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JPL Contract 951709

STERILIZABLE LIQUID PROPULSION SYSTEM

Fifth Quarterly Progress Report

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January 1968

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This work was performed for the Jet Propulsion Laboratory, California Institute of Technology, sponsored by the National Aeronautics and Space Administration under Contract NAS7-100.

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MARTIN MARIETTA CORPORATION
DENVER DIVISION

FOREWORD

This document is the fifth issue of the Quarterly Progress Report and is submitted in accordance with Article 1(a)(1)(v)(E) and 2(b)(5) of JPL Contract 951709.

CONTENTS

I. INTRODUCTION

This is the fifty quarterly progress report submitted in accordance with JPL Contract 951709. The report covers the period from 1 October 1967 through 31 December 1967.

The program involves the exposure of an assembled and fueled bipropellant liquid propulsion system to the ethylene oxide (ETO) and heat sterilization environments specified by JPL Specification VOL 50503 ETS. After exposure, the system will be fired for 300 seconds.

The program plan includes a design and component selection phase during which the propulsion system design was evolved. A second phase involved the procurement of components for both a component test series and for assembly into the complete system. The third phase of the program, which was carried on in parallel with the design phase, was a materials investigation. The fourth phase of the program involves the assembly and test of the complete propulsion system.

Twelve cycles of heat sterilization of the components have been completed and post-test functional tests were done. All components with the exception of the thrust chamber valves and the solenoid valve have been disassembled and inspected for degradation.

Considerable progress has been made in the buildup of the test firing cell. During the early portion of the next period, this work will be completed in line with the schedule for module firing.

During this report period the module assembly was completed, propellants were loaded and the module was started into the decontamination and sterilization exposures. At the end of the period six cycles of ETO decontamination and four cycles of heat sterilization had been completed. At the completion of ETO testing, it was found that the nitrogen pressurant tank pressure had decayed from 1540 psig to 1120 psig due to a leak. The leak was found in a test instrumentation port cap. A repair was made and testing progressed without further problems.

II. CONCLUSIONS

As a result of the work performed during this period, the following conclusions were reached:

1) Based upon the component test results, sterilization of typical liquid propulsion system components poses no significant problems. Although some degradation did occur, no unsolvable problems were experienced. A flight system could be designed using state-of-the-art components to meet the sterilization requirements with a high degree of confidence even though some slight modifications may be required.

2) Although humidity and ETO concentration control in a chamber is difficult, it can be accomplished with currently available equipment. In addition automatic control is relatively simple once the characteristics of the chamber are known.

III. RECOMMENDATIONS

As a result of the work performed during this period, the following recommendations are made:

1) Additional work should be initiated, in particular, on the components which showed degradation during component sterilization. In addition more experience should be accumulated on the module in order to lend credence to the test results and to allow better system and component reliability estimates. Specifically, the following items are recommended:

    a) Additional sterilization cycles and firing of the existing module.

    b) Modify the existing module to accept a monopropellant test of hydrazine-hydrazine nitrate.

    c) Use different components in the existing module including a beryllium engine.

    d) Develop an improved Teflon diaphragm.

    e) Develope a tantalum screen trap.

    f) Do additional material and propellant compatibility testing.

    g) Develop and test a system to sterilize propellants separate from the module followed by sterile transfer to the module and firing.

IV. GENERAL REPORT

A. COMPONENT DESIGN

As the program has progressed and component problems have occurred during testing, design changes have been made where possible to the module components. At the completion of component testing, a leakage problem had been experienced with the hand valve. Stem leakage and leakage through the stem cap seal were above the allowable of 1×10^{-8} scc/sec of He at 1000 psi. A program was initiated to improve the stem cap seal, Figure 1a, since some leakage of the Teflon stem packing must be expected with operation. As a first step the 1000 aluminum alloy gaskets were annealed to the soft condition (1100-O) and maximum allowable torque was applied to the cap. The test unit still leaked under these conditions. Examination of the unit indicated a combination of effects were probably preventing the use of a metallic seal. Surface finish on the valve body and inside the cap along with "out-of-parallel" seal surfaces were the main contributors.

The valve was reassembled using thin (.010) Teflon gaskets on each surface of the soft aluminum gasket. Although leakage was decreased considerably, it was not eliminated. In addition the Teflon sheet was extruded from between the aluminum washer and the valve body and the valve cap. This extrusion process would probably continue until virtually no Teflon remained between the metal surfaces at which time the seal loading would be equal to the compressive yield strength of the Teflon. With heating and cooling, this seal load would be reduced until the seal would be ineffective.

