Autogenous Ignition Temperature Test (ASTM G72-82)

! This Instruction Contains Descriptions of **HAZARDOUS OPERATIONS** !

Materials and Processes Laboratory
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	[s] Gail H. Gordon	Materials Test Branch Chief	EM10	11/15/05
	[s] Dennis Davis	Industrial Safety	QD50	11/16/05

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This document baselines the Organizational Work Instruction (OWI) for the Autogenous Ignition Temperature Test. Any change to this OWI must be submitted to and approved by the Materials Test Branch Chief, EM10. 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 EM10.

Concurring organizations:
Building 4623 Contracting Officer's Technical Representative
Building 4623 Contractor
Environmental Health, AD60M

Contents

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1.0 Scope

1.1 Scope

The scope of this operating instruction is ASTM G 72-82, Autogenous Ignition Temperature (AIT) of Liquids and Solids in a High-Pressure Oxygen-Enriched Environment, as defined in ASTM Fire Test Standards, Fifth Edition, and as performed in Building 4623 at Marshall Space Flight Center (MSFC).

1.2 Purpose

The purpose of the AIT test is to determine the temperature at which liquids and gases will ignite spontaneously. These materials must ignite without application of spark or flame in a high-pressure oxygen-enriched environment.

1.3 Applicability

This instruction applies to the Chemistry Team, Materials Test Branch, of the Materials and Processes Laboratory.

2.0 Applicable Documents

ASTM 74-82. *Standard Test Method for Ignition Sensitivity of Materials to Gaseous Fluid Impact.*

BB-O-925. *Oxygen, Technical, Gas and Liquid.*

Compressed Gas Association Booklet G-1. *Acetylene.*

Compressed Gas Association Booklet G-4.*Oxygen.*

EM10-OWI-CHM-042. *Test Sample Preparation for Testing in Building 4623.*

EM10-OWI-CHM-050. *Building 4623 Guidelines for Test Operations*.

EM10-OWI-CHM-051. *Receipt, Handling, Prioritizing, and Data Requirements of Samples Submitted for Testing in Building 4623 of the Materials and Processes Laboratory*.

EM10-OWI-CHM-057. *Building 4623 Oxygen Supply System.*

EM10-OWI-CHM-058. *Chemical Hygiene Plan for Building 4623.*

MIL-C-81302. *Cleaning Compound, Solvent, Trichlorotrifluoroethane.*

MPR 1840.2. *MSFC Hazard Communications Program*.

MPR 1840.3. *MSFC Hazardous Chemicals in Laboratories Protection Program*.

MPR 8715.1. *Marshall Safety, Health, and Environmental (SHE) Program*.

MPR 8823.2. *Pressure Systems Guidelines and Certification Requirements*.

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

MWI 8715.2B. *Lockout/Tagout Program*.

MWI 8715.4. *Personal Protective Equipment (PPE)*.

MWI 8715.6. *Hazardous Operations*.

MWI 3410.1. *Personnel Certification Program*.

Note: Personnel **shall** always **refer** to the current version of each applicable document.

3.0 Definitions

3.1 Definitions

NASA	Marshall Space Flight Center EM10 responsible personnel
Lockout/Tagout	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 authorized person who placed it there
Oxygen Enriched	Oxygen level in air is greater than 23.5%
Test Area	The portion of Building 4623 and fenced area of the north wall of Room 112
Test Cell	The room in which the AIT tester is located (Room 112)
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 the test under the guidance of the test engineer

3.2 Acronyms

AIT	Autogenous Ignition Test
M&P	Materials and Processes
MSFC	Marshall Space Flight Center
NASA	National Aeronautics and Space Administration
OWI	Organizational Work Instruction

4.0 Instructions

All operations of this equipment shall be conducted using the applicable documents referenced above (section 2). All critical measuring devices shall be in current calibration (section 9.4). All data and test results shall be recorded on form EM10-F-CHM-070, the Autogenous Ignition Temperature Test data sheet (section 7.2, Figure 7-1). A summary of pertinent test information and test results shall be compiled in a NASA memorandum, signed by the test organization management, and mailed to the test requester.

4.1 Sample Preparation

The *sample preparation technician* **shall prepare** test samples according to EM10-OWI-CHM-042, *Test Sample Preparation for Testing in Building 4623*. 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 and **await further instruction**. The *test operator* shall also:

- **Verify** that the test request number and material designation are identical on all paperwork.
- **Confirm** that the prepared sample agrees 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 and the original test request before proceeding. *If the test plan and the test request do not agree,* **request** clarification from the test engineer.

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** this photograph and **place** three copies of the photograph in the test folder before proceeding with the test. The entire sample shall be visible in the photo. Steps for photographing samples are outlined in the *Photography Operating Guide*, located in Room 126.

4.3 Equipment Checkout

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

4.3.1. **Glance** at the oxygen monitor station located in Room 116. **Locate** the monitor for Room 112. **Ensure** that the LED readout indicates a normal oxygen level (19.5 to 23.5%) before entering the test cell.

4.3.2. **Ensure** all test equipment, electrical components, wiring, *etc.,* are in good condition and properly connected and grounded. **Notify** the test engineer of any faulty wiring.

