

Temperature Oxygen Index Test

(BS EN ISO 4689-3: 1996)

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**Materials, Processes, and Manufacturing Department
Materials Combustion Research Facility, Building 4623**

**National Aeronautics and Space Administration
George C. Marshall Space Flight Center
Marshall Space Flight Center, AL 35812**

Release Authority	Name	Title	Organization	Date
Office of Primary Responsibility	_____	Chemistry Group Leader	ED36 _____	
	_____	Industrial Safety	QS10 _____	



Revision	Date	Originator	Description	Affected Pages
Baseline	xx/xx/06	Eddie Davis		

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This document revises the Organizational Work Instruction (OWI) for performing oxygen index at elevated temperature tests in the Materials Combustion Research Facility (MCRF). Any deviation to this procedure must be approved by the test engineer via an approved test plan. Any changes to the test equipment shall be noted on the tester maintenance log and approved by the test engineer. It is the responsibility of the test engineer to obtain NASA Contracting Officer's Technical Representative (COTR) approval where necessary for changes to the test equipment.

Any change to this OWI must be submitted to and approved by the Chemistry Group Leader, ED36. Revisions may be also be submitted to the concurring organizations listed below for review and concurrence by memo. The original OWI and all changes shall be maintained by ED36. Any change to materials used requires a change to mechanical drawings, in addition to ED36B team lead approval. All documentation must be approved by the appropriate persons mentioned above and incorporated into the OWI before operation of the reconfigured test equipment can resume.

Concurring organizations:
MCRF Test Operations Contractor
ED36B Team Leader
Environmental Health, AD02M

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1.0 Scope

1.1 Scope

The scope of this operating instruction is BS EN ISO 4689 Temperature Oxygen Index Test, as performed in Marshall Space Flight Center's (MSFC's) Materials Combustion Research Facility. This test method covers a fire-test-response procedure. This test method describes a procedure for measuring the minimum concentration of oxygen that will just support flaming combustion in a flowing mixture of oxygen and nitrogen.

1.2 Purpose

Methods are provided for testing materials that are structurally self-supporting in the form of vertical bars or sheet up to 10.5 mm thick. These methods are suitable for solid, laminated, or cellular materials characterized by an apparent density greater than 15 kg/m³. The methods may also be applicable to some cellular materials having an apparent density of less than 15 k kg/m³. A method is provided for testing flexible sheet or film material while supported vertically.

1.3 Applicability

This instruction applies to the Chemistry Group of the Materials, Processes, and Manufacturing Department. The test method may be used to measure and describe the properties of materials, products, or assemblies in response to heat and flame under controlled laboratory conditions and shall not be used to describe or appraise the fire hazard or fire risk of materials, products, or assemblies under actual fire conditions. Results of this test may be used, however, as elements of a fire-risk assessment that takes into account all of the factors pertinent to an assessment of the fire hazard of a particular end use.

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2.0 Applicable Documents

- ASTM D 2512-82. *Standard Test Method for Compatibility of Materials with Liquid Oxygen (Impact Sensitivity Threshold and Pass-Fail Techniques)*. 1982.
- ASTM D 618. *Practice for Conditioning, Plastics and Electrical Insulating Materials for Testing*. Current revision.
- ASTM D 1071. *Test method for Volumetric Measurement of Gaseous Fuel Samples*. Current revision.
- ASTM D 1622. *Test Method for Apparent Density of Rigid Cellular Foam Plastics*. Current revision.
- ASTM D 2444. *Test Method for Impact Resistance of Thermoplastic Pipe and Fittings by Means of a Tup (Falling Weight)*. Current revision.
- ASTM D 4802. *Specifications for Poly (Methyl Methacrylate) Acrylic Plastic Sheets*. Current revision.
- ASTM E 176. *Terminology of Fire Standards*. Current revision.
- ED36-OWI-042. *Test Sample Preparation for Testing in the Materials Combustion Research Facility*. Current revision.
- ED36-OWI-050. *Materials Combustion Research Facility Guidelines for Test Operations*. Current revision.
- ED36-OWI-051. *Receipt, Handling, Prioritizing, and Data Requirements of Samples Submitted for Testing in the Materials Combustion Research Facility of the Materials, Processes, and Manufacturing Department, Building 4623*. Current revision.
- ED35-OWI-058. *Chemical Hygiene for Building 4623*. Current revision.
- ISO 4589-2. *Plastics-Determination of Flammability by Oxygen Index-Part 2, Ambient Temperatures*. Current revision.
- ISO 7823-1. *Poly (Methylmethacrylate) Sheets-Types, Dimensions and Characteristics-Part 1-Cast Sheets*. Current revision.
- MPD 1840.3. *MSFC Respiratory Protection Program*, December 1999.
- MPG 1040.3H. *MSFC Emergency Plan*, June 2001.

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MPG 1840.2A. *MSFC Hazard Communication Program*, September 2000.

MPG 8715.1A. *MSFC Safety, Health, and Environmental (SHE) Program*, August 2001.

MSFC-SPEC-164B. *Specification for Cleanliness of Components for Use in Oxygen, Fuel, and Pneumatic Systems*. 1994.

MWI 3410.1D. *Personnel Certification Program*, July 2001.

MWI 8621.1A. *Close Call and Mishap Reporting and Investigation Program*, August 2001.

Note: Always reference the current version of an applicable document.



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3.0 Definitions

3.1 Definitions

Elevated temperature oxygen index: The minimum concentration of oxygen, by percent volume, in a mixture of oxygen and nitrogen, introduced into an agreed test temperature of greater than 25°C, that will just support combustion of a material under specified test conditions.

Flammability temperature: The temperature at which combustion of a material is just supported in air under specified test conditions

Ignition. The initiation of flaming combustion.

Limited access. A term meaning, “Only the test operator may enter the test cell with appropriate personal protective equipment.”

NASA. Marshall Space Flight Center EM10 responsible personnel.

No access. A term meaning, “No one may enter the test cell.”

Oxygen index. The minimum concentration of oxygen determined by the method described in section 9.1, expressed as volume percent, in a mixture of oxygen and nitrogen that will just support flaming combustion of a material initially at 23 ±2 °C under the conditions of this test method

Tag out. Placement of a tag-out device on an energy-isolating device to indicate that the energy-isolating device and equipment being controlled must not be operated until the tag-out device is removed by the person who placed it there.

Test area. The portion of Building 4623 and fenced area south of the north wall of Room 126D.

Test cell. Room 126D of Building 4623, which contains the temperature oxygen index testing equipment.

Test engineer. The person responsible for correctly following the approved test plan for a specific test -- from sample receipt to test data evaluation.

Test operator. The person responsible for conducting a test under the guidance of the test engineer.

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3.2 Acronyms

<i>MCRF</i>	Materials Combustion Research Facility
<i>MSDS</i>	Material Safety Data Sheet
<i>MSFC</i>	Marshall Space Flight Center
<i>OI</i>	Oxygen Index
<i>OWI</i>	Organizational Work Instruction
<i>PMMA</i>	Poly(methyl methacrylate)
<i>RH</i>	Relative Humidity

3.3 Symbols

C_F	Oxygen concentration of final test [% (V/V)]
d	Oxygen concentrations increment [% (V/V)]
D	Internal diameter (mm) of glass chimney
FT	Flammability temperature ($^{\circ}C$)
k	Factor to be determined from Table 3 (?)
l	liter
mm	millimeter
N_L	Series of tests giving the same response as the oxygen index determination
N_T	Series of tests in the oxygen index determination (includes the last 5 tests and the N_L series of tests in the determination)
OI	Oxygen index at elevated temperature [% (V/V)] (temperature must be specified)
s	second
u	Linear flow rate [40 (± 0.8) mm/s]
V	Volume flow rate (l/min)
σ^*	Standard deviation [% (V/V)]

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4.0 Instructions

All operations of this equipment will be conducted using the applicable documents referenced above (section 2). All data and test results will be recorded on form _____, the Temperature Oxygen Index Test Data Sheet (section 7.x, Figure X). A summary of pertinent test information and test results will be compiled in an NASA memo, signed by the test organization management, and mailed to the test requester.

4.1 Sample Preparation

The *sample preparation technician shall prepare* temperature oxygen index samples according to ED36-OWI-042, *Test Sample Preparation for Testing in the Materials Combustion Research Facility* (current version). When non-standard samples are required, the *sample preparation technician shall follow* the directions written in the test plan for that test request. *If this information is not provided with the test plan, the sample preparation technician shall seek clarification* from the test engineer.

Before testing begins, the *test operator shall review* the information supplied on the test data sheet (prepared by the sample preparation technician) to make certain the information is complete and appears sound. *If a problem is identified, the test operator shall notify* the test engineer. The *test operator* shall also:

- **Verify** that the test request number and material designation are identical on all paperwork.
- **Confirm** that the prepared samples agree with the test request.
- **Verify** that the sample preparation technician has noted if the sample has been cleaned or if the sample does not require cleaning.
- **Note** any flaws or imperfections in the sample, and **record** these on the test data sheet.
- **Review** the test plan signed by NASA and the original test request before proceeding. *If the test plan and the test request do not agree, request clarification* from the test engineer, who shall query NASA.

4.2 Pre-Test Photography

The *sample preparation technician shall take* a pre-test photograph of at least one of the samples and **place** three copies of the photograph in the test folder. *If the pre-test photograph has not been taken, the test operator shall take* the photograph and **place** three copies of the photograph in the test folder before proceeding with the test. The entire sample must be visible in the photo. Steps for photographing samples are outlined in the *Photography Operating Guide*.

