AFRPL-TR-71-113

AD735288

LONG-TERM STORABILITY OF PROPELLANT TANKAGE

H. M. WHITE, 1st Lt, USAF

TECHNICAL REPORT AFRPL-TR-71-113

NOVEMBER 1971

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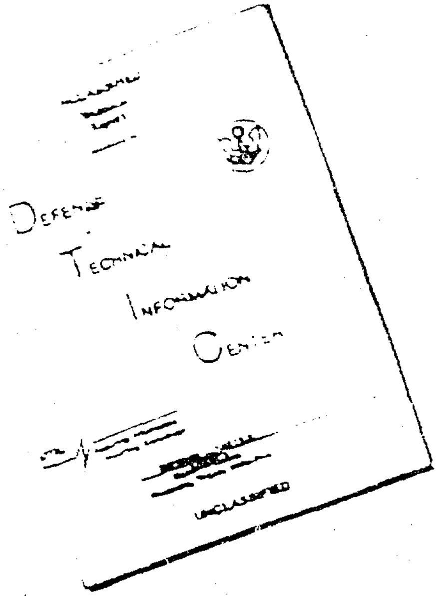

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DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author) Air Force Rocket Propulsion Laboratory, Edwards, California	2a. REPORT SECURITY CLASSIFICATION Unclassified 2b. GROUP
3. REPORT TITLE Long-Term Storability of Propellant Tankage	
4. DESCRIPTIVE NOTES (Type of report and inclusive dates) Technical (through June 1971)	
5. AUTHOR(S) (First name, middle initial, last name) Howard M. White, Lt, USAF	
6. REPORT DATE November 1971	7a. TOTAL NO OF PAGES 112 & viii — 7b. NO. OF REFS 4
8a. CONTRACT OR GRANT NO. b. PROJECT NO. 305805FRJ c. d.	9a. ORIGINATOR'S REPORT NUMBER(S) AFRPL-TR-71-113 9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)
10. DISTRIBUTION STATEMENT This document has been approved for public release and sale; its distribution is unlimited.	
11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY Air Force Rocket Propulsion Laboratory, Air Force Systems Command, USAF, Edwards, California 93523

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The Air Force Rocket Propulsion Laboratory (AFRPL) is performing a program to investigate the storability of liquid system components and tankage under severe conditions of relative humidity and temperature. A variety of system components and tankage materials is being evaluated for long-term storability with liquid rocket fuels and oxidizers. Storage conditions are +85°F temperature and 85 percent relative humidity for oxidizer systems, and +65°F to +165°F temperature and uncontrolled humidity for fuel systems. The propellants under test are N_2O_4, ClF_5, N_2H_4 and MHF-5. Tankage materials under test are various alloys of aluminum, steel and titanium. Tankage is joined by automatic and manual TIG, EB and solid-state bonding techniques.

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DD FORM 1 NOV 65 1473

Unclassified
Security Classification

NOTICES

14 KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Propellant storage						
Environmental effects						
Propellant leakage						
Corrosion						
Oxidizer leakage						
Stress corrosion						
Integranular corrosion						
Hydrazine blend storage						
Packaged propulsion system storability						
Stress corrosion cracking						
Rocket propellants						
Leak detection						
Propellant tank quality control						

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ABSTRACT

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FOREWORD

This report covers the testing of liquid rocket propellant tankage and propellant subsystems to evaluate their long-term storage characteristics. The testing is being conducted by the Air Force Rocket Propulsion Laboratory, Edwards, California, under Project No. 305805FRJ. Testing is being conducted in test areas 1-40 and 1-36. The project engineer is Lt Howard M. White; the test engineer is Mr. Clifford T. Hurd. This report covers all work done under Project 305805FRJ through June 1971. Previous reports written on this project are: AFRPL-TR-69-82, "Long Term Storability of Propellant Tankage and Components," AFRPL-TR-70-43, "Long-Term Storability of Propellant Tankage and Components Interim Report No. 2," and AFRPL-TR-71-20, "Long Term Storability of Propellant Tankage." This report will be presented at the 1971 JANNAF Combined Propulsion Meeting 1 November 1971 through 4 November 1971.

This technical report has been reviewed and is approved

JERRY N. MASON, Capt, USAF
Chief, Subsystems Branch
Air Force Rocket Propulsion Laboratory

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SECTION I

INTRODUCTION

Experience with liquid propellant rocket feed systems has shown that the leakage of oxidizers can occur and constitute a difficult problem under certain environmental conditions. In propellant tankage and certain types of feed systems, leakage is most frequently observed at or near weldments. It has been shown experimentally for N_2O_4 that when a vapor leak occurs (through a weldment microcrack for example), the result is drastically influenced by the relative humidity of the atmosphere surrounding the tank (Reference 1). If the relative humidity is on the order of 30 percent or lower, the vapor from the leak (principally NO_2) will dissipate into the atmosphere and does nothing to aggravate the leakage. If the environment surrounding the tank has a relative humidity of greater than 30 percent, the vapor from the leak will not dissipate into the atmosphere, but rather the vapor will hydrolyze with the water vapor in the air, forming dilute nitric acid on the exterior surface in the immediate vicinity of the original leak. Figure 1* clearly shows the resultant corrosion and discoloration that results from this process. The nitric acid has a further effect in that it will enlarge the original leak path by working inward toward the source of the leak. In time, small or even minute vapor leaks can become large liquid leaks, if they are allowed to proceed. Although a similar detailed experimental program has not been performed with the storable interhalogen oxidizers such as ClF_5, an analogous process would be expected with hydrogen fluoride as the hydrolysis product. Failures of tankage with the above propellants lend credence to the foregoing hypothesis of the interhalogen oxidizers.

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* Figures and tables are presented sequentially beginning on pages 26 and 47, respectively.

In the past, the selection of materials for system applications has been based upon conventional fluid/material compatibility testing to determine discoloration pitting, weight gain or loss, notch sensitivity, stress corrosion cracking susceptibility, potential degrading effects on the propellant, and to a certain extent, a particular system contractor's experience with the fabrication of various materials likely to be used on the system in question.

Even after this thorough analysis and selection process, the material and/or processing used in the propellant tankage may not function properly or leaks may develop during the extended time required of many current liquid rocket systems. it is readily apparent that the use of conventional compatibility criteria, while certainly a part of the material selection process, is not in and of itself suitable for the selection of materials for the extended storage of liquid rocket propellants when fabricated into system tankage.

The major limitation on interpreting long-term storability effects in a realistically severe storage environment is the inability of conventional fluid/material compatibility criteria to predict leakage. Small, undetected inholes or microcracks could be formed by an attack by the propellant or grain boundary precipitates and weld inclusions that might never be detected through weight gain or loss calculations. Furthermore, the possibility of such defects forming is greater in the high-strength, limited weldability materials frequently used in liquid rocket propellant tankage. The size and methods of producing test specimens used in compatibility work eliminates many of the manufacturing and quality control procedures associated with production systems. Smooth, polished samples, welded or unwelded, are not comparable to fabricated tankage material. The experience of the Titan II weapons system is an excellent example of the inability to translate basic fluid/material compatibility data to fabricated tankage material. In that case, the tank ge material, 2014-T6 aluminum, is compatible with the oxidizer, N_2O_4 MIL-P-265398; however, in the field, th missile was plagued by leakage, frequently occurring in the tankage weldments or heat-affected weld zone, in a humid environment (< 30 percent relative humidity).

Long periods of storage may affect the functional performance and system reliability of prepackaged propulsion systems. To factor the storability variable in an adequate manner, many areas should be considered. Storage conditions must be selected that are representative of operational system conditions. Such factors as temperature and humidity play an important role. A detailed propellant analysis before and after testing is required to evaluate the effects of storage on the propellant. The cleanliness levels of the test articles must be known for reasons of safety, but equally important, for evaluating the process which was used to effect this cleaning level. Materials and chemicals used for cleaning may have an effect upon system life. In the same manner, manufacturing processes and quality control standards may impose many unforeseen conditions which vary from one manufacturer to another. throughout the fabrication of tankage (i.e., during forming, welding, inspection and testing), all data should be available for a meaningful post-test failure analysis in the event of a test article failure. Metal preparation prior to welding may make the difference between a satisfactory or unsatisfactory weld with regard to its ability to contain propellant without leakage. Helium leak testing of systems and the technique of leak testing are very important since small leakage which cannot be detected by X-ray or dye-penetrant inspection can lead to propellant leakage under adverse environmental conditions. These very small leaks can be detected through helium leak testing. The above variables must be known and controlled in a meaningful storability investigation.

Although there has not been a storability problem of the magnitude of the oxidizer storability problem on the Titan II, present and future monopropellant satellite systems require long-term storability data so their system designers can design systems with confidence with 5- to 10-year mission lives. In the long-term storability of hydrazine, the failure mode is one of propellant decomposition rather than leakage. This decomposition is catalyzed by impurities in the materials in contact with the propellant; therefore, the tanks must be prepassivated or, in the extreme, be allowed to self-passivate when loaded with propellant. The use of standard fluid/material compatibility tests will demonstrate basic propellant/material compatibility. The premise in this fuel storability program is that completely fabricated tankage must be loaded with propellant and placed in extended storage to permit evaluation of fabrication variables in determining those tankage materials that are suitable for long-term storage of hydrazine-type fuels with negligible pressure rise.

SECTION II

PROGRAM STRUCTURE

To bridge the gap between laboratory compatibility samples and the long storability required of operational liquid systems, the Air Force Rocket Propulsion Laboratory (AFRPL) has been conducting, for the past 5 years, a program entitled "Packaged Systems Storability." This program deals with the long-term (5 to 10 years) storage of tankage, components and integrated propulsion feed systems with earth-storable fuels and oxidizers. Tankage materials under investigation include aluminum, steel and titanium alloys. Test systems include tankage; and complete feed systems including tankage, components, expulsion devices and gas pressurization systems. Previously tested under this program were integrated systems consisting of tankage and feed system components.

The test systems encompassed by the program are divided into three basic groups: (1) small containers, (2) representative-type tankage, and (3) tankage with associated expulsion devices and/or feed system components.

GROUP I: SMALL CONTAINERS

All tankage in this group is of approximately 1-quart capacity manufactured from aluminum and steel alloys. The purpose for using this tankage group is to evaluate a particular problem or to evaluate a promising material. These test articles are relatively cheap and serve as excellent "screening" devices. Because of their small size, these containers cannot duplicate the manufacturing and quality control problems associated with larger-size tanks. There are three types of tankage in this group:

1. 3- by 6-Inch Containers

There are 28 containers of this type in the program. All containers are manufactured of 2014-T6 aluminum. Containers manufactured by McDonnell Douglas, General Dynamics, Martin and North American Rockwell (seven from each firm), were tested. These were a direct offshoot of the leakage problem encountered with the Titan II weapons system, and were indicated to determine if N_2O_4 (Specification MIL-P-26539) and 2014-T6 aluminum was an "unstorable" propellant/material combination, or if the Titan II leakage problem was solely a Martin fabrication/quality control problem. Figure 2 shows tanks of this type.

2. Alcoa 1-Quart Containers

These tanks are Alcoa standard containers for material compatibility testing and are used to evaluate the storability of various aluminum alloys with N_2O_4 and ClF_5. The aluminum alloys are: 2014-T6, 2021-T6, 2219-T81, 3003, 5456 T-6 and 7007-T6. Tankage of this type is shown in Figure 3.

3. Arde Cylinders

These are small containers developed by Arde, Inc., as high-pressure CO_2 cylinders of AISI 301, cryogenically stretch formed stainless steel. They are used to evaluate the storability of this material in both aged and unaged condition with N_2O_4, ClF_5 and N_2H_4. They are illustrated in Figure 4.

GROUP II: REPRESENTATIVE TANKAGE

The tankage in this group varies in size from 10-gallon capacity up through a full-scale Agena tank, and encompasses tankage fabricated solely for use as test articles in this program as well as surplus tankage from actual operational systems. The tankage in this group is fabricated through current or advanced state-of-the-art methods, and the types of fabrication and quality control problems encountered during the course of manufacture of this tankage group would likely be encountered during the manufacture of an operational liquid rocket system. There are three basic types of tanks in this tankage group:

1. Storability Test Articles

These are tanks of 10- to 15-gallon capacity procured especially for use in this program. These are tanks which were either manufactured by Convair (Figure 5) or Martin (Figure 6) as a part of several procurements over the course of several years. The tankage is manufactured from several aluminum, steel and titanium alloys. It was manufactured using large-scale production methods, and includes dome, girth, cylindrical, and longitudinal welds characteristic of large tankage design. Manufacturing process records, X-ray, photographs, inspection logs and metallurgical samples of welded and unwelded materials were delivered with the tanks to serve as documentation. The tanks are loaded with N_2O_4 (Specification MSC-PPD-2A), ClF_5 and N_2H_4.

2. Existing Tanks

These are tanks that were donated or were surplus to other AFRPL programs, or tankage from operational liquid rocket systems. The tanks are as follows:

a. Bullpup Tanks. These are three 2014-T6 aluminum tanks (Figure 7) manufactured by the Reaction Motors Division of the Thiokol Chemical Corp., and are loaded with N_2O_4 (Specification MSC-PPD-2A)

b. Minimum Cost Design Tanks. These are four tanks of HY-140 steel and six tanks of Maraging-200 steel (Figure 8). They were designed to demonstrate 90-day storability of N_2O_4 (Specification MIL-P-26539) and UDMH as part of the AFRPL Minimum Cost Booster Program.

c. ULPR Tanks. These tanks were surplus from the AFRPL Ultra Low Pressure Rocket (ULPR) Program (Figure 9). They are two 2219-T81 tanks and are loaded with N_2O_4 (specification MSC-PPD-2A).

d. Agena Tank. This was a tank utilized to demonstrate 90-day storability of N_2O_4 (Specification MSC-PPD-2A) as part of the Agena E program (Figure 10). The standard Agena oxidizer is IRFNA.

3. Solid State Bonded Tanks

These tanks were hardware delivered under an AFRPL program with Martin to demonstrate the explosive bonding technique in the fabrication of tankage. Two configurations (Figure 11) of tankage were fabricated from A286 stainless steel and one configuration was fabricated of 65A titanium. The A286 tankage is loaded with N_2O_4 (Specification MSC-PPD-2A) and ClF_5, while the Ti-65A tankage is loaded with N_2O_4 (Specification MSC-PPD-2A).

GROUP III: EXPULSION SYSTEMS AND COMPONENTS

In an operational system, an expulsion device is often integrated into the tankage to ensure that single-phase liquid is fed to the engine. Since this is the case, the storability of this combination must be evaluated. Also, any liquid rocket feed system has components associated with it, and an assessment of the component storage characteristics is necessary to design properly a liquid rocket propulsion system. Test articles in this group represent an attempt to assess the storability of components and expulsion systems. The test articles in this group are:

1. Metallic Reversing Diaphragms

There are two types of tankage in testing associated with its expulsion device (Figures 12 and 13). In all cases the tankage is AISI 301, cryogenically stretch-formed stainless steel. One group of six tanks 12 inches in diameter has a 304L stainless steel reversing diaphragm and is similar to that developed for LITVC tankage on the third stage of Minuteman III. These test articles are loaded with N_2O_4 (Specification MSC-PPD-2A), ClF_5 and N_2H_4. Two 28-inch-diameter conospheroid tanks are also being tested with N_2O_4 (Specification MSC-PPD-2A). These tanks have an AISI 321 stainless steel expulsion diaphragm. All of this tankage was manufactured by Arde, Inc.

2. Rolling Diaphragm

These are three tanks fabricated by the Reaction Motors Division of Thiokol Chemical Corp. (Figure 14). These tanks have an 1100-0 aluminum expulsion diaphragm bonded to a Maraging-200 steel shell and are 30 inches in diameter. Test articles are loaded with N_2O_4 (Specification MSC-PPD-2A).

3. AFRPL Integrated Systems

The tanks here are similar to those described under Group II Storability Test Articles, but, associated with the tank on tubing located on the top and bottom, are fluid components normally found in liquid rocket systems. The tankage is either 2219-T81 aluminum or AM350 steel. Fluid components consist of a pressure switch, explosive valve and burst disk. Fittings are AFRPL mechanical fittings (MIL-F-27417) and TIG welded joints. Since tankage material and component materials are of both aluminum and steel, intermetal transitums are made using both mechanical fittings and solid state bonded transition joints. These systems are shown in Figures 15 through 19.

4. Prepackaged Feed Systems

These are test articles developed by General Dynamics Corp., and consist of 2219-T86 EB welded tankage, with either a rolling diaphragm or surface tension screen expulsion device, and either a liquid propellant gas generator (LPGG), solid propellant gas generator (SPGG) or high-pressure stored gas device (GD) pressurization systems. Systems are loaded with N_2O_4 (both MIL and MSC specifications), ClF_5 and MHF_5. They are shown in Figure 20.

SECTION III

TEST FACILITIES

Storage testing of tankage loaded with oxidizer is conducted in a metal Quonset hut storage test building equipped to provide a constant controlled environment of 85 $\pm$ 5°F and 85 $\pm$ 5 percent relative humidity. The oxidizer storage test facility is insulated by a spray-in-place foam (polyurethane). Environmental conditions are maintained by two evaporative coolers and immersion water heaters. Safety provisions in this facility consist of a firex-type water deluge system, large water drain piping, fire detectors, a continuous toxic vapor detector and closed-circuit television monitoring. The oxidizer storage facility is presently being modified by the addition of an automatic conditioner shutdown and scrubbing system, which will be operated when an excess of oxidizer vapor is detected by the facility toxic vapor detector.

The storage testing of fuels is conducted in a building equipped to provide controlled temperatures and uncontrolled relative humidity. The temperature inside the fuel facility can be controlled at any temperature between +65° and +165°F. Temperature conditioning is maintained by a heating and refrigeration system. The fuel storage building was originally insulated with a spray-in-place polyurethane type of form insulation. This installation has subsequently been shown to be a fire hazard. At the present time, testing is suspended pending replacement of the insulation with a fire-retardant variety. It is expected that fuel testing will resume in November 1971.

SECTION IV

PROCEDURES

The test articles utilized in this program are procured from aerospace contractors and are fabricated with tooling and fabrication methods currently in use in liquid rocket systems. The primary responsibility for quality control and quality assurance of the test articles is vested in the manufacturer of the test article. To ensure high-quality test articles for use in this program, procedure specifications governing all aspects of the test article manufacture, inspection and cleaning were either generated or identified for use in the procurement of all test articles in this program.

All test articles with the exception of the integrated pressurization/ tankage/ expulsion systems procured from General Dynamics, are leak checked by helium mass spectrometer and verified to be clean at the AFRPL to ensure against the development of leaks and the introduction of contamination during shipment of the test articles from the manufacturer.

Following this, the test articles are loaded with propellant and placed in the appropriate storage facility for storability testing. The oxidizer tankage is montored for leakage. The fuel tankage is monitored for excessive pressure rise.

Oxidizer tankage is removed when evidence of leakage is found. This leakage is detected either through observation of an actual liquid leak, or the detection and location of a vapor leak by means of the facility toxic vapor detector. This instrument can also be configured as a "sniffer" to pinpoint leakage.

In the event of excessive pressure rise in a fuel tank, the tank is vented and propellant and ullage gas samples are taken. Tanks which exhibit continued pressure rise are removed from testing and analyzed to determine whether the pressure was due to an isolated instance or is indicative of a lack of storability of the material.

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SECTION V

DISCUSSION OF RESULTS

A summary of all test articles and the results to date are presented in Tables I through IIIA. Detailed analysis of all test article failures in this program are presented in the appendices of the reports published during this program (References 2 through 4). The failure analyses in this report cover those analyses performed from January to July 1971. A general discussion of the results obtained from each tankage group is presented below.

GROUP I: SMALL CONTAINERS

1. 3-by 6-Inch Containers

This type of tankage is no longer in test. Of the 28 containers that were placed in storage, 23 were still loaded with propellant when the test was terminated. The failure of four of the five tanks that were withdrawn from storage testing can be attributed to poor container end-plate joint design, which in turn resulted in poor weld penetration (Reference 2). The failure of the other vessel was due to nitric acid attack on the exterior surface which led to eventual development of stress corrosion cracking and vessel failure. The failure analysis performed on this vessel does not indicate whether the initial nitric acid attack resulted from N_2O_4 vapor leak in this vessel or from N_2O_4 leaking from some other vessel in the storage facility and then condensing on the vessel in question.