4.3.3. **Ensure** that the test cell floor and the room behind the test cell are visibly clean before testing begins each day. *If conditions warrant*, **scrub** the floor of the cell with a detergent solution, and **rinse** with water. **Verify** that the test cell is neat and free of excess materials, spare parts, *etc.*

4.3.4. **Turn on** the video monitor and camera, and **verify** that both are working properly.

4.4 System Setup and Sample Loading

The test operator **shall perform** the following steps:

4.4.1. **Clean** all components.

CAUTION: Review section 6.2 for precautions to observe in handling chromic acid cleaning solution and 1,1,2-trichlorotrifluoroethane.

4.4.1.1. **Soak** glass parts in chromic acid cleaning solution; **rinse** parts in distilled water; **dry** parts.

4.4.1.2. **Rinse** metal components in 1,1,2-trichlorotrifluoroethane. **Polish** parts until clean and bright with steel wool. **Rerinse** with 1,1,2-trichlorotrifluoroethane.

CAUTION: Steel wool is highly flammable in oxygen. **Remove** all visible traces of steel wool from metal parts.

4.4.2. **Weigh** a 0.20- (±0.03-) g sample, either in liquid or solid form, into the sample holder.

4.4.3. **Assemble** the inner reaction vessel and sample holder as shown in Figure 9-3.

4.4.4. **Install** the inner reaction vessel into the AIT chamber using two brass bushings, one on the bottom and one on top of the inner reaction vessel.

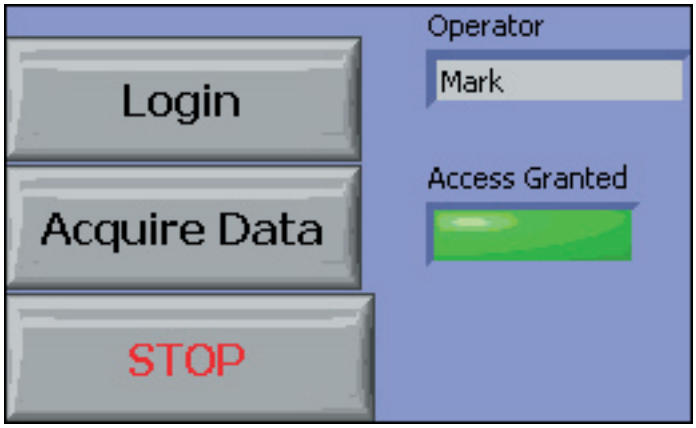
4.4.5. **Assemble** the reaction chamber as directed by the reaction vessel manufacturer. (**See** section 7.1.)

4.5 Detailed Test Procedure

The test operator **shall perform** the following steps:

4.5.1. **Turn** on the computer, and **load** the LabVIEW™ AIT control program. **Press** the **Login** button on the startup interface (Figure 4-1), and **log** into the system by entering the operator's first name and the login password (ait). After successful login, **press** the **Acquire Data** button on the startup interface to activate the system and open the LabVIEW™ front panel.

Figure 4-1.
AIT Login Screen



4.5.2. The program will request the following data; filename for data and pressure transducer calibration data.

- For filename, **enter** the folder number and sample number for the data filename, *i.e.,* 107888sample1.
- For pressure transducer calibration, **enter** the voltage range, the maximum pressure, and the offset parameters. **Contact** the test engineer for these data if they are not known.

4.5.3. **Flush** the system three times with oxygen. First, **press** the **Vent** button on the LabVIEW™ front panel to close the vent valve. Next, **press** the **GOX Supply** button to open the GOX supply and pressurize the system to 5.0 MPa (725 psi). Release the pressure by pressing the **Vent** button to open the valve. **Perform** this entire procedure three times.

Note: The text on the **Vent** button will give the current state of the valve (open or closed) (Figure 4-2).