Note: we need to provide a section for the Sample Prep OWI also.

4.3 Equipment Checkout

At the beginning of the test day, the *test operator* shall perform the following steps:

4.3.1. **Glance** at the oxygen monitor station located in the break room. **Locate** the monitor for Room 126D. **Ensure** that the digitized LED readout indicates a normal oxygen level (19.5 to 23.5%) before entering the test cell.

4.3.2. **Ensure** that the test cell floor and the room behind the test cell are visibly clean. *If conditions warrant*, **scrub** the floor of the cell with a detergent solution, and **rinse** with water.

4.3.3. **Ensure** that the fume hood over the tester is operating correctly.

4.3.4. **Ensure** that gas lines are securely attached to the rear panel ports.

4.3.5. **Ensure** that the control unit selection switch is set to 110 V.

4.3.6. **Press** the **POWER** button to turn on the unit. **Ensure** that the **POWER** button lights up and that the temperature and oxygen concentration displays are functioning.

4.4 Sample Loading and Marking

4.4.1. **Inspect** the contents of the current test folder to ensure that the following information is provided:

- Test request
- Signed test plan
- Test data sheets
- Sample preparation sheet
- Test material's MSDS or the Exclusion Statement for the material/component being tested
- Pre-test photographs (See section 4.2.)
- Oxygen Index Pre-Test Checklist.

WARNING: Read the test material's MSDS to ensure familiarity with all safety precautions associated with the material. Verify that the test engineer is aware of all highly hazardous, reactive, or toxic components of the test material. The *test engineer* shall direct the test operator in proper safety procedures concerning these test materials.

4.4.3.2. **Turn on** warning lights to indicate that hazardous testing is in progress. This places the test area in a *limited-access* condition.

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4.4.3.3. **Ensure** that the surfaces of the specimens are clean and free from flaws that could affect burning behavior, *e.g.*, peripheral moulding flash or burrs from machining.

4.4.2. *If a test specimen is not self-supporting at the temperature of the test, support* it externally using 0.55- (± 0.05 -) mm diameter nickel-chromium alloy wire with a maximum working temperature of 1100°C . **Secure** the samples with ties of copper wire of 0.20- (± 0.02 -) mm diameter.

4.4.3. Marking of the test specimens. To monitor the distance over which a specimen burns, the specimen may be marked with transverse lines at one or more levels, depending on sample form and ignition procedure.

4.4.3.1. **Mark** self-supporting samples on at least two adjacent faces. *If using wet ink, ensure* that the marks are dry before the specimen is ignited.

4.4.3.2. **Mark** specimens of Types I, II, III, or IV that will be tested in accordance with Procedure A (top surface ignition) 50 mm from the end to be ignited.

4.4.3.3. **Mark** specimens of Types I, II, III, or IV that will be tested in accordance with Procedure B at 10 mm and at 60 mm from the end to be ignited.

4.4.3.4. Reference marks for Type V specimens are on the supporting frame; however, for convenience, these may be marked at 20 mm and at 100 mm from the end to be ignited when testing heat-stable materials. **Ensure** that, when mounting a specimen in the holder, the marks on the frame correspond with those on the specimen.

4.4.4. **Condition** the test specimens for at least 88 hours at 23 (± 2) °C and 50 (± 5)% relative humidity (RH) immediately before testing.



Note: For cellular materials that may contain volatile flammable material, **precondition** the samples in suitable ventilated ovens for 168 hours before conditioning them at 23 °C and 50% RH.

4.5 Detailed Test Procedure

4.5.1. **Maintain** the ambient temperature for the test apparatus at 23 (± 2) °C. *If necessary, keep* the test specimens in an enclosure at 23 (± 2) °C and 50 (± 5)% RH from which each test specimen may be taken when required.



Note: Ensure the nitrogen/oxygen gas mixture temperature is 23 (± 2) °C.

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4.5.2. Ensure that the Temperature Index control unit is connected to the heated glass chimney correctly and that the Oxygen Index control unit is connected to the oxygen and nitrogen gas supplies.

4.5.3. Ensure that the oxygen and nitrogen ball valves located on the front of the Oxygen Index Unit are both in the **OFF** position. Set the nitrogen and oxygen gas regulators to 2.0 bar (29 psig) *if the instrument has a standard flowmeter (1-12 l/min air)* or 2.75 bar (39.9 psig) *if a 2-25 l/min air flowmeter is fitted*. **Do NOT allow** the line pressure to exceed 3 bar (43.5 psig), as damage to the oxygen analyzer may result. **Ensure** that both the Oxygen Index and Temperature Index control units are connected to the main power supply, and **press** the power button on both units to turn the instruments on. **Ensure** that the power buttons light and that the temperature and oxygen concentration displays are functioning on both units.



4.5.4. Position the heated glass chimney on the Oxygen Index control unit, and **turn on** the extraction system.

4.5.5. Position the sample thermocouple inside the heated chimney at the position to be occupied by the *top* of the test specimen.

4.5.6. Press the pump button to turn the air pump on. **Adjust** the needle valve on the flowmeter to obtain a flow rate equivalent to 40 (± 0.8) mm/s flow through the chimney.

Note: For a glass chimney of internal diameter 75 mm, 40 mm/s linear flow is indicated by 10.6 l/min.



4.5.7. Set the Preheat and Column temperature controller set points to the temperatures required to obtain the temperature at the specimen to conduct the oxygen index tests. (**Refer** to **Figure 7-x** for guidance on the appropriate settings.)

4.5.8. Press the Preheat and Column buttons to power the heating elements.

4.5.9. Monitor the temperatures on all three displays (Preheat, Column, and Sample). **Wait** for the temperatures to stabilize.

Note: The test standard specifies that the temperature in the vicinity of the test specimen must be maintained at ± 2 °C. **Monitor** the temperature, measured by the sample thermocouple, over the region from 30 mm above the top of the test specimen to 70 mm below the top of the test specimen, *i.e.*, a 100-mm region. This temperature must be maintained at ± 2 °C of the temperature measured at the top of the specimen. **Adjust** the preheat and column temperatures as required, allowing the system to reach equilibrium.





Note: The maximum sample temperature at which elevated temperature oxygen index tests can be conducted is 400 (± 3) °C [752 (± 5.4) °F].



Note: The adjustment of the independent Preheat and Column controls should be such that the preheater raises the incoming gas mixture temperature at the base of the column to approximately that of the column itself.

4.5.10. Turn the nitrogen valve on the Oxygen Index control unit to the **ON** position.

4.5.11. Ensure that the nitrogen needle valve is fully open, and **adjust** the Flostat to obtain a flow rate equivalent to 40 (± 0.8) mm/s flow through the chimney. **Press** the pump button to turn the pump off.



Note: For a glass chimney of internal diameter 75 mm, 40 mm/s linear flow is indicated by 10.6 l/min.

4.5.12. Allow the oxygen analyzer to settle; then **use** a screwdriver on the zero adjust (on the side of the Oxygen Index control unit) to obtain a reading of 0.0%.

4.5.13. Ensure that the oxygen needle valve is fully open. **Turn** the oxygen valve to the ON position and the nitrogen valve to the OFF position.

4.5.14. Assuming the same line pressure for nitrogen and oxygen and about 30 to 40 s after opening the valve, the flow rate indicated should be that shown for the nitrogen in step 4.5.13. *If it is not*, **adjust** the pressure regulator on the oxygen bottle until the required flow rate is obtained. This ensures that the two gases are at the same pressure.

4.5.16. Using the span adjust on the front panel of the Oxygen Index control unit, **set** the display to read the oxygen concentration specified on the supply bottle (99.5% for commercial grade, 99.9(5)% (?) for high-purity oxygen). **Turn** the oxygen valve to the **OFF** position, and **press** the pump button to turn the pump on.



Note: The column and preheat elements must not be used without a gas flow through the system. Always **ensure** that either the pump is on or the oxygen/nitrogen mixture is flowing through the chimney. To conserve the bottled gases, only **use** the oxygen and nitrogen while testing a specimen. **Revert** to having air pumped through the system between tests.

4.5.17. Select an initial concentration of oxygen to be used. When possible, this may be based on experience of results for similar materials. Alternatively, **try to ignite** a test specimen in air, and **note** the burning behavior. *If the specimen burns rapidly*, **select** an initial concentration of ~18% oxygen; *if the test specimen burns gently or unsteadily*, **select** an initial concentration of ~21%; *if the*

specimen does not continue to burn in air; **select** an initial concentration of at least 25%, depending on the difficulty of ignition or the period of burning before extinguishment in air.

4.5.18. **Turn** the nitrogen valve slowly to the ON position; then, **turn** the oxygen valve to the ON position. **Press** the pump button to turn the pump off. **Turn** the nitrogen valve slowly to the **ON** position; then **turn** the oxygen valve to the **ON** position. Using the oxygen gas needle valve, **obtain** the required oxygen concentration at the test flow rate of 40 (± 0.8) mm/s.

Note: For oxygen concentrations in excess of ~50 %, the oxygen needle valve should be fully open and the nitrogen valve used to obtain the required concentration. **Always ensure** that one of the needle valves is fully open, using the other valve to adjust the concentration. The valves should be turned on slowly to avoid an initial surge of flow, a result of the back pressure in the system. When turning the valves on, **always turn** the nitrogen valve on fully before turning on the oxygen valve.



Note: When the oxygen/nitrogen mixture is flowing through the chimney, **ensure** that the pump is off to avoid generating incorrect results.