At this time other approaches were tried, Figure 1b and 1c. Soft aluminum seals with single and double serrations and a combination "O" ring and aluminum spacer ring were used. The aluminum seals with serrations did not solve the problem because of the surface finishes involved. The combination "O" ring and spacer ring, however, proved to be tight under hand torques. Several installations were tried in two different test units with no leakage detected in either case. The valves were soaked in an oven at approximately 250°F for 24 hours followed by a mass spectrometer leak check using helium at 950 psig with no indication of leakage. The "O" rings selected for the installation were of Viton rubber, good for operating temperatures up to 400°F. This material, while not completely compatible with the

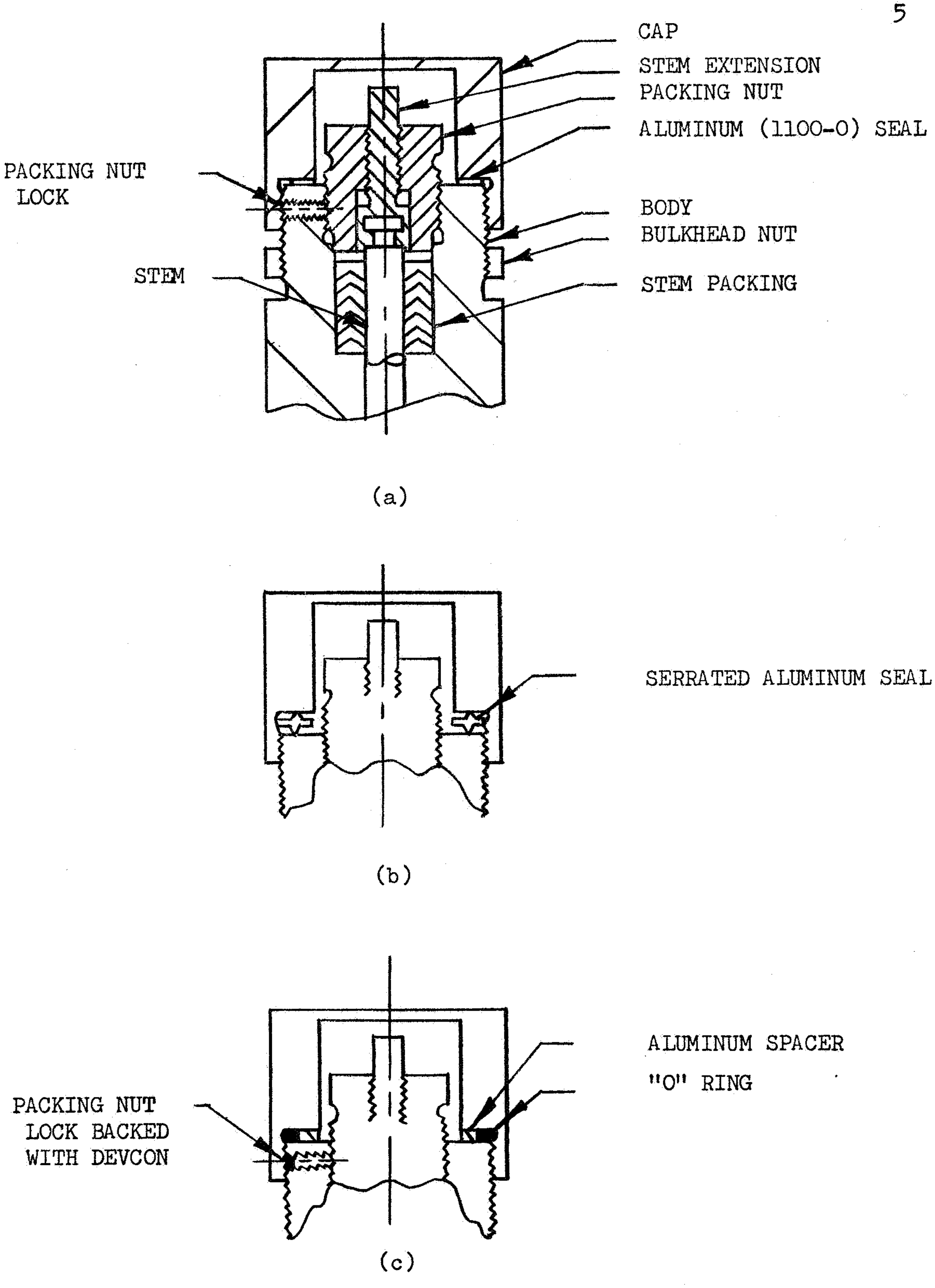


Figure 1 HAND VALVE STEM CAP SEAL CONFIGURATIONS

with the propellants, resists attack of both oxidizer and fuel. It was decided that the module units would be modified to use the "O" ring seal since the stem cap seal is a secondary seal and at worst would see dilute propellant vapors.

An additional problem which had occurred involved leakage from the stem packing nut lock device, Figure 1c. This locking device consists of a Teflon plug backed up by a set screw. During the stem cap seal tests this lock frequently leaked. As a repair for the two valves in test, a new Teflon plug was cut and installed with the set screw. The screw was torqued into the body until it was well below the external body threads. The cavity behind (on the outside of) the set screw was packed with Devcon WR. Devcon WR is a suspension of metal particles in an epoxy resin and is generally used as metal patching compound. Although the epoxy resin is probably not compatible with either the fuel or oxidizer, it was used due to the particular application. The area of possible contact is the clearance area between the set screw threads and the valve body. In addition only a dilute mixture of propellant vapor and air trapped in the cap volume would contact the material. Although some degradation might take place at the surface, complete breakdown is not anticipated. This modification which proved to be adequate for the test units was also added to the module valves.