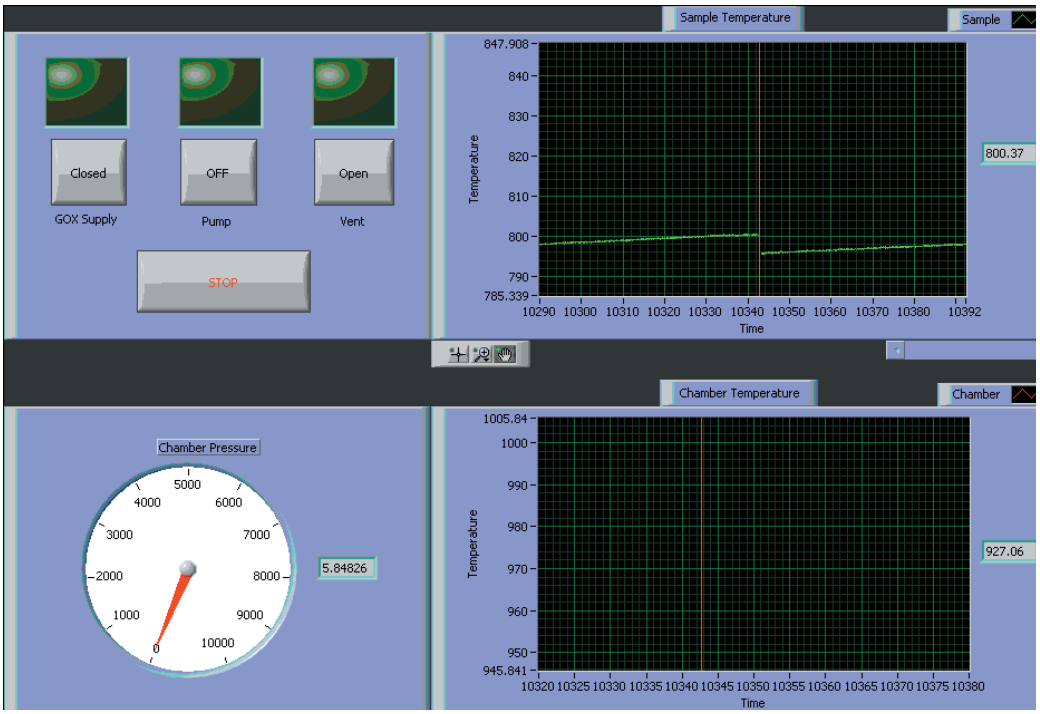


Figure 4-2.
LabVIEW™ Front Panel

4.5.4. **Fill** the reaction vessel with oxygen to a pressure of 11.5 MPa (1650 psi) or to the pressure requested by the test plan. **Allow** the vessel to stand at room temperature for 15 min to ensure there are no leaks.

Note: The pressure will drop approximately 0.5 MPa (45 psi) while the gas cools; however, it remains nearly constant thereafter. A steady pressure drop indicates a system leak that shall be corrected before proceeding.

4.5.5. **Adjust** the pressure in the vessel to 10.3 MPa (1500 psi) or to the pressure requested by the test plan.

4.5.6. **Turn** on the vessel heating jacket. **Heat** the reaction vessel at a rate of 5 (±1) °C [9 (±1) °F]/min. **Maintain** this rate of heating from 60 to 260 °C (140 to 500 °F).

Note: Above 260 °C (500 °F), a heating rate of 5 (±1) °C [9 (±1) °F]/min may be difficult to obtain; however, a heating rate higher than 3 °C (5 °F)/min shall be maintained.

4.5.7. **Monitor** the sample temperature on the LabVIEW™ front panel. Ignition of the sample is indicated by a rapid temperature rise of at least 20 °C (36 °F). When ignition is complete but not less than 3 min after it has begun, **turn off** the heater, and **stop** the recorder by pressing the **Stop** button on the front panel.

4.5.8. **Release** the reaction vessel pressure by opening the vent valve from the LabVIEW™ front panel.

CAUTION: Avoid making physical contact with or breathing the decomposition products that are vented as pressure is released. These products may be toxic.

4.5.9. *If no ignition occurs up to the maximum safe operating temperature of the reaction vessel [425 °C (800 °F)],* **stop** the heating, and **release** pressure into the system vent.

4.5.10. **Vent** the system to ambient pressure, and **set** the vessel heating jacket control to **0**, the off position. **Close** the oxygen and nitrogen supply valves located in Room 112.

4.5.11. **Remove** the sample holder, and **load** a new sample into a clean sample holder.

WARNING: The system will be very hot and could cause serious injury. **Use** thermal gloves if opening system when chamber temperature is above 50 °C (122 °F).

4.5.12. **Repeat** steps 4.5.3 through 4.5.11 until three samples have been tested, unless a different number of samples is specified by the test plan.

4.6 Shutdown Procedure

The test operator **shall perform** the following steps:

4.6.1. **Vent** the system to ambient pressure by pressing the **Vent** button on the front panel to the open position. **Set** the vessel heating jacket control to **0**, the off position. **Press** the **Stop** button on the front panel, and **press** the **Stop** button on the startup interface to close the software. **Close** the oxygen and nitrogen supply valves located in Room 112.

Note: Closing the software places all remotely controlled valves in the "safe" position.

4.6.2. **Allow** the chamber to cool below 50 °C (122 °F), as indicated by the system thermocouples.

4.6.3. **Disconnect** the fill/vent line, and **remove** the reaction vessel from the heating jacket.

4.6.4. **Open** the reaction vessel, and **remove** the inner reaction vessel and sample holder for cleaning.

4.6.5. **Inspect** the reaction vessel for debris or damage. **Clean** or **repair** as necessary.

4.6.6. **Shut down** the control computer by using the Microsoft shutdown function. **Press** the **Start** button on the lower left of the screen, **press** the **Shutdown** button, **select Shut Down** from the pull down menu, and **press OK**.

4.6.7. **Shut down** power to the control console by pushing the **EMERGENCY STOP** button.

4.7 Data Recording and Reduction; Post-Test Photography

The test operator **shall perform** the following steps:

4.7.1. **Fill out** the test report data sheet (section 7.2, Figure 7-1) completely. **Place** the test data sheet, the completed Pre-Test Checklist (section 7.2, Figure 7-2), and the LabVIEW™ printout in the work folder, and **return** the folder to the test engineer for evaluation.

