

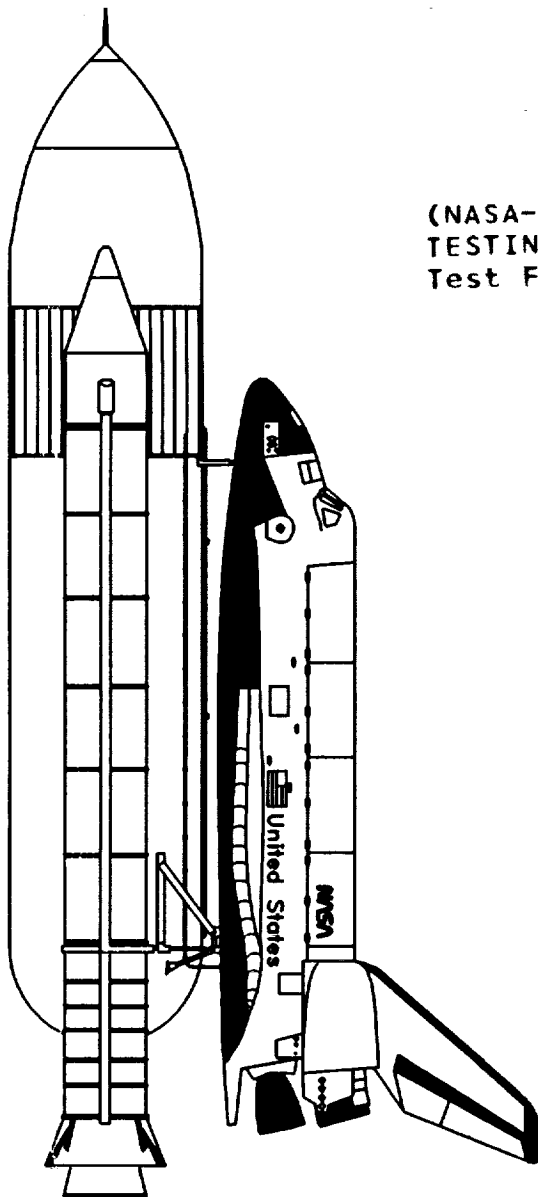


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Test Plan

APU Diaphragm Testing



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Lyndon B. Johnson Space Center
White Sands Test Facility
P. O. Drawer MM
Las Cruces, NM 88004
(505) 524-5011

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Test Plan

APU Diaphragm Testing

Issued By
National Aeronautics and Space Administration
Johnson Space Center
White Sands Test Facility
Laboratories Office

Prepared By: Richard M. Shelley
Richard Shelley
Lockheed-ESC

Reviewed By: [Signature]
John Morelli
Lockheed-ESC

Concurred By: Raymond Reynaud 1/13/92
Raymond Reynaud
NASA WARSO

Approved By: [Signature]
David L. Baker
NASA Laboratories Office

Concurred By: David W. Young 1-7-92
David W. Young, APU Subsystems Manager
NASA Johnson Space Center

Concurred By: [Signature] 1/10/92
Tibor Farkas
Rockwell International, Power Systems

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Introduction

Auxiliary Power Unit (APU) fuel (hydrazine) tanks have had to be removed from the Columbia Shuttle (OV-102) because they have been in service for 11 years, which is the limit of their useful life.

As part of an effort to determine whether the useful life of the fuel tanks can be extended, examination of the ethylene propylene rubber (EPR) diaphragm and the metal from one of the APU tanks is required. Johnson Space Center (JSC) Propulsion and Power Division has requested White Sands Test Facility (WSTF) to examine the EPR diaphragm thoroughly and the metal casing generally from one tank.

Objective

The objective is to examine the EPR diaphragm for signs of degradation that may limit the life of its function in the APU propellant tank. The metal casing will also be examined for signs of surface corrosion.

Background

WSTF has received the three old tanks from OV-102. One tank will be tested to determine any measurable degradation in the diaphragm material and any signs of surface corrosion in the metal casing. The other two tanks will be cycle-type tested under a separate test plan.

The examination results will be useful because the other orbiters have tanks that are generally newer than OV-102's, and the tanks might not have to be replaced as soon as presently planned if insufficient signs of degradation are found in the OV-102 tank. Two OV-103 tanks are 7 years old and one tank (S/N 0004) is 14 years old (6 years of which have been dry). The OV-104 tanks are 6 years old.

