
- 3.1.5 *igniter*, *n*—a material used to ignite the promoter that can burn under an electrical influence, such as a small-diameter wire.
- 3.1.6 *lowest burn pressure*, *n*—the minimum gas pressure (at a specified oxygen concentration and fixed sample temperature) at which a material burns more than 3 cm (1.2 in.) above the promoter for one or more tests specimens.
- 3.1.7 lowest burn temperature, n—the minimum sample temperature (at a specified oxygen concentration and pressure) at which a material burns more than 3 cm (1.2 in.) above the promoter for one or more tests specimens.

<sup>&</sup>lt;sup>1</sup> This test method is under the jurisdiction of ASTM Committee G04 on Compatibility and Sensitivity of Materials in Oxygen Enriched Atmospheres and is the direct responsibility of Subcommittee G04.01 on Test Methods.

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<sup>&</sup>lt;sup>2</sup> For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>&</sup>lt;sup>3</sup> The boldface numbers in parentheses refer to a list of references at the end of this standard.



- 3.1.8 *promoter*; *n*—an optional material that can add supplemental heat and increase the temperature to start burning of the metallic material being tested.
- 3.1.9 regression rate of the melting interface, n—the average rate at which the solid-liquid metal (melting) interface advances along the test sample length during a test.
- 3.1.10 sample temperature, n—the initial temperature of the test sample being evaluated.
- 3.1.10.1 *Discussion*—Various methods of measuring sample temperatures are used. The method selected must be reported with test data.
- 3.1.11 standard rod test sample, n—a 3.2 mm (0.125 in.) diameter rod with a minimum length of 101.6 mm (4 in.).
- 3.1.12 *threshold pressure*, *n*—This term is historically used to represent the definitions of either the lowest burn pressure or the highest no-burn pressure.
- 3.1.12.1 *Discussion*—In this standard, it represents the lowest burn pressure, which is used as the new term throughout.
- 3.1.13 *valid test, n*—a test in which the igniter and/or promoter combination has melted the bottom section of the test sample where the igniter and/or promoter is located.

#### 4. Summary of Test Method

- 4.1 A standard rod sample of the material to be tested is vertically suspended in a chamber filled with pressurized test gas. The chamber contains sufficient oxygen so that not more than 10 % of the oxygen will be consumed if the sample completely burns. A promoter (aluminum is most common, however titanium, carbon steel and magnesium are also used) may be applied to the bottom of the rod to start burning of the material in conjunction with the igniter (typically Pyrofuse or Nichrome wire)<sup>4</sup>. The test chamber is pressurized to the required test pressure and the sample is heated to the required test temperature (if elevated temperature is one of the parameters).
- 4.2 The test is initiated by ignition of the igniter wire/promoter (typically through resistive heating) so that the end of the test sample is melted away to produce a valid test with relevant data collected, as specified.

Note 1—In 4.3 as subsequent samples are tested, only one parameter of temperature or pressure is varied and the other held constant within the tolerance allowed by this test method. It is up to the user to determine if the purpose of the test is to determine burn/no-burn pressure or burn/no-burn temperature. Only one of these variables should be changed during a series of tests.

4.3 If the sample is flammable, another standard sample rod is tested at a reduced test pressure or temperature. If the sample is not flammable, testing continues until the sample is not flammable in a minimum of fivetests at one set of conditions. It has been shown, for a burn probability of less than 10 %, 5 no burn results provides a 41 % confidence level in the (no burn) result, whereas twenty-two no burn results provides a 90 % confidence level (for the same burn probability of 10 %).

<sup>4</sup> The trade name for aluminum-palladium wire is Pyrofuze. It is a registered trademark of the Pyrofuze Corp., 121 S. Columbus Ave., Mt. Vernon, NY 10553, and is available from them.

A thorough discussion of the burn probabilities and associated confidence levels is given in Ref (3).

Note 2—Increasing the number of samples will always give a higher level of confidence and is recommended when possible. This method defines the highest no-burn pressure or temperature and the lowest burn pressure or temperature. The maximum no-burn (and burn) temperature and pressure and regression rate of the melting interface can be determined from the test data.