The 3- by 6-inch container testing was terminated for three reasons. First, at the time of testing at which these containers were terminated (5 March 1971), a total of 1522 days of testing was accumulated on the 23 containers under test. It was felt that this was enough storage time to demonstrate the storability of the N_2O_4/2014-T6 aluminum combination. Secondly, the floor space taken up in the facility by these test articles was needed for other more representative types of test articles. Finally, the testing of the 3- by 6-inch containers was terminated because the basic design of the containers was a poor one, and as a result, few data pertinent to flight-type systems were gathered.

2. Alcoa 1-Quart Containers

Testing of containers loaded with N_2O_4 was suspended 5 March 1971. As of that date, no leaks had been detected in any of the 16 containers being tested with N_2O_4. The principal reason for withdrawing these containers from testing was to utilize the floor space in the facility taken up by these containers for more advanced test articles. A secondary consideration was that all but two of the aluminum alloys, 5456 and 7007, were represented in other test articles in this program.

Testing of Alcoa containers loaded with ClF_5 continues; of the 37 test articles originally put in storage, 25 are still being tested. Failure analysis of those tanks withdrawn from testing indicated that the failure mechanism was one of stress corrosion cracking initiated by the presence of dilute HF on the external surface of the test article. As with the above 2014-T6 aluminum 3- by 6-inch container, the failure analysis cannot indicate whether the HF resulted from a ClF_5 vapor leak in some nearby tank/container with the HF condensing on the container which leaked or from the leaking container itself. In 1970, four 2014-T6 aluminum tanks were withdrawn from storage testing with bad cracks in the fitting boss weld, but prior to actual failure, as evidenced by leakage. The analysis of the cracking (Reference 4) in these tanks would seem to lend credence to the argument that the cause of cracking (determined to be stress corrosion cracking) may have been due to the HF condensed on the surface or that the HF was from some tank other than the one in which the stress corrosion cracking developed. The foregoing argument is discussed further in the failure analysis report of a Group II tank.

3. Arde Cylinders

Of the 59 test articles placed into storage, N_2O_4 (5 aged, 5 unaged), ClF_5 (5 aged, 5 unaged) and N_2H_4 (15 aged, 14 unaged), only one, an aged vessel load with ClF_5 has been removed from testing. This failure was the result of an environmentally induced stress corrosion crack, and occurred over a 2-day period when approximately 7.5 gallons of ClF_5 were released into the oxidizer test facility. This large release of ClF_5 resulted in a high concentration of HF buildup in the facility and was at least partially responsible for the leakage of this test article. The reason for this large release of ClF_5 will be discussed later under Group III tankage, ARRPL Integrated Systems. Discounting this one leak, it would appear that the 301 cryo-stretched material is an excellent material for use in liquid rocket tankage.

GROUP II: REPRESENTATIVE TANKAGE

1. Storability Test Articles

The tankage failures encountered during testing of this group of test articles are probably the most significant of the entire problem. These failures give a firm indication as to the areas where improvements can be made to increase the storability of various propellant/material combinations.

During this program, five titanium vessels (three of 6Al-4V titanium and two of 5Al-2.5Sn titanium) failed as a result of loading with "brown" N_2O_4 (MIL-P-26539B). All of the titanium tanks leaked within 35 days after loading with N_2O_4. Both the 6Al-4V and the 5Al-2.5Sn titanium alloys were in the annealed condition when tested. The use of "green" N_2O_4 (Specification MSC-PPD-2A) was considered at the time of loading the tanks; however, the stress levels in the tankage, based on the nominal loads and thickness, were considerably below the threshold for the stress corrosion cracking reported (16 ksi nominal stess versus 40 ksi reported threshold). Also, the test temperature was significantly below the temperatures at which problems were encountered (85° versus 110°F.) On the basis of the above considerations, stress corrosion cracking was not thought to be significant. Failure analysis of the five tanks (References 2 and 3) indicates that stress corrosion cracking in the weld area and heat-affected zone was responsible for the failure of these tanks. Currently, there are three 6Al-4V tanks, similar in design to the 5Al-2.5Sn tanks described above, in the program that are loaded with "green" N_2O_4 (Specification MSC-PPD-2A). These tanks have been tested for approximately 2 years with no indication of leakage. Based on the foregoing, the use of "green" N_2O_4 is encouraged for all systems utilizing titanium.

A second type of failure encountered with high frequency during the storage testing of these test articles is hot-short cracking in and around the area of double-pass welds. These may either be start-stop zones or repair welds. In either case, there is a high probability of hot-short cracks which lead to vessel failure. To compound the problem further, these defects are often missed during the course of normal quality control operations. Failures of this type are indicated in the failure analyses presented in References 2 and 3. Quality control operations should be structured so as to decrease the chances of such a defect slipping through.

One failure encountered in this program indicates poor design on the part of the test article manufacturer. This is documented in Reference 3. In this case, 7039-T6 aluminum vessel with N_2O_4(MIL-P-26539 specification) failed because of stress corrosion cracking along the short transverse grain direction. After failure, a stress analysis was performed, and excessive stress was found to exist along that grain orientation, thereby allowing a predication of the vessel failure mode prior to loading of the tank with propellant. A careful review of this design would have prevented this vessel failure.

The final and most common type of failure encountered in this program and also with a group of test articles is failure due to environmentally induced stress corrosion cracking. This failure mode has been documented in failure analysis performed by Martin-Marietta Corporation and in-house by the AFRPL Metallurgical Laboratory (References 3 and 4). This is the failure mode alluded to earlier in discussing the failure of 1-quart Alcoa containers loaded with ClF_5. In the above vessel failure mode, stress corrosion cracking is induced by diluted acid on the exterior surface. This acid comes from the hydrolysis of oxidizer vapors whose source is a small leak in a vessel in the storage facility. Whether the source of the leak is the actual vessel that fails or some other vessel is open to question. There is some experimental evidence to support both conclusions. The work done by Martin (Reference 1) as part of the Titan II leakage problem would indicate that vapor leakage in a vessel would in time lead to a liquid leakage in that vessel. Failure analysis done by Martin-Marietta on two vessels containing N_2O_4 revealed the presence of fluorides on the exterior surface (Reference 2). The only source of fluorides in the storage facility was the ClF_5 stored in the building. This would in turn indicate that ClF_5 vapor, whose source was a ClF_5 vapor leak in another test article, hydrolized in the humid environment of the test facility to form HF, which in turn condensed on the N_2O_4 vessels and initiated stress corrosion cracking which led to test article failure.

Of the failures classed as due to environmentally induced stress corrosion cracking, a majority occurred in tankage fabricated from either 17-7PH or AM350 steel and loaded with ClF_5. In no case, were nitrates, indicating nitric acid/N_2O_4 attack, found. This would indicate that the use of either 17-7PH or AM350 steels with ClF_5 would be unwise.

The only way to eliminate this type of failure would be to isolate each tank from every other tank. In view of the extensive facility modification required to do this, this alternative is not being considered at this time. It is hoped that instead, the installation of toxic vapor detectors (as discussed in Section III) will substantially reduce the incidence of this type of failure by clearing the facility whenever a concentration of oxidizer vapor is detected.

Although hydrazine testing is currently suspended pending a facility modification, almost a year of testing was completed prior to the termination of testing. While firm conclusions cannot be drawn on the basis of pressure rise data collected to date, it appears that 17-7PH and AM350 are unsuitable for high-temperature (>100°F) storage.

2. Existing Tanks

a. Bullpup Tanks. All three continue in storage. They have accumulated over 3 years of storage.

b. Minimum Cost Design Tanks. Of the ten tanks of this type, two of the HY-140 tanks were loaded with N_2O_4 and two with UDMH. Three of the Maraging-200 tanks were loaded with N_2O_4 and three with UDMH. The storage testing was to demonstrate a 90-day loaded padlife without leakage or excessive pressure rise. This 90-day padlife was demonstrated and the tankage removed from testing with the exception of two maraging steel tanks which were retained for continued evaluation of the storability of this material with N_2O_4. Approximately 2 years of storage time have been accumulated on the two tanks still being tested.

c. ULPR Tanks. Testing of these tanks was terminated after approximately 3 years in storage. At the time the tanks were removed from testing, no leaks had developed. These tanks were removed to provide floor space for more advanced test articles.

d. Agena Tank. This tank was tested to demonstrate a 90-day storability with N_2O_4, in support of the Agena E (Advanced Agena) Program which contemplated a change from IRFNA to N_2O_4. The requirement was met and the tank was removed from testing at the end of that time.

GROUP III: EXPULSION SYSTEMS AND COMPONENTS

1. Metallic Reversing Diaphragms

All test articles of this type loaded with oxidizers are still being tested with no leakage observed. Those test articles loaded with hydrazine have been withdrawn from testing and will be returned to testing when the modifications to the fuel storage facility are completed. During the storage testing of these articles with hydrazine, no excessive pressure rise was noted.

2. Rolling Diaphragm

Of the three test articles initially placed in storage testing, two remain. The third developed a leak after 3 months of storage testing. The leak was due to failure of a hub-to-diaphragm weld. This failure points to an area where increased quality control inspection would be in order.

3. AFRPL Integrated Systems

Testing of these articles has been suspended and will not be resumed. This action is a result of the extensive damage sustained by the fluid components when 7.5 gallons of ClF_5 were released into the facility. The ClF_5 was released when a manual weld in the tubing on the bottom of an AM350 steel tank failed because of a tungsten inclusion in the weld. This release of ClF_5 caused leaks in the above AM350 tank and an Arde cylinder also loaded with ClF_5. Following the leak, all test articles of this type were removed from testing and examined. It was then determined that the fluid components, particularly the pressure switches and transition joints, sustained unacceptable damage. At this time, the installation was reconfigured to allow testing of the tanks alone. The fluid components were retained for analysis. Appendix I presents the results of post-storage tests of squib valves associated with the test articles. The tanks associated with these test articles are now reported in Group II-Storability Test Articles. Testing of these tanks will resume in September of 1971.

4. Prepackaged Feed Systems

To date, there have been no failures in those systems loaded with MHF-5. Failures that have occurred in systems loaded with ClF_5 have been the result of propellant leakage. Leakage has occurred through either the fill tube, which was welded shut (Reference 2), or through voids in the gas side burst disk (Reference 4). There are no more systems loaded with ClF_5 under test.

There has been one leak in an N_2O_4 system due to a fill tube leak. Also, seven systems have been withdrawn from testing because of a failure in a regulator. The failure was due to environmentally induced stress corrosion cracking. Both N_2O_4 and ClF_5 hydrolysis products were found on the surface of the regulator.

SECTION VI

CONCLUSIONS

The Package Systems Storability Program has accumulated a significant amount of storage time, and sufficient data have been collected so that tentative conclusions and recommendations can be made. The conclusions and recommendations are based on failure analysis reported in earlier progress reports and general observations made during the program.

It has been observed that double heat welds which occur at start/stop points and at weld intersections or at weld repairs lead to a high incidence of hot short cracks. This condition is especially prevalent in manual repair welds because of poor control of heat input. It is therefore concluded that quality control criteria for acceptance of welds be made stringent enough, especially in the case of repair welds, to preclude the acceptance of defects.

This program has demonstrated the influence of propellant chemistry on storability. In five separate cases, tankage fabricated from titanium experienced failure due to stress corrosion cracking (at stress levels below the generally accepted threshold for stress corrosion cracking) in 1 month or less when loaded with "brown" N_2O_4 (MIL-P-26539 Specification Grade). At the present time, there are three titanium test articles with more than 2 years of successful storage time, loaded with "green" N_2O_4.

In one instance, it was noticed that because of poor tank design, excess stress levels existed in the short transverse direction of the material. This led to tank failure due to stress corrosion cracking, indicating that tank design must be carefully scrutinized to preclude significant stress levels along stress corrosion sensitive grain orientations.

The presence of trace amounts of tungsten resulted from inclusions produced by the tungsten inert gas (TIG) or heliarc welding process. This in turn resulted in the rapid development of weld leakage in welded tube joints used with ClF_5. This is because the tungsten was removed in the form of gaseous tungsten fluoride, and in turn resulted in a leak path. The process is somewhat analogous to intergranular corrosion. The problem of tungsten in fluoride service points up the need for stict quality control and the rejection of any weld showing traces of tungsten inclusions.

SECTION VII

RECOMMENDATIONS

In line with the conclusions presented in the preceding section of this report, tentative recommendations can be made with regard to improving the storability characteristics in liquid rocket propellants.

It is recommended that quality control systems be reviewed to preclude the possibility of the acceptance of tankage with poor design characteristics (i.e., excessive stress along sensitive grain orientations) or questionable welds (i.e., hot short cracks in double-pass regions, or trace inclusion).

It is also recommended that in the case of titanium tankage loaded with N_2O_4, the propellant have sufficient NO content to prevent initiation of stress corrosion cracking.