The diaphragm in the orbiter fuel tank has to be flexible, thus an elastomeric material was chosen. Several studies were performed on different elastomers for their suitability for use with hydrazine (Takimoto and Denault 1969; Repar 1970 and 1973; Sheets 1974; Coulbert, Cuddihy, and Fedors 1973; Martin and Sieron 1977; and Yee and Etheridge 1985). Takimoto and Denault (1969) found that elastomers containing carbon black increased degradation of hydrazine. Repar (1970, 1973) found that silica (SiO₂) fillers produced compounds that were more suitable for use in hydrazine. The base materials used in the test compounds were butyl and ethylene propylene terpolymer rubbers.

Sheets (1974) tested EPR AF-E-332, a newly developed polymer, that was based on an ethylene propylene terpolymer. This compound was found to be more resistant to hydrazine than the previously tested compounds.

Further testing of EPR AF-E-332 with hydrazine was performed (Coulbert, Cuddihy, and Fedors 1973; Martin and Sieron 1977; Yee and Etheridge 1985). These works tested for permeation of propellant, compression set of seal bead, swelling, and tensile property retention, as well as posttest appearance of the diaphragm, potential leakage or pull-out of

different seal bead designs, pressure fluctuations, and chemical composition of the posttest propellant.

Lifetime predictions for EPR in hydrazine have been attempted using Arrhenius and Williams Landel Ferry (WLF) models (Coulbert, Cuddihy, and Fedors 1973; Martin and Sieron 1977), but none has been thoroughly demonstrated as correlating and predicting long-term behavior.

The EPR diaphragm material is given a shelf life of up to 10 years (MIL-HDBK-695C 1985; MIL-STD-1523A 1984), although the shelf life is not based on scientific data (Boyum and Rhoads 1990). Reports indicate that EPR AF-E-332 is unaffected by up to 10 years of exposure to hydrazine (Gill 1986; Repar 1973). The design lifetime of the APU tanks was previously 10 years, but it has been extended by 2 years to 12 years.*

Aging of an elastomer material is usually caused by several mechanisms: chemical attack resulting in crosslinking or chain scission of the polymer chains, physical relaxation of the polymer arising from stress, and change in the compound when ingredients bleed to the surface or are leached out by contacting fluids.

Increased crosslinking has the reverse effect of chain scission; it causes the polymer to become harder, with a higher tensile strength and lower elongation (less flexible, more brittle). Relaxation of the polymer will increase compression set. Change in the compound changes the properties of that compound; for example, removal of a plasticizer from an elastomer compound would result in a material with higher hardness, higher tensile strength, and lower elongation.

Because one of OV-103's tanks has been dry for six years, it is important to know whether significant diaphragm degradation occurs in air. All the aging mechanisms except leaching could occur in air as well as in hydrazine, so it is possible that the elastomer, if degraded by the hydrazine, could undergo similar degradation in air. However, previous studies and case histories have shown that many elastomers (including EPR) either do not require a shelf life or do not age detectably in air for up to 22 years (Boyum and Rhoads 1990; Rubber Manufacturer's Association 1966; Bellanca and Harris 1967; Young 1960; Sullivan 1966; House 1972).

The most important properties of the diaphragm are compression set, hardness, specific gravity, tensile properties, and chemical content.

Compression set is important to the diaphragm because the seal bead holds the hydrazine; therefore sealing ability is important. Hardness is a sign of change in the elastomer properties caused by contact with the hydrazine. Specific gravity indicates changes in the elastomer ingredients. An elastomer compound consists of materials of different specific gravities. By knowing the amount and specific gravity of each ingredient in the compound, the compound specific gravity can be calculated fairly accurately. Any ingredients lost during exposure to hydrazine may result in a detectable change in specific gravity. Tensile properties are important to the diaphragm because it is under tensile forces during use. Chemical content, like specific gravity, indicates changes in elastomer ingredients and thus changes in elastomer

*Presentation by W. Scott. "OV-102 APU System Hardware Disposition." NASA, Propulsion and Power Division, May 1, 1991.

properties. To determine changes in elastomer ingredients another way, thermal analysis determines both ingredient change/loss and embrittlement or hardening.

The effect of hydrazine on metals and metal surfaces is generally analyzed by determining the corrosion product on the metal surface. Specifically for Ti-6Al-4V (the material from which the metal casing is constructed), tin nitride, nitrogen, ammonia, etc., usually would be deposited. Auger Electron Spectroscopy studies on the sample surface could reveal some of these deposits. However, this detection technique has some inherent problems. The principle problem is that the Auger peaks for titanium, titanium nitride, and nitrogen overlap and are extremely difficult to decipher. Therefore, other surface techniques may need to be applied to determine the corrosion products.