4.7.2. **Photograph** any visual changes, and **document** these in writing on the test report data sheet. **Do not photograph** post-test samples that did not react. **Take** the required photographs as close to the sample as possible. More than one sample or reaction in each photo is acceptable, as long as the details of reactions are visible. Procedures for taking photographs are outlined in the *Photography Operating Guide*. **Place** three copies of each post-test photograph in the test folder before returning the folder to the engineer. Photographs shall be retained indefinitely.

5.0 Notes

Custodians for EM10-OWI-CHM-062	
Master List and Document Control	EM10 Management Support Assistant
Alternate Document Control	EM10 ISO Representative
Records	Materials Test Branch ISO Representative
Memoranda	Materials Test Branch ISO Representative

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 autogenous ignition test system involves several hazards to the system operator and facility. These hazards include:

- Pressurized systems (oxygen)
- Potential for exposure to oxygen-enriched environment
- Possible shock hazard from electrical components
- Potential for chemical burns, exposure to carcinogens
- Potential for thermal burns from heated chamber.
- Potential fire.

6.2 Safety Precautions

The test operator **shall perform** the following procedures:

CAUTION: The AIT chamber is extremely hot. **Do not touch** the chamber between samples without wearing thermal gloves rated for at least 800 °F (1,425 °C).

6.2.1. **Plan** oxygen supply activities so at least one other person is in Building 4623 during normal business hours. After business hours or on weekends, a test engineer shall be present during all activities.

6.2.2. **Wear** appropriate PPE during chemical handling and transfer:

- Goggles or full face shield
- Rubber gloves
- Long-sleeved shirt/jacket or apron
- Acid gas respirator.

6.2.3. **Read** the MSDS for the test material and for the cleaning agents chromic acid and 1,1,2-trichlorotrifluoroethane.

6.2.3.1. Chromic acid is a human carcinogen that enters the body through ingestion. *If ingested,* **drink** large amounts of water; **do not induce** vomiting. Chromic acid is also a corrosive irritant that can cause severe burns to the eyes and skin. *If the acid contacts the eyes,* **flush** the eyes with water for at least 15 minutes. For contact with skin, **wash** thoroughly with soap and water. **Seek** medical treatment for all exposures.

6.2.3.1. Although the toxicological properties of 1,1,2-trichlorotrifluoroethane have not been investigated, **wear** PPE to prevent opportunities for direct contact with the skin and eyes, and **do not inhale**.

6.2.4. **Activate** the building warning system, including warning lights, for the duration of the test, in accordance with EM10-OWI-CHM-050. **Evacuate the test cell immediately if the oxygen warning systems sound and lights flash**: **close** the door between the control room and the test cell, and **turn on** the test cell ventilation. **Leave** the control room until the oxygen in the area has balanced or Facilities has checked the system.

6.2.5. **Do not try to extinguish** any fire in the test cell. **Evacuate** the area immediately, and **call 911** to notify the fire department.

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

6.2.7. Before exposure to GOX, **clean** all equipment that will contact GOX as described in section 9.3, Required Tester Maintenance.

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

6.2.9. **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 or high-temperature source for 30 minutes after exposure to solvents and oxygen-enriched gasses.

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

6.2.11. **Check** the building warning lights daily for proper operation.

6.3 Emergency Shutdown

The test operator s**hall shut down** the tester by depressing the red **EMERGENCY STOP** button on the test console.

6.4 Accident Reporting

The test operator **shall perform** the following procedures:

6.4.1. In case of fire, **pull** the alarm, and **exit** to a safe location.

6.4.2. From a safe location, **immediately call 911** (**544-4357** if using a cell phone) if using a cell phone and **notify** the EM10 Branch Chief.

6.4.3. From a safe location, the *EM10 Branch Chief* **shall immediately report** the accident to the NASA Safety Monitor and the appropriate supervisor(s).

6.5 Emergency Response Plan

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

6.6 Mishap Reporting

The test operator **shall report** all mishaps occurring in Building 4623 to the test engineer, who shall report the mishap to the area coordinator/EM10 Branch Chief. An initial verbal report shall be made within 8 hours, followed by a written report within 3 days. The EM10 Branch Chief shall prepare a managerial report within 7 days. Both reports shall be reviewed by the technician's supervisor and by the NASA Safety Monitor. The detail and extent of the mishap report shall depend on the nature and extent of the damage. *If personal injury or equipment damage does occur,* the mishap report shall be completed in accordance with MWI 8621.1 *Mishap/Incident*

7.0 Attachments, Data, Reports, and Forms

7.1 Attachment - Access to Reaction Vessel

The test operator **shall gain access** to the reaction vessel in either of two ways: **remove** the vessel from the heating jacket and **disassemble** it, or **disassemble** the vessel while it is installed in the heating jacket. (Refer to Figure 9-1, Assembly Drawing, as needed.)

The test operator **shall perform** the following procedures:

7.1.1. **Remove** vessel from the heating jacket and **disassemble**.

7.1.1.1. **Unscrew** the gland nut, and **remove** tubing from the head.

7.1.1.2. **Unscrew** the tailbolt (end of vessel opposite tubing connection). *If instrument is Aminco-pencil type,* **remove** the thermocouple by unscrewing the gland nut, and **remove** leads from the binding posts on heating jacket.