# 5. Significance and Use

5.1 This test method will allow comparisons of the burning characteristics of various metallic materials. The burning characteristics that can be evaluated include (1) burn and no-burn pressure, (2) burn and no-burn temperature, (3) regression rate of the melting interface, and (4) visual evaluation of the burning process of the test sample.

# 6. Interferences

- 6.1 Any materials inside the test chamber that may bake out, ignite/burn, or vaporize during the burning process at test temperature/pressure may interfere with the chemistry of the fire propagation and subsequently affect burning.
- 6.2 The specific atmosphere in the test chamber can have a severe chemical or thermodynamic effect, or both. Therefore, test gas contamination or diluents (such as argon, nitrogen, carbon dioxide, water vapor, and others) can be important factors, so the oxygen gas purity and quantities and types of diluents should be specified in the data sheet.
- 6.3 The standard test is conducted under non-flowing conditions. Depending on the final gas velocity, tests conducted under flowing oxygen conditions may dramatically affect the test results.

#### 7. Apparatus

- 7.1 System—A schematic of a typical system is shown in Fig. 1. Other designs may also be used if they fulfill the following requirements.
- 7.2 Test Chamber—A cross-section of a typical test chamber is shown in Fig. 2. Appendix X1 provides criteria for establishing the lowest test pressures that meet the stated criterion of using no more than 10 % of the available oxidizer for various vessel volumes. If the chamber cannot be made sufficiently large, an accumulator can be attached between the test chamber and the chamber isolation valve that contains more test gas. The test chamber (and accumulator if used) shall not contribute any chemical interference to testing.
- 7.3 *Sample Holder*—capable of securing the sample at the top and supporting it in a vertical position.
- 7.4 Temperature Sensor—used to measure gas or sample temperatures in the chamber, accurate to within  $\pm 1\,\%$  of reading or accuracy otherwise noted.
- 7.5 Pressure Transducer—used to measure gas pressure in the chamber, accurate to within  $\pm 1$  % of reading or accuracy otherwise noted.
- 7.6 *Liner (optional)*—a burn-resistant (for example, copper or ceramic) liner is recommended in the test chamber to serve as an internal shield to protect the chamber and components

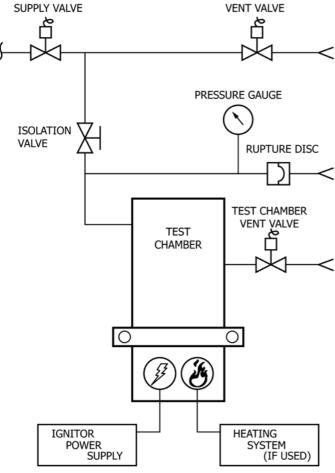


FIG. 1 Schematic of Typical System

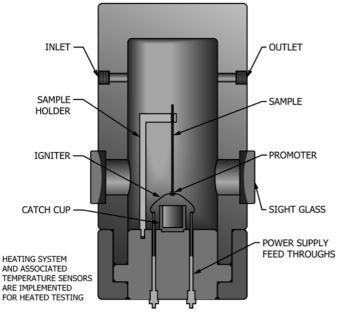


FIG. 2 Typical Stainless Steel Test Chamber Cross-Section

from the burning, molten slag, and other reaction products produced during sample burning.