Figure 1. Hygroscopic Action of N_2O_4 Vapor

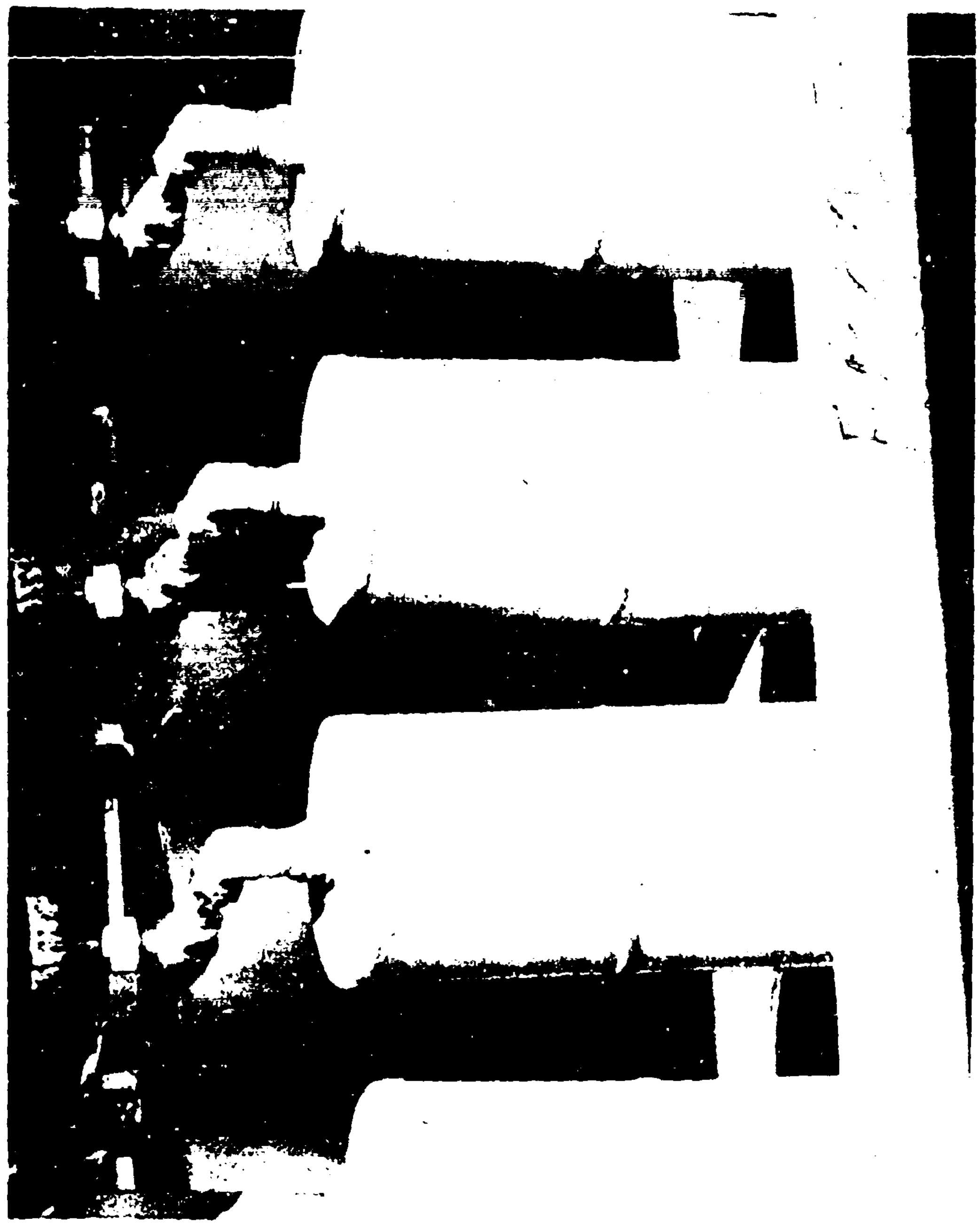

Figure 2. 3- by 6- Inch Containers

Figure 3. One-Quart Alcoa Containers

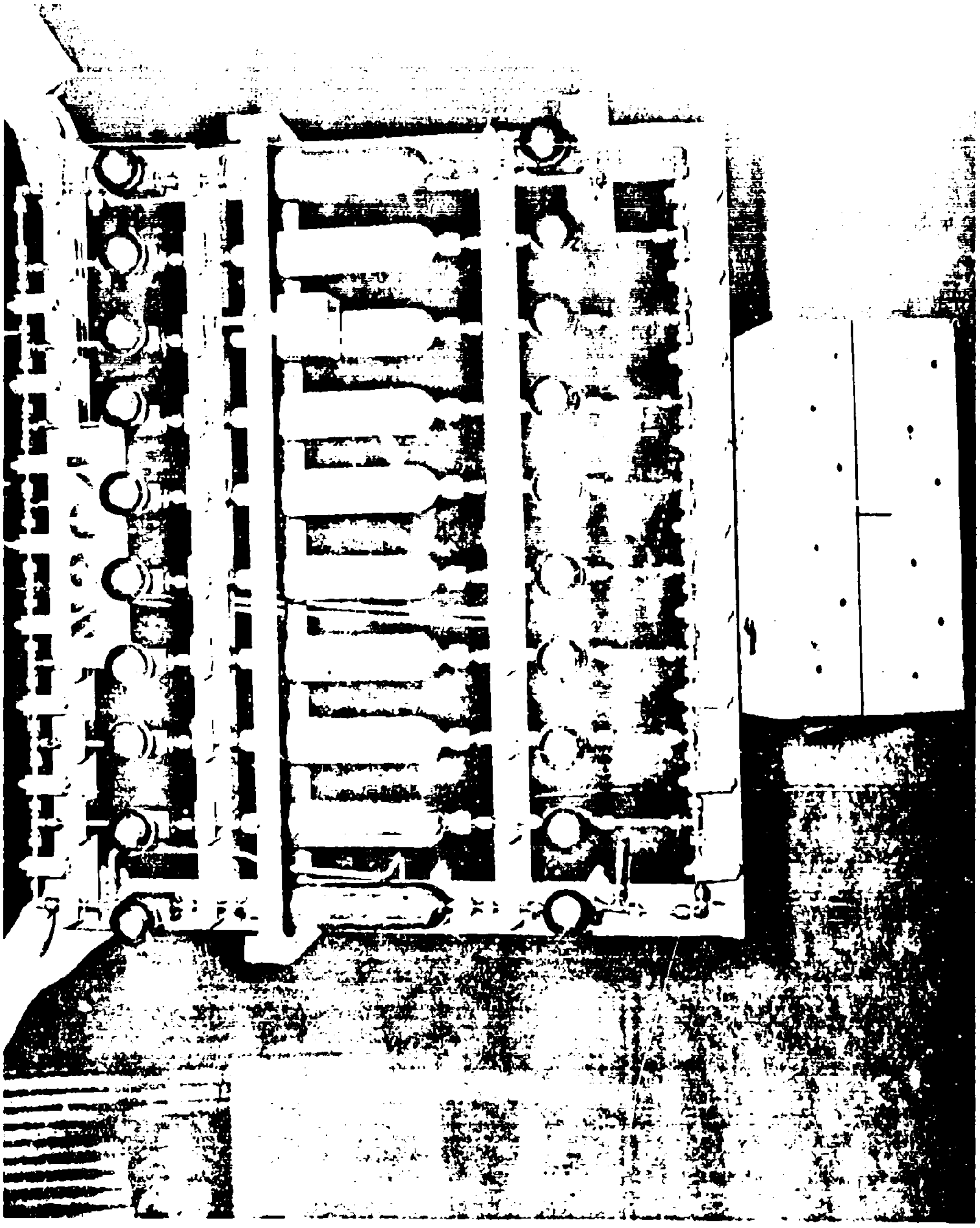

Figure 4. Arde Cylinders

Figure 5. Convair Storability Test Articles

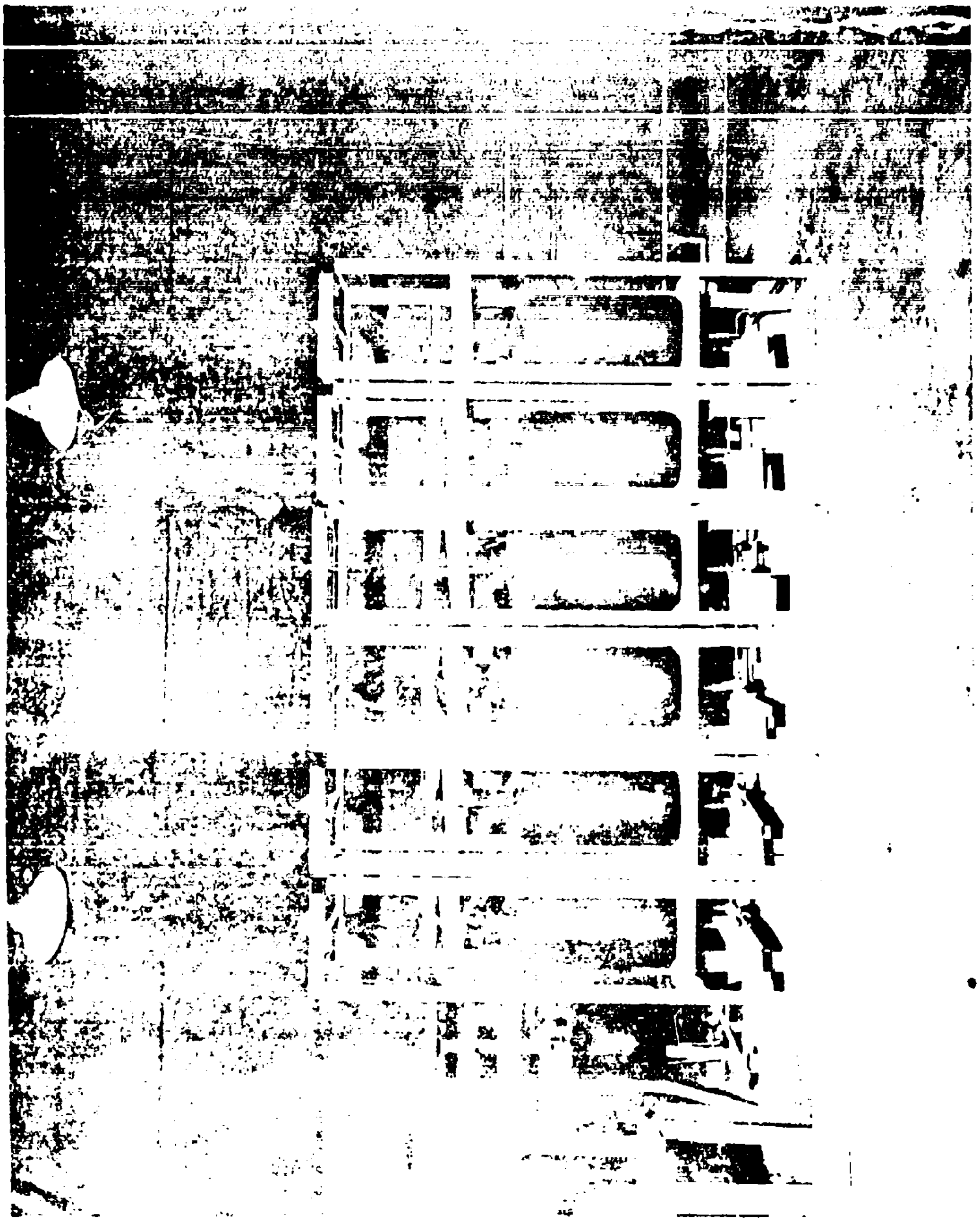

Figure 6. Martin Storability Test Articles

NOT REPRODUCIBLE

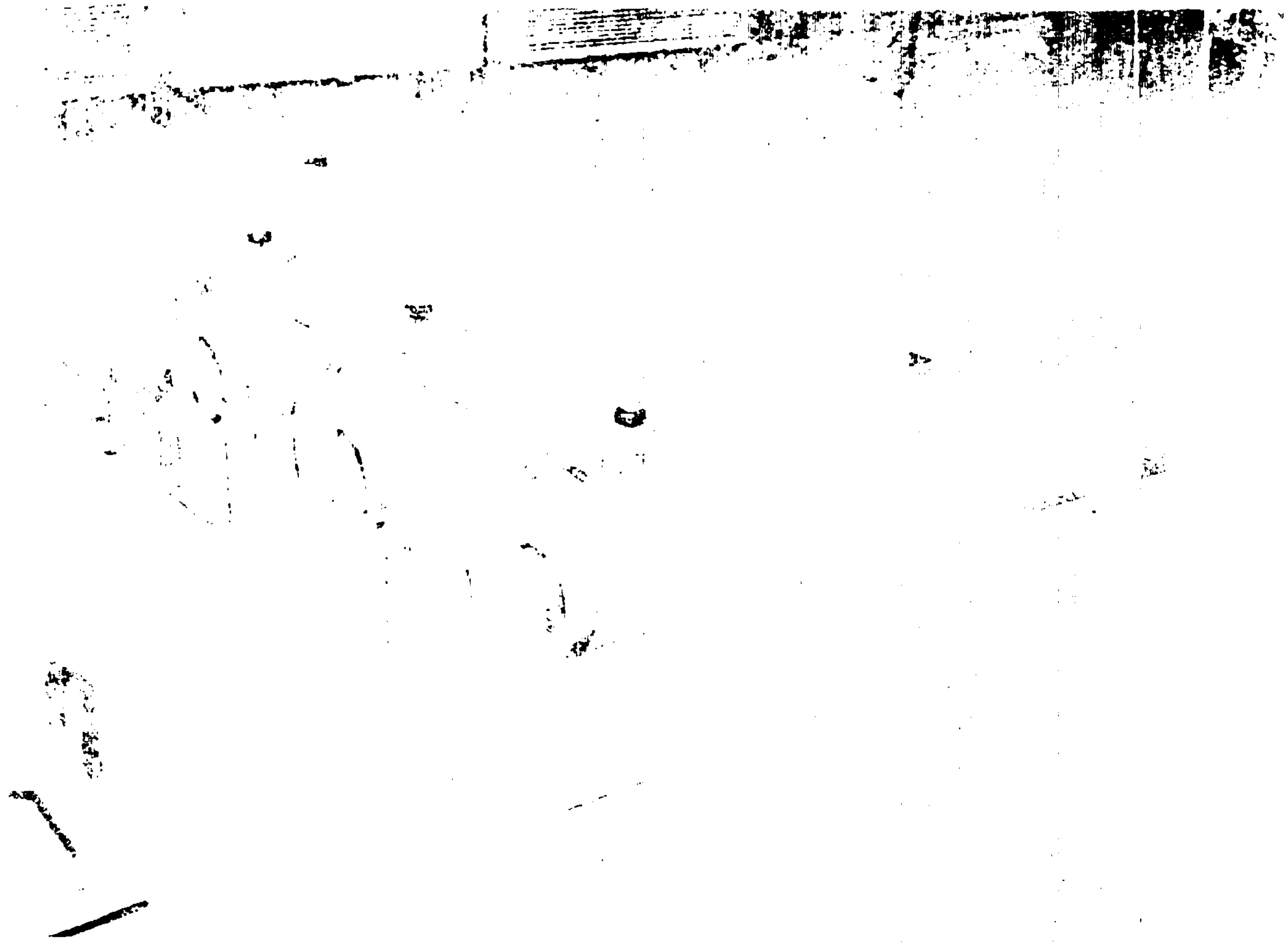

Figure 7. Bullpup Tanks

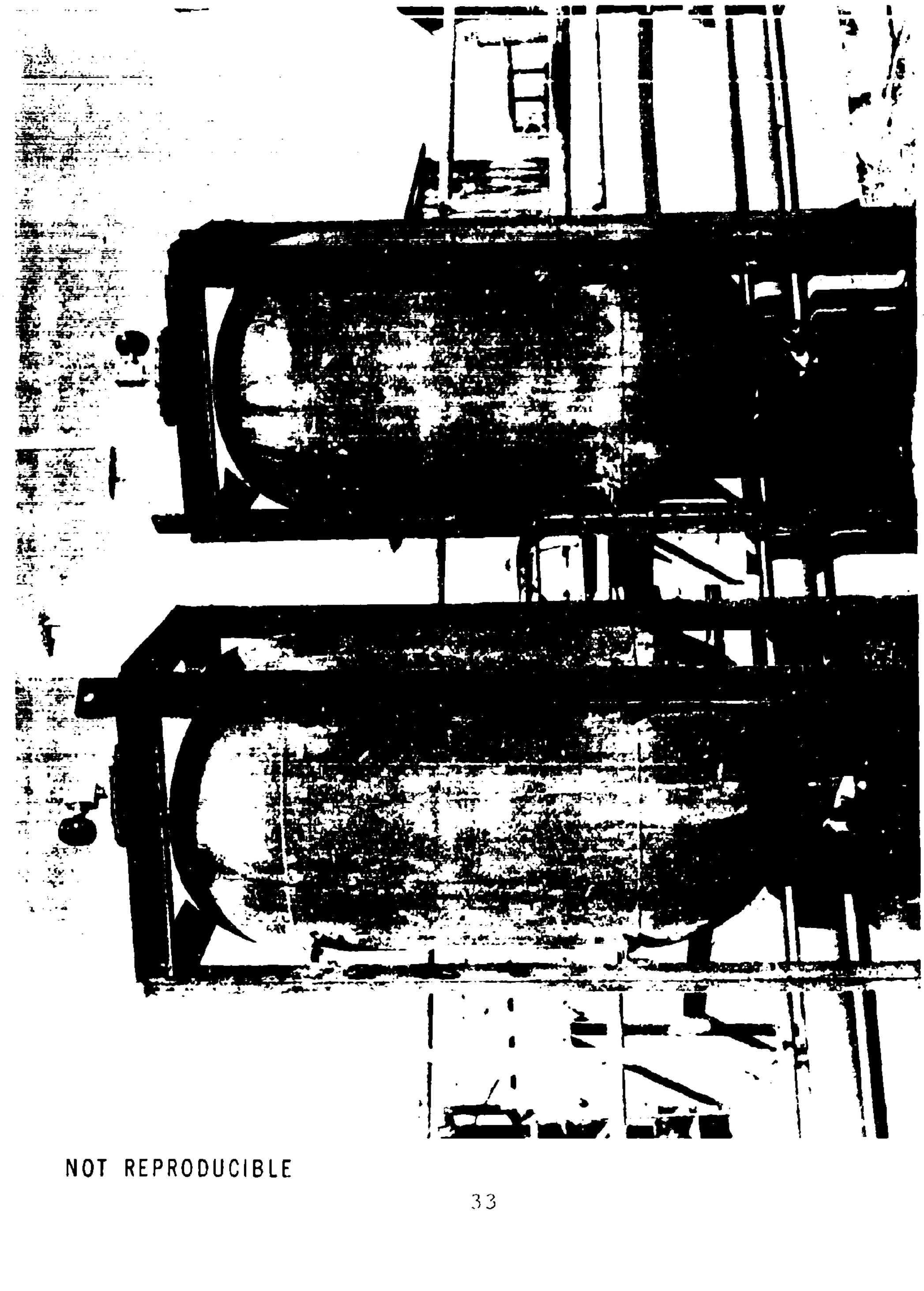

Figure 8. Minimum Cost Design Tanks

NOT REPRODUCIBLE

Figure 9. ULPR Tanks

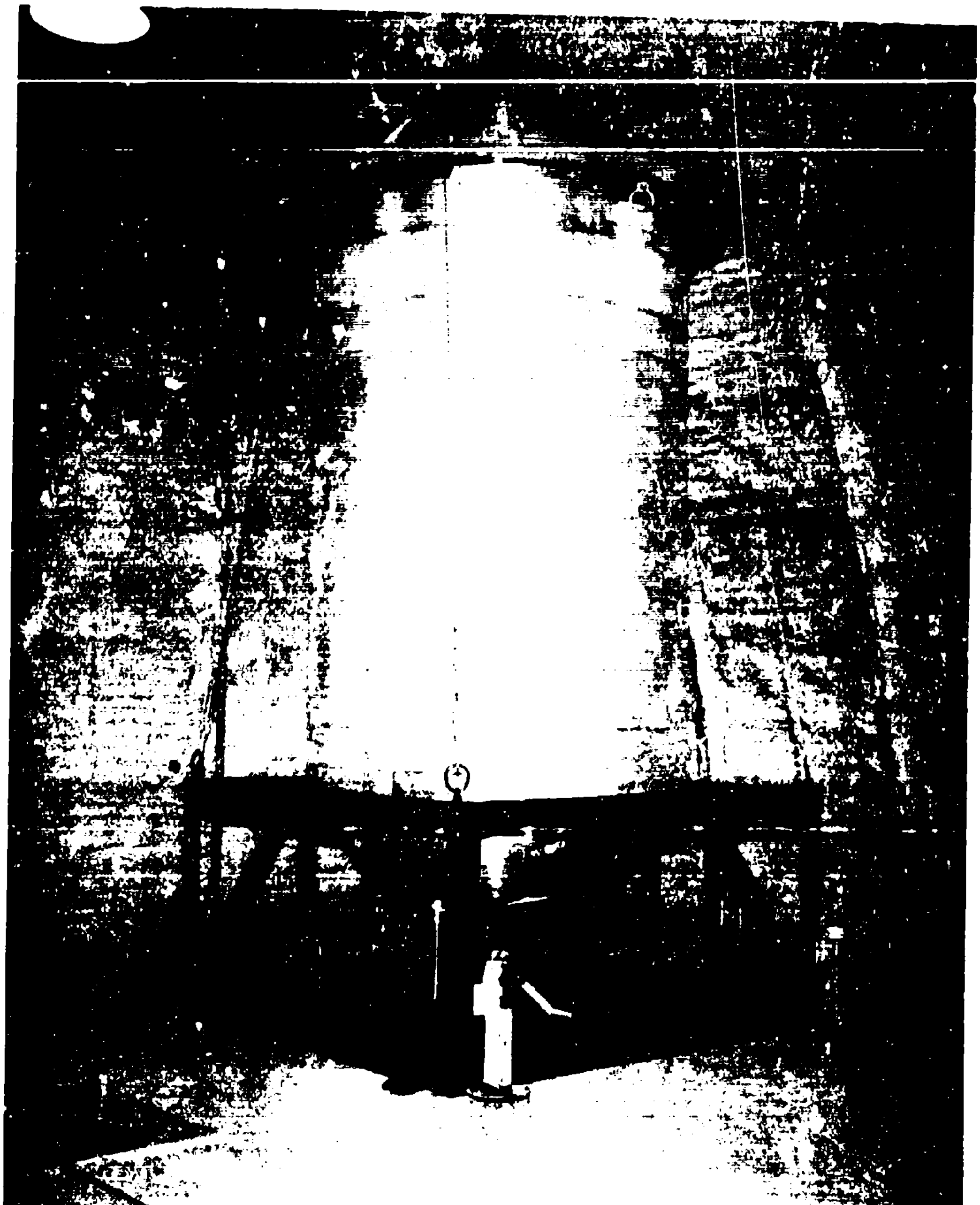

Figure 10. Agena Tank

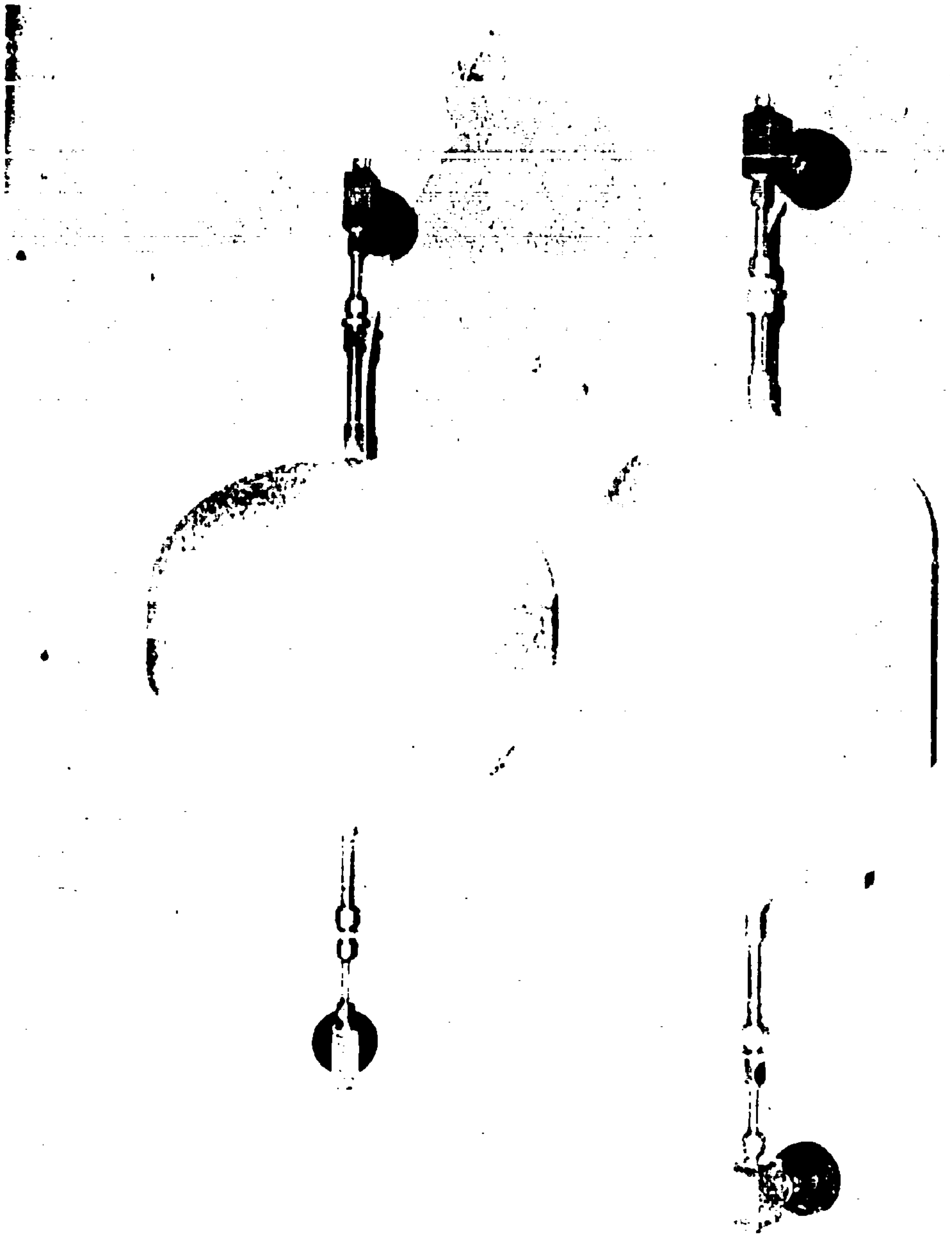

Figure 11. Solid State Bonded Tanks

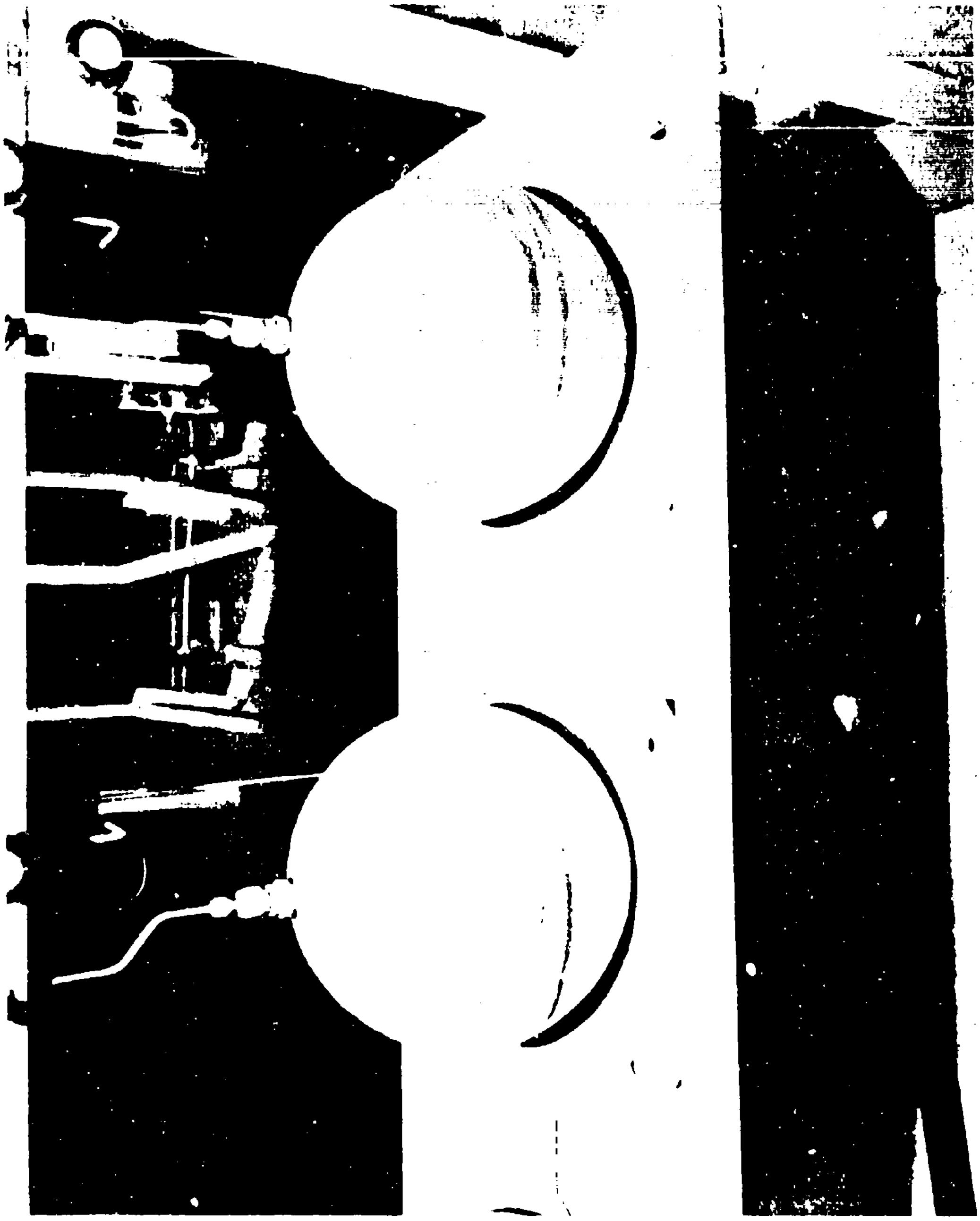

Figure 12. Twelve-Inch Arde Reversing Diaphragm Tank

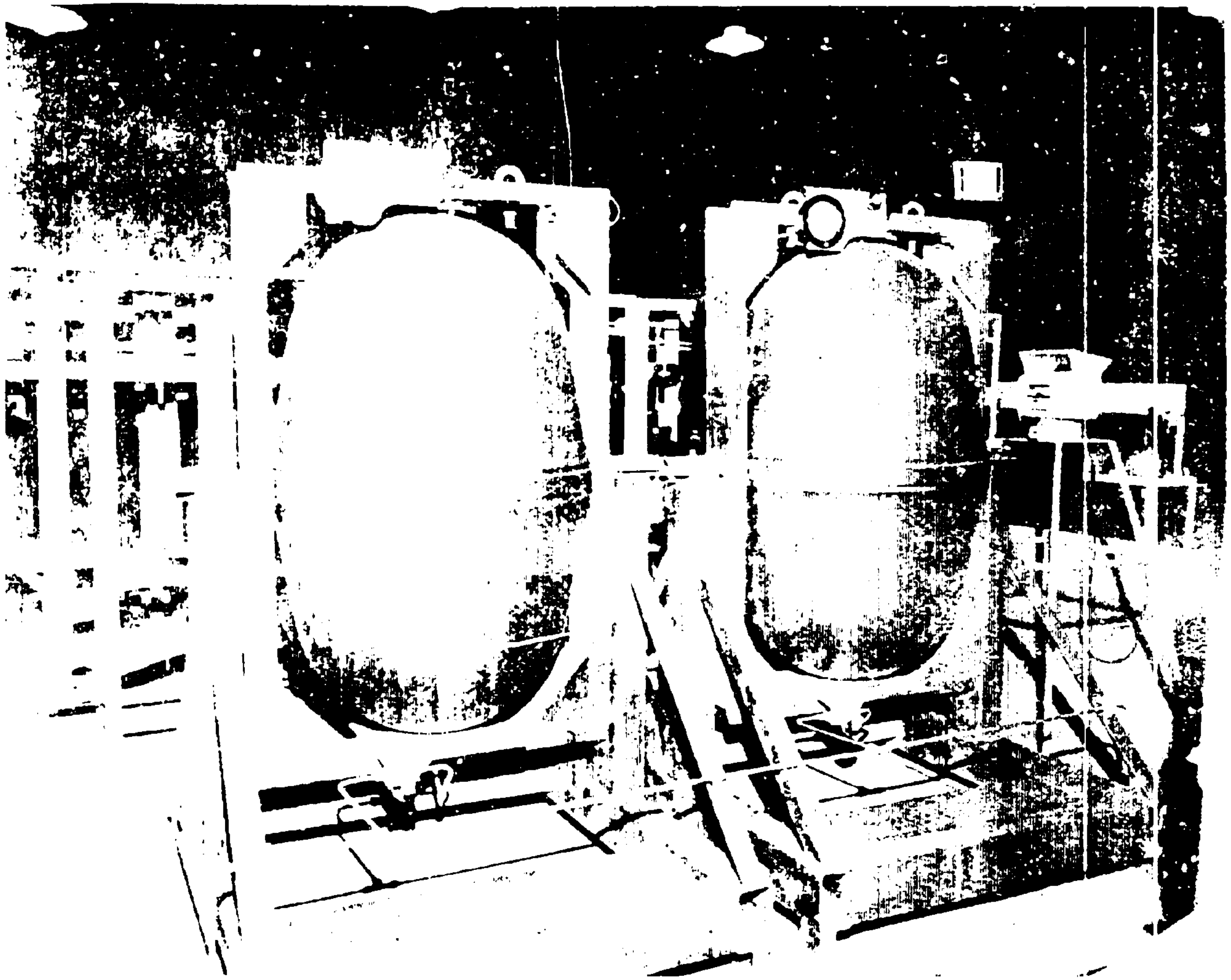

Figure 13. Arde Conospheroid

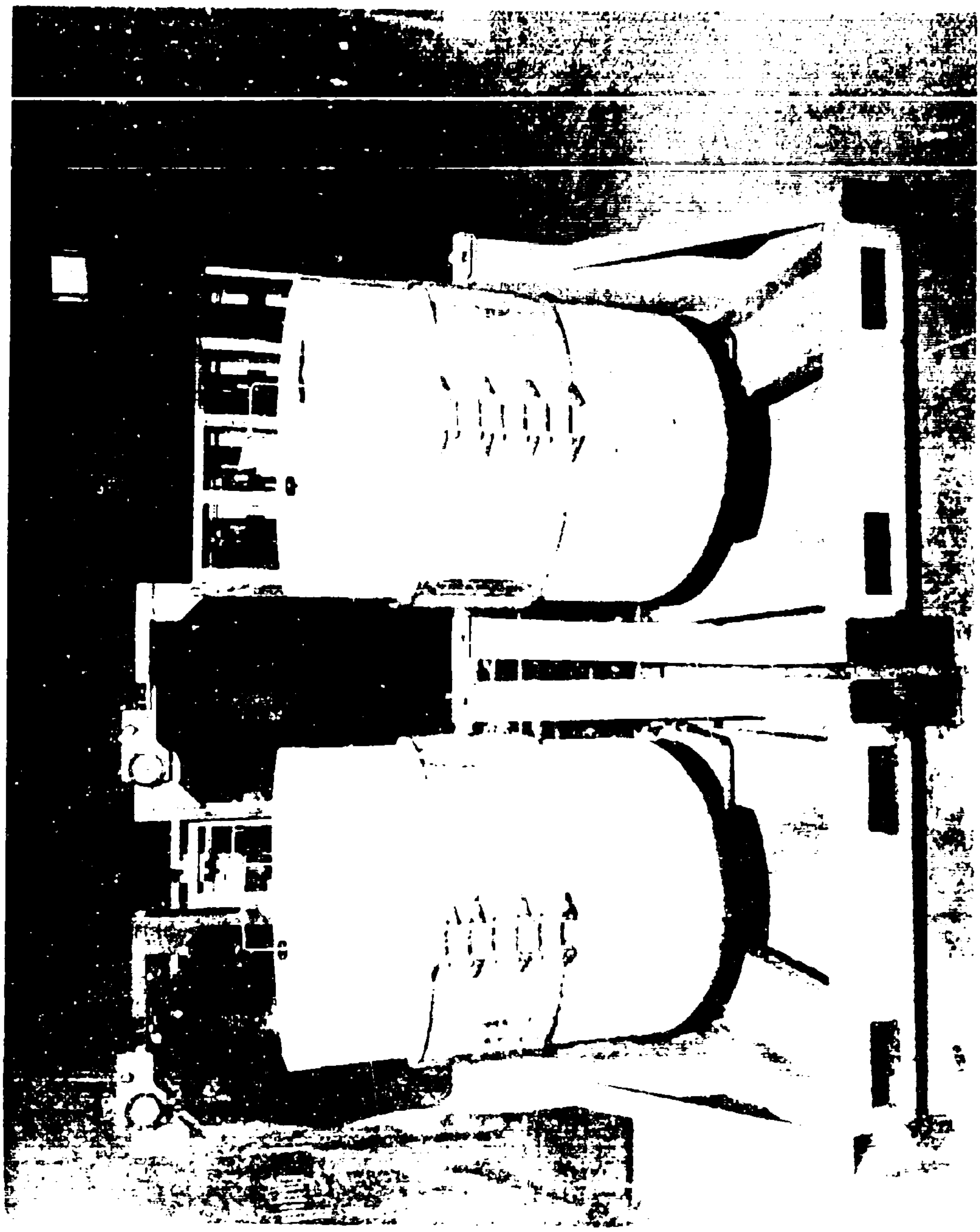

Figure 14. Thiokol Rolling Diaphragm Tanks

NOT REPRODUCIBLE

PART	MATERIAL
Tank	AM350
Transition Joint	347SST/6061-T6 Al
Pressure Switch	347 SST
Explosive Valve	347 SST
Burst Disk (100 psig)	6061-T651 Al
Burst Disk (120 psig)	6061-T651 Al
Hoke Hand Valve	347 SST
1/2 in. by 0.035 in. Tubing	347 SST
1/2 in. by 0.035 in. Cross	347 SST
1/2 in. by 0.035 in. Tee	347 SST
1/2 in. by 0.065 in./0.035 in. Tubing	347 SST

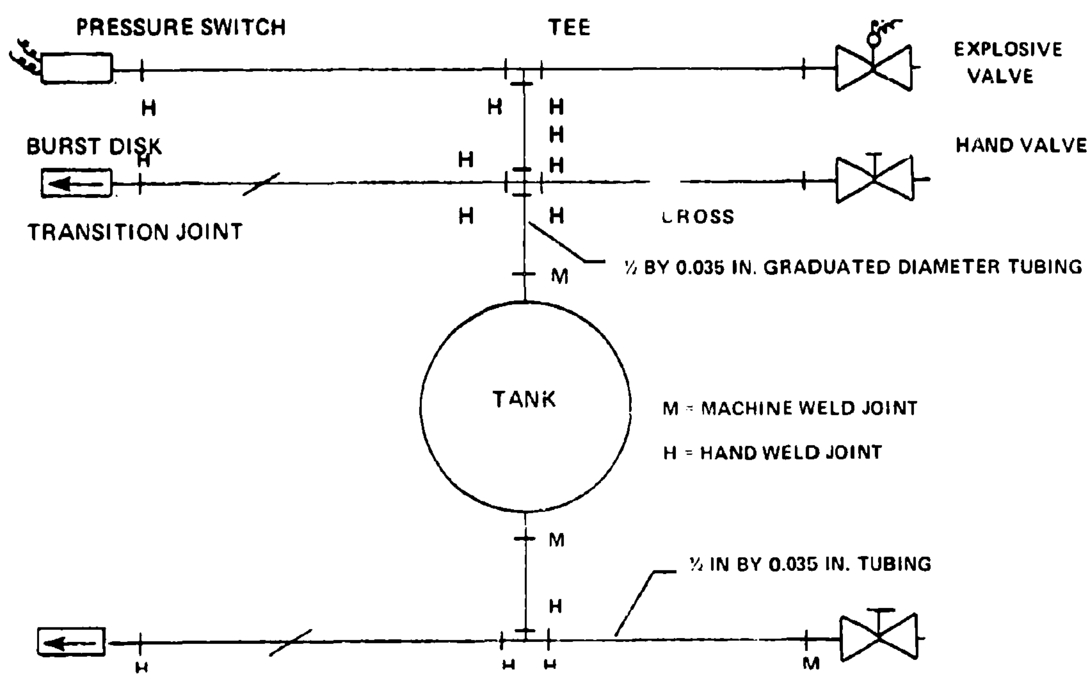

Figure 15. All-Welded Stainless Steel Systems for ClF_5 and N_2O_4 Application

PART	MATERIAL
Tank	6061-T6 Al
Transition Joint	347 SST/6061-T6 Al
Pressure Switch	347 SST