7.1.1.3. **Slide** the vessel from the heating jacket by grasping the eyebolts and pulling.

7.1.1.4. **Grip** the vessel body across the flats in a vise, and **loosen** the cap screws on the vessel cap. **Unscrew** the vessel cap, and **lift off**. *If the cap resists unscrewing,* **use** a pry bar to loosen the cap. **Insert** the bar in the 3/8-in. diameter holes in the cap periphery.

7.1.1.5. **Lift** the thrust ring, head, and thermowell or liner from body.

Note: The flat gasket will come off with the head.

7.1.2. **Disassemble** the vessel in the heating jacket.

7.1.2.1. **Unscrew** the gland nut, and **remove** tubing from the head.

7.1.2.2. **Loosen** the cap screws, and **unscrew** the cap from the vessel.

7.1.2.3. **Remove** the thermocouple by unscrewing the gland nut, *if the instrument is Aminco-pencil type.* **Remove** the leads from the binding posts on the heating jacket.

7.1.2.4. **Remove** the thrust ring and head with thermowell and flat gasket from vessel.

Note: The thermowell need only be removed (by unscrewing) for replacement or when using special Pyrex® liners, in which case the thermowell opening shall be compatible.

7.1.3. Reassemble Reaction Vessel

7.1.3.1. **Place** the sample in the vessel, and **assemble** in the reverse order of disassembly (section 7.1.1.2).

7.1.3.2. *If the reaction vessel has been removed from the heating jacket,* **slide** the vessel into the jacket, and **turn** it until the locating pins engage the corresponding holes in the bottom of the reaction vessel.

7.1.3.3. **Apply** thread lubricant to the tailbolt. (The tailbolt holds the vessel in the heating jacket.) **Tighten** the tailbolt hand-tight.

7.1.3.4. Before replacing the cap, **apply** thread lubricant to the vessel body threads and cap screw threads.

7.1.3.5. When installing the cap over the head and thrust ring, **perform** the following:

a. **Unscrew** all cap screws until they do not protrude beyond the inner face of the cap.
b. **Place** flat gasket in groove. No additional gasket is needed. **Center** the head, and **slip** on thrust ring.
c. **Screw** cap on until it just seats on the thrust ring. **Screw** cap on hand-tight only.
d. **Screw** in each cap screw finger-tight,
e. With a torque wrench, sequentially **tighten** opposing cap screws 1/16 turn.
f. After tightening all screws, **repeat** step (e).

Note: Proper torque is needed to seal the vessel. This information is marked on the tag attached to all vessels and recorded in the AIT maintenance log.

7.2 Forms

Figure 7-1 shows a representative Autogenous Ignition Temperature Test Data Sheet (EM10-F-CHM-070), Figure 7-2 shows a representative AIT Pre-Test Checklist (EM10-F-CHM-071), and Figure 7-3 shows a representative Calibration Sheet (EM10-F-CHM-018).

Figure 7-1.
AIT Data Sheet

Autogenous Ignition Test Data Sheet

Test No.: _____ Date: _____ Project: _____
Request ID No.: _____ Requester: _____
Manufacturer Designation: _____
Manufacturer: _____
Composition: _____
Specification: _____

Sample #	Test Atmosphere Composition	Sample Wt. (g)	Ignition Temp*		Temp Rise on Ignition		System Gas Pressure (psi)		Pressure Rise on Ignition (psi)	Appearance of Residue
			(°C)	(°F)	(°C)	(°F)	Initial	Final		
1.										
2.										
3.										

*If no ignition occurs up to 425 °C (800 °F), report ignition temperature as >425 °C (>800 °F).

Volatility: ☐ Yes ☐ No
Low heat release: ☐ Yes ☐ No

Remarks _____

Excess Sample: ☐ Y ☐ N Quantity of Excess _____ Storage Box _____

Test Conductor _____

1/05 EM10-F-CHM-070

Note: Illustration is representative. Actual appearance may vary.

Autogenous Ignition Temperature Test Pre-Test Checklist

Request No. _____

	Initial Yes	Initial No
1. AIT OWI reviewed?	☐	☐
2. All safety equipment available and working properly?	☐	☐
3. Test equipment checked out per OWI?	☐	☐
4. Test material's MSDS read?	☐	☐
5. Test plan read and changes noted and approved by engineer?	☐	☐
6. Pre-test photograph taken?	☐	☐
7. Oxygen monitor checked to ensure normal O_2 level?	☐	☐
8. O_2 and GN_2 supplies turned on?	☐	☐
9. All test components cleaned per OWI?	☐	☐
10. Inner reaction vessel and sample holder assembled correctly?	☐	☐
11. Inner reaction vessel installed in AIT chamber correctly?	☐	☐
12. Thermocouples checked and installed per OWI?	☐	☐
13. Operation of video monitor and camera checked?	☐	☐
14. Test sign and door barricade put up?	☐	☐
15. Facility warning beacon activated?	☐	☐

Remarks/Discussion of Discrepancies:

_____

_____

_____

_____

_____

_____

_____

_____ _____

Test Operator Date

1/05 EM10-F-CHM-071

Figure 7-2.
AIT Pre-Test Checklist

Note: Illustration is representative. Actual appearance may vary.