- 7.7 Sight Glass—(optional for tests not determining either the regression rate of the melting interface or visual evaluation of the burning process), capable of withstanding the maximum test pressure anticipated (initial pressure plus pressure rise due to heating during burning). Other methods of observing the test may be possible, though direct observation is most common.
- 7.8 *Igniter Power Supply*—electrically isolated and capable of providing adequate current to initiate the ignition within 3 s of the application of power.
- 7.9 Test Cell—a room to house the test chamber, constructed of non-flammable material (such as concrete or metal) with sufficient strength to provide protection from explosion, pneumatic release or fire hazards. A continuous ventilation system shall circulate fresh air in the test cell. The test cell shall be cleaned periodically to avoid contamination of the sample and equipment and minimize fire hazards.
- 7.10 *Piping System*—which purges, pressurizes, and vents the test chamber. The piping system shall be designed to permit remote test chamber purge, pressurization, and venting without unsafe exposure of personnel. It is recommended the test chamber be purged and pressurized through one line and vented through a separate line to minimize the chances of a contaminant migrating into the pressurization line, which might influence subsequent tests. It is also recommended that a pressure relief device with an appropriate setting be fitted to the piping system and be able to communicate to the test chamber.

Note 3—Although the use of separate lines is preferred it is not a requirement. Periodic inspection and cleaning of lines and valves should be done to decrease the risk of cross contamination. A typical piping system for this test is shown in Fig. 1.

- 7.11 Control Area—which will isolate test personnel from the test cell during tests. This control area shall be provided with the necessary control and instrumentation features to perform test chamber purge, pressurization and venting operations, and monitoring of the test chamber instrumentation during the test.
- 7.12 Data Acquisition System—capable of recording, storing, and accessing the pressure, temperature and regression rate data at a rate of ten samples per second (minimum). It may also include a video recording device that displays the "real-time" burn phenomenon. The video recording with embedded timer, thermocouple sensor arrays, and ultrasonic rod length measurements are some of the methods available for determination of the regression rate of the melting interface (see Annex A1).
- 7.13 Heating System (for elevated temperature testing only)—which will heat the sample to the required initial test sample temperature range, without interfering with the other functions of the test system or the test chamber integrity. The heating system is required to evaluate burning characteristics at elevated temperatures above ambient. (No heating system is required if testing is to be done at ambient temperature only.) The method used can include, but is not limited to, localized heating methods including induction heating, resistive heating, and radiant heating. Heating of the entire system has also been successfully used, however the vessel pressure rating must be considered due to the temperature dependency of the chamber

material strength (see 9.4) and any non-metallic materials exposed to elevated temperatures should be used in accordance with Guide G63.

#### 8. Reagents and Materials

8.1 Gaseous Oxygen—Oxygen purity equal to or greater than that of practical systems is preferred for the standard test, and an analysis of the test oxidant is required. Other oxygen/diluents mixtures may be used and it is recommended that the exact oxygen purity used be specified with the test results. (4)

Note 4—Oxygen purity has been shown, for certain materials to significantly affect the results. Extremely high purity or low purity oxygen (with diluents present) should be avoided unless conducting special studies using gas mixtures (5) and in all cases the purity should be specified along with any diluents present

8.2 *Promoter*—The promoter shall provide sufficient energy to melt the end of the test sample to produce a valid test and, if flammable at test conditions, ignite the test specimens. Some examples of promoter material include aluminum, Pyrofuze, magnesium, titanium and carbon steel (6). In some cases, a promoter may not be necessary when the igniter itself can provide sufficient energy to produce a valid test. Nonmetallic promoters may be used; however, the combustion products of such promoters might contaminate the test media and care must be taken to ensure that the use of nonmetallic promoters produces a valid test result. Other ignition sources, such as laser or electrical, may also be used. In selecting the promoter material, the possibility of a chemical reaction between the test material and the promoter should be considered.

8.3 *Igniter*—The igniter shall have sufficient energy to ignite the promoter or the sample and produce a valid test result. Some examples of the igniter wire material are nickel/chromium (Nichrome) or aluminum/ palladium (Pyrofuze).<sup>4</sup> The electrical system that supplies current to the wire should provide sufficient current to melt and ignite the igniter in 1-2 s. A slow heat-up can increase the amount of pre-ignition energy loss to the sample rod, which will increase the heat affected zone of the sample and potentially produce an invalid test.

Note 5—Rapid heating of the sample may result in damage to, or ignition of, the igniter wire and either prevent ignition of the promoter or ignite it prior to establishment of required test conditions.