4.5.19. **Ensure** that the temperature at the specimen is as set in step 4.5.9. **Move** the sample thermocouple away from the position where the specimen will be placed.

4.5.20. **Ensure** that the neck valve on the igniter (just above the handle) is closed, and **turn** on the propane bottle. **Open** the neck valve only a crack, and **ignite** the gas at the end of the tube. **Place** the tube vertically in the chimney, and **adjust** the neck valve to obtain a flame that projects 16 (± 4) mm vertically downward.

4.5.21. **Remove** the propane igniter, and **mount** a specimen vertically and centrally in the appropriate specimen holder. Using the specimen holder insertion device, **place** the specimen holder on the supporting stem. *If the cap at the top of the heater chimney is removed while positioning the specimen, replace the cap immediately.*

Note: The top of the specimen must be at least 100 mm below the open top of the chimney and the lowest exposed part of the specimen must be at least 100 mm above the top of the debris tray.



4.5.22. **Allow** the specimen to preheat at the test temperature for 240 (± 10) s. **Adjust** the oxygen concentration, *if necessary*, to ensure that the oxygen concentration remains at the required value.

4.5.23. **Lower** the flame into the chimney (with the tube vertical), and **ignite** the specimen.



Note: The procedure chosen determines how the flame must be applied to the specimen. Procedure A is used for specimens of Types I, II, III, and IV; Procedure B is used for specimens of Type V.



Note: For tests on materials that exhibit steady burning and spread of combustion in oxygen concentrations at or close to the oxygen index value or for self-supporting specimen ≤ 3 mm thick, Procedure B may give more consistent results than Procedure A. Procedure B may then be used for specimens of Type I, II, III, or IV.

4.5.23.1. Procedure A (top surface ignition): **Apply** the lowest visible part of the flame to the top of the specimen using a sweeping motion, *if necessary*, to cover the whole surface. **Take care** not to maintain the flame against the vertical faces or edges of the specimen. **Remove** the flame every 5 s just long enough to see whether the entire top surface of the specimen is burning. *If the entire top surface of the specimen is burning, remove* the igniter from the chimney, **turn off** the igniter, and **start** the timer. *If the specimen is not burning, reapply* the igniter to the top of the specimen, and **remove** it after another 5 s. **Repeat** this procedure for a maximum of 30 s, after which, *if the material has not ignited, deem* it “non-ignitable,” and **record** an “O” response.

4.5.23.2. Procedure B (propagating ignition): **Use** the igniter to apply the visible flame to the end face of the specimen and to a depth of ~6 mm to its vertical faces. **Apply** the flame for up to 30 s, and **remove** it every 5 s to observe the specimen for ignition, as in Procedure A. **Start** the timer when any part of the visibly burning portion reaches the level of the upper reference mark.



Note: Ignition is defined as occurring when the vertical faces are burning steadily or the visibly burning portion first reaches the level of the upper reference mark.



Note: Ignition implies the initiation of flaming combustion; however, some materials may exhibit a non-flaming type of combustion instead of (or at a lower oxygen concentration than that required for) flaming combustion. When testing such materials, **identify** the type of combustion for which the oxygen index is required or measured.

4.5.24. **Observe** the burning behavior, and **note** the duration of burning. *If neither the period nor the extent of burning exceeds the relevant limit specified in Table 4 for the appropriate specimen, note* the duration and extent of burning, and **record** an “O” response for the specimen. *If either the period or extent of burning exceeds the relevant limit specified in Table 4, note* the burning behavior accordingly, e.g., >180 s or >50 mm, and **record** an “X” response for the specimen.

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Table 4.
Criteria For Oxygen Index
Measurements

Specimen Type	Ignition Procedure	Alternative Criteria	
		Period Of Burning After Ignition/s	Extent Of Burning*
I, II, III, IV	A: Top Surface	180	50 mm below top of specimen
	B: Propagating	180	50 mm below upper reference mark
V	B: Propagating	180	80 mm below upper reference mark

*The extent of burning is exceeded when any part of the visibly burning portion of the specimen, including burning drips descending down the vertical faces, passes the level defined in the table

4.5.25. Extinguish the burning specimen by turning the oxygen valve to the **OFF** position.

4.5.26. Press the pump button to turn on the pump; then, **ensure** both gas ball valves are in the **OFF** position. **Remove** the specimen, and **clean** any surfaces within the chimney or on the igniter that have become contaminated with soot, *etc.*

4.5.27. Repeat the test with a new specimen and a new temperature value determined by the response of the preceding specimen. **Determine** the new value by (a) decreasing the oxygen concentration *if the burning behavior of the preceding specimen gave an "X"* or (b) increasing the oxygen concentration *if the burning behavior of the preceding specimen gave an "O."*

4.5.27.1. Determine the preliminary oxygen concentration. Oxygen concentration changes are of any convenient step size until two oxygen concentrations have been found that differ by $\leq 1\%$ and of which one gave an "O" response and the other an "X" response. These need not be from consecutive specimens. The preliminary oxygen concentration is taken to be the concentration that gave the "O" response.

4.5.27.2. Determine the oxygen index based on the preliminary oxygen concentration. **Test** another specimen at the preliminary oxygen concentration, and **record** its response. (This need not be the same response as the run previously performed at this concentration.) This test is the first in the N_L and N_T series of tests.

4.5.28. Test a new specimen with the oxygen concentration change step size, $d = 0.2\%$ in accordance with step 4.5.27. **Record** the oxygen concentration and the response. **Repeat** until a different response to that observed in the first test is obtained. Tests giving the same response constitute the N_L series.

4.5.29. Once a different response has been recorded, **test** four more specimens with a step size of 0.2% and in accordance with step 4.5.27. **Record** the oxygen concentration and test response for all the tests. The last 5 tests and the tests in the N_L series constitute the N_T series. ($N_T - N_L = 5$).

4.6 Shutdown Procedure for Elevated Temperature Oxygen Index

- 4.6.1. When testing has been completed, **press** the Preheat and Column buttons to turn off the heating elements.
- 4.6.2. **Press** the pump button to turn the pump on, and **check** the flow rate.
- 4.6.3. **Turn** off both gas supply isolation valves, and **allow** the lines connecting to the Oxygen Index control unit to bleed before turning the nitrogen and oxygen ball valves to the OFF position.
- 4.6.4. **Allow** air to pump through the heater chimney for 10 minutes; then **turn off** the pump.
- 4.6.5. **Press** the power buttons on the both the Oxygen Index and Temperature Index to turn the units off. **Disconnect** the main power cables.
- 4.6.6. **Ensure** that the propane bottle is turned off, and **flare off** the excess propane in the line by opening the neck valve and igniting the gas.
- 4.6.7. **Clean** the units, chimney, debris tray, and specimen holders using non-abrasive cleaners.



Note: Do not clean until it has been disconnected from the main power supply.

4.7 Detailed Test Procedure - Flammability Temperature

- 4.7.1. **Maintain** the ambient temperature for the test apparatus at $23 (\pm 2) ^\circ\text{C}$. *If necessary, keep* the test specimens in an enclosure [at $23 (\pm 2) ^\circ\text{C}$ and $50 \pm 5\%$ RH] from which each test specimen may be taken when required.
- 4.7.2. **Ensure** that the Temperature Index control unit is connected to the heated glass chimney correctly.
- 4.7.3. **Connect** the Temperature Index control unit to the main power supply, and **press** the power button to turn on the instrument. **Ensure** that the power button lights and that the temperature displays are functioning.
- 4.7.4. **Position** the heated glass chimney under an extraction hood, and **turn on** the extraction system.

4.7.5. Position the sample thermocouple inside the heated chimney at the position to be occupied by the *top* of a test specimen.

4.7.6. Press the pump button to turn on the air pump. **Adjust** the needle valve on the flowmeter to obtain a flow rate equal to 40 (± 0.8) mm/s flow through the chimney. **Maintain** this flow for the duration of the tests.

Note: For a glass chimney of internal diameter 75 mm, 40 mm/s^l linear flow is indicated by 10.6 l/min.

Note: The Column and Preheat elements must not be used without air flow through the system. **Always ensure** that the pump is on.

4.6.7. Set the Preheat and Column temperature controller set points to the temperatures required to obtain the temperature *at the specimen* to conduct the oxygen index tests. (**Refer** to Figure 7-x for guidance on the appropriate settings.)

4.7.8. Press the Preheat and Column buttons to power the heating elements.

4.7.9. Monitor the temperatures on all three displays. **Wait** for the temperatures to stabilize.

4.7.10. Adjust the Preheat and Column temperatures as required to obtain a temperature gradient of $<5^{\circ}$ C between the position of the top of the specimen and 50 mm below this position. **Position** the thermocouple 25 mm below the position of the top of the test specimen. **Allow** the temperature to stabilize.

Note: The maximum sample temperature at which flammability temperature tests can be conducted is 400 °C (752 °F) .

Note: **Adjust** the independent Preheat and Column controls such that the preheater raises the incoming air temperature at the base of the column to approximately that of the column itself. The use of widely differing settings between preheater and column temperatures can contribute to variation in the Flammability Temperature value obtained.

4.7.11. Ensure that the neck valve on the igniter (just above the handle) is closed. **Turn on** the propane bottle. **Open** the neck valve *only a crack*, and **ignite** the gas at the end of the tube. **Place** the tube vertically in the chimney, and **adjust** the neck valve to obtain a flame that projects 16 (± 4) mm vertically downward. **Remove** the propane igniter.