Explosive Valve	6061-T6 Al
Burst Disk (100 psig)	6061-T6 Al
Burst Disk (120 psig)	6061-T6 Al
Hoke Hand Valve	347 SST
1/2 in. by 0.035 in. Tubing	6061-T6 Al
1/2 in. by 0.035 in. Cross	6061-T6 Al
1/2 in. by 0.035 in. Tee	6061-T6 Al
1/2 in. by 0.065 in. Tubing	6061-T6 Al

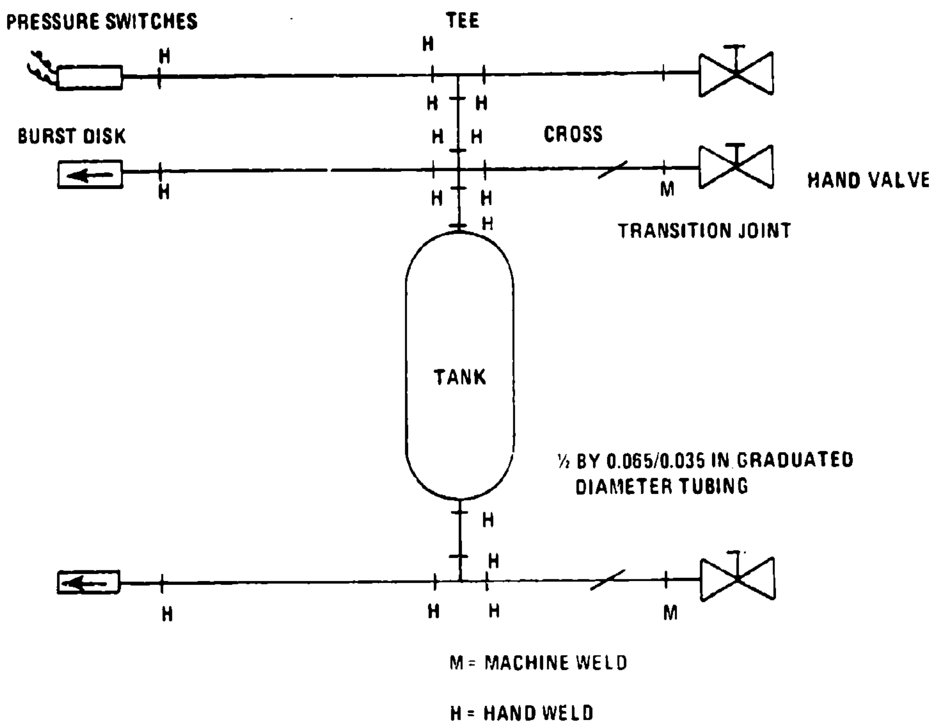

Figure 16. All-Welded Aluminum Systems for ClF_5 and N_2O_4 Application

PART	MATERIAL
Tank	347 SST
Hoke Hand Valve	347 SST
Burst Disk	6061-T6 Al
Transition Joint	347 SST/6061-T6 Al
AFRPL (Connector Elbow (MS27866--8)	347 SST
Bobbin Seal (Unplated)	304-L SST
Plain Flange (MS27853-08)	CRES AMS5646
0.035 Plain Flange (MS27853-08)	CRES AMS5646
Nut (MS27852-08)	A-286
AFRPL Connector Tee (MS27863-08)	AMS4127 Al Alloy
0.065 in. Plain Flange (MS27853-08)	CRES AMS5646
Plain Flange (MS27858-08)	AMS4127 Al Alloy
Bobbin Seal (MS27860-08)	AMS4127 Al Alloy
0.035 in. AFRPL Connector Union (MS27851-08)	

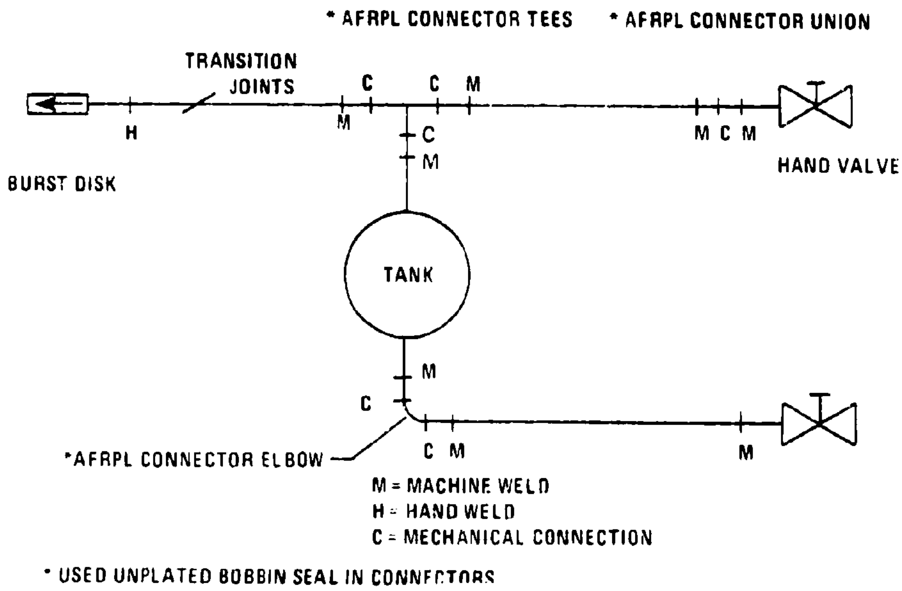

Figure 17. Separable Connector Stainless Steel System for N_2O_4 Application

PART	MATERIAL
Tank	347 SST
Hoke Hand Valve	347 SST
Burst Disk	6061-T6 Al
Transition Joint	347 SST/6061-T6 Al
AFRPL Connector Elbow (MS27866-08)	347 SST
Bobbin Seal (Ni Plated - MS27855-08)	304-L
Plain Flange (MS27853-08)	CRES AMS5646
0.035 Plain Flange (MS27853-08)	CRES AMS5646
Nut (MS27852-08)	A-286
AFRPL Connector Tee (MS27864-08)	AMS4127 Al
0.065 in. Plain Flange (MS27853-08)	CRES AMS5646
Plain Flange (MS27858-08)	AMS4117 Al Alloy
Bobbin Seal (MS27860-08)	AMS4127 Al Alloy
0.035 in. AFRPL Connector Union (MS27851-08)	