# 9. Hazards

- 9.1 *High-Pressure Oxygen System*—**Warning:** There are hazards involving the use of a enriched-oxygen systems. The following guidelines will reduce the dangers:
- 9.1.1 Personnel should be isolated from the test system when it is pressurized. Preferably, personnel should be shielded by both physical protection (for example, the test cell) and distance.
- 9.1.2 The test system itself should be isolated to prevent danger to people not involved in the test.
- 9.1.3 The test system should incorporate equipment able to handle the maximum operating pressure safely, including an appropriate safety-factor.
- 9.1.4 The test system should be kept clean to prevent unintentional ignition.
- 9.1.5 The test system should be double-isolated from the test gas supply system.

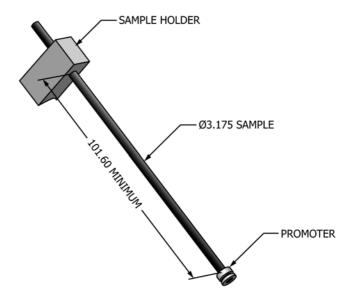


FIG. 3 Typical Test Sample Dimensions

- 9.1.6 Remote readout devices should be provided so personnel do not have to approach the test system to obtain operating data or test results, or both.
- 9.2 Oxygen—Warning: Oxygen enrichment accelerates combustion vigorously. Care should be taken at all times when working with oxygen. CGA Pamphlets G-4.0 (7) and G-4.1 (8), Guide G88, Practice G93, and ASTM Manual 36<sup>5</sup> provide further details.
- 9.3 *Metal Oxides*—**Warning:** Toxic metal and oxide dusts may be produced when using this test method; safety procedures appropriate to the hazard must therefore be followed.
- 9.4 Vessel Failures—Warning: The vessel described in this standard shall contain the burning of various metallic materials successfully. Molten metal slag produced in these tests can be very destructive, and as pressure or oxygen purity increases, the intensity and risk of uncontrolled burning increases, so the possibility of a burn-through or failure of the vessel or associated piping cannot be ruled out. Additionally, care should be taken to consider the effects of the sample heating method selected on the structural strength of the vessel.

# 10. Sampling, Test Specimens, and Test Units

10.1 Preparation of Samples—Typically, samples shall be prepared as cylindrical rods, 3.2 mm diameter and 101.6 mm long ( $\pm 5\%$ ) minimum (see Fig. 3). The sample length available for burning does not include the part of sample where the a) igniter and promoter or b) sample support is interfaced to the test sample. Samples of other sizes and configurations may also be used and this should be noted on the data sheets and reported. Test results will vary with the specific configuration selected.

<sup>&</sup>lt;sup>5</sup> ASTM Manual 36, Safe Use of Oxygen and Oxygen Systems. Available from ASTM International Headquarters.

- 10.2 Preparation of Promoters—The promoter shall provide sufficient energy to melt the material in contact with the promoter in order to be considered a valid test. See Fig. 4 for a typical promoter design. The promoter selected must be capable of burning at the test pressure.
- 10.3 Cleaning—Samples and promoters shall be cleaned and dried as they would be in the end-use application, if known. If end use cleaning spec is not known or specified then samples and promoters should be cleaned and dried to be free of hydrocarbons or other flammable species that could potentially introduce interferences. Practice G93 provides further details related to cleaning.
- 10.4 Assembly—If an ignition promoter is used, it shall be coupled to the bottom-end of the sample when it is ready to be installed in the test system and coupled to the igniter wire chosen. As shown in Fig. 4, the promoter has a groove in it to allow coupling of the igniter wire.

# 11. Preparation of Apparatus

11.1 General—Maintain the test chamber, its accessories, and the test cell in a visibly clean condition in order to ensure reproducibility of the results, to provide personnel, system and facility safety, and to meet the requirements of calibration and standardization as described in Section 12. Clean the sample preparation equipment as required to prevent the crosscontamination of test materials.