4.7.12. Preheat a specimen holder to the temperature measured by the sample thermocouple, and **mount** the specimen vertically and centrally in the appropriate specimen holder. Using the specimen holder insertion device and wearing heat protection gloves, **place** the holder on the supporting stem.





Note: The top of the specimen must be at least 100 mm below the open top of the chimney, and the lowest exposed part of the specimen must be at least 100 mm above the top of the debris tray. **Reposition** the specimen *if necessary*.

4.7.13. With the sample thermocouple positioned immediately adjacent to the test specimen, **leave** the apparatus for 240 (± 10) s to establish temperature stabilization. **Record** the indicated air temperature at the sample. *If the recorded air temperature after stabilization is >10 °C different from the temperature set in step 4.7.10, **discard** the test specimen, and **repeat** steps 4.7.10 and 4.7.12. If otherwise, **move** the sample thermocouple away from the specimen, and **proceed** to ignite the specimen immediately.*



Note: It may be helpful to preheat the specimen holder insertion device *if an excessive temperature difference is induced by the cooling effect from that tool*.



Note: Use of periods other than 240 (± 10) s between introduction of the test specimen and the initial application of the igniter may lead to non-comparable results when testing materials for which the Flammability Temperature test is sensitive to the period of exposure to elevated temperature.

4.7.14. **Lower** the flame into the chimney with the tube vertical, and **apply** ~6 mm of the flame on the top of the test specimen. As the test specimen burns, **lower** the burner to maintain the flame impingement of ~6 mm. **Apply** the flame for 15 (± 1) s to ignite the test specimen. Upon removal of the flame, **start** timing.

4.7.15. **Observe** the burning behavior, and **note** the duration of burning. **Record** whether the flame is extinguished before either of the relevant criteria from Table 4 are satisfied. Alternatively, when either of the criteria are exceeded, **snuff out** the flame, and **record** the response of the specimen.

4.7.16. **Remove** the specimen, and **clean** any surfaces within the chimney, or on the igniter that have become contaminated with soot, *etc.*

4.7.17. **Reposition** the sample thermocouple. Depending on the behavior of the preceding test specimens, **select** the temperature for the next burn and **adjust** the Column and Preheat controllers as necessary to obtain the required temperature at the sample thermocouple, in accordance with the criteria in step 4.7.10. **Reduce** the temperature *if the burning of the previous specimen exceeded the criteria*, or **increase** the temperature *if neither criterion was exceeded*.

4.7.18. **Repeat** the test with a new specimen once the sample temperature has stabilized.

4.7.19. **Continue** repeating the procedure until the Flammability Temperature is established to within an increment of 5 °C as the lowest column temperature as recorded in step 4.7.13, at which the test specimen exceeds at least one of the

test criteria. Increments of >5 °C may be used to establish an approximate value of the Flammability Temperature.

4.7.20. Determine at least three values for the Flammability Temperature of the material by conducting the test procedure at least three times.

4.8 Shutdown Procedure for Flammability Temperature

4.8.1. Press off the Preheat and Column buttons.

4.8.2. Allow air to pump through the system for 10 minutes; then **press** off the **Pump** button.

4.8.3. Press the **Power** button to turn off the control unit.

4.8.4. Disconnect the main power cable.

4.8.5. Ensure that the propane bottle is turned off, and **flare off** the excess propane in the line by opening the neck valve and igniting the gas.

4.8.6. Clean the unit, chimney, debris tray, and specimen holders using non-abrasive cleaners.

Note: Do not clean the unit until it has been disconnected from the main power supply.



4.9 Data Recording and Reduction; Post-Test Photography

4.9.1. Data Recording

4.9.1.1. *If neither the period nor extent of burning exceeds the relevant limit specified in Table 7.x for the applicable specimen, note* the duration and extent of burning, and **record** as an “O” response.

4.9.1.2. *If either the period or extent of burning exceeds the relevant limit specified in Table 7.x, note* the burning behavior, **extinguish** the flame, and **record** as an “X” response.

4.9.1.3. Note the burning characteristics of the material, *e.g.*, dripping, charring, erratic burning, glowing combustion, or after-glow.

4.9.1.4. Complete the test report data sheet (section 7.x, Figure 7-1). **Place** these sheets and the completed Pre-Test Checklist (section 7.x, Figure x) in the test folder.

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4.9.2. Photograph reacted samples, charred or melted test equipment, or any anomalies. **Document** these in writing on the test report data sheet. **Do not photograph** post-test samples that did not react. **Take** photographs as close to the samples as possible. More than one sample or reaction per photograph is acceptable, if the details of reactions are visible. **Refer** to the *Photography Operating Guide* for procedures for taking photographs. **Place** two copies of each post-test photograph in the test folder before returning the folder to the engineer. Photographs shall be retained indefinitely.



Note: *If there are several reactions and samples are hard to handle, representative photos may be taken and labeled as such. The test engineer, in consultation with NASA, will decide whether to make representative photos on a case-by-case basis.*

Package samples and inserts from reacted samples in clear photography slide sleeves. **Label** the protector with the test request number, NASA-STD-6001 test type, temperature at which the test was performed, reactions per number of samples tested, pressure at which the test was performed, and the date. **Use** a red pen to label reacted samples; **use** a black pen to label all other samples. **Identify** the sample by sample number. **Return** the samples with the test folder to the test engineer for evaluation. The *test engineer shall return* samples to the *sample preparation technician* who **shall store** them for future reference.

4.9.3. Include the following information in the test report:

- A reference to this test method
- Date of testing
- A statement that test results relate only to the behavior of the test specimens under the condition of this test method and that these results must not be used to infer the fire hazards of the material in other forms or under other fire conditions
- Identification of the material tested, including (where relevant) the type of material, density, previous history, specimen orientation with respect to any anisotropy in the material or sample, and the date of manufacture with lot number
- The oxygen index as determined in 7.1.1
- The test specimen type or dimension
- The gas measurement and control device accuracy
- The ignition procedure used [Test Method A (step 4.5.8) or Test Method B (step 4.5.9)]
- When Procedure B is used, the relevant specified minimum oxygen index of the material and if the material tested had a lower or higher oxygen index
- If applicable, the estimated standard deviation of the oxygen concentration increment used, if other than 0.5 percent
- A description of any relevant characteristics or behavior, such as charring, dripping, severe shrinkage, erratic burning, after-glow
- Any variations from the requirements of this test method.

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5.0 Notes

Custodians for EM10-OWI-CHM-063

Master List and Document Control	EM10 Management Support Assistant
Alternate Document Control	EM10 Group ISO Representative
Memos	Materials Compatibility Team ISO Representative

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6.0 Safety Precautions and Warning Notes

6.1 Hazards

Warning

Death, severe personal injury, or loss of major equipment may result if maintenance or operating procedures, techniques, restrictions, etc., are not followed exactly.

Safety shall have precedence over all activities. The testing system involves several hazards to the operator and facility, including exposure to the following hazards:

- Exposure to an oxygen-deficient environment
- Flammability and health risks from volatile cleaning solvents
- Handling and moving of heavy parts of the test apparatus on a regular basis
- Potential of explosion and hazardous fume by-products from burning materials in a pure oxygen or oxygen-enriched environment
- Electrical shock related to operation of equipment
- Potential touch temperature risks from hot surfaces when handling test equipment
- Pressurized systems with nitrogen.

6.2 Safety Precautions

6.2.1. Plan test setup, testing, and shutdown so that at least one test operator is in the test area and one other person is in Building 4623 during normal business hours. After normal business hours and on weekends, a test engineer must be in Building 4623 during all test activities. **No more than five personnel** shall be in the test area at any given time. Operation of tests shall comply with ED36-OWI-050, *Materials Combustion Research Facility Guidelines for Test Operations*.

6.2.2. Do not operate controls when personnel are working with the tester. **Place** on the control console the sign warning that personnel are working in the test cell.

6.2.3. Refer to the MSDS for information on personal protective equipment required for materials being handled (sample materials, solvents used, gaseous nitrogen, liquid nitrogen, and liquid oxygen.) **Wear** safety apparel appropriate for test specimens and conditions:



- Safety shoes when there is a danger of foot injuries from falling or rolling objects, objects piercing the sole of the shoe, or when feet may be exposed to an electrical hazard
- Clean laboratory coats when working with enriched oxygen or other oxidizers, combustion by-products, compressed gases, or flammable solvents
- Chemically resistant goggles and gloves while cleaning test equipment and while working with solvents

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- Clean thermal gloves and goggles when pouring, handling, or transferring cryogenic fluids. Hydrocarbon residue can contaminate equipment and affect oxygen compatibility test results or damage the tester.
- A respirator when working with solvents in closed or poorly ventilated spaces. **Note** that the appropriate respirator must be worn as indicated on the MSDS. Cartridge respirators are only good for the constituents listed on the filtration cartridge and for dust particle filtration. Personnel must be qualified to use the respirator, and the respirator must be supplied by MSFC.
- Hearing protection during testing
- Safety glasses at all times while in the test cells.

6.2.4. Serious tissue damage can occur on exposure to cryogenic LOX or LN₂, cold vapors, or cold equipment. *If injury occurs, call 911 and ask for medical assistance.* Bystanders can (**but are not required to**) do the following:

- *If it is safe to do so, remove the person* from the source of cold.
- *In the event of full-body cryogenic exposure and if it is safe to do so, remove the person* from the exposure atmosphere, and **keep the person's airway open. Loosely wrap the person** in a blanket until medical personnel arrive.
- **Do not remove** frozen gloves, shoes, or clothing.
- **Do not massage** affected part(s).
- **Do not expose** affected part(s) to temperatures above 112 °F (45 °C).
- **Do not apply** ice, snow, or ointments to affected part(s).