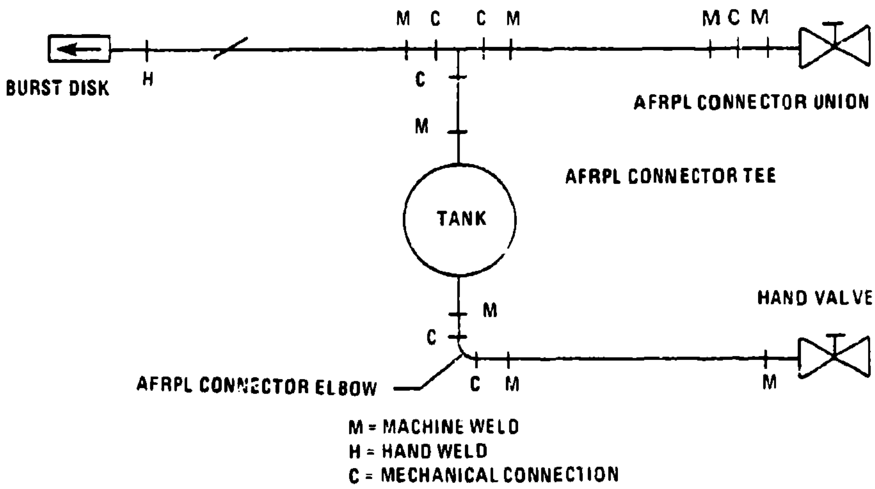

Figure 18. Separable Connector Stainless Steel System for ClF_5 Application

PART	MATERIAL
Tank	2219 Al
Hoke Hand Valve	347 SST
Burst Disk	6061-T6 Al
Transition Joint	347 SST/6061-T6 Al
AFRPL Connector Elbow (MS27862-08)	AMS4127 Al Alloy
Bobbin Seal (MS27860-08)	AMS4127 Al Alloy
Nut (MS27857-08)	AMS4117 Al Alloy
AFRPL Connector Tee (MS27863-08)	AMS4127 Al Alloy
0.065 in. Plain Flange (MS27858-08)	AMS4117 Al Alloy
0.035 in. Plain Flange (MS27858-08)	AMS4117 Al Alloy
0.035 in. AFRPL Connector Union (MS27856-08)	

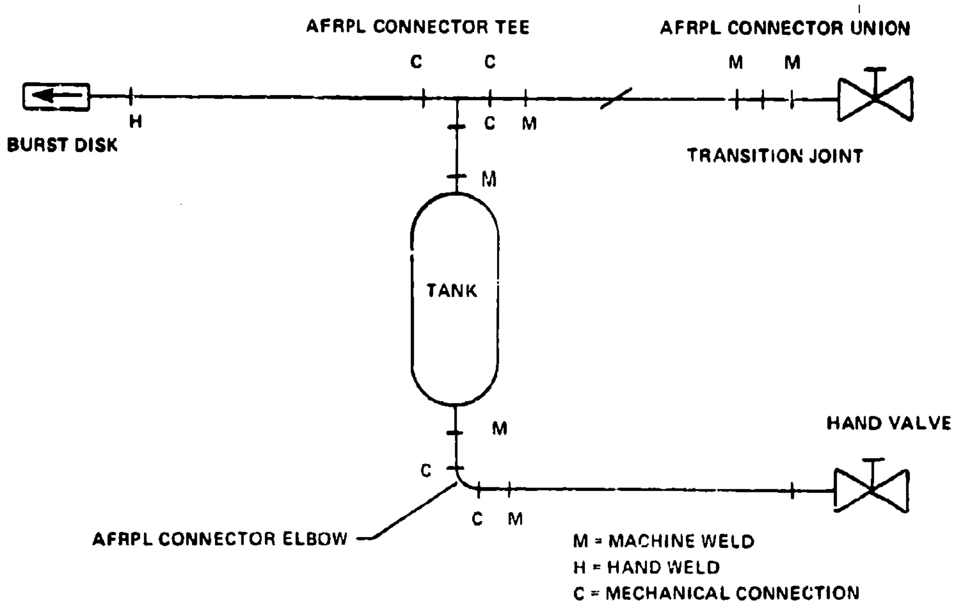

Figure 19. Separable Connector Aluminum Systems for ClF_5 and N_2O_4 Application

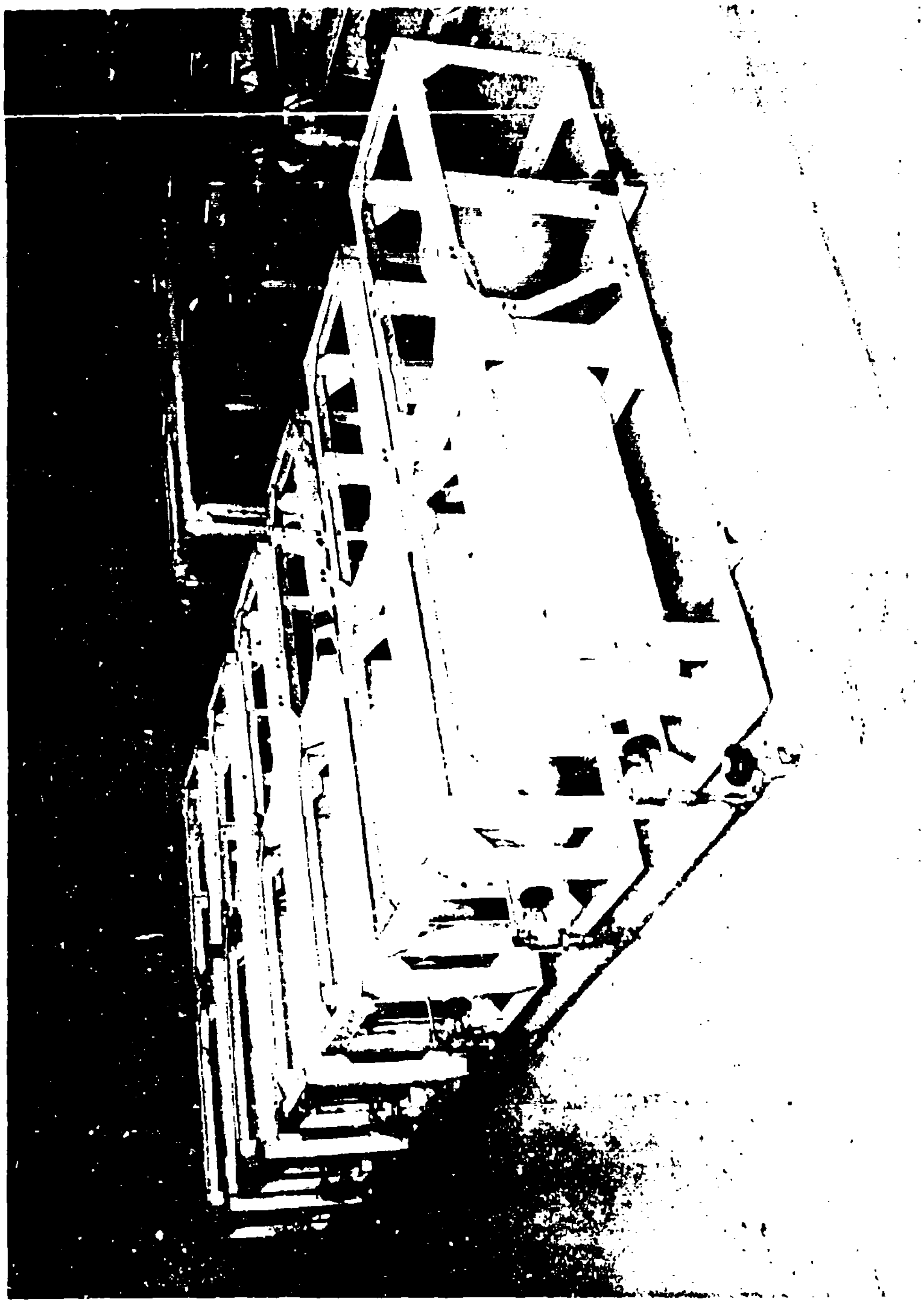

Figure 20. Prepackaged Feed Systems

TABLE I. GROUP I: SUMMARY OF RESULTS

Propellant	Tank	Quantity	Tank Material	Test Initiated	Test Terminated	Days in Test
N_2O_4 *	3- by 6-inch	4	2014-T6	9-7-66	9-12-66	5
N_2O_4 *	3-by 6- inch	1	2014-T6	1-3-67	1-5-67	2
N_2O_4 *	3- by 6-inch	23	2014-T6	1-3-67	13-5-71	1522
N_2O_4 *	Alcoa 1 quart	6	2014-T6	12-5-66	3-5-71	1548
N_2O_4 *	Alcoa 1 quart	3	6016-T6	12-5-66	3-5-71	1548
N_2O_4 *	Alcoa 1 quart	2	2219-T6	12-5-66	3-5-71	1548
N_2O_4 *	Alcoa 1 quart	1	7007-T6	12-5-66	3-5-71	1548
N_2O_4 *	Alcoa 1 quart	2	2021-T6	12-5-66	3-5-71	1548
N_2O_4 *	Alcoa 1 quart	2	5456-T6	12-5-66	3-5-71	1548
N_2O_4 *	Arde 1 pint Cryo Form	5	AISI 301 aged	6-21-67		In test
N_2O_4 *	Arde 1 pint Cryo Form	5	AISI 301 unaged	6-21-67		In test
ClF_5	Alcoa 1 quart	1	6061-T6	9-7-66	7-16-68	973
ClF_5	Alcoa 1 quart	1	6061-T6	9-7-66	5-14-69	616
ClF_5	Alcoa 1 quart	1	6061-T6	9-7-66	1-17-69	524
ClF_5	Alcoa 1 quart	3	6061-T6	4-7-66		In test
ClF_5	Alcoa 1 quart	8	2014-T6	9-7-66		In test
ClF_5	Alcoa 1 quart	4	2014-T6	9-7-66	2-27-70	655
ClF_5	Alcoa 1 quart	1	2014-T6	9-7-66	5-22-67	57

* MIL-P-26539 Specification N_2O_4

TABLE I. GROUP I: SUMMARY OF RESULTS (Continued)

Propellant	Tank	Quantity	Tank Material	Test Initiated	Test Terminated	Days in Test
ClF_5	Alcoa 1 quart	4	2219-T6	4-7-66		In test
ClF_5	Alcoa 1 quart	4	2021-T6	9-7-66		In test
ClF_5	Alcoa 1 quart	3	3003-T6	9-7-66		In test
ClF_5	Alcoa 1 quart	3	5456-T6	9-7-66		In test
ClF_5	Alcoa 1 quart	1	5456-T6	9-7-66	2-27-70	[illegible]
ClF_5	Alcoa 1 quart	1	7007-T6	9-7-66		In test
ClF_5	Alcoa 1 quart	1	7007-T6	9-7-66	8-11-69	[illegible]
ClF_5	Arde 1 pint Cryo Form	1	AISI 301 aged	8-25-67	9-14-69	[illegible]
ClF_5	Arde 1 pint Cryo Form	4	AISI 301 aged	8-23-67		In test
ClF_5	Arde 1 pint Cryo Form	[illegible]	AISI 301 unaged	8-23-67		
N_2H_4	Arde 1 pint Cryo Form	15	AISI 301 aged	7-1-69	5-4-70	No excessive pressure rise
N_2H_4	Arde 1 pint	14	AISI 301 unaged	7-1-69	5-4-70	No excessive pressure rise

TABLE II. GROUP II: SUMMARY OF RESULTS

Propellant	Tank	Quantity	Tank Material	Test Initiated	Test Terminated	Days in Test
N_2O_4 *	Martin	1	2014-T6	1-3-67	1-25-67	22
N_2O_4 *	Martin	1	2014-T6	1-3-67		In test
N_2O_4 *	GD/C	2	2014-T6	1-3-67		In test
N_2O_4 *	Martin	1	6Al-4V	1-3-67	1-13-67	10
N_2O_4 *	Martin	1	6Al-4V	1-3-67	2-7-67	34
N_2O_4 *	Martin	1	6Al-4V	1-3-67	2-8-67	35
N_2O_4 *	GD/C	1	5Al-2.5Sn	1-3-67	1-17-67	14
N_2O_4 *	GD/C	1	5Al-2.5Sn	1-3-67	1-19-67	16
N_2O_4 *	GD/C	2	6061-T6	1-3-67		In test
N_2O_4 *	Martin	1	7039-T6	1-3-67	7-11-68	555
N_2O_4 *	Martin	1	7039-T6	1-3-67		In test
N_2O_4 *	GD/C	1	AM350	1-3-67	10-24-67	294
N_2O_4 *	Martin	1	17-7PH	1-3-67	10-25-67	295
N_2O_4 *	GD/C	3	2021-T6	8-4-69		In test
N_2O_4 **	GD/C	3	6Al-4V	8-4-69		In test
ClF_5	Martin	1	2014-T6	1-3-67	3-6-67	58

* MIL-P-26539 Specification N_2O_4

** MSC-PPC-2A Specification N_2O_4

TABLE II. GROUP II: SUMMARY OF RESULTS (Continued)

Propellant	Tank	Quantity	Tank Material	Test Initiated	Test Terminated	Days in Test
ClF_5	GD/C	1	2014-T6	1-3-67		In test
ClF_5	GD/C	1	AM1350	1-3-67	10-24-67	294
ClF_5	GD/C	1	AM1350	1-3-67	10-25-67	295
ClF_5	GD/C	1	6061-T6	1-3-67		In test
ClF_5	Martin	1	7039-T6	11-28-68	12-29-70	460
ClF_5	Martin	1	17-7PH	1-3-67	3-9-67	64
ClF_5	Martin	1	17-7PH	1-3-67	10-23-67	293
N_2O_4**	Bullpup	3	2014-T6	6-10-68		In test
N_2O_4**	ULPR	2	2219-T81	5-21-68	2-25-71	1011
N_2H_4	Martin	5	17-7PH	5-22-69	5-4-70	Some pressure rise
N_2H_4	Martin	5	AM350	5-22-69	5-4-70	
N_2H_4	Martin	5	A286	5-22-69	5-4-70	
N_2H_4	Martin	5	2021-T6	5-22-69	5-4-70	No excessive pressure rise
N_2H_4	Martin	5	2014-T6	5-22-69	5-4-70	
N_2H_4	Martin	5	2219-T8	5-22-69	5-4-70	
N_2H_4	Martin	5	6Al-4V	5-22-69	5-4-70	
N_2H_4	GD/C	3	6Al-4V	6-4-69	5-4-70	No excessive pressure rise
N_2H_4	GD/C	3	2021-T6	6-4-69	5-4-70	

TABLE IIIA. GROUP III: SUMMARY OF RESULTS

Propellant	Number	Pressure System	Expulsion System	Test Initiated	Test Terminated	Days in Test
MHF-5	2	LGG	RD	6-9-67		In test
MHF-5	2	SGG	RD	6-9-67		In test
MHF-5	1	H	RD	6-9-67		In test
MHF-5	2	LGG	ST	6-9-67		In test
MHF-5	2	SGG	ST	6-9-67		In test
MHF-5	2	H	ST	6-9-67		In test
N_2O_4	2	LGG	RD	5-22-67	3-24-71	1402
N_2O_4	2	SGG	RD	5-22-67		In test
N_2O_4	2	H	ST	5-22-67	12-4-70	1292
ClF_5	1	SGG	RD	5-20-67	10-7-67	20
ClF_5	1	H	ST	8-4-67	10-23-67	80
ClF_5	1	H	ST	8-4-67	8-18-70	1110
N_2O_4 *	1	LGG	RD	5-10-67	12-20-70	941
N_2O_4 *	1	LGG	RD	5-10-67	12-4-70	1304
N_2O_4 *	1	SGG	RD	5-10-67		In test
N_2O_4 *	1	H	RD	5-10-67	2-3-71	1367

* MSC-PPC-2A Specification N_2O_4

NOTE: LGG = liquid gas generator; SGG = solid gas generator; H= stored helium; ST = surface tension.
RD = rolling diaphragm.

TABLE IIIB. GROUP III: SUMMARY OF RESULTS

Propellant	Tank	Quantity	Expulsion Device	Material	Test Initiated	Test Terminated	Days in Test
N_2O_4 *	Ardé	2	Ring-stiffened diaphragm	AISI 301 Cryo Form	7-3-69		In test
N_2O_4 *	Ardé	2	Ring-stiffened diaphragm (conospheroid)	AISI 301 Cryo From	12-23-70		In test
N_2O_4 *	Thiokol	2	Rolling diaphragm	Shell-200 Maraging Diaphragm-1100-0	1-5-71		In test
N_2O_4 *	Thiokol	1	Rolling diaphragm	Shell-200 Maraging Diaphragm-1100-0	1-5-71	3-15-71	[illegible]
ClF_5	Ardé	2	Ring-stiffened diaphragm	AISI 301 Cryo Form	7-3-69		[illegible]

* MSC-PPC-2A Specification N_2O_4

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4. H. M. White, 2nd Lt, USAF, Long-Term Storability of Propellant Tankage, AFRPL-TR-71-20, Air Force Rocket Propulsion Laboratory, March 1971.

AUTHOR'S BIOGRAPHY

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Lt. White was born in Philadelphia, Pennsylvania, in 1947. He attended Lehigh University in Bethlehem, where he graduated in 1969 with honors in Chemical Engineering.

Prior to entering the Air Force he was employed as a process design engineer by Betz Laboratories in Trevose, Pennsylvania.

Lt. White is currently serving as a project engineer in the Liquid Rocket Division at the Air Force Rocket Propulsion Laboratory. He is responsible for the areas of propellant/material compatibility, liquid rocket tankage and pressurization.

APPENDIX I

RELIABILITY AND QUALITY CONTROL

REPORT QC & R 6-030

10025 SHOEMAKER AVE
SANTA FE SPRINGS CA 90670

PYRONETICS, INC.
SUBSIDIARY OF COSMODYNE
10025 Shoemaker Avenue, Santa Fe Springs, California 90670

REPORT NUMBER QC & R 6-030

FINAL TEST REPORT

FOR

PYRONETICS MODEL 1242 EXPLOSIVE ACTUATED VALVE AND PYRONETICS MODEL 1243 EXPLOSIVE ACTUATED VALVE

PREPARED FOR

EDWARDS AIR FORCE BASE

PREPARED BY

E. Avalos

EDWARD AVALOS
Reliability Engineer

APPROVED BY

Brian H. Lundquist

BRIAN H. LUNDQUIST
Reliability Manager

Number of Pages: 25

BOOK COPY NO. 02
ISSUED TO Edwards AFB

26 February 1971

10025 SHOEMAKER AVE
SANTA FE SPRINGS CA 90670

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SANTA FE SPRINGS, CA 90670

INTRODUCTION AND SUMMARY

The Test Program, described herein, for the Model 1242 and 1243 Explosive Actuated, 1/2 inch Line Size, Normally Closed Valves is the final series of tests which the valves were subjected to, after an Edwards Air Force Base Test Program to test the valves under long term storage conditions while the units were pressurized with N_2O_4 or CLF1.5.

The objective of the Test Program was to determine if the valves were still operable after the long term storage. Although the Edwards Air Force Base Test Program was initially intended to last five (5) years, the test program was cut short in late 1969 due to system failure. (Non-valve related.)

A total of fourteen (14) Model 1242 Valves and fourteen (14) Model 1243 Valves were originally sent to Edwards for the test program in November 1966. Of this quantity a total of ten (10) Model 1242 Valves and eleven (11) Model 1243 Valves were returned.

The units were subjected to electrical tests and subsequent actuation testing. There were no test failures.

Capability of the units to properly function after being subjected to the test program has successfully been demonstrated.