#### 12. Calibration and Standardization

12.1 The test facility shall demonstrate successfully the ability to obtain repeatable data when testing a reference material. The user should purchase a sufficiently large quantity of a single batch of material and verify the repeatability of the test system before it is placed into service and periodically thereafter. A frequency of once per year is recommended. It is

possible that the data may not be reproduced exactly. The user should determine the repeatability that is acceptable for their applications.

# 13. Conditioning

13.1 Pressurize and vent the chamber a sufficient number of times to ensure that no more than 0.01% of the original atmosphere in the vessel remains. If the vessel is pressurized on each cycle to the absolute pressure,  $P_h$ , and then vented to atmospheric pressure,  $P_a$ , the minimum number of cycles, n, required is equal to or greater than that given by the following relationship (9):

$$n = \frac{-4}{\log_{10}(P_a/P_h)} \tag{1}$$

Note 6—It is recommended that the vent gas be analyzed to confirm Eq 1 is producing the required results.

Alternately, the vessel may be purged or evacuated and the vent gas analyzed to confirm oxygen purity.

#### 14. Procedure

- 14.1 Attach the promoter or igniter, or both, to the bottom of the prepared test sample.
- 14.2 Measure the length of the test sample. The portion of the test sample included in the sample mount and the promoter is to be excluded in this measurement.
- 14.3 Install the test sample in the chamber coupled to the sample support.
  - 14.4 Attach the igniter to the power supply.
- 14.5 Seal the chamber and condition in accordance with Section 13.
- 14.6 Pressurize with test gas to the test pressure. Astute use of the existing data given in Guide G94, Table X1.1, may

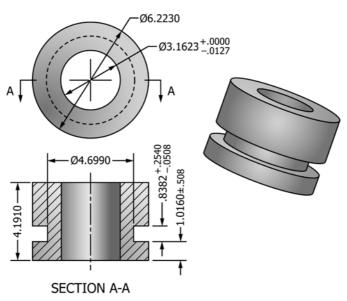


FIG. 4 Typical Promoter Dimension (dimensions in mm)

enable the initial selection of test pressures near the ultimate threshold and reduce the amount of testing required.

- 14.7 Activate the data acquisition system to record all necessary data, including the video recorder, if being used.
- 14.8 If performing heated testing, activate the sample heating system until the target test temperature is reached.
- 14.9 Apply electrical power to ignite the igniter (and promoter, if used).
- 14.10 When the igniter has stopped burning, turn off power to the igniter.
- 14.11 Turn off the test sample heating system (if used) when the test sample has stopped burning.
- 14.12 Turn off the data acquisition system when the test sample burning has ceased completely and adequate post test data has been collected.
- 14.13 Vent the gases in the test chamber and verify that the chamber is depressurized. Ensure that no personnel are exposed to the vent gases. The test chamber can be purged with an inert gas before opening or appropriate respiratory personal protective equipment (PPE) should be used.
- 14.14 Open the chamber and remove the test sample from the sample mount. Be careful since the test sample remaining or chamber parts, or both, may be hot.
- 14.15 Measure the length of the remaining sample to determine the burn length, being careful to not include the igniter and test sample support lengths.

#### 15. Interpretation of Results

- 15.1 If the standard rod test sample is flammable at the initial test pressure and temperature, the lowest burn pressure and lowest burn temperature is at or below this test condition and the highest no-burn pressure and highest no-burn temperature is below this test condition. Testing would then be continued at a lower pressure or lower temperature to determine the lowest burn pressure and highest no-burn pressure and lowest burn temperature and highest no-burn temperature.
- 15.2 If the standard rod test sample is not flammable at the starting test pressure and temperature, the test shall be repeated at least four more times. If the standard test sample is flammable (that is, burns more than 3 cm (1.2 in.) above the promoter) during any of those five tests, the test shall be repeated at the next lower pressure or temperature, or both (suggested test intervals are given in Table 1).