6.2.5. **Smoking is not permitted** in Building 4623. The test area is generally an oxygen-enriched environment. Open flame or other high-temperature sources are not permissible in the testing area while enriched-oxygen conditions exist. **Do not smoke or expose clothing** to an open flame for 30 minutes after handling liquid or gaseous oxygen.



6.2.6. **Activate** the building warning system for the duration of all testing, including pre- and post-test procedures. **Evacuate** the test area immediately when the oxygen alarm sounds and lights flash.

6.2.7. *In case of an uncontrollable LOX fire, do not try to extinguish the fire.* **Evacuate** the area immediately. **Call 911** to notify the fire department.

6.2.8. **Do not store** anything in the test cell area other than parts or components of the testing apparatus that are designated as spare parts and the tools necessary for routine equipment maintenance. **Remove** all other materials from the test area. **Place** any spare parts that will be exposed to an enriched-oxygen environment in the secured inventory area.

6.2.9. **Ensure** that the ventilation system fan is on continuously during testing, including pre-test and post-test activities, to bring in fresh air and remove fumes and other combustion by-products from the test cell.

6.2.10. Perform all testing **remotely**. No one is allowed in the test cell during a test. Only the test operator(s) shall be allowed in the test cell during pre-test and post-test activities. Sample technicians are allowed, as required, to prepare liquid samples for testing. Other people shall be allowed in the test cell before and after testing as allowed by the test engineer.



6.2.11. Ensure all electrical components, wiring, etc., are in good condition and properly connected and grounded. **Do not operate** electrical devices when floors in the test cell are wet. **Use caution** when operating any electrical equipment.

6.2.12. Do not use glass dewar flasks for the transfer of cryogenic fluids. (**Refer** to step 4.4.8 of this work instruction.)

6.2.13. Clean all equipment that will contact LOX as described in section 9.3, Required Tester Maintenance, and section 4.3, Equipment Checkout, before the equipment is exposed to LOX.

6.2.14. When handling cylinders and dewars or making connections for compressed gases and/or liquids, **refer** to *Working Safely with Compressed Gases and Cryogenics* and *NSTC 313-Cryogenics Safety*. (**See** the test engineer for these resources.) **Comply** with the suggestions inside these presentations.

6.2.15. Check the building warning lights daily for proper operation.

6.3 Special Hazards Associated with Compressed Gases and Liquids

6.3.1. All operations involving compressed gases and liquids shall be conducted with at least 2 people, in visual contact, in the facility.

6.3.2. All operating personnel shall be instructed on the nature of hazards associated with compressed gases and liquids.

6.3.3. Before removal of any component of the system for servicing, the *operator shall secure and inspect* the system to ensure that no unsafe condition exists.

6.3.4. Personnel shall perform continuous monitoring, *e.g.*, check operating pressures, look for leaks, listen for unusual noises, during all operations. Personnel shall ensure that oxygen leak levels are adequate throughout operations.

6.4 Emergency Shutdown Procedure

The tester does not have to be shut down to be considered safe in an emergency situation.

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6.5 Accident Reporting

6.5.1. From a safe location, the *test operator* shall call 911 immediately and notify the EM10B team lead.

6.5.2. From a safe location, the *EM10B team lead* shall immediately report the accident to the NASA Safety Monitor and the appropriate supervisor(s).

6.6 Emergency Response Plan

Emergency procedures and plans for Building 4623 are incorporated into this OWI and are stated in MPG 1040.3H, *MSFC Emergency Plan*. Plans will be modified if operations change in a significant manner.

6.7 Mishap Reporting

Report all mishaps occurring in Building 4623 to the test engineer, who shall report the mishap to the area coordinator/Safety Monitor. An initial verbal report must be made within 8 hours, followed by a written report within 3 days. The EM10B team lead must prepare a managerial report within 7 days. Both reports must be reviewed by the test operator's supervisor and by the NASA Safety Monitor. The detail and extent of the mishap report will depend on the nature and extent of the damage. *If personnel injury or equipment damage does occur*, the mishap report will be completed in accordance with MWI 8621.1A, *Close Call and Mishap Reporting and Investigation Program*.

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7.0 Attachments, Data, Reports, and Forms

7.1 Calculations

7.1.1. Determination of Elevated Temperature Oxygen Index (OI)

Calculate the *OI* from the following equation:

$$OI = C_F + kd \quad (2)$$

where:

C_F = the final value of oxygen concentration (in percent volume to one decimal place) used in the N_T series, *i.e.*, that of the last test

k = a factor to be obtained from Table 7-1 as described in section 7.1.1.1

d = the step size between oxygen concentration levels used in the evaluation part of the procedure.

7.1.1.1. Determination of k . The value and sign of k are dependent on the pattern of the responses of specimens in the N_T series.

7.1.1.1.1. *If the response of the first specimen tested in the N_T (and N_L) series gave an “O” response so that the first contrary response was an “X,” refer to column 1 of Table 7-1 to select the row for which the last five response symbols correspond to those found when testing the last five specimens. The value and sign of k will be that shown in column 2, 3, 4 or 5 for which the number of “Os” shown in row (a) of the table corresponds to the number of “O” responses found for the N_L series.*

7.1.1.1.2. *If the response of the first specimen tested in the N_T (and N_L) series gave an “X” response so that the first contrary response was an “O,” refer to column 6 of Table 7-1 to select the row for which the last five response symbols correspond to those found when testing the last five specimens. The value of k will be that shown in column 2, 3, 4 or 5 for which the number of “Xs” shown in row (b) of the table corresponds to the number of “X” responses found for the N_L series; however, the sign of k must be reversed, *i.e.*, negative values shown in the table for k become positive and vice versa.*

7.1.1.2. For a valid result, the following condition must be satisfied:

$$\frac{2\hat{\sigma}}{3} < d < \frac{3\hat{\sigma}}{2} \quad (3)$$

where:

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Table 7-1.
Determination of k

Response for the Last 5 Measurements	Values of k for which the First N_T Determinations Are:				Response for the Last 5 Measurements
	(a) 0	00	000	0000	
X0000	-0.55	-0.55	-0.55	-0.55	0)000X
X000X	-1.25	-1.25	-1.25	-1.25	0)00X0
X00X0	0.37	0.38	0.38	0.38	0)X0X0
X00X0	-0.17	-0.14	-0.14	-0.14	0)X000
X0X00	0.02	0.04	0.04	0.04	0)X0X0
X0X0X	-0.50	-0.46	-0.45	-0.45	0)X0X0
X0X00	1.17	1.24	1.25	1.25	0)X00X
X0X0X	0.61	0.73	0.76	0.76	0)X000
X0000	-0.30	-0.27	-0.26	-0.26	00)00X
X000X	-0.83	-0.76	-0.75	-0.75	00)X00
X0X0X0	0.83	0.94	0.95	0.95	00)X0X
X0X0X0	0.30	0.46	0.50	0.50	00)X00
X00X00	0.50	0.65	0.68	0.68	000)X0
X00X0X	-0.04	0.19	0.24	0.25	000X00
X000X0	1.60	1.92	2.00	2.01	0000X0
X0000X	0.89	1.33	1.47	1.50	000000
	Values of k for which the First N_T Determinations Are:				Response for the Last 5 Measurements
	(b) X	X0X	X00X	X000X	
are as given in the above table opposite the appropriate response in column 6 but with the sign of k reversed					

$\hat{\sigma}$ = the standard deviation of the oxygen concentrations from the last six measurements in the N_T series, from the calculated oxygen index

d = the step change in oxygen concentration used (usual value = 0.2%)

7.1.1.2.1. Calculate the estimated standard deviation ($\hat{\sigma}$) of oxygen concentration measurements using the following equation:

$$\hat{\sigma} = \left[\frac{\sum (c_i - OI)^2}{n - 1} \right]^{1/2} \tag{4}$$

where:

c_i represents, in turn, each of the percent oxygen concentrations used during measurement of the last six responses in the N_T series of measurements, including c_F

OI = the oxygen index value calculated from equation (2)

n = the number of measurements of oxygen concentration contributing to the summation in the numerator of equation (4) [Here n is 6; however for $n < 6$ the method loses precision and for $n > 6$, alternative statistical criteria would apply.]

7.1.1.2.2. If $d < 2\hat{\sigma}/3$, repeat the oxygen index determination from step 4.5.21 using an increased value of d . **If $d > 3\hat{\sigma}/2$, repeat** the oxygen index determination from step 4.5.21 using a decreased value of d , except that d shall not be reduced below 0.2 %. (In the case where d exceeds the limit given and it is equal to 0.2 %, the oxygen index value may be reported.)

7.1.2. Determination of Flammability Temperature: Calculate the Flammability Temperature: **CHECK THE MASTER LIST -- ONLY THE LATEST VERSION IS VALID**

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ity Temperature of the material as the mean value of the three or more values determined. **Express** it as the mean value rounded to the nearest 1 °C, with an exactly intermediate result being rounded downward.

$$V = 1.5 \times 10^{-5} \cdot \pi \cdot u \cdot D^2 \tag{1}$$

7.2 Forms

Figure 7-1 show a representative **Oxygen Index at Elevated Temperature Pre-Test Checklist**. Figure 7-2 contains a representative sample of an Oxygen Index at Elevated Temperature Data Sheet. Figure 7-3 is a chart that provides guidance for setting the Preheat and Column temperature controller set points. Figure 7-4 shows a typical calibration sheet.