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VALVE DESIGN PARAMETERS

MODEL 1242

Primary Material	**347 Stainless Steel**
Pressure	
Operating	0-3000 PSIG
Proof	4500 PSIG
Burst	6000 PSIG
Estimated Weight	0.80 lbs.
Cartridge Data	
Bridgewire Resistance	1.1 ± 0.2 ohms
No Fire Current	1 amp or 1 watt
All Fire Current	4.0 amps
Mating Connector	Bendix PT 06P-12-8S
Firing Circuits	Pin B •∿∿∿• C
	Pin E •∿∿∿• F
Flow Rate	4 GPM of N_2O_4 at 1.0 PSID

MODEL 1243

Primary Material	6061-T6 Al Alloy
Pressure	
Operating	0-3000 PSIG
Proof	4500 PSIG
Burst	6000 PSIG
Estimated Weight	0.45 lbs.
Cartridge Data	
Bridgewire Resistance	1.1 ± 0.2 ohms
No Fire Current	1 amp or 1 watt
All Fire Current	4.0 amps
Mating Connector	Bendix PT 06P-12-8S
Firing Circuits	Pin B •∿∿∿• C
	Pin E •∿∿∿• F
Flow Rate	4 GPM of N_2O_4 at 1.0 PSID

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1.0 PURPOSE OF TEST

The objective of the test program was to determine if the Model 1242 and 1243 Valves would successfully function after being subjected to long term storage testing while pressurized with either N_2O_4 or $CLFL_5$.

1.1 Description of the Model 1243 Valve

The Model 1243 Valve is a normally closed, cartridge actuated valve of 1/2 inch line size. Upon actuation, the valve allows the pressurization fluid to flow through the valve and perform its function. Valve actuation is accomplished by explosive energy generated when the cartridge is ignited by application of electrical power. The valve body and nipples are made up of 6061-T6 aluminum alloy of welded construction.

The Cartridge, Model 3545, is an electrically initiated pyrotechnic device. It is comprised of a cylindrical housing, an explosive main charge (lead azide), redundant initiating circuits which ignite two (2) initiating charges (zirconium potassium perchlorate), an electrical connector which mates with a Bendix PT06P-12-8S connector and a threaded fitting for mating with the valve. The cartridge is hermetically sealed for long term storageability. The Model 3545 Cartridge is the identical cartridge which Pyronetics utilized on many successful space programs, among which the most significant was the Gemini program.

The valve is mounted in a system by welding the nipple protrusions to mating lines. In the normally closed position, internal leakage is prevented by integral nipples. The nipples are sheared and retained within the valve body by a ram propelled by the actuation of the cartridge. A flow passage is opened through the valve by the shearing of the nipples. Valve configuration is presented in Figure 1.

1.2 Description of the Model 1242 Valve

The Model 1242 Valve is virtually identical to the Model 1243 Valve, except that the valve body and nipple material is 347 stainless steel instead of 6061-T6 aluminum. Both valves employ the same cartridge (Model 3545).

1.3 Disposition of Test Samples

The test samples have been retained in Pyronetics bonded stores.

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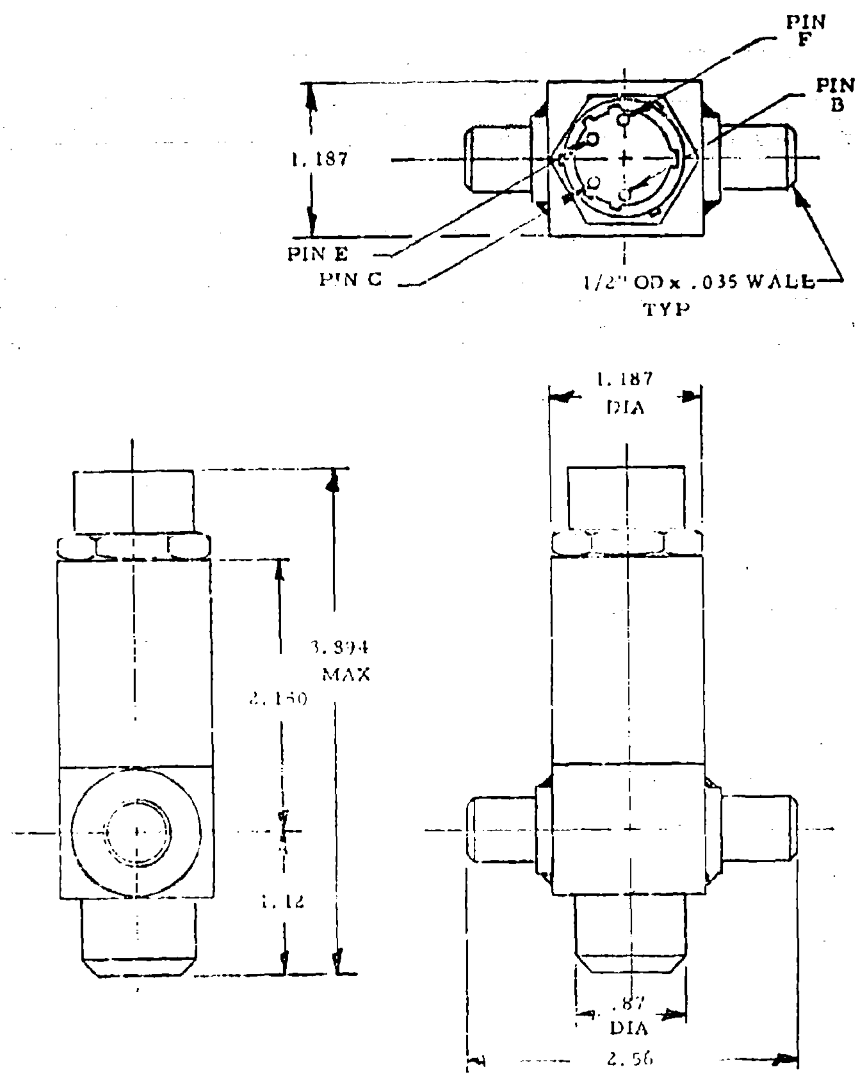

FIGURE 1 - MODEL 1242 and MODEL 1243 VALVE CONFIGURATION

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2.0 REFERENCE DOCUMENTS

The following documents comprise the criteria for this test program to the extent specified herein:

2.1 Military

MIL-C-45662A	Calibration System Requirements 09 February 1962

2.2 Pyronetics

Drawing 1242	Valve N.C. Explosive Actuated, 1/2" Openings
Drawing 1243	Valve N.C. Explosive Actuated, 1/2" Openings
TS 1242 TS 1243	Acceptance Test Specification Model 1242 and 1243 19 September 1966

3.0 TEST DESCRIPTION AND RESULTS

Ten (10) Model 1242 Valves and eleven (11) Model 1243 Valves were subjected to testing as described herein. Because of hardware damage (caused by installation and removal from the Edwards test system) proof pressure testing and helium leakage testing which would have been desirable for this test program, could not be performed.

The following valve serial numbers were returned from Edwards AFB for the test program:

Model 1242

Valve	Cartridge	Valve	Cartridge
S/N 626-003	640	S/N 626-014	680
626-004	623	626-015	653
626-005	625	* 626-UNK	636
626-010	631	* 626-UNK	655
626-011	610	* 626-UNK	687

Model 1243

Valve	Cartridge	Valve	Cartridge
S/N 627-002	734	S/N 627-008	743
627-003	702	627-010	714
627-004	704	627-011	700
627-005	738	627-012	746
627-006	684	627-014	709
627-007	703		

* Serial numbers eradicated by exposure to test fluid media at Edwards AFB.

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3.0 TEST DESCRIPTION AND RESULTS (CONTINUED)

The tests performed within this test program were (1) Visual Examination, (2) Bridgewire Resistance Testing, (3) Insulation Resistance Testing, (4) Actuation Testing (with measurement of bridgewire burn-out time), and (5) Visual determination of proper actuation.

Prior to the test units being returned to Pyronetics they were exposed to either N_2O_4 or $CLFL_5$ as is described in paragraph 3.1.

The test descriptions and results are given in the following paragraphs.

3.1 Long Term Storage -- Propellant Exposure

Data furnished by Edwards AFP indicates that the valves which Pyronetics supplied were welded into a test system and exposed to pressurization with either N_2O_4 or $CLFL_5$. The units remained in the system for a maximum period of sixteen (16) months prior to a system malfunction which cut the storage test short of its anticipated five (5) year term. The information, further relates, that termination of the test was not caused by a valve failure. Test unit serial numbers, propellant exposure media and start and stop time of the tests are given below.

Model 1243
(Aluminum Valve)

Serial Number	Test Fluid	Exposure Start	Exposure Stop
S/N 626-003	$CLFL_5$	11/68	9/69
626-004	$CLFL_5$	11/68	
626-005	N_2O_4	6/68	
626-010	N_2O_4	6/68	
626-011	$CLFL_5$	11/68	
626-014	$CLFL_5$	11/68	
626-015	$CLFL_5$	11/68	
626-UNK	N_2O_4	6/68	
626-UNK	N_2O_4	6/68	
626-UNK	N_2O_4	6/68	9/69

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3.1 Long Term Storage -- Propellant Exposure (continued)

Model 1242
(Stainless Steel Valve)

Serial Number	Test Fluid	Exposure Start	Exposure Stop
627-002	N_2O_4	6/68	9/69
627-003	N_2O_4	6/68	9/69
627-004	N_2O_4	6/68	9/69
627-005	N_2O_4	6/68	9/69
627-006	$CLFL_5$	9/68	9/69
627-007	N_2O_4	6/68	9/69
627-008	$CLFL_5$	11/68	8/69
627-010	$CLFL_5$	11/68	9/69
627-011	$CLFL_5$	11/68	9/69
627-012	$CLFL_5$	11/68	9/69
627-014	$CLFL_5$	11/68	9/69

As was reported above, no test failures were experienced.

3.2 Visual Examination

All units were subjected to a visual examination upon receipt at Pyronetics. Results are given in Table I for the Model 1242 Valve and Table II for the Model 1243 Valve. Although most units had some corrosion, discoloration, rust (stainless steel valve only), and some white residue (unknown substance), all valves were deemed to be structurally sound and capable of retention of test fluids. Unfortunately, the nipple weldment stubs were in such a condition that leakage testing could not be performed without extensive valve modification.

The heat discoloration found in the nipples was attributed to system welding operations. Likewise the minor rust found was determined to have been caused by welding and removal operations which were probably not passivated.

The white residue is of an unknown substance. The etching which was found on the exterior surfaces of the valve bodies is attributed to direct exposure to atmosphere and propellants when the system malfunction occurred.

In summation, no visual damage could be determined as having been caused by the long term storage tests prior to the system malfunction.

[illegible]netics Inc.
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TABLE 1 - MODEL 1242 VISUAL EXAMINATION RESULTS

SER. NO.	VISUAL INSPECTION ELECTRICAL CONNECTOR		VISUAL INSPECTION INLET & OUTLET		VISUAL INSPECTION BODY
	INTERNAL	EXTERNAL	LEFT	RIGHT	
626-003	Clean, O-RING INTACT	MINOR DISCOLORATION	CLEAN	MINOR RUST OVER [illegible] SURF	1) MINOR ETCHING - IDENTIFICATION ALMOST UNREADABLE 2.) AREA UNDER TAPE (HOLDING ON SHORTING PLUG) IS CLEAN.
626-004	CLEAN, O-RING INTACT	MINOR DISCOLORATION	CLEAN	FAINT RUST OVER [illegible] SURFACE	SAME AS S/N 626-003
626-005	CLEAN, O-RING INTACT	MINOR DISCOLORATION	CLEAN EXCEPT FOR WELD HEAT DISCOLORATION	MINOR SPOTTY RUST	SAME AS S/N 626-003
626-010	CLEAN, O-RING INTACT	MINOR DISCOLORATION	MEDIUM SPOTTY RUST	CLEAN	1) SAME AS S/N 626-003 2) SAME AS S/N 626-003 3) EXTERNAL RUST ON NIPPLE STUB
626-011	CLEAN, O-RING INTACT	CLEAN	WELD HEAT DISCOLORATION	WELD HEAT DISCOLORATION	1) CLEAN EXCEPT FOR SPOTTY RUST ON NIPPLE STUBS.
626-014	CLEAN, O-RING INTACT	MINOR DISCOLORATION	CLEAN	1) WELD HEAT DISCOLORATION 2) SOME RUST (SPOTTY)	SAME AS S/N 626-003
626-015	Clean, O-RING INTACT	CLEAN	1) WELD HEAT DISCOLORATION 2) MINOR RUST	1.) WELD HEAT DISCOLORATION 2) MINOR RUST	SAME AS S/N 626-011
CART S/N 626	CLEAN, O-RING INTACT	MINOR DISCOLORATION	HEAT DISCOLORATION	1) WELD HEAT DISCOLORATION 2) MINOR RUST	1.) SAME AS S/N 626-003 2.) SAME AS S/N 626-003 3) SOME IDENTIFICATION COMPLETE [illegible] (i.e. S/N)
CART S/N 655	Clean, O-RING INTACT	MINOR DISCOLORATION	HEAT DISCOLORATION	1) WELD HEAT DISCOLORATION 2) MINOR RUST	SAME AS S/N 636 (CARTRIDGE)
CART S/N 687	Clean, O-RING INTACT	MINOR DISCOLORATION	HEAT DISCOLORATION	1) SPOTTY RUST 2) WHITE RESIDUE 3) HEAT DISCOLORATION	SAME AS S/N 636 (CARTRIDGE)

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TABLE 2. - MODEL 1243 VISUAL EXAMINATION RESULTS

Serial No.	Visual Inspection Electrical Connector: Internal	Visual Inspection Electrical Connector: External	Visual Inspection Inlet & Outlet: Left	Visual Inspection Inlet & Outlet: Right	Visual Inspection Body
627-002	Clean, O-ring intact	Minor discoloration	Heavy white residue (C.R.T.)	Medium white residue (C.R.T.(TYP))	Part of electroetching removed from body
627-003	Clean O-ring intact	Some indications of burning	Minor white & black residue	Minor white residue	1) Most of body etched 2) Area under tape (holding shorting plug to body) is clean
627-004	Cruddy, O-ring reduced in diameter (O.D.) some rust	Minor discoloration	Minor white & black residue	Faint white residue	Same as S/N 627-003
627-005	Clean, O-ring intact	Minor discoloration	Heavy white residue	Medium white residue	Same as S/N 627-003
627-006	Clean, O-ring intact	Minor discoloration	Clean	Faint white residue	Same as S/N 627-003
627-007	Clean, O-ring intact	Minor discoloration	Heavy white residue	Minor white residue	1) Same as S/N 627-003 2) Same as S/N 627-003 3) Some indications of burning on tape.
627-008	Clean, O-ring intact	Clean	Faint white residue	Clean	Clean
627-00	Area discolored 100% of surface	Minor discoloration	Faint white residue	Faint white residue	Same as S/N 627-003
627-011	Clean Electrical conn. damaged - won't accept mate. conn.	Clean	Clean	Clean	Same as S/N 627-003
627-012	Clean	Indications of burning	Clean	Clean	Minor discoloration mostly clean
627-014	Clean	Minor discoloration	Clean	Clean	Same as S/N 627-003

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3.3 Bridgewire Resistance Testing

Test Requirements:

The resistance of each bridgewire circuit shall be measured at laboratory ambient conditions using a 10 milliampere maximum test current. The resistance of each circuit shall be 1.1 ± 0.2 ohms after the unit has been stabilized at laboratory ambient temperature for one hour minimum.

Test Description

The units were assembled in the test setup shown in Figure 2 and the electrical continuity of each bridgewire circuit was checked at laboratory ambient conditions with an Allnco Ohmmeter, having a test current of less than 10 milliamperes.

Test Results

Each cartridge checked passed the bridgewire resistance requirement of 1.1 ± 0.2 ohms. The minimum and maximum resistance recorded were 0.98 ohm to 1.22 ohms. These results confirm that no evidence of degradation was sustained as a result of the Edwards test and none of the units was rendered inoperative. Reference test results in Appendix 1.

3.4 Insulation Resistance Testing

Although no insulation resistance test requirements were imposed by Edwards AFB, the Model 3545 Cartridge is designed to have a minimum insulation resistance of 500 megohms when tested at 500 VDC on a megohmmeter.

The units were assembled in the test setup shown in Figure 3. For information, all twenty-one (21) units were checked and the resistance recorded. All cartridges checked passed the 500 megohm requirement. The minimum and maximum resistances recorded were 4000 megohms and 1,100 K megohms. Reference test results in Appendix 1.

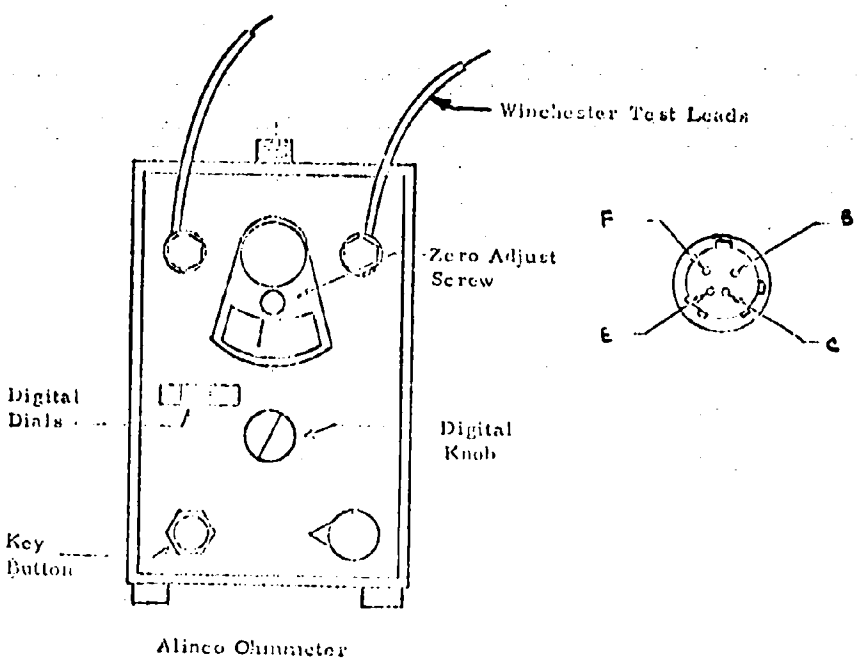

FIGURE 2 -- BRIDGEWIRE RESISTANCE TEST SETUP

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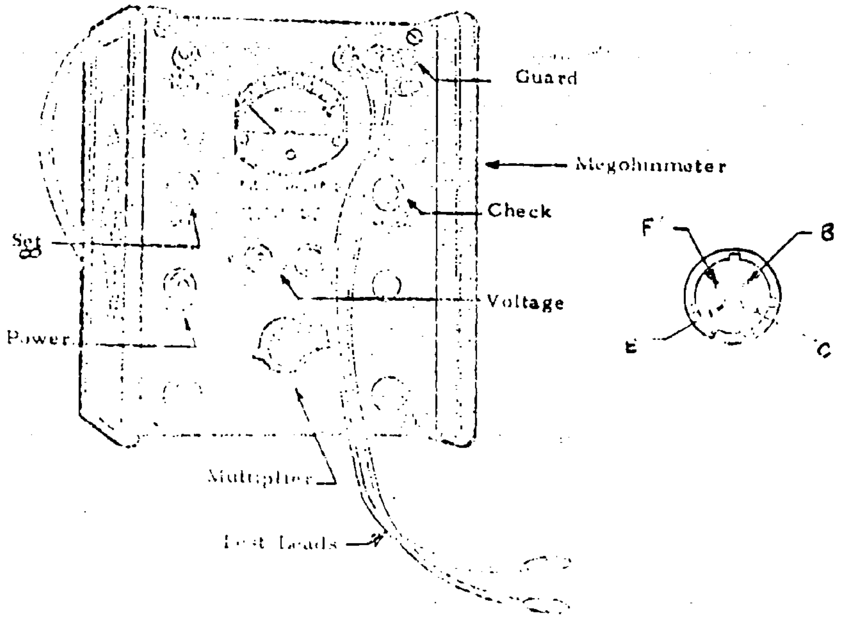

FIGURE 5 -- INSULATION RESISTANCE TEST SETUP

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3.5 Actuation Testing

Test Requirement

Each valve shall be actuated by applying 4.0 ± 0.1 amperes to one of the cartridge bridgewires. No response time shall be measured since no valve pressurizing can be effected. The bridgewire burnout time shall not exceed 10 milliseconds.

Test Description

The twenty-one (21) units were assembled in the test setup as shown in Figure 4. Each valve was actuated by applying 4.0 ± 0.1 amperes to one of the cartridge bridgewires. Upon actuation, the normally closed nipples were sheared and the valve was opened. Bridgewire burnout time was recorded.