# 16. Report

- 16.1 Record and report the following data during the test. Fig. 5 depicts an example of a typical data sheet that can be used
  - 16.1.1 Material identification (chemical analysis optional);
  - 16.1.2 Preparation history (if any);
  - 16.1.3 Sample dimensions;
  - 16.1.4 Igniter/promoter information;
  - 16.1.5 Sample configuration;
- 16.1.6 Initial and final chamber pressures and chamber volume;
  - 16.1.7 Final chamber temperature (optional);
- 16.1.8 Sample temperature (including description of the method used for this measurement);
  - 16.1.9 Burn length;
  - 16.1.10 Initial and final weight of test sample (optional);
- 16.1.11 Regression rate of the melting interface (apparent burn rate), if measured. See Annex A1 for procedures typically used to calculate the regression rate of the melting interface and the associated error;
- 16.1.12 General observations and description of any unusual behavior such as rapid or erratic burning, production of burning particles or ejecta, and the like phenomena; and
  - 16.1.13 Composition of test gas.
- 16.1.14 Incorporate the following caveat in its entirety in the test report: These data are dependent on the precise test configuration and other test variables, and no criteria are implied for relating them to the suitability of materials for use in any actual system. The application of data obtained from this test method is discussed in Guides G88 and G94 and Manual 36.5

# 17. Precision and Bias

17.1 The precision and bias of this test method have not been determined.

# 18. Keywords

18.1 burning; burning rate; consumption rate; highest noburn pressure; highest no-burn temperature; high-pressure; igniter; lowest burn pressure; melting rate; metal burning; metal combustion; metallic materials; metals ignition; oxygen; oxygen-enriched; promoted combustion; promoted ignition; promoter; regression rate of the melting interface lowest burn temperature; threshold pressure

TABLE 1 Suggested Intervals for Pressure/Temperature to Determine Lowest Burn or Highest No-Burn Pressure and/or Lowest Burn or Highest No-Burn Temperature

Test Pressure (MPa)	Pressure Interval (MPa)	Test Temperature (°C)	Temp Interval (°C)
0 – 0.01	0.02	Ambient – 260	25
0 - 0.69	0.14	260 - 538	50
0.69 - 3.45	0.34	538 - 815	80
3.45 - 6.90	0.69	815 - 1093	100
6.90 - 13.80	1.40	1093 – 1371	130
13.80 - 34.50	3.50	1371 – 1649	150
34.50 - 69.00	6.90	>1650	Not Recommended

# **Promoted Combustion Data Sheet**

				Mater	ial Iden	tificatio	n			
Materia	al Desigr	nation								
Materia	al Manuf	acturer_								
Compo	sition_									
Specifi	cation_									
Heat N	lo			Lot		_	E	Batch_		
Promo	ter Mate	rial			Promote	er Wt		_ (gm)	)	
Igniter	· Materia	l								
	Test Conditions									
Pressu	re	(	Pa)		Cham	ber Volui	me		$(m^3)$	
Test Te	emperati	ıre								
			_ O <sub>2</sub> F							
Name	Seconda	ıry Gas_			Percer	nt Second	dary Gas	S		
	Name Secondary Gas Percent Secondary Gas Percent Tertiary Gas									
Results										
Sample #	Sample Length (mm)	Sample Width (mm)	Sample Dia/Thick (mm)	Pre-test Weight (g)	Post-test Weight (g)	Ignition Pressure (Pa)	Burn Length (mm)	Burn Time (sec)	Regression Rate of Melting Interface (mm/sec)	Sample Temp (°C)
1										
2										
3										
4										
5										
6										
7										
8										
10										

FIG. 5 Typical Data Sheet

Remarks

# **ANNEX**

(Mandatory Information)

# A1. MEASUREMENT OF THE REGRESSION RATE OF THE MELTING INTERFACE (10, 11)

#### A1.1 Visual Method

A1.1.1 The measurement of the regression rate of the melting interface by visual methods typically requires a video recording of the test and a post-test analysis of the video using a predetermined scale factor and imbedded timer. Visual access to the test sample rod is typically obtained through a sight glass in the test chamber.