For example, some metals are known to exhibit stress corrosion cracking when exposed to hydrazine for an extended period of time. To determine evidence of stress corrosion cracking, samples need to be prepared for microstructure evaluation. Intergranular or transgranular cracking may be easily visible with this technique. Both corrosion of the metal surface and stress corrosion cracking can be observed by using optical and scanning electron microscopy.

Approach

A literature search will be conducted to obtain the most current information on testing that has been performed on EPR and similar materials in hydrazine. If recent literature dictates, adjustments can be made to the following tests.

The EPR tests will be based on tests for immersion of elastomer materials and literature on EPR in hydrazine (Takimoto and Denault 1969; Repar 1970 and 1973; Sheets 1974; Coulbert, Cuddihy, and Fedors 1973; Martin and Sieron 1977; Yee and Etheridge 1985). The metal casing will be tested by analyzing the sample surfaces for corrosion.

Nondestructive evaluation, dissection, visual examination, microscopic analysis, thickness measuring, hardness testing, specific gravity measuring, tensile testing, chemical analysis, and thermal analysis will be performed on the old EPR diaphragm and on samples of unexposed EPR material.

Tested properties of the tested diaphragm will be compared with the tank manufacturer's design criteria, shown in Table 3. If the diaphragm properties have changed from those of the unexposed material, the change will be calculated as a percentage difference.

Any changes will be compared with changes reported in the literature. For example, tensile strength of material has been reported to decrease upon hydrazine exposure by 15 to 30 percent (Martin and Sieron 1977; Yee and Etheridge 1985). Recommendations will be made, based upon the results of the tests and comparisons, as to whether or not the expected life of the APU propellant tanks can be extended.

Experimental Materials

The system to be tested consists of an APU tank from OV-102, with an approximately 71-cm-diameter elastomeric diaphragm manufactured by Pressure Systems, Inc. (PSI). Three elastomeric materials will be tested: material from the 11-year-old tank, 11-year-old material that has not been in service and thus has not been exposed to hydrazine, and newer material that also has not been in service. All three materials are designated EPR AF-E-332 compound that meets MIL-R-83412A specifications (1977).

The EPR AF-E-332 compound is based on ethylene-propylene terpolymer rubber (Nordel 1635 EPT), with a theoretical compound specific gravity of 1.05. The ingredients are listed in Table 1.

Table 1
EPR AF-E-332 Ingredients

Ingredients	Manufacturer	Parts by Weight	Specific Gravity
Nordel 1635 EPT	DuPont	100	0.86
Aerosil R972	Degussa	30 ± 1.5	1.95
B-3000 Resin	Dynachem Corp.	20 ± 1.0	0.90
Teflon Powder T-8A	DuPont	10 ± 0.3	2.15
Zinc Oxide Reagent	Baker	5 ± 0.2	5.57
Calcium Oxide Reagent	Baker	5 ± 0.2	2.20
Luper 101 Peroxide (Curing Agent)	Wallace and Tiernan	0.9 ± 0.2	1.00

The 11-year-old diaphragm material will be obtained from the tank from OV-102, and the 11-year-old, unexposed material will be obtained from the WSTF bonded storage, where it is held under WSTF Number 91-25134. The newer, unexposed sample of EPR AF-E-332 supplied by PSI was cut from P/N 80-228007, S/N 0071 and was molded in July 1984. PSI will also test samples of this newer material so both testing facilities can ensure consistent testing results.

The literature properties of the unexposed material are given in Table 2 (Sheets 1974; Yee and Etheridge 1985).

Table 2
Mechanical Properties of the Virgin Material

Property	Value
Tensile Strength	1799 MPa
Elongation	253%
Specific Gravity	1.08
Compression Set	17.3%

Table 3 lists the manufacturer's design criteria of the diaphragm material.

Table 3
Manufacturer's Design Criteria of Diaphragm Material

Property	Required Value	Standard Used For Testing
Compression Set	22% Max	ASTM D395, Part B ^a
Hardness	90 ± 5	Shore A
Specific Gravity	1.07	Not Applicable
Tensile Strength	11.4 MPa, min.	ASTM D412 ^b
Elongation	230% min.	ASTM D412
100% Modulus	6.6 MPa, min.	ASTM D412
Tear Strength	525 N/cm, min.	ASTM D624 ^c

^a22 hours at 70°C
^bDie D, 50.8 cm/minute
^cDie B, nicked crescent

The metal casing of the APU tank is constructed of Ti-6Al-4V; this is an alpha beta titanium alloy containing aluminum and vanadium.

Experimental Procedure

The following tests will be conducted in this order. The 11-year-old and newer unexposed materials will be tested by the same tests the diaphragm will undergo. The location of all samples removed from the materials will be noted.