Test Results

All twenty-one (21) valves actuated to the open mode with no restriction in the flow passage and without any detectable evidence of damage to the structural integrity of the valves. Bridgewire burnout occurred between 2.40 and 3.29 milliseconds. The actuation test was considered successful since the performance parameters and subsequent visual examination proved satisfactory. Reference actual test results in Appendix 1 and Appendix 2.

3.6 Post Actuation Visual Examination

All twenty-one (21) units were visually examined after actuation testing for proper actuation. All the units exhibited no restriction in the flow passage indicating proper actuation.

4.0 CONCLUSIONS

Accomplishment of the test program specified herein, signified acceptance of the Model 1242 and Model 1243 Valves. The units fulfilled all the test requirements which were capable of being performed.

Examination of the data indicated excellent repeatability of all the functional characteristics, i.e., ignition time and proper actuation.

Therefore based on the data obtained during the test program, it can only be concluded that the valves suffered no degradation due to the Edwards AFB test program of long term storage/propellant exposure.

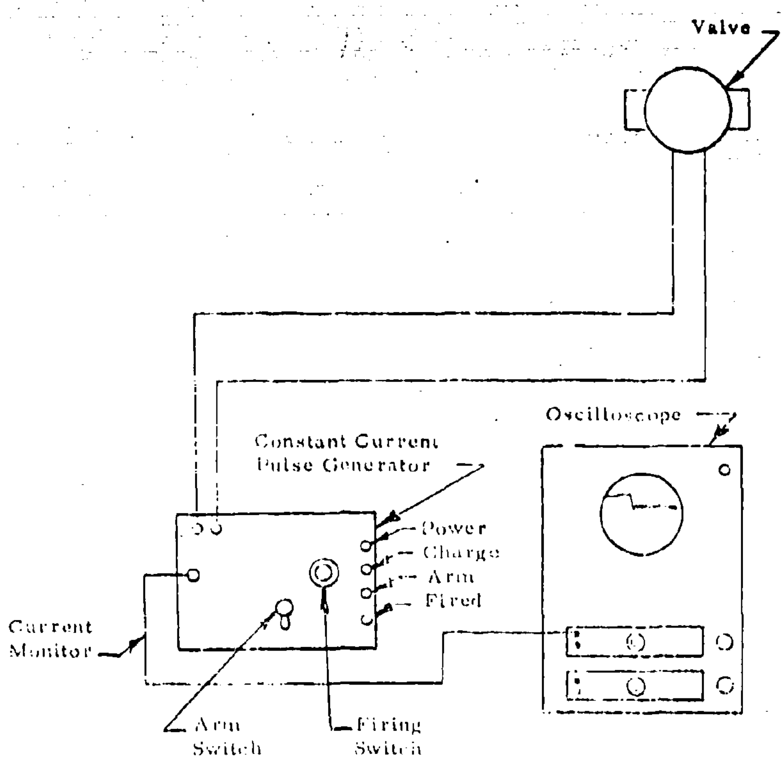

FIGURE 1 -- ACTUATION TEST SETUP

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5.0 TEST EQUIPMENT

The test equipment and apparatus employed in the performance of the various tests described herein, are listed below. All equipment was checked for reliable performance prior to initiation of specific tests. Accuracy and capability is as specified and all calibrations meet the requirements of MIL-C-45662A and are traceable to the National Bureau of Standards.

5.1 Test Equipment - Bridgewire Resistance Test

Instrument	Ignition Circuit Tester
Manufacturer	Alinco
Model No.	1015-AF, S/N-501
Range	0 - 10, 0 - 20 ohms
Accuracy	± 0.02 ohms
Calib. Frequency	90 days
Calib. Due	17 March 1971

5.2 Test Equipment - Insulation Resistance Test

Instrument	Megohmmeter
Manufacturer	General Radio Co.
Model No.	1862 B
Range	0.5 - 2 x 10^6 megohms
Accuracy	± 5%
Calib. Frequency	Annual
Calib. Due	29 December 1971

5.3 Test Equipment - Actuation Test

Instrument	Oscilloscope
Manufacturer	Tektronix
Model No.	502, S/N 009513
Range	100 v to 20 v/cm
Accuracy	± 3%
Calib. Frequency	90 days
Calib. Due	18 March 1971

Instrument	Constant Current Pulse Generator
Manufacturer	E & R Development Co.
Range	0 - 10 amps, 0 - 100 ms
Accuracy	± 0.5%
Calib. Frequency	6 months
Calib. Due	July 1971

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APPENDIX I

LABORATORY TEST REPORTS

Electronics Inc. 10025 SHOEMAKER AVE. SANTA FE SPRINGS, CALIF.

S/M

LABORATORY TEST REPORT

JOB NO X 626 MODEL 1242 Tests Conducted By EAE BHL

Instruments and Procedure Per: TS 1242 MODIFIED DATE 12-21-70

Witnessed By: — Source Insp. —

* ORIGINAL JOB NUMBER UNITS WERE SHIPPED TO EDWARDS ON

STAINLESS STEEL VALVE.

PARAGRAPH NO

P/N 1242 SERIAL NUMBER	*Ordnance Serial Number	Bridgewire Resistance Test [illegible]	[illegible]	Actuation Testing: Response [illegible] (in ms)	Firing Current (amps)	Bridgewire Burnout (in ms)	Circuit Fired On		Visual Exam of Flow Passage		
[illegible]-003	6??	1.15	1.13	[illegible] ms	4.[illegible]	2.5 ms	E-F		NO OBSTRUCTION		
[illegible]-004	625	1.09	1.10	[illegible]	4.[illegible]	2.4 ms	E-F		NO OBSTRUCTION		
[illegible]-005	[illegible]	1.11	1.13	[illegible]	5.[illegible]	2.0 ms	E-F		NO OBSTRUCTION		
[illegible]-010	[illegible]	1.1[illegible]	1.04	[illegible] ms	5.0	[illegible] ms	E-F		NO OBSTRUCTION		
[illegible]-011	6[illegible]	1.10	1.06	[illegible]	4.[illegible]	2.1 ms	E-F		NO OBSTRUCTION		
[illegible]-014	8[illegible]	1.1[illegible]	1.13	[illegible]	4.[illegible]	2.4 ms	E-F		NO OBSTRUCTION		
[illegible]-015	853	1.1[illegible]	1.12	5[illegible] ms	[illegible]	2.5 ms	E-F		NO OBSTRUCTION		
[illegible]	[illegible]	1.1[illegible]	[illegible]	5[illegible] ms	4.[illegible]	2.6 ms	E-F		NO OBSTRUCTION		
[illegible]	6[illegible]	[illegible]	1.06	[illegible]	[illegible]	2.6 ms	E-F		NO OBSTRUCTION		
[illegible]	687	1.10	1.13	[illegible]	4.0	2.6 ms	E-F		NO OBSTRUCTION		

*(1) DUE TO SEVERE ETCHING ON EXTERIOR OF [illegible], VALVE SERIAL NUMBERS WERE INDISTINGUISHABLE

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S/N

LABORATORY TEST REPORT

SANTA FE SPRINGS, CALIF.

JOB NO. [illegible] MODEL [illegible] Tests Conducted By [illegible]

Tests were run Procedure Per. [illegible] MODIFIED A DATE [illegible]

Witnessed By ________ Source Insp. ________

[illegible] NUMBER UNITS WERE SHIPPED TO [illegible] ON:

ALUMINUM VALVE

PARAGRAPH NO. [illegible]	[illegible]	[illegible]	[illegible]	[illegible] (OHMS)	ACTUATION TESTING [illegible] (AMPS)	[illegible] (MS)	CIRCUIT FIXED ON		VISUAL EXAM. OF FLOW PASSAGE		
[illegible] - 002	[illegible]	[illegible]	[illegible]	[illegible]	4.0	3.0 ms	B-C		NO OBSTRUCTION		
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	4.0	2.0 ms	B-C		NO OBSTRUCTION		
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	4.0	2.2 ms	B-C		NO OBSTRUCTION		
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	4.0	2.0 ms	B-C		NO OBSTRUCTION		
621 - 00[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	4.0	[illegible] ms	B-C		NO OBSTRUCTION		
621 - 00[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	4.0	[illegible] ms	B-C		NO OBSTRUCTION		
621 - 0[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	4.0	2.0 ms	B-C		NO OBSTRUCTION		
621 - 010	[illegible]	[illegible]	[illegible]	[illegible]	4.0	[illegible] ms	B-C		NO OBSTRUCTION		
621 - 011	[illegible]	[illegible]	[illegible]	[illegible]	4.0	2.1 ms	B-C		NO OBSTRUCTION		
621 - 012	[illegible]	[illegible]	[illegible]	1,800 [illegible]	4.0	2.0 ms	B-C		NO OBSTRUCTION		
621 - 014	[illegible]	[illegible]	[illegible]	[illegible]	4.0	2.8 ms	B-C		NO OBSTRUCTION		

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Page [illegible]

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APPENDIX 2

TEST DATA RECORDS

QC & R 6-030

Pyronetics Inc.

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Santa Fe Springs, Calif.

S/N
SHEET 1 OF 3

TEST DATA RECORD

TEST TITLE Actuation Test PART NAME Valve N.C., Explosive Actuated, 1/2 in. Openings
CUSTOMER P/N N/A PYRONETICS P/N 1242
TEST SPEC NO. N/A TEST NAME Actuation Test
TESTING PER (PARA) N/A TEST BY E. Avalos
APPROVED BY [illegible] DATE 12-21-70

NOT REPRODUCIBLE

QC & R 6-030

Pyronetics inc.
10025 Shoemaker Ave.
Santa Fe Springs, Calif.

S/N
SHEET 2 OF 3

TEST DATA RECORD

TEST TITLE Actuation Test PART NAME Valve N.C., Explosive Actuated 1/2 in. Openings
CUSTOMER P/N N/A PYRONETICS P/N 1242
TEST SPEC NO. N/A TEST NAME Actuation Test
TESTING PER (PARA) N/A TEST BY E. Avalos
APPROVED BY B. Lundquist DATE 12-21-70

Pyronetics Inc.

10025 Shoemaker Ave., Santa Fe Springs, Calif.

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S/N

SHEET 3 OF 3

TEST DATA RECORD

TEST TITLE Actuation Test

PART NAME Valve N. C., Explosive Actuated, 1/2 in. Openings

CUSTOMER P/N N/A

PYRONETICS P/N 1242

TEST SPEC NO. N/A

TEST NAME Actuation Test

TESTING PER (PARA) N/A

TEST BY E. Ayalos

APPROVED BY B. Lundquist

DATE 12-21-70

NOT REPRODUCIBLE

QC & R 6-030

Pyronetics Inc.
10025 Shoemaker Ave.
Santa Fe Springs, Calif.

S/N
SHEET 1 OF 3

TEST DATA RECORD

TEST TITLE Actuation Test
PART NAME Valve N. C., Explosive Actuated, 1/2 in. Openings
CUSTOMER P/N N/A
PYRONETICS P/N 1243
TEST SPEC NO. N/A
TEST NAME Actuation Test
TESTING PER (PARA) N/A
TEST BY E. Avalos
APPROVED BY B. Lundquist
DATE 12-21-70

PYRONETICS S/N ... RESPONSE TIME

PYRONETICS S/N ... RESPONSE TIME

CALIBRATION

Pyronetics Inc.
10025 Shoemaker Ave.
Santa Fe Springs, Calif.

QC & R 6-030

S/N
SHEET 2 OF 3

TEST DATA RECORD

Valve N. C., Explosive

TEST TITLE Actuation Test PART NAME Actuated, 1/2 in. [illegible]

CUSTOMER P/N N/A PYRONETICS P/N 1243

TEST SPEC NO. N/A TEST NAME Actuation Test

TESTING PER (PARA) N/A TEST BY [illegible]

APPROVED BY B. Lundquist DATE 12-21-70

PYRONETICS S/N ... RESPONSE TIME ...

PYRONETICS S/N ... RESPONSE TIME ...

PYRONETICS S/N ... RESPONSE TIME ...

PYRONETICS S/N ... RESPONSE TIME ...

CALIBRATION

HORIZONTAL SCALE ... MILLISECONDS PER CENTIMETER

VERTICAL SCALE (UPPER) ... AMPS PER CENTIMETER

VERTICAL SCALE (LOWER) ... PSIG PER CENTIMETER

... PER CENTIMETER

Pyronetics inc.
10025 Shoemaker Ave.
Santa Fe Springs, Calif.

QC & R 6-030

S/N

SHEET 3 OF 3

TEST DATA RECORD

TEST TITLE Actuation Test

PART NAME Valve N. C., Explosive Actuated, 1/2 in. Openings

CUSTOMER P/N N/A

PYRONETICS P/N 1243

TEST SPEC NO. N/A

TEST NAME Actuation Test

TESTING PER (PARA) N/A

TEST BY E. Avalos

APPROVED BY B. Lundquist

DATE 12-21-70

IGNITION TIME

RESPONSE TIME

NOT REPRODUCIBLE

APPENDIX II

PROJECT 305805FRJ

PACKAGE SYSTEM STORABILITY REPORT

LABORATORY TEST REPORT	Report Nr. 104M	Date 4 Mar. 1971
Requesting Organization (Symbol and/or Name) LKIC	Name of Requestor Lt. H. White	Phone Number 32282

Sample, Test or Project

305805FRJ Package System Storability

Work Required

Determine Cause of Rupture of Regulator Casing

TEST DATA

I. MATERIAL: All exposed parts are of stainless steels, except the tag on the sealing wire which is an aluminum alloy.

II. BACKGROUND: The casing of the regulator (Fig.s 1,2,3) ruptured during storage testing of a N_2O_4/N_2H_4 system. The environment was 85°F and 85% relative humidity. The exterior of the regulator was coated with reddish-brown corrosion products. The N_2O_4/N_2H_4 system had not leaked.

III. CONCLUSIONS: Three undesirable factors contributed to rupture of the thin wall portion of the regulator casing: 1) a corrosion medium of chloride and fluoride ions in the warm, moist environment; 2) a helical, thread-like groove (Fig. 3) and 3) a zinc coating on the thin wall portion. The compressed heavy spring inside the regulator (Fig. 3) was the final factor contributing to rupture.

IV. TESTS & RESULTS:

1. Extensive x-ray fluorescence examination was completed on exposed and unexposed sections of the regulator. Corrosion products, carefully scraped from the thin wall section, were also examined by x-ray fluorescence. The metals thus detected are as follows, listed by diminishing peak intensity:

Casing	outside	inside (unexposed)	
	Fe	Fe	
	Cr	Cr	
	Zn	Mo	(note absence of Zn)
	Cu, Mo	Cu	
	Ni	Ni	

It is certified that this is an accurate report of test or analysis performed by the Chemical & Materials Branch.

Performed By		Signature of Approving Official
Name G. S. Whiting G. S. Whiting, AIC	Name	Name H. Rede, Capt.
Name	Name	Title Chief Metallurgy Section
		Chemical & Materials Branch

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Sealing wire	tag	Corrosion products
Fe	Al	Fe
Cr	Cr	Cr
Ni	Fe	Zn
Cu	Cu	Cu
	Zn	Mo
	Ni	Ni

2. X-ray diffraction of the corrosion products indicated a predominance of amorphous material with small amounts of some crystalline compounds (namely, iron oxides, chromium oxide, zinc chloride, and zinc fluoride).

3. A specific ion analysis determined the presence of chlorides and fluorides in the corrosion products. The results were:

4.03% F^-

.59% Cl^-

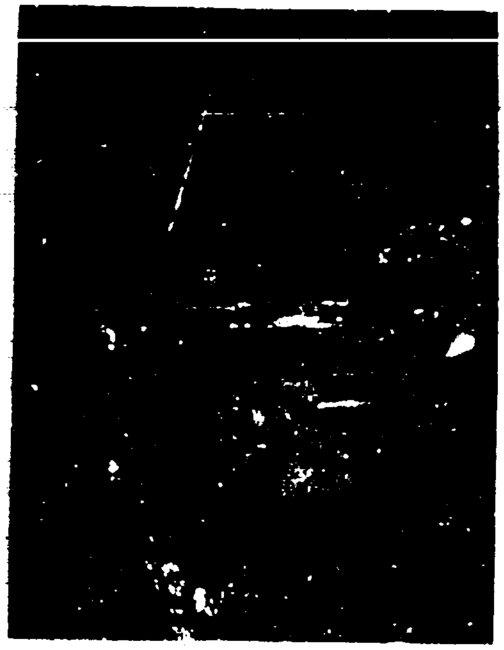
V. DISCUSSION: The thin wall portion of the casing (Fig. 3) ruptured due to corrosion cracks and flues (Fig. 4) along the thread-like groove (which greatly reduced the strength of that region) and the pressure of the compressed heavy spring.

The utility or purpose of the thread-like groove (Fig. 3) mentioned above is not obvious. However, in a corrosive medium the groove would behave as an anode (that which would corrode) and the adjacent metal as a cathode. Thus, cracks did penetrate the wall at the groove (Fig. 4), while only light pitting corrosion occurred elsewhere (Fig. 5).

The presence of zinc as found on the outside wall of the regulator is curious. Why the zinc is there is unknown. Zinc is frequently plated on non-stainless grade steels, where it intentionally serves as a sacrificial coating for corrosion protection. The origin of the zinc on the casing was in question, so the sealing wire and end tag (Fig. 3) were examined expressly for zinc content. Since no detectable zinc content was detected on the sealing wire or tag, the zinc on the casing must have been deliberately plated, instead of chemically washed onto that area.

The determination of chlorides, fluorides, and zinc in the corrosion product opens more objections to the zinc, apparently present as a cladding material. Fluoride ions are deleterious to metals like zinc. In addition, zinc chloride in the presence of moisture is acidic and aggressively corrosive. So, if this regulator was zinc plated, it is not apparent to this metallurgy lab why it should have been used in this particular environment.

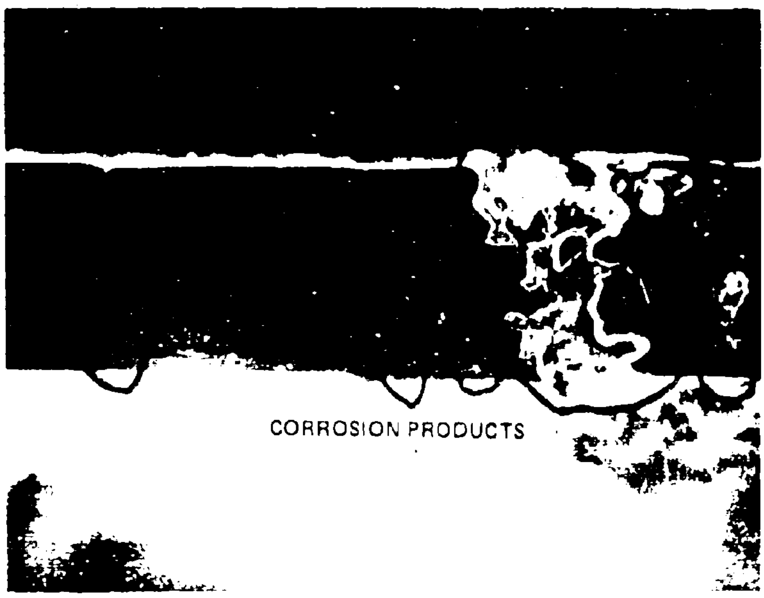
Figure 1. Rupture of Regulator in Situ



NOT REPRODUCIBLE

Figure 2. Rupture of Regulator in Situ

Figure 3. Regulator as Ruptured (3/4 X)
1. Thin wall portion of casing
2. Thread-like groove
3. Sealing wire
4. Sealing wire end tag


CORROSION PRODUCTS

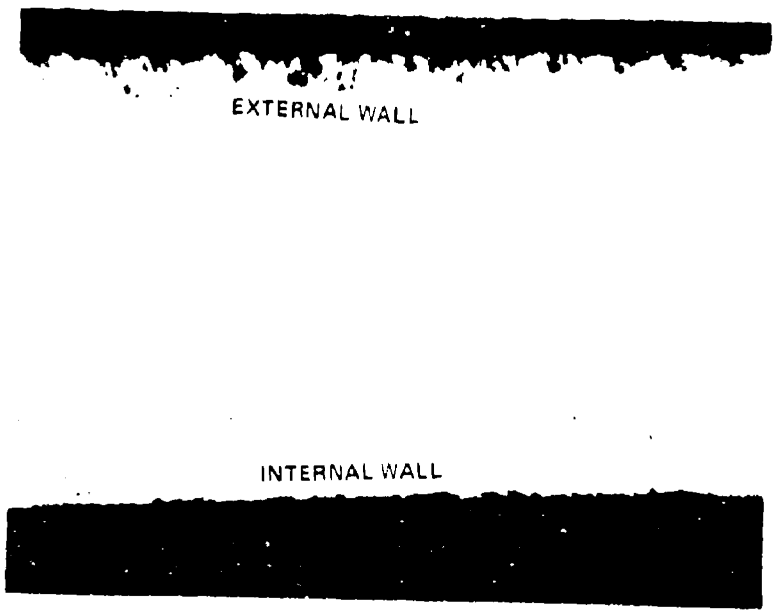


Figure 5. Cross Section of Thin Wall of [illegible] Casing Showing Pitting Corrosion on Exterior Wall (200X)

APPENDIX III

PROJECT 305805FRJ

REPORT ON EXAMINATION OF 5456

ALUMINUM ALLOY TANK WELDS

LABORATORY TEST REPORT	Report Nr. 156	Date 29 June 1971
Requesting Organization (Symbol and/or Name) LKFC	Name of Requestor Lt. H. White	Phone Number 32282

Sample, Test or Project
305805FRJ

Work Required
Examination 5456 Aluminum Alloy Tank Welds

TEST DATA

I. MATERIALS: 5456-F Aluminum alloy 1-qt. Alcoa tank; 5556 Aluminum alloy lug-to-tank weld filler.

II. BACKGROUND: Tank (S/N 79) exhibited external cracks in the lug-to-tank weldments during storage testing in an environment of 85°F and 85% relative humidity. The tank successively contained two fluids: ClF_3 for 479 days, followed by ClF_5 for 655 days. These cracks were noted in the lug-to-tank weldments after each test period, although the tank did not leak (as verified with GN_2 at 40 psi).