A1.1.2 The lens of the video recording camera should be positioned to optimize the length of test sample rod viewed through the sight glass. Once the camera is in a fixed position and before conducting a test, video of a calibrated length (that is, ruler) should be recorded as part of the scale factor determination during post-test analysis. The calibrated length should be positioned in the same position and direction as the axis of the test sample rod otherwise an extra trigonometric factor should be incorporated (for parallax). If the width of the test sample rod is used for the calibrated length and the axis of camera lens is not perpendicular to the axis of the test sample rod then an extra trigonometric factor must also be incorporated.

A1.1.3 If necessary, light filters can be used to reduce the luminosity of the burning event and improve the ability to identify and track the melting interface. Alternately, a camera with an automatic iris or internal filtering can be used. The intensity of the light released depends on the metallic material being evaluated.

A1.1.4 Once the video of the burning event is obtained, the regression rate of the melting interface should be determined during review and analysis. If the recording medium is film, then the scale factor should be determined on the playback screen by viewing the recording of the calibrated length and using the embedded timer. If the recording medium is digital, then the scale factor should be determined based on the number of pixels viewed for the calibrated length and the embedded timer.

A1.1.5 The playback of the test event should be performed by tracking and progressively marking the regression of the melting interface along the rod with intervals of specified frame numbers. The tracking should begin above the position of the igniter or promoter, or both. The regression rate of the melting interface is the slope of a line fitted to the melting interface movement per unit time.

A1.1.6 Errors associated with the visual method calculation are uncertainties in the time resolution, identifying the location of the melting interface, and vertical scale factor obtained from

the playback medium. The following summarizes these errors, their sources and their calculation.

A1.1.6.1 Uncertainty in the time resolution of data points  $(\Delta F/F)$ , where  $\Delta F$  is half the inverse of the frame rate (number per second) and F is the total time over the spread of data points tracking the melting interface.

A1.1.6.2 Vertical scale factor uncertainty ( $\Delta S/S$ ) based on the standard deviation ( $\Delta S$ ) from the average value of (S) of many scale factor measurements.

A1.1.6.3 Uncertainty of locating the melting interface ( $\Delta X/X$ ), where X is a length measurement of the range of data points recorded on the playback medium.  $\Delta X$  is the absolute difference between the "pen mark" (analysis) position and the true position of the melting interface. This value can be estimated based upon the thickness of marking instrument, quality of the picture clarity (that is, screen resolution), obscuration of the interface from burning by-products, and accuracy of measurement tool used.

A1.1.6.4 The regression rate of the melting interface is a linear function of the above independent errors. Therefore, the root-sum-square method for calculating the uncertainty in the final value obtained is used;  $\Delta R_M = [(\Delta F/F)^2 + (\Delta S/S)^2 + (\Delta X/X)^2]^{0.5}$ .  $R_M$ , where  $R_M$  is the regression rate and  $\Delta R_M$  is the associated absolute error.

### A1.2 Thermocouple Sensor Method

A1.2.1 This method consists of thermopiles (many thermocouples) positioned at the end of copper tubes that are located along the walls of the combustion chamber. As the luminous region of the burning test sample rod passes the end of the copper tubes the thermopiles provide a real-time response. Knowing the fixed distance between the copper tubes and the time between adjacent thermopiles' responses, the regression rate can be calculated. Other temperature/luminosity sensors (such as infrared) are possible using this method.

# A1.3 Ultrasonic Method

A1.3.1 The ultrasonic method is a high resolution approach that uses a pulse-echo technique to measure the regression rate of the melting interface. In this method an ultrasonic transducer is attached to the top end of the test sample rod and transmits and receives ultrasonic structural waves that reflect off the melting interface. The changing length of the rod is determined using the material's predetermined sound velocity. The changing rod length, as a function of time, corresponds to the regression rate and the method, therefore, provides a real-time measurement.