Figure 7-3.
Calibration Sheet

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:

_____

_____

_____

_____

_____

_____

_____

_____

1/05 EM10-F-CHM-018

Note: Illustration is representative. Actual appearance may vary.

8.0 Records

Records for Autogenous Ignition Testing shall consist of (a) memoranda that contain test results and that are stored electronically in the Materials and Processes Technical Information System (MAPTIS) and (b) calibration records.

8.1 Memoranda

Memoranda containing test results shall be retained indefinitely by EM10. These memoranda shall be stored electronically in the MAPTIS database and shall be accessible by test request number or memorandum number.

8.2 Calibration Records

8.2.1. All equipment requiring calibration shall be in current calibration, in accordance with EM10-OWI-CHM-050, *Building 4623 Guidelines for General Operations*.

8.2.2. The Calibration Sheet (Figure 7-3) shall be used to document the calibration of AIT equipment.

8.3 Maintenance of Records

8.3.1. Memoranda less than 10 years old shall be maintained in ready-access files in MAPTIS; memoranda 10 years old or older shall be 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.

8.3.3. The original test records shall be saved for a minimum of 5 years.

9.0 Tools, Equipment, and Materials

9.1 Equipment

Most organic liquids and solids will ignite in a pressurized oxidizing gas atmosphere if heated to a sufficiently high temperature and pressure This test provides a numerical value for the temperature at the onset of ignition under carefully controlled conditions. The temperature at which material spontaneously ignites varies greatly with the geometry of the test system and the rate of heating.

In the AIT test, the solid or liquid sample is exposed to increasing temperature in a high-pressure reaction vessel (bomb). The bomb, including a sample-holding assembly, is pressurized in an oxygen-enriched environment. An electric furnace heats the bomb at a predetermined rate, and the temperature of the sample-holding assembly is monitored as a function of time by a thermocouple and a recording potentiometer. The minimum temperature required to cause the sample to ignite is determined at any selected pressure. The point at which spontaneous ignition occurs is denoted by a sudden rise in temperature and the destruction of the sample. The amount of rise in temperature is related to sample size. The system is pressurized to the desired test pressure. During the test, as the temperature is increased, the pressure increases. No effort is made to control pressure, but this parameter is monitored to ensure that it does not exceed a safe limit for the equipment.

While the test method can be used at pressures of 2.1 to 20.7 MPa (300 to 3000 psi), the pressure used in this OWI is 10.3 MPa (1500 psi). The method is for liquids or solids with ignition temperatures in the range from 60 to 425 °C (140 to 800 °F). The procedure may be used in atmospheres from 0.5% to 100% oxygen.

The stainless-steel reaction vessel is cylindrical [approximately 65 mm (2.56 in.) OD and 298 mm (11.75 in.) in length] and is bored to a depth of 20.9 cm (8.25 in.), producing a reaction chamber volume of approximately 110 milliliter. The vessel weighs 9.75 kg (21.5 lb). The maximum working pressure at 427 °C (800 °F) is 82.7 MPa (12,000 psi).

A borosilicate glass test tube (15x125 mm), stoppered with 12.5-mm borosilicate glass tubing, forms the inner reaction vessel. A borosilicate glass culture tube (10x7mm) placed inside the inner vessel holds the sample material. Sample size is selected to prevent damage to the equipment that could be caused by exceeding safe system pressure or temperature limits.

The inner reaction vessel is supported in the bomb cavity by a No. 21 AWG chromel A wire, wound into a spring-type shape to fit snugly around the glass test tube. A loop of wire positions the thermocouple in the mouth of the sample holder. A bushing fits into the bomb cover and supports the entire sample-holding assembly.

A 230-V, 1000-W fitted heating jacket surrounds the vessel, and a 203-mm (8-in.) chromel-alumel thermocouple extends into the reaction chamber and sample holder. The tester is equipped with a strip chart recording pyrometer.

The entire AIT assembly is equipped with a stainless-steel pressure-relief blowout with rupture discs [48.3 MPA (7000 psi) at 22.2 °C (72 °F)].

Figures 9-1, 9-2, and 9-3 illustrate the AIT system.