B. COMPONENT TESTING

During the quarter ending 31 December 1967, the Phase II component testing was completed. The second series of six cycles of sterilization were completed and functional testing was finished. At the end of the period disassembly and inspection of components was in progress.

A brief summary of heat sterilization cycles 7 through 12 is presented as follows:

Cycle Number 7, 22 September 1967 to 25 September 1967

This sterilization cycle was started after all components had been subjected to the mid-sterilization functional tests. The oxidizer tank test item was not installed, having been removed from sterilization testing for failure analysis during sterilization Cycle Number 6. The customer-furnished throttling valve was installed in the chamber to undergo six cycles of sterilization testing. The fuel and oxidizer passages in the throttling valve were half-filled with MMH and N_2O_4, respectively. The remaining components were installed in the sterilization chamber as they had been installed during the first series of six sterilization cycles.

The chamber reference temperature and fuel tank pressure histories during this cycle are shown in Figure 2. The cycle was uneventful with the exception of a chamber shutdown at T + 17 hours which was caused by spurious signals from the fuel vapor detector. Chamber temperature was restored in approximately 2 hours. Equilibrium fuel vapor pressure (fuel temperature) was re-established eight hours after the shutdown, at which time testing was resumed at the T + 17 hour mark.

Cycle Number 8, 26 September 1967 to 29 September 1967

It will be noted that there was an unusually wide excursion in chamber reference temperature during the first half of the run. This was caused by slippage of a shim under the cam follower of the temperature controller. During the latter half of the cycle, the temperature set-point was increased to compensate for the lower temperatures experienced during the earlier part of the cycle. The controller problem was rectified at the conclusion of the cycle. The chamber reference temperature and fuel tank pressure histories are shown in Figure 3.