#### **APPENDIX**

(Nonmandatory Information)

#### X1. CALCULATIONS FOR LOWER PRESSURE LIMITS FOR TESTING

X1.1 When burning occurs during a test, the pressure may rise (due to temperature increase) or fall (due to oxygen consumption) and any inert impurities will increase in concentration by virtue of their not participating in the reaction. In other words, if half of the oxygen is consumed, the concentration of the unaffected inert diluents in the remaining oxygen will be at least double the initial value. To limit the effects of this mechanism, the oxygen available should be sufficient to maintain a reasonably uniform test environment.

X1.2 This test method recommends that no more than 10 % of the available oxygen be consumed during any one test. This will prevent the pressure from falling below 90 % of the initial value. This criterion also limits any increase in trace impurities to about 10 % greater than their initial level. Indeed, in many tests, pressure may, in fact, rise significantly due to temperature increase and the possible production of volatile species. However, a rise in pressure is less of a concern because it increases flammability and leads to conservative results in a safety regard.

X1.3 One can approximate the minimum recommended test pressure for alloys by summing the fractional minimum pressures for each constituent in terms of its mass fraction in the alloy. For example, in a 7.37 3 10<sup>-4</sup> m<sup>3</sup> (0.026 ft<sup>3</sup>) vessel, an alloy of 60 % copper and 40 % iron will have an approximate minimum recommended test pressure of 2337 kPa (339 psi), which is the sum of 60 % of the minimum recommended test pressure for copper, that is, 1689 kPa (245 psi) and 40 %

of the minimum recommended test pressure for iron, that is, 648 kPa (94 psi).

X1.4 If a stoichiometric metal oxide product that is generated during burning can be identified, the equation to calculate the minimum suggested test pressure, P, for a given vessel of volume,  $V^{\nu}$ , specimen of metal, M, having atomic weight, AW, and density, D and which reacts according to the following:

$$XM + YO_2 \rightarrow M_XO_{2Y}$$
 (X1.1)

is given by the following:

$$P = \frac{(10)(Y)(P_a)(MW(O_2))(V_m)(D_m)}{(X)(V_w)(D_a)(AW(M))}$$
(X1.2)

where:

P = minimum suggested test absolute pressure to

limit O<sub>2</sub> loss to 10 %,

 $P_a$  = absolute atmospheric pressure, 101.35 kPa

(14.7 psia),

 $MW(O_2)$  = molecular weight of oxygen, 32,

 $V_m$  = volume of metal specimen,

 $V_{\nu}$  = test vessel volume,

 $D_m$  = density of metal,

 $D_o$  = density of oxygen at 1 atm, room-temperature,

1.33 kg/m<sup>3</sup> (0.08281 lb/ft<sup>3</sup>),

X, Y = reaction coefficients given in Table X1.1, and AW(M) = atomic weight of metal (AW in Table X1.1).

TABLE X1.1 Data for Estimating Minimum Recommended Test Pressure for Testing Ambient Temperature Samples

Element	Oxide	Х	Υ	Atomic Weight, AW	Specific Gravity, sp gr
Al	Al <sub>2</sub> O <sub>3</sub>	2	1.5	26.98	2.6989
Cr	Cr <sub>2</sub> O <sub>3</sub>	2	1.5	51.996	7.19
Co	CoO	1	0.5	58.93	8.9
Cu	CuO	1	0.5	63.54	8.96
Fe	Fe <sub>3</sub> O <sub>4</sub>	3	2	55.847	7.874
Li	Li <sub>2</sub> O	2	0.5	6.939	0.534
Pb	PbO	1	0.5	207.19	11.35
Mg	MgO	1	0.5	24.312	1.738
Mn	MnO <sub>2</sub>	1	1	54.938	7.21-7.44
Mo	MoO <sub>3</sub>	1	1.5	95.94	10.22
Ni	NiO	1	0.5	58.71	8.902
Sn	SnO <sub>2</sub>	1	1	118.69	5.75, 7.31
Ti	Ti <sub>2</sub> O <sub>5</sub>	2	2.5	47.9	4.54
W	WO <sub>3</sub>	1	1.5	183.85	19.3
Zn	ZnO	1	0.5	65.37	7.133
Zr	$ZrO_2$	1	1	91.22	6.53



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