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**Figure 7-1.
Typical Oxygen Index at Elevated
Temperature Pre-Test
Checklist**

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Figure 7-2.
Typical Oxygen Index at Elevated
Temperature
Data Sheet

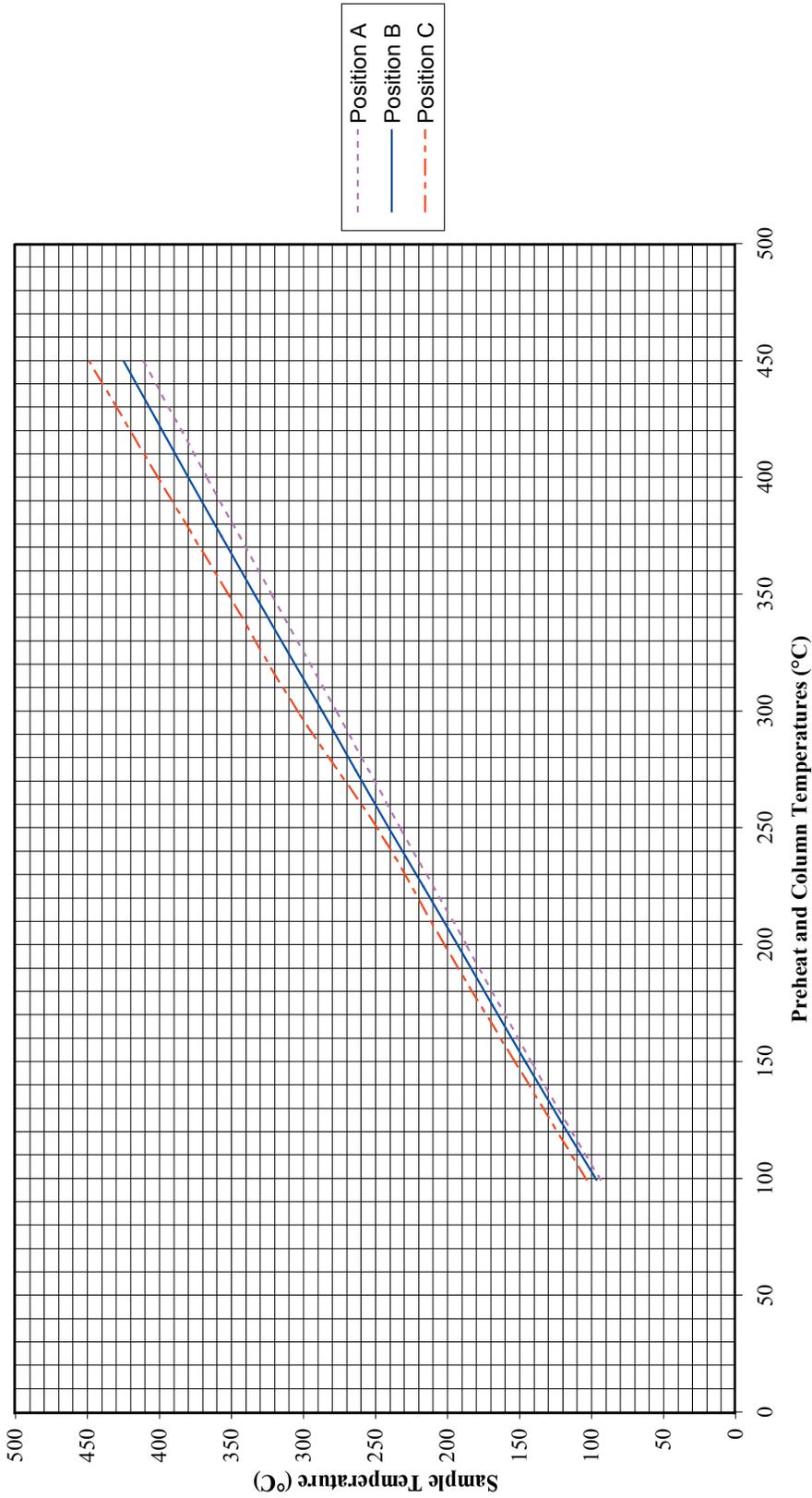
OXYGEN INDEX AT ELEVATED TEMPERATURE DATA SHEET											
Test Number:				Date:							
Material:				Ignition procedure (circle one): A B							
Density (kg/m ³):				Oxygen Concentration Increment, <i>d</i> (%):							
Specimen Type (circle one): I II III IV V				Oxygen Index (%):							
Specimen Dimensions (mm)				Standard Deviation, $\hat{\sigma}$ (%):							
Length:				Temperature of Gas Mixture (°C):							
Width:				Conditioning Procedure:							
Thickness:											
PART 1. Determination of the preliminary oxygen concentration for one pair of "X" and "O" response at <1% O ₂ concentration interval											
Oxygen concentration (%)											
Burning period (s)											
Length burned (mm)											
Response (X or O)											
Preliminary oxygen concentration (%):											
PART 2. Determination of oxygen index value											
N _T series measurements											
										C _F	
N _L series measurements											
Oxygen concentration (%)											
Burning period (s)											
Length burned (mm)											
Response (X or O)											
						Column (2, 3, 4 or 5):			Row (1 to 16):		
						k value from Table 7-1:					
						k value, with correct sign:					
O _I = C _F + <i>kd</i> =											
(To 1 decimal place for reporting O _I ; to 2 decimal places for calculating the standard deviation)											
PART 3: Verification of the oxygen concentration increment, <i>d</i>											
Last 6 Results		Oxygen Concentration (%)									
		c _i	O _I	c _i -O _I	(c _i -O _I) ²						
C _F	1										
	2										
	3										
	4										
	5										
n	6										
										Total Σ(c _i - O _I) ²	
Standard deviation ($\hat{\sigma}$): $\left[\frac{\hat{\sigma} = \Sigma(c_i - O_I)^2}{n - 1} \right]^{1/2} =$											
$\frac{2\hat{\sigma}}{3} =$			$d =$			$\frac{3\hat{\sigma}}{2} =$					
If $2\hat{\sigma}/3 < d < 3\hat{\sigma}/2$ OR if $d > 3\hat{\sigma}/2$ and $d = 0.2\%$, then O _I is valid.											
Otherwise,											
if $d < 2\hat{\sigma}/3$, repeat Part 2 using an increased value of <i>d</i> .											
if $d > 3\hat{\sigma}/2$, repeat Part 2 using a decreased value of <i>d</i> .											
PART 4: Additional Information											
Test Conductor:											

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Note: Representative Data Sheet. Refer to Forms Master list for current version. ED36-F-xxx

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Preheat and Column Temperature Guidance



Position A: 180 mm from the base of the chimney
Position B: 270 mm from the base of the chimney
Position C: 370 mm from the base of the chimney

Figure 7-2.
Guide for Setting Preheat and
Column Temperature Control-
ler Set Points

Figure 7-4.
Typical Calibration Statement.

Calibration Statement: Categories IV and V Equipment

Calibration is required before use per MPR-8730.5.

(Calibration before use for each test series and periodic testing
by the Using Line Organization)

Calibration Contacts: EM10/James Perkins, EM10/Mark Griffin

User Name: _____

Equipment Description: _____

(attach multiple components sheets if necessary)

Manufacturer: _____

ECN: _____ Serial No.: _____ Model No.: _____

Date of Calibration: _____

Type of Software and Version: _____

Listing of Standards Associated with Calibration:

Are standards National Institute of Standards and
Technology (NIST) traceable?

Y N

Did calibration meet equipment manufacturer's
specifications?

Y N

Calibration was performed by: _____

Remarks:

8.0 Quality Records

Quality Records for Temperature Oxygen Index testing consist of memoranda that contain test results and that are stored electronically in the Materials and Processes Technical Information System (MAPTIS) and calibration records.

8.1 Memoranda

Memoranda containing test results are stored electronically in the MAPTIS database by test request number.

8.2 Calibration Records

8.2.1. All equipment requiring calibration shall be in current calibration, in accordance with EM10-OWI-CHM-050 (current revision), *Materials Combustion Research Facility Guidelines for General Operations*.

8.2.2. Quality Record form EM10-F-018, current revision (Figure 7-3) will be used to document the calibration of all Category IV and Category V equipment.

8.3 Maintenance of Quality Records

8.3.1. The MAPTIS database is backed up both daily to disk and weekly to tape. In addition, the data are protected by the Oracle security system, which requires authorization to access and change data. Memoranda less than 10 years old are maintained in ready-access files in MAPTIS; memoranda 10 years old or older are automatically transferred to historical files.

8.3.2. Calibration records are maintained on site for a minimum of 10 years, filed, and indexed by test request number. These are stored in a manner that will protect them, *e.g.*, in a test folder stored in a metal file cabinet. After 10 years, calibration records are transferred to historical files.

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9.0 Tools, Equipment, and Materials

9.1 Standard Configuration of Temperature Oxygen Index Tester

The Temperature Oxygen Index tester consists of a control unit, a separate heated glass test chimney, two types of specimen holders, and a hand-held propane igniter. The entire assembly, including the chimney, stands 1200 mm high, 800 mm wide, and 365 mm deep. (A bench depth of 630 mm is required for making gas and power connections.) The mass of the assembly is ~50 kg. This tester complements the Oxygen Index control unit and can also be used for stand-alone flammability temperature testing.