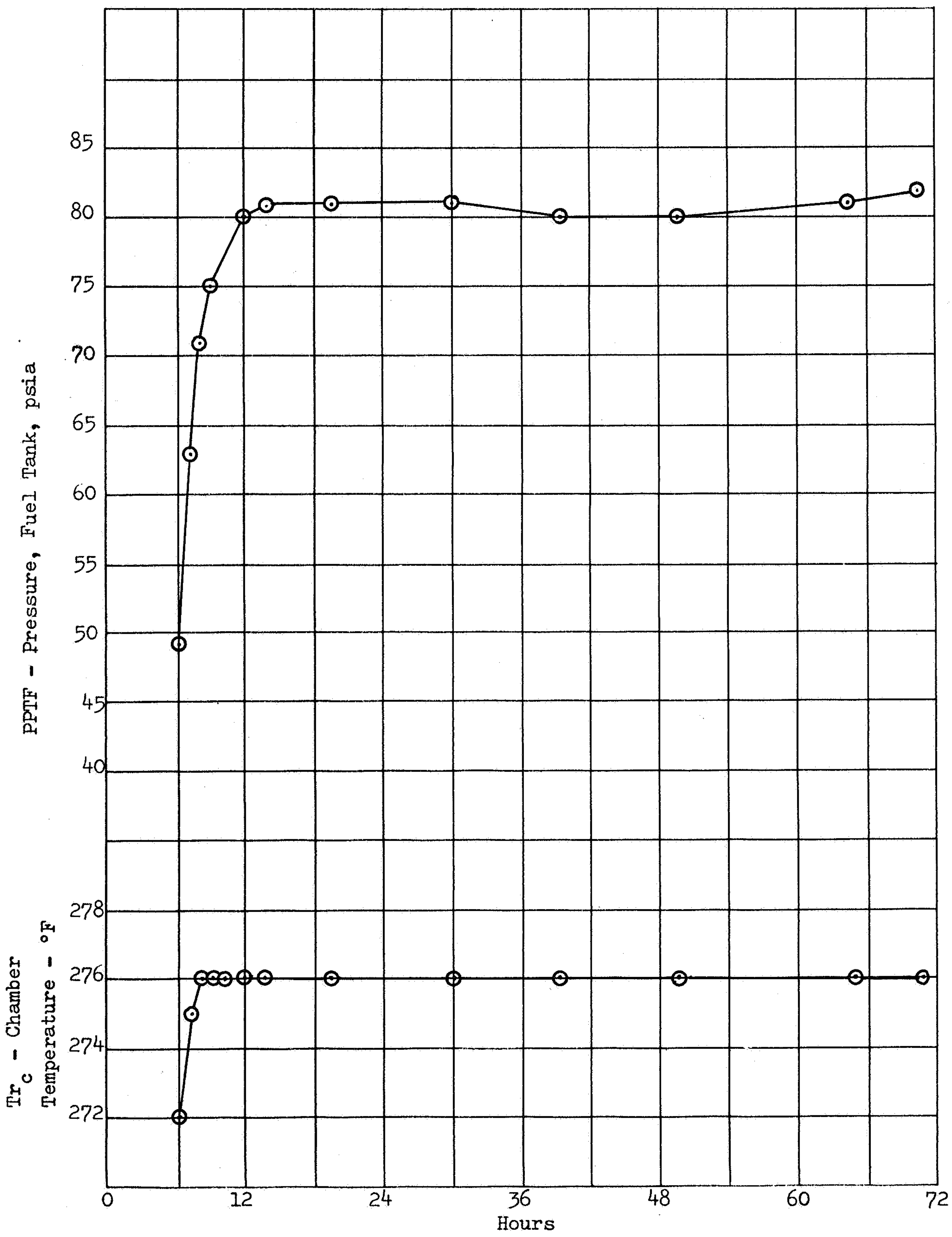


Fig. 2 Sterilization Cycle No. 7

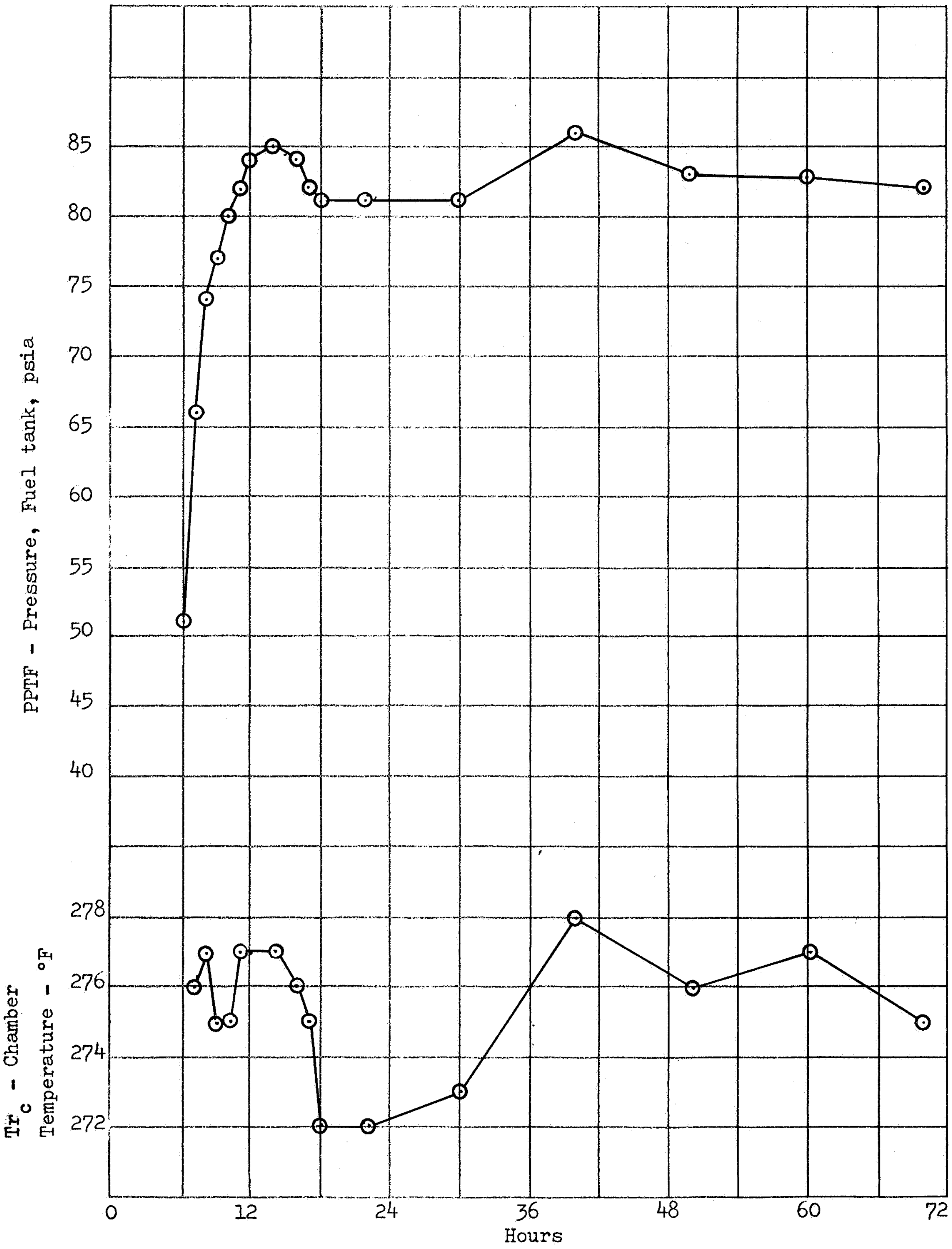


Fig. 3 Sterilization Cycle No. 8

Cycle Number 9, 29 September 1967 to 3 October 1967

This cycle was interrupted at T + 48:50 hours by loss of facility GN_2 pressure in the test area, which caused the chamber temperature controller to shut off the chamber heaters. This condition occurred during unattended chamber operation on Sunday, 1 October, and the shutdown was of a type which could not activate the chamber kill alarm system. The shutdown condition was, therefore, not detected until the following morning, at which time the chamber had been at under-temperature conditions for approximately 18 hours. Equilibrium chamber temperature and fuel vapor pressure (fuel temperature) were re-established at 16:50 on 2 October 1967, at which time the cycle timing was resumed at T + 48:50 hours elapsed time. The cycle was concluded at 20:00 on 3 October without further incident. The chamber reference temperature and fuel tank pressure histories are presented in Figure 4, exclusive of the transient histories during the above-described interruption.

Cycle Number 10, 3 October 1967 to 6 October 1967

This cycle was completed without incident. The chamber reference temperature and fuel tank pressure histories are presented in Figure 5.

Cycle Number 11, 7 October 1967 to 10 October 1967

This cycle was completed without incident. Data histories are shown in Figure 6.

Cycle Number 12, 10 October 1967 to 13 October 1967

This cycle was completed without incident. Histories of chamber reference temperature and fuel tank pressure are presented in Figure 7.