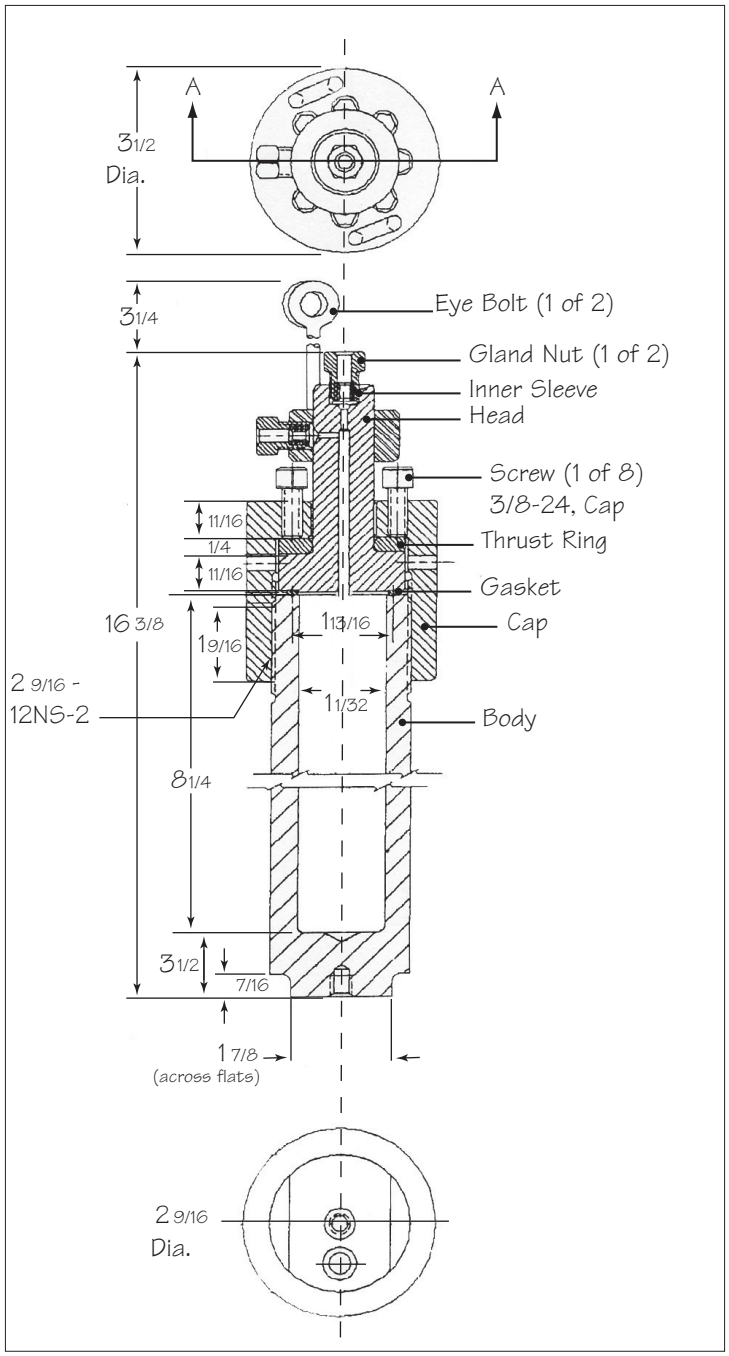


Figure 9-1.
AIT Tester

Note: Illustration is representative. Actual appearance may vary.