The Temperature Index apparatus can be used to measure two fire parameters: the oxygen index at a temperature greater than 25°C and the flammability temperature. To measure the flammability temperature only the Temperature Index apparatus is required. To measure the elevated temperature oxygen index both the Temperature Index apparatus and the Oxygen Index apparatus are required. (The MSFC Organizational Work Instruction for the Oxygen Index apparatus is EM10-OWI-CHM-059.) The Temperature Oxygen Impact tester components are described below.

9.1.1. Control Unit

9.1.1.1. Front Panel (**Figure 9-x**): Located on the the front panel of the unit are the following controls:

- Temperature controllers for column and preheater assembly (for type K thermocouples)
- Sample temperature indicator (type K thermocouple)
- Power, pump, preheate, and column ON/OFF push button switches, which operate by firm pressure and lights up when activated
- Flowmeter with needle valve control, scale: 1-12 l/min air (or a 2-25 l/min air at ATP)

9.1.1.2. Rear Panel (**Figure 9-x**): Located on the the rear panel of the unit are the following items:

- Preheat, sample, column type K thermocouple sockets
- Preheat supply and column supply socket
- Fuses for controllers (0.6A F), pump (1A T), preheat (3.15A FF), and column (8A FF)
- Fused 10-A (Type T), 3-pin mains inlet for earthed 230 V/50 Hz supply
- Column air supply
- Cooling fan cover.

9.1.2. Heated Glass Test Chimney (**Figure 9-X**): The glass chimney, manufactured from 2.5-mm thick borosilicate glass, consists of two concentric glass columns

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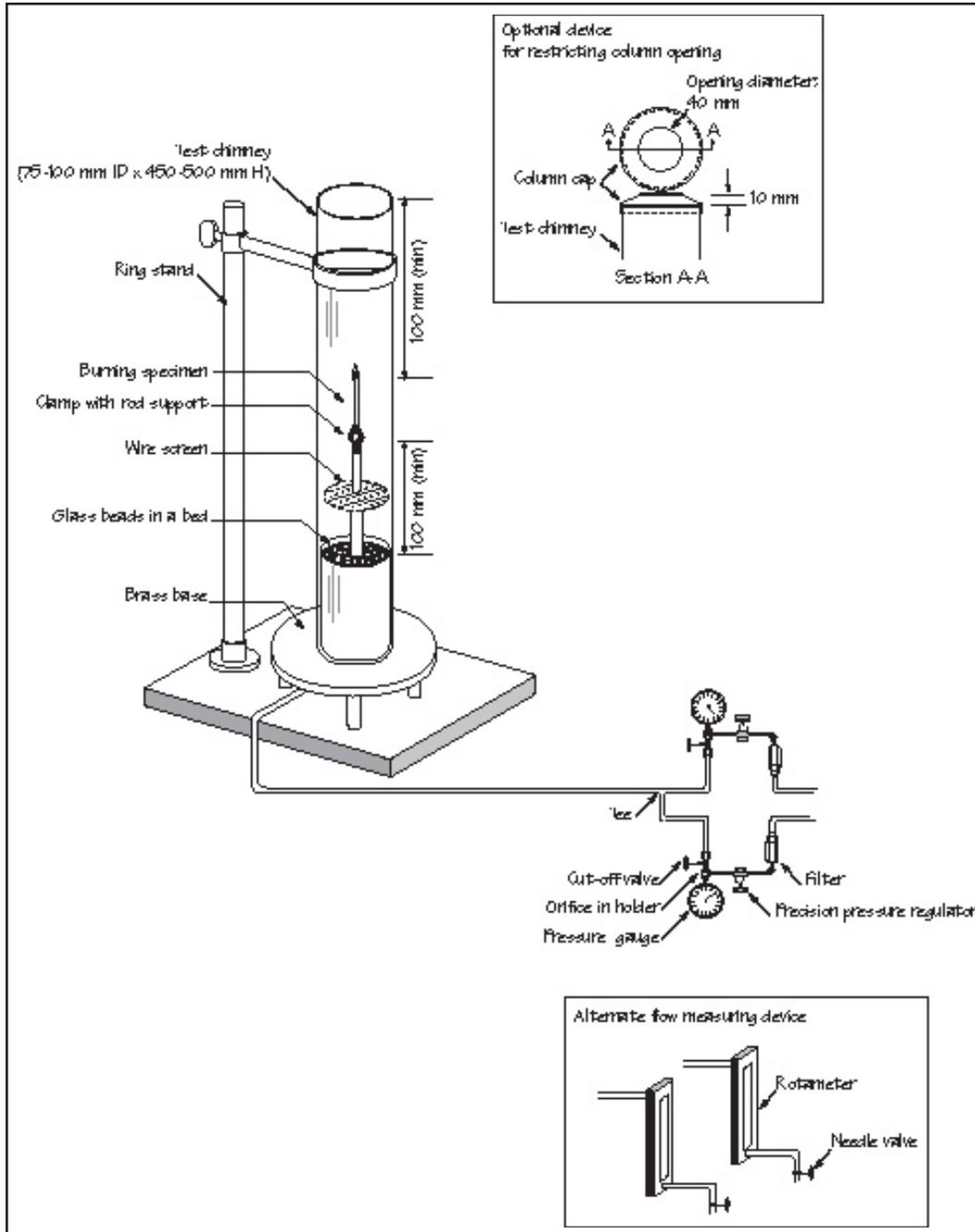


Figure 9-1.
Typical Equipment Layout.

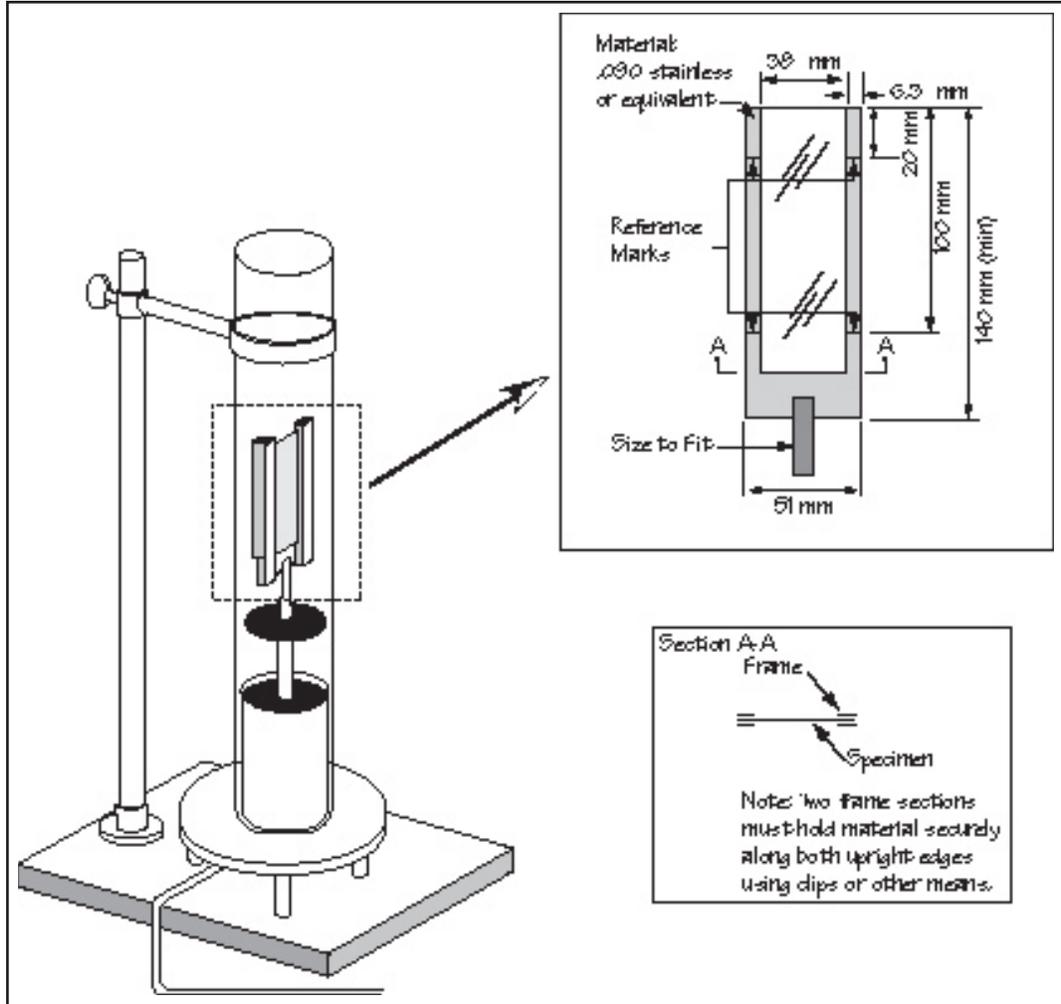
Note: Illustration is representative. Actual appearance may vary.

supported vertically between insulating top and base plates. The chimney has a heating element around the inner glass column and a preheater at the base of the chimney for heating the incoming gas mixture. The heated chimney is fixed to the preheater assembly by four screws accessed from the underside of the ceramic base plate. This allows for access to both ends of the chimney for cleaning.

The test chimney configuration maintains the test atmosphere within the inner column in the vicinity of the test specimen within $\pm 2^\circ\text{C}$ of any specific test temperature $\geq 125^\circ\text{C}$ and at within $\pm 3^\circ\text{C}$ of any higher test temperature.