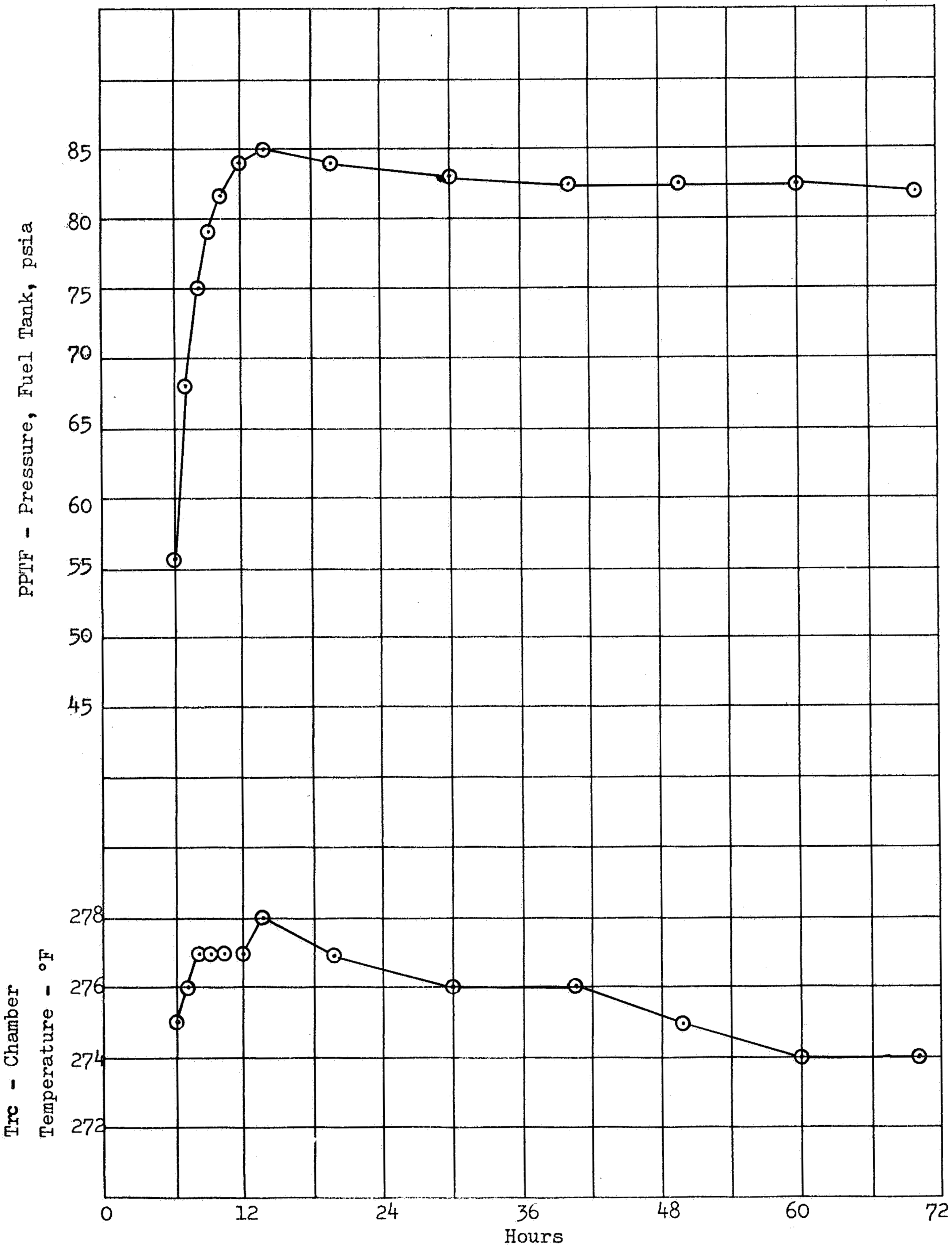


Fig. 4 Sterilization Cycle No. 9

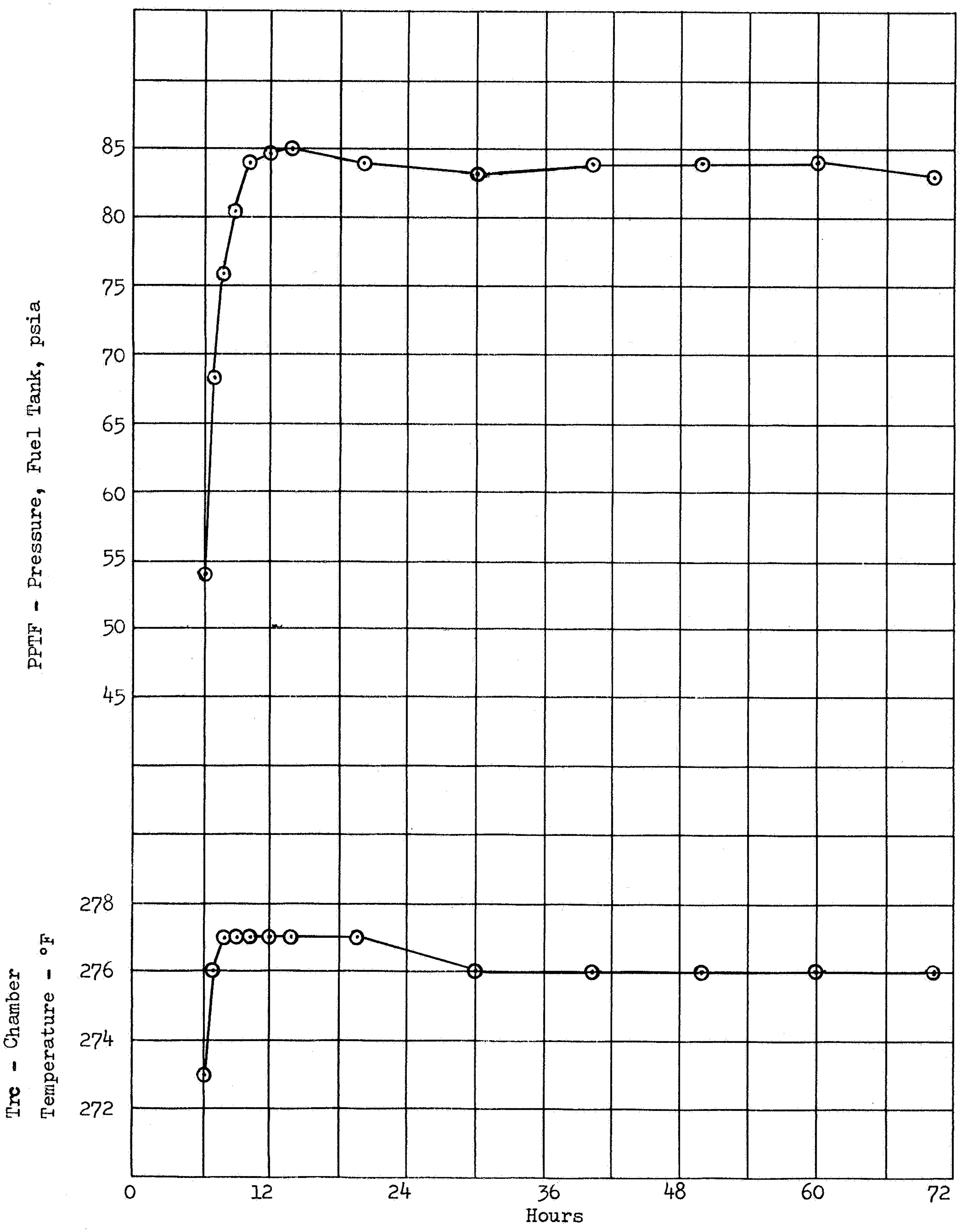


Fig. 5 Sterilization Cycle No. 10

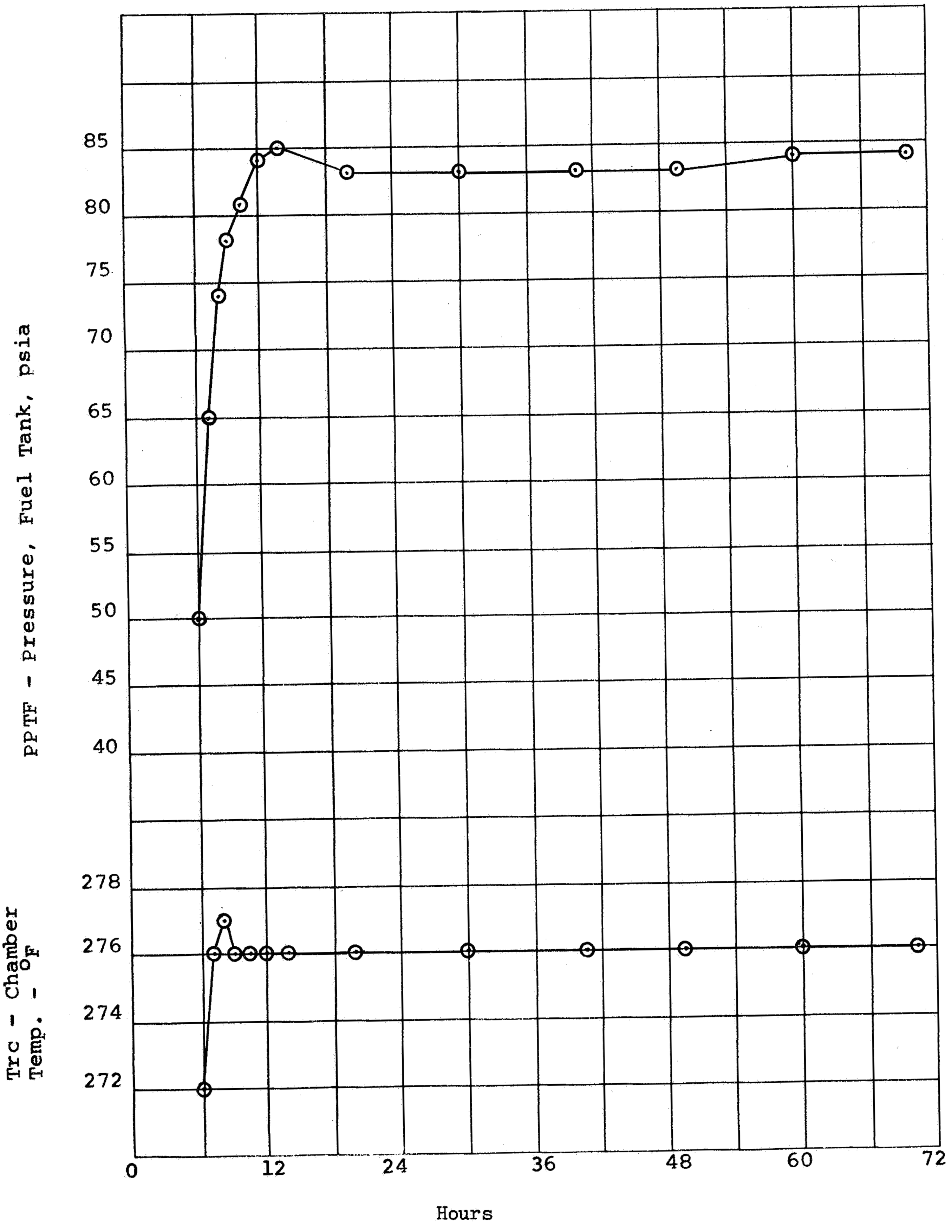


Fig. 6 Sterilization Cycle No. 11

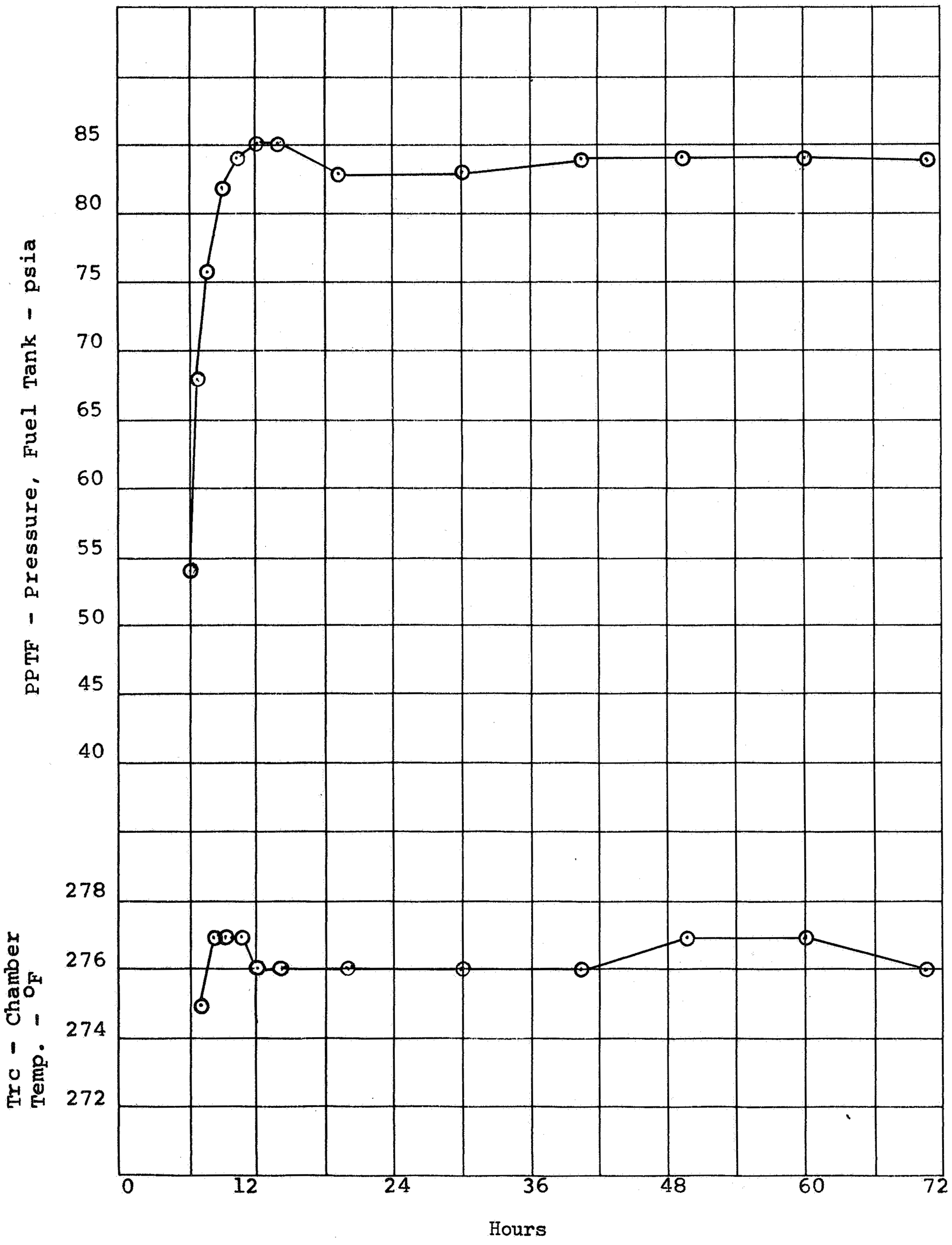


Fig. 7 Sterilization Cycle No. 12

Phase II Post-Sterilization Functional Tests

The post-sterilization functional tests were completed on all test components with the exception of the oxidizer tank (removed from testing during Cycle Number 6) and the customer-furnished throttling valve. The throttling valve was decontaminated and shipped to JPL for functional tests. Results of the post-sterilization functional tests on each of the components are presented in the Summary Data Sheets of Figures 8, 9, 10, 12, 16, 17, and 20, together with the data from the pre-sterilization and mid-sterilization functional tests.

In general, there was no significant change in the performance of the test components after exposure to the twelve sterilization cycles with the exception of the pressure regulator and the hand valve. The solenoid valve showed a marked degradation in dielectric strength; however, the performance of the valve was not noticeably impaired. The degradation noted