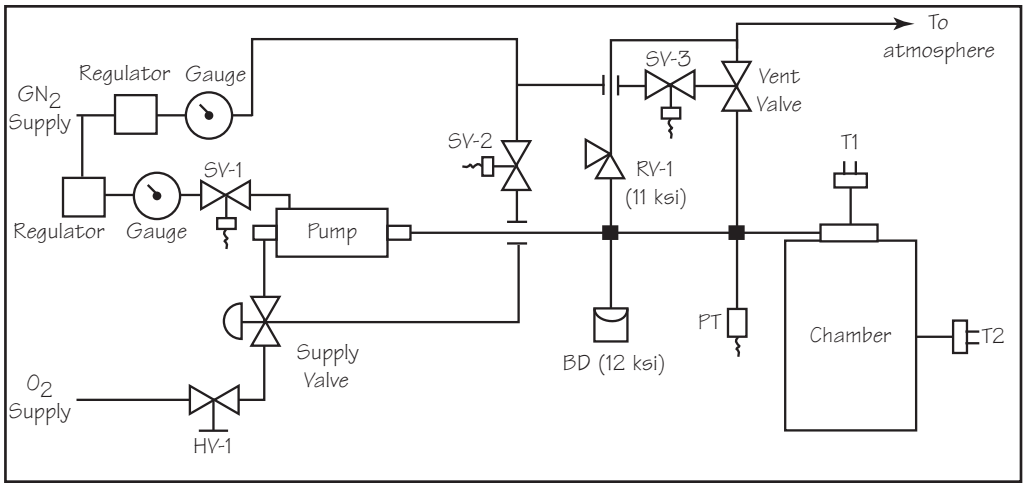


Figure 9-2.
AIT System

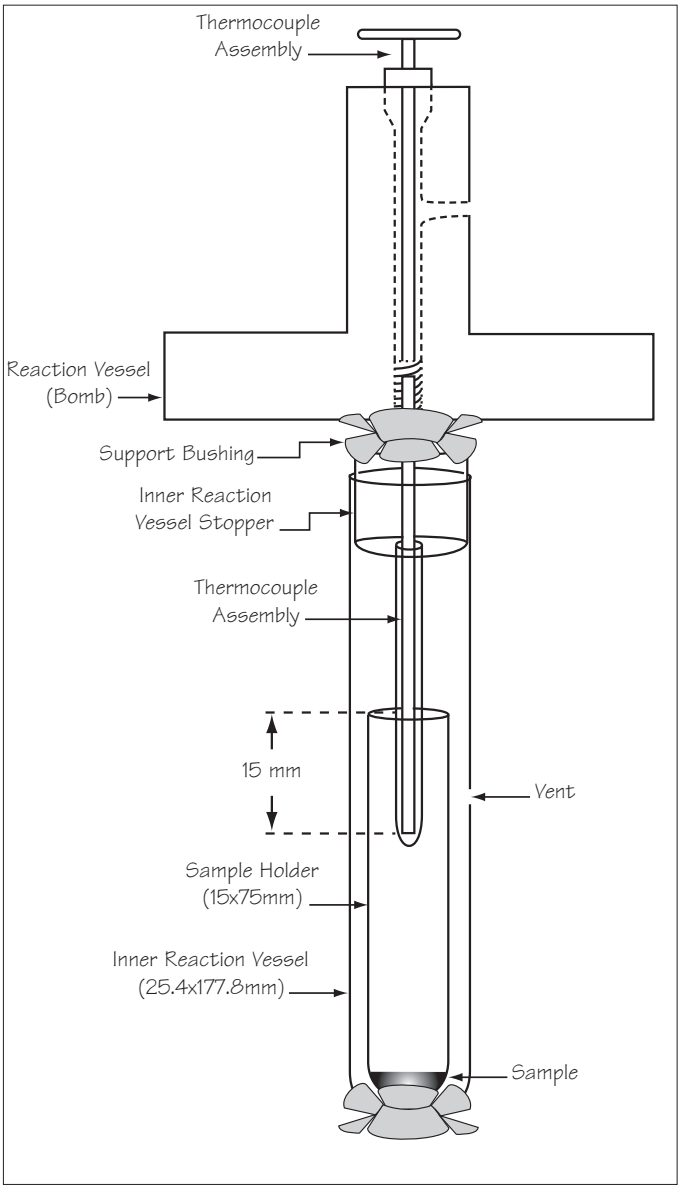


Figure 9-3.
AIT Sample Holder

Note: Illustrations are representative. Actual appearance may vary.

9.2 Procedure for Deviations

Deviations to the baselined AIT system configuration require NASA written approval. The test engineer shall obtain the appropriate approval from the NASA COTR. After written approval is received, the change shall be added to the *AIT Configuration Control Book*.

9.3 Required Maintenance

The standard maintenance program for the AIT system is divided into weekly, bi-weekly, and monthly service. In addition, the program involves a maintenance log, calibration, and a required parts inventory. The test operator **shall perform** the following maintenance procedures and **shall refer** to the drawings in the *AIT Configuration Control Book*, as needed.

9.3.1. Weekly Maintenance and Before Use

Verify thermocouple calibration with ice bath and boiling water.

9.3.2. Monthly Maintenance

Check tubing for leaks.

9.3.4. As-Needed Maintenance

Whenever a gasket becomes nicked or work hardened, **replace** the gasket, according to the following procedure:

9.3.4.1. **Spring** the gasket from the groove by striking it sharply several times with a cape chisel around the inner and outer circumferences. **Do not chisel down** the gasket face as this will damage the head.

9.3.4.2. **Place** a new gasket into the groove in the head. There will be a slight amount of clearance between the gasket and the groove. After several installations and removals of the cap, the gasket will expand into the recess. At the same time, a ridge will appear in the face of the gasket, caused by its being forced into the groove on the vessel body.

9.4 Calibration

Calibrate thermocouples before each use.

9.5 Required Spare Parts Inventory

Before beginning each test request, the test operator shall **ensure** that there are spares for all pieces of test equipment listed in Table 9-1.

Table 9-1.
Spare Parts Inventory

Part		Quantity
41-125SS	Vessel Assy 2-9/16” OK - 316 SS Type B	1
43-12111	2-9/16” Heating Jacket - 115 V	1
01148A17200	Spring for micro vessel	2
42-12581	Pyrex liner (110 ml) for Thermocouple	1
	Shielded Type-K Thermocouples	2
	Solenoid Valves	3
	Relief Valve (11 ksi)	1
	Burst Disk (12 ksi)	1

10.0 Personnel Training

The nature of testing that occurs in Building 4623 is complex and involves potential hazards; therefore, all test operators shall complete the requirements for Category 1 Credentials before conducting any test

To obtain Category 1 Credentials, the test operator shall complete training in following areas:

- High Pressure Systems Safety
- Oxygen Compatibility.
- Good Laboratory Practices.

Category 1 Credentialling also requires:

- Successful completion of an annual physical examination conducted by the medical facility at Marshall Space Flight Center (or equivalent), including a hearing exam
- A demonstration of knowledge of the test and equipment by the completion of two successful test sets under the supervision of the test engineer.
- A demonstration of knowledge of the OWI. Candidate test operators shall thoroughly read the test OWI and sign a statment confirming that they have read and understand the OWI. Each shall be issued a personal copy of the OWI.
- Passing of a written test covering the OWI. The test shall be administered by the test engineer.

A copy of the written test, along with the signed statement and the training record, shall constitute verification of credentials. Training records shall be kept on file as proof of training. These records shall include training expiration dates and required refresher courses.

Category 1 Credentials shall expire after a period of 2 years. After that time, recredentialling shall be required.

EMERGENCY PHONE NUMBERS

Emergency..... 911
Cell Phone..... 544-4357

Medical Center..... 4-2390

Industrial Safety..... 4-0046

Chemical Spills..... 4-4357

Safety Monitor
Building 4623..... 5-0358