Nondestructive Evaluation

Any hydrazine remaining between the diaphragm and the metal casing will be measured before the nondestructive evaluation by emptying the tank at WSTF and measuring the hydrazine in a measuring cylinder. The tank will then be flushed with nitrogen for 2 hours. The hydrazine in the measuring cylinder will be chemically analyzed. Nondestructive testing of all three tanks will be performed using neutron radiography to examine signs of inhomogeneity, pull-out, etc, at the seal bead.

Dissection

After the nondestructive evaluation is completed, one of the tanks will be dissected to remove the diaphragm.

Visual Examination

The diaphragm will be examined inside and outside for blemishes, cracks, discolorations, and any other distinguishing features. Cracks may have formed at or near the weld bead or at other places of high stress concentrations, or the material may have discolored on exposure to hydrazine. Color, frequency, size, shape, position, and any other noticeable characteristics of any distinguishing features will be noted.

Microscopic Analysis

Sections of the diaphragm will be examined with the stereo microscope at magnifications of 10 to 15 times. Other sections of the diaphragm will be microtomed in liquid nitrogen and examined under the transmission optical microscope to identify ingredient dispersion characteristics. The diaphragm material will be compared with samples of the unexposed materials. If further resolution is needed, samples will be prepared and observed with Scanning Electron Microscopy.

Thickness Measuring

To calculate a range of compression sets, measurements of thickness around the seal bead of the diaphragm (5 cm apart) and calculations of data will be made according to ASTM D395 (1986), sections 12 through 14. The original thickness of the material will be obtained from historical records.

Hardness Testing

The type A durometer will be used to perform hardness tests on areas throughout the inside and outside of the diaphragm, according to ASTM D2240 (1986).

Specific Gravity Measuring

Ten samples will be taken from the diaphragm and measured for specific gravity by the water immersion method according to ASTM D792 (1986).

Tensile Testing

Because the diaphragm is under tensile loading while in use, tensile properties were chosen as the main mechanical property to evaluate.

Dogbone samples will be tested for tensile strength. ASTM D412 will be used as the standard (1986), with Die D defining the dogbone sample size. The grip separation rate is 500 mm/minute. Tensile strength, elongation, and 100 percent modulus of samples from the diaphragm will be calculated according to the standard and compared with values from the unexposed materials.

Chemical Analysis

Material from the EPR diaphragm will be microtomed to obtain samples at various distances from the surface in contact with hydrazine. These samples will then be analyzed with transmission infrared spectroscopy. This study might also be performed with the aid of Auger Electron Spectroscopic techniques.

Diaphragm material will be analyzed for any chemical change that might have occurred as a result of exposure to hydrazine, for example, loss of plasticizer or ingredient during exposure. Plasticizers, oils, or other solvent-compatible ingredients content will be evaluated according to ASTM D3421 (1986) and ASTM D297 (1986).

Thermal Analysis

Thermal Gravimetric Analysis (TGA) gives weight loss versus temperature and, combined with FTIR, will give an idea of which, the quantity of, and at what temperature products are being lost by the material. Samples for thermal analysis will be taken from representative areas of the diaphragm and will be heated at 10 K per minute in ambient air to 973 K.

Material embrittlement will be measured as a function of temperature using Thermal Mechanical Analysis (TMA). Glass transition temperature of EPR also changes as a function of the plasticizer content; glass transition temperature will be measured with TMA at subambient temperatures.

Metal Testing

Two samples will be cut from the tank. One (Sample 1) will be cut from the metal surface exposed to hydrazine for an extended period, and Sample 2 will be cut from a fresh surface of the same metal unexposed to the hydrazine. The samples will then be prepared for metallographic observation in the optical microscope. Samples 1 and 2 will be compared to assess any changes. Oblique illumination or other special techniques to differentiate between the two samples might be necessary. Scanning electron microscopy may also be used to provide a much finer scale of the same effects.

Schedule

Table 4 shows the schedule for this test program.

Table 4
Test Schedule

Task	Completion Date
Receive Tanks	8/16/91
Nondestructive Evaluation and Dissection	10/5/91
Visual Examination	10/18/91
Microscopic Analysis	10/18/91
Thickness Measuring	10/18/91
Hardness Testing	10/18/91
Specific Gravity Measuring	10/18/91
Tensile Testing	11/1/91
Chemical Analysis	
Transmission Infrared Analysis	11/8/91
Volatiles/Extractables	11/8/91
Thermal Analysis	11/8/91
Metal Testing	11/8/91
Formal Report	11/15/91

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