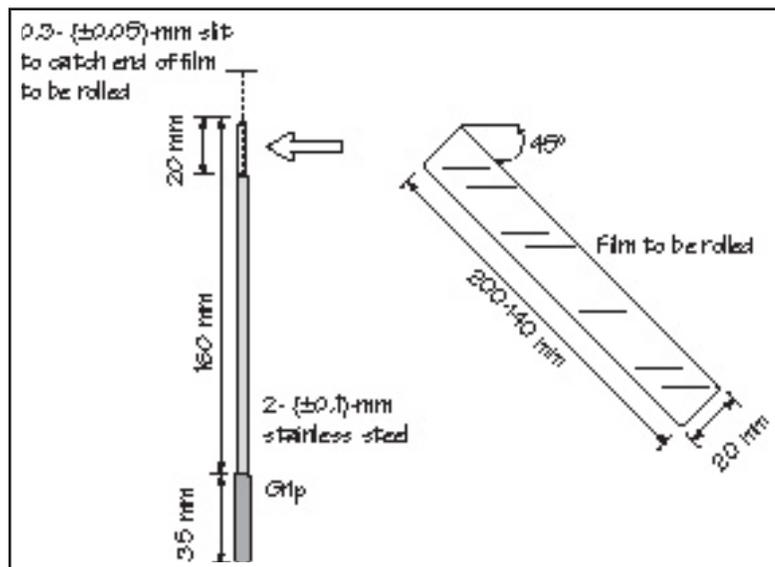
CHECK THE MASTER LIST -- ONLY THE LATEST VERSION IS VALID

Figure 9-2.
Typical frame design for thin
film and sheet specimens.



Note: Illustrations are representative. Actual appearances may vary.

Figure 9-3.
Typical thin film rolling tool.



Note: Illustrations are representative. Actual appearances may vary.

The upper outlet of the chimney is restricted by a cap that tapers to a diameter of ~40 mm. The internal diameter of the inner glass column is ~75 mm. The chimney assembly stands ~590 mm high.

9.1.3. Specimen Holder: Two types of specimen holders are supplied: one for materials of Types I, II, III, and IV and one for materials of Type V, which includes reference marks.

9.1.4. Flame Igniter (Figure 9-x): The igniter system consists of a propane gas cylinder connected by via flexible tubing to a hand-held neck valve, which has a stainless steel tube [ID 2 (± 1) mm] screwed into it. The tubing is shaped to allow a small flame to be inserted easily into the chimney from its top end while holding the handle (below the neck valve) in a vertical orientation. The assembly is can produce a flame that will project 16 (± 4) mm vertically downward from the outlet when the stainless-steel tube is vertical within the chimney and the flame is burning in the chimney atmosphere.

9.1.5. Gas Circuit (Figure 9-x)

9.1.5.1. For Elevated Temperature Oxygen Index Test: A mixture of oxygen and nitrogen passes through the Oxygen Index control unit (supplied separately) into the chimney. Both the nitrogen and oxygen are filtered before passing through a needle valve and an ON / OFF ball valve. The gases are then combined at an equal tee, and full mixing is ensured by the presence of a 7-mm filter. The majority of the gas mixture passes through the floatat, flowmeter, and the chimney to atmosphere. A small portion is taken by the oxygen analyser and vented to atmosphere through the bypass and cell ports on the rear of the unit. The pressure regulator upstream of the oxygen analyser ensures that the maximum operating pressure of the analyser (10 psi) is not exceeded. Any excess pressure is automatically vented to atmosphere at the regulator.

CAUTION: Under no circumstances must the setting of the pressure regulator be altered.



9.1.5.2. For Flammability Temperature Test: Ambient air is pumped from the Temperature Index control unit when the pump button is pressed into the chimney. The air is drawn into the pump through a silencer, which contains a filter, and is pumped through the flowmeter. It then passes through a solenoid valve, opened by pressing the pump button, and exits the Temperature Index control unit through the Column Air Supply outlet. The air then passes along a connection tube to the base of the heated glass test chimney and through the preheater arrangement.

The hardware standard configuration is controlled by the *Temperature Oxygen Index Tester Configuration Control Book*, which is controlled by NASA.

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9.2 Equipment and Support Services

The following equipment and services are required in support of the Temperature Oxygen Index Tester:

- Extraction: A hood system with flow rate of 50 l/s and that will allow the chimney to be positioned on the Oxygen Index control unit (see ED36-OWI-059) with the sample thermocouple in its uppermost position. This can best be achieved by having a mobile hood on a flexible ducting that can be moved into place above the chimney when required, or by situating the unit in a fume cupboard.
- Electrical power: Electrical power providing either 230VAC at 50 to 60 Hz or 110 VAC at 50-60 Hz at the instrument must be available via one wall socket.

Note: The Temperature Index control unit can only be used with a 110 VAC 50/60 Hz supply, if it is first passed through a 110–230 V transformer.

To conduct elevated temperature oxygen index tests, the Oxygen Index control unit (see ED36-OWI-059) must have electrical power providing either 230 VAC. at 50 Hz or 110 VAC. at 50/60 Hz, as specified by the label on the rear of the control unit.

- Gas Supplies: Bottled propane under ~7 bar of pressure is required to be connected to the supplied igniter. To conduct elevated temperature oxygen index tests, bottled nitrogen (oxygen free) and oxygen, capable of producing a flow of 14 l/min at a maximum pressure of 2.5 bar (36 psi), are required.
- Environment: **Ensure** that the tester is located in a draft-free environment at a temperature of $23 \pm 2 \text{ }^\circ\text{C}$ [$73.4 (\pm 3.6) \text{ }^\circ\text{F}$].

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9.3 Procedure for Deviations

Deviations to the baselined tester configuration require NASA written approval. It is the responsibility of the test engineer to obtain the written approval. After written approval is received, the change shall be added to the *Oxygen Index Tester Configuration Control Book*.

9.4 Required Tester Maintenance

The standard maintenance program for the Temperature Oxygen Index tester is divided into *regular cleaning and periodic and yearly service (correct?)*. In addi-

tion, the program involves a maintenance log, calibration, and a required spare parts inventory.

9.3.1. Regular Cleaning: **Keep** the tester clean, especially the glass chimney, using non-abrasive cleaners.

9.3.2. Periodic Maintenance

9.3.2.1. Leak Checks: **Perform** leak checks on all internal pipe connections, valves, and the glass chimney assembly, especially around its O-ring . After performing these checks and when replacing the top cover, **ensure** that the ground wire is connected to the underside of the cover. *(Are leak checks considered maintenance or standard checks before performing the test?)*

9.3.2.2. Pump Check: **Check** the pump inside the control unit after every ~9,000 hours of use. *If a deterioration of pump performance is noticed, check pump filters and/or diaphragm to determine if they should be replaced.*

9.3.2.3. Debris Tray: **Remove** the debris tray, and **clean** it to remove any material trapped in the holes.

9.3.3. Yearly Maintenance: **Check** the filter on the silencer of the air intake every year. **Replace** the filter *if necessary*.

9.3.4. Maintenance Log. **Document** any maintenance to the OI tester or setup in the *Temperature Oxygen Index Tester Maintenance Log* to provide a history of the tester. Any deviation to standard maintenance shall be documented by the test operator and approved on the maintenance log by the responsible test engineer.

9.5 Calibration

any needed?

9.6 Required Spare Parts Inventory

Verify that the spare parts listed in Table 2 are available at the beginning of each test request, so that the testing of a material can be completed as close to within 1 working day as possible.

Table 2.
Required Spare Parts Inventory for Temperature Oxygen Impact Test.

<u>Part</u>	<u>Quantity</u>	<u>Drawing #/Description</u>

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10.0 Personnel Training and Certification

The nature of testing that occurs in the MCRF, Building 4623, is complex and involves potential hazards; therefore, all Temperature Oxygen Index test operators must be Category 1 certified before conducting any test, and all Temperature Oxygen Index tester maintenance personnel must be Category 2 certified. This section describes the two levels of certification:

- **Category 1 Certification** qualifies personnel to perform basic test operations.
- **Category 2 Certification** qualifies personnel to maintain and modify testing apparatus.

Category 1 - Basic Operations

To be certified, all Temperature Oxygen Index test operators must complete training in the following areas:

- Handling of Compressed Gas Cylinder
- Oxygen Compatibility
- Use of Personal Protective Equipment
- General Safe Laboratory Practices
- Hazardous Waste Disposal.

Category 1 Certification also requires an annual physical examination conducted by the medical facility at Marshall Space Flight Center (or equivalent), including a hearing exam.

The operator must demonstrate knowledge of the test and equipment by completing two successful test sets under the supervision of the test engineer. There is no emergency shutdown procedure for this tester.

Test operators shall thoroughly read the test OWI as part of the certification process. They shall sign a statement that they have read and understand the OWI and shall be issued personal copies of the OWI. The test engineer shall give the candidate a written test covering the OWI. A copy of this test, along with the signed statement and the training record, shall constitute verification of certification. Training records shall be kept on file as proof of training. These records shall include training expiration dates and required refresher courses.

These certifications shall expire after a period of 2 years. After that time, recertification shall be required.

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Category 2 - Tester Maintenance and Modifications

Personnel seeking **Category 2 Certification** must become qualified and certified in the following areas:

- *Compressed Gases and Working with Compressed Gas Lines and Fittings*
- *Basic Electrical Wiring.*

This qualification/certification may be achieved through training classes approved by the candidate's supervisor or through training classes completed during previous employment.

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EMERGENCY PHONE NUMBERS

Emergency..... 911

Medical Center..... 4-2390

Industrial Safety..... 4-0046

Chemical Spills..... 4-4357

Safety Monitor

Building 4623..... 4-3571

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