III. CONCLUSIONS: The acidic environment caused inter-granular corrosion cracking on the crown of the welds. Two microstructural feactures (heavy coring and large dendrites in the cracked weld region) indicated excessive heating of the weld filler during welding.

IV. OBSERVATIONS & DISCUSSION:

1. Observation: A vertical cross-section of the lug-to-tank region was polished and examined metallographically up to 400X. Fig. 1 shows the crown of the stop-pass weld and two cracks, above and below the crown. The dendrite size in the crown of the stop-pass region was very fine. (Fig. 2) The region underneath the crown (which would be the first weld pass) had a dendrite size double that of the stop-pass. (Fig. 2) Other weld regions, with cracks, had dendrite sizes three- to five-fold that of the stop-pass. (Fig. 3; note same magnification)

It is certified that this is an accurate report of test or analysis performed by the Chemical & Materials Branch.

Performed By		Signature of Approving Official
Name G. S. Whiting, A1C	Name	Name H. Rede, Capt
Name	Name	Title Chief Metallurgy Section
		Chemical & Materials Branch

RPL FORM 31

Discussion: The gross dendrites in the cracked weld versus the fine dendrites in the crown of the stop-pass weld yields considerable information. The larger dendrites resulted from the weld filler cooling over a longer time. One assumption is made: no devices were applied locally around the perimeter of the weld to provide a non-uniform heat sink. Therefore, the difference in dendrite sizes must have resulted from excessive heating in the weld zone where the larger dendrites are seen. Overheating in welding is known to be deleterious, because the material cannot cool rapidly enough to prevent coring and grain growth. (Coring is the rejection of impurity and alloying constituents to the grain boundaries or interdendritic spaces.) Longer cooling time permits greater coring, which in turn creates purer aluminum dendrite matrices and purer non-aluminum interdendritic material, which in turn makes a galvanic couple for corrosion. This type of galvanic corrosion, which resembled intergranular attack, was observed in the examined welds; the larger dendrite material corroded, which on the surface looked like cracks and beneath the surface looked like exfoliation.

2. A cross-section of the vertical strip on the girth weld was metallographically mounted and examined. (Fig. 4) The dendrite size was approximately one and one-half times that of the stop-pass. The interdendritic material was broken up or not as continuous as in the lug-to-tank weld. A tiny corrosion pit (Fig.s 4 & 5) was observed on the crown of the weld. The crown of the stop-pass on the lug-to-tank weld had the smallest dendrite size observed and exhibited no pits or cracks. However, no certain limiting dendrite size can be recommended by this lab below which corrosion would not have occurred in this environment. The pit was examined at higher magnification (Fig. 6) to find that interdendritic material remained standing in the corroded area. This observation indicated that the interdendritic material was cathodic to the dendrite matrix (which was anodic and corroded). This corrosion relationship was preferred for optimum corrosion resistance, since the dendrite matrix had the larger surface area.

Attachment 1

Microprobe Analysis Work

Ref AFRPL (RTCA) Lab Report No. 156, dtd 16 Dec 1970, para V.5, concerning the microprobe analysis work which was requested, the following information is submitted.

Magnaflux Corporation, Los Angeles, California, performed the work. Their findings showed that there was no abnormal amount of $CuAl_2$ precipitates at the grain boundaries as had been suspected. Microprobe analysis did reveal, however, that there were grain boundary precipitates rich in iron, manganese, and aluminum. It could not be determined through the literature whether this iron-maganese-aluminum precipitate was anodic or cathodic to the parent metal grains. However, discussion with Mr. Leonard W. Boyd, Jr., Supervisor Metallurgical Laboratory, Magnaflux Corporation, indicated that either of the two conditions mentioned are deleterious to the metal, i.e., would enhance corrosion at the grain boundaries. If the precipitates were anodic to the parent metal, they would preferentially corrode. If they were cathodic, then the parent metal immediately adjacent to the precipitates would preferentially corrode. The latter statements would hold true when an appropriate electrolyte is present, in this case, chloride and fluoride ions in the 85% relative humidity.

Summarizing, the cause of the cracks forming in the weld beads of the 2014-T6 aluminum alloy tanks with the 4043 aluminum weld filler metal was intergranular corrosion. (Ref Met Lab Report No. 156, dtd 16 Dec 70) A probable secondary cause was the presence of iron-manganese-aluminum precipitates which microprobe analysis showed to occur discontinuously at the grain boundaries. This precipitate was found primarily at the center of the weld bead, which was where the cracks were propogating, rather than in the heat-affected zones (HAZ) of the weld.

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Figure 1. Crown of Stop-Pass Weld on Boss Weld (5X)

Figure 2. Dendrite Size of Stop-Pass (Lower) and First-Pass (Upper) Welds Below Crown on Boss Weld (425 X)

Figure 3. Dendrite Size Below Crown in Crack Region of Boss Weld (425 X)

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Figure 4. Corrosion Pit in Cross Section of Vertical Strip Weld Across Girth Weld (15 X)

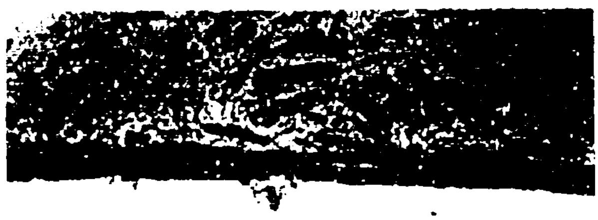

Figure 5. Corrosion Pit Shown in Figure 4 Enlarged to 95X

Figure 6. Interdendritic Material Still Standing in Corrosion Pit (1350 X). See Figures 4 and 5

March 9, 1971

Rocket Propulsion Laboratory
Edwards Air Force Base, California 93523
Attention: Captain Hector Rede, Chemistry Branch (RPTC)

Dear Captain Rede:

This will acknowledge your letter of October 26, 1970, requesting our comments on the welded liquid rocket propellant vessel. We apologize for the length of time it has taken for our evaluation.

You will find attached Alcoa Research Laboratories' Report No. 13-51747 which discusses the cause of the cracking.

The unusual corrosion effects have been discussed further with our Chemical Metallurgy Division and the interdendritic corrosion and cracking that resembles exfoliation of certain of the welds is unique in our experience. It appears to be related to welds that were alloyed with the 2014 base alloy to an unusual degree. We do not know the cause. The highly alloyed condition did not exist in the sheet to sheet welds in the wall vessel, so we could surmise that in making the forging-to-sheet welds, a greater amount of heat may have been put into the weld to insure proper fusion and penetration. All of the welds were sound, had good penetration, and appeared to be normal except for the extensive alloying and coring in the cracked forging-to-sheet welds. The atmospheric environment in which the exterior of the vessels was exposed, apparently was not abnormally corrosive as the 2014-T6 sheet revealed general intergranular attack like one might expect in a seacoast atmosphere.

If there is an uncorroded vessel like this one available we would be interested in trying to duplicate this form of attack in some of our accelerated exfoliation tests.

... some additional comments in the future on welding techniques ... this problem.

I ... this information will be of assistance and if you have any further ... or comments, please feel free to call.

Sincerely,

Thomas R. Brodrick
Gov't. Market Development Division

TRB/1d

Attach.

cc: Mr. John Brannigan, (RPRPT)

ALUMINUM COMPANY OF AMERICA

ALCOA RESEARCH LABORATORIES

NEW KENSINGTON, PA.

PHYSICAL METALLURGY DIVISION REPORT

FROM W. R. GRAFF
ALCOA RESEARCH LABORATORIES
NEW KENSINGTON

TO MR. J. A. DICKSON
APPLICATION ENGINEERING DIVISION
NEW KENSINGTON

February 28, 1971

No. 13-53747

WELDED LIQUID ROCKET PROPELLANT VESSEL

REFERENCE

Job order from J. A. Dickson to Mr. R. H. Stevens dated November 30, 1970.

DESCRIPTION OF MATERIAL

One small welded vessel exhibiting cracks in the weldments which attaches the end lugs to the tank was submitted for examination. The circumferential and longitudinal welds in the tank body did not reveal the presence of cracking. The tank and lug materials were 2014-T6 alloy and the filler material was 4043 alloy.

OBJECT OF EXAMINATION

The samples were examined to determine the cause of the weld cracking.

RESULTS OF EXAMINATION

Examination of sections from the welded tank-lug regions revealed that the cracks in the weld beads resulted from corrosive attack.

Interdendritic type of attack in each instance extended in from the crown of the bead and then spread out causing an exfoliated condition as illustrated by Figures 1 and 2. The microstructure of the weld metal in these corroded regions revealed a large amount of coring indicating a significant amount of solution of the 2014 [illegible] tank and lug sections during welding. The dilution of the filler alloy and the cored structure of the weld bead is [illegible] apparent in the microstructure shown in Figure 3. A [illegible] section from one relatively large welded tank to lug [illegible] not reveal any evidence of cracking, exhibited some [illegible]

[illegible] but also showed a surface layer having a typical 4043 alloy weld structure as illustrated by Figure [illegible]. This uncorroded region may have been in a start-stop region since one area showed a sharp line of demarcation between the body of weld with coring and a cover area of 4043 alloy as illustrated by Figure 5.

With the absence of cracking in the welds made in the vessel body it was expected that very little dilution of the 4043 filler had occurred, however, when these welds were metallographically examined, a significant amount of coring was observed. Since the composition of the metal could not be determined metallographically, a [illegible] analysis of the weld beads was made. The results of this analysis in Table I show that although the copper and magnesium content was about the same in both welds, a much higher percent silicon was present in the welds that did not fail.

The examined samples also revealed the presence of intergranular corrosive attack in the 2014-T6 alloy tank body extending to a depth of .0135".

From this examination it is evident that the weld failures resulted from corrosive attack. It is believed that in the large weld when the bead was partially solidified, much of the aluminum-silicon eutectic is forced by the arc to the edge of the bead leaving the crown and/or body of the weld metal low in silicon. If the excessive dilution of the filler alloy cannot be corrected by a change in welding practice, it may be possible to increase corrosion resistance of the diluted bead by placing a cover pass of 4043 or 1100 type alloy over the original weld bead.

Report by W. R. Graff — Approved by R. H. Stevens

ip

cc: H. Y. Hunsicker, ARL
ARL TID

TABLE I

WELD BEAD COMPOSITION

Spec. No.	Weld	Si	Cu	% Mg	Fe	Mn
243092	Circumferential	2.50	1.88	.12	.34	.36
243092	Lug End	.83	1.87	.10	.33	.21

Fig. 1 Spec.No. 243092 Neg. 181226A Mag. 20X Etch: Keller's

Shows the corroded condition of one of the cracked welds.

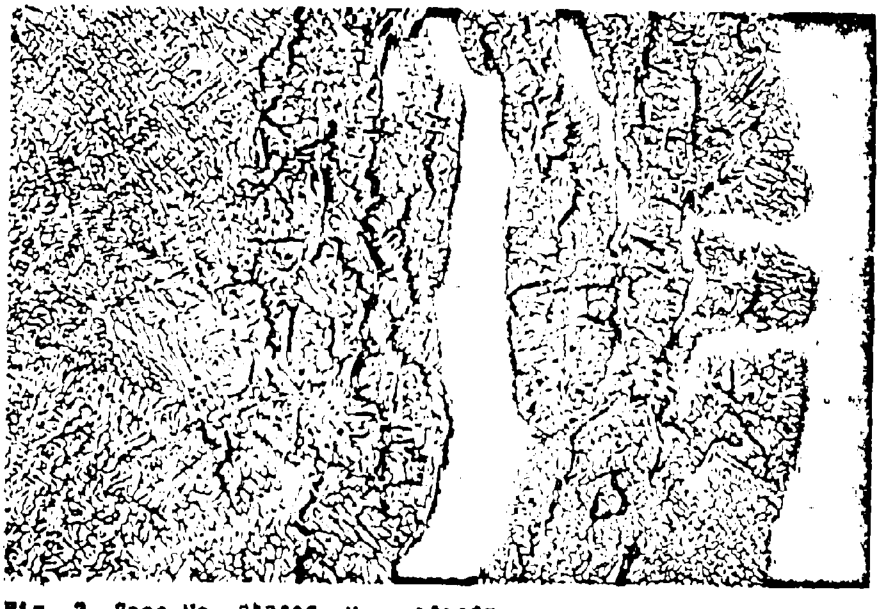

Fig. 2 Spec.No. 243092 Neg. 181287A Mag. 100X Etch: Keller's

Shows the attack, at a higher magnification, extending along the dendritic boundaries.

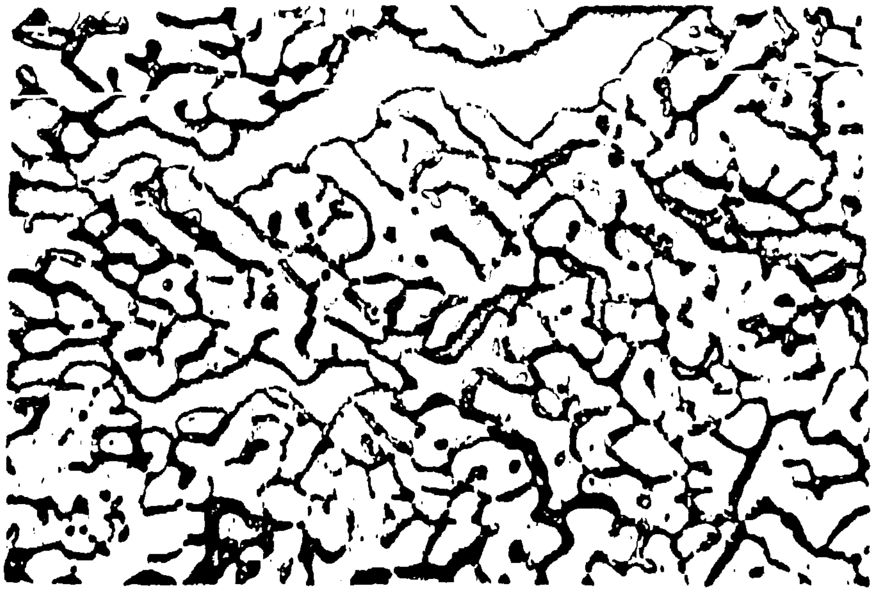

Fig. 3 Spec.No. 243092 Neg. 181288A Mag. 500X Etch: Keller's

Shows the coring present in the corroded region of the lug end weld.

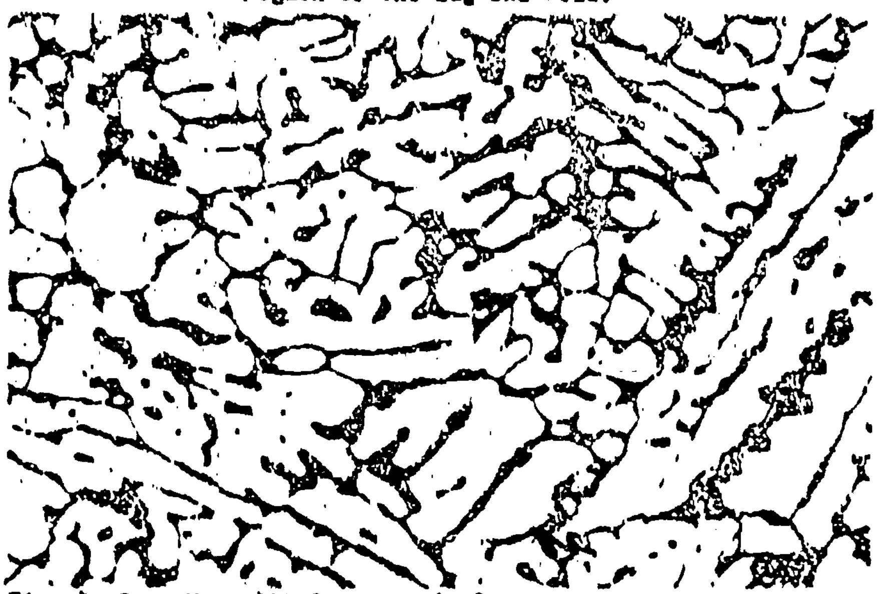

Fig. 4 Spec.No. 243092 Neg. 181289A Mag. 500X Etch: Keller's

Shows a weld structure representative of a typical 4043 alloy bead.

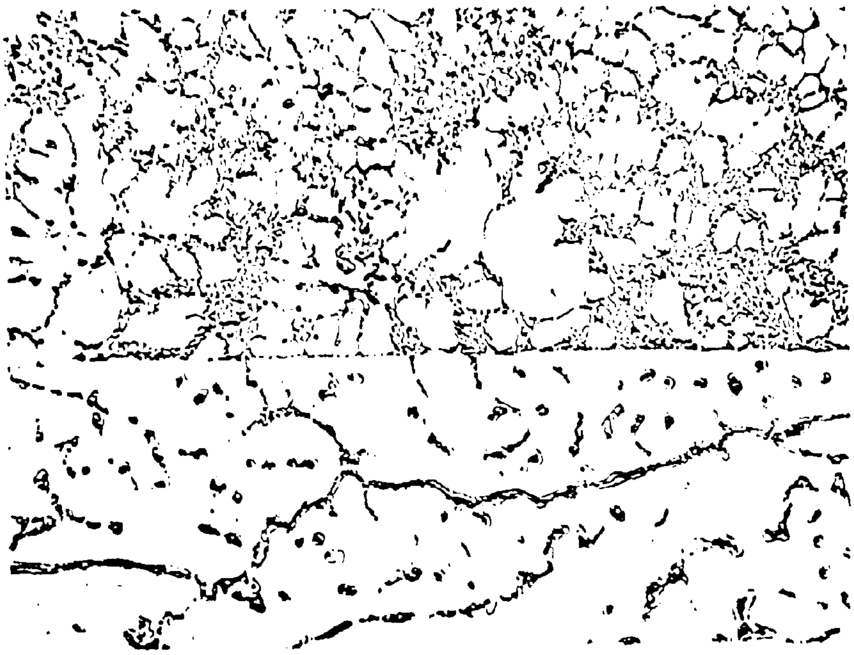

Fig. 5 Spec.No. 243092 Neg. 181290A Mag. 500X Etch: Keller's

Shows aluminum-silicon alloy structure
covering a portion of the dilute 4043 weld metal.