in the performance of the regulator and hand valve occurred during the first series of six sterilization cycles, while the degradation in the condition of the solenoid valve occurred during the second series of six sterilization cycles. A review of the performance of each of the test components is given below:

Ordnance Valve: There was no change in the performance (i.e., leakage characteristics) of the ordnance valve after exposure to the 12 sterilization cycles. The valve exhibited zero internal and external leakage when checked with the helium mass spectrometer leak detector. Following the leakage tests, the normally-closed portion of the valve was fired open with the same squib which had been exposed to all sterilization tests in the valve. The pressure drop of the valve was then measured at the design conditions for the propulsion module pressurant gas supply at the propellant tanks. Following the flow capacity test, the normally-open section of the valve was fired closed for the final leakage test. Internal leakage was again zero; however, external leakage of helium in the area of the inlet flange mechanical seal had increased from zero to 7.3×10^{-7} scc/sec.

The flanged joint of the unit was disassembled. The three holding screws required considerable torque to loosen. The aluminum gasket showed a good imprint of the circular ridges and was spread out tightly against the body section.

Task II

SUMMARY DATA SHEET

Component Name:	Ordnance Valve
Part Number:	J315-7853
Serial No:	015

	Pre-Sterilization	Mid-Sterilization	Post-Sterilization	
			Pre-Firing	Post-Firing
A. Leakage Rate, Helium (scc/sec)				
Internal:	Zero	Zero	Zero	Zero
External:	Zero	Zero	Zero	7.3×10^{-7}
B. Response (dP_o/dt psi/sec)	N/A	N/A	N/A	42,500
C. Pressure Drop, Design GN_2 Flow (at 260 psia(psi))^2	N/A	N/A	N/A	1.4

Fig. 8 Performance Data, Ordnance Valve

Another line joint approximately one inch from the flanged joint had been a source of leakage in post tests and it is felt that the indicated leakage was from this joint rather than the flange joint.

Solenoid Valve: There was no measurable change in the performance of the solenoid valve; however, the dielectric strength of the solenoid coil showed degradation during the last six heat sterilization cycles. This has been discussed with the Vendor and it was suggested that moisture had condensed inside the potted area since the valve is not hermetically sealed. To check this theory the unit was placed in a vacuum oven at a pressure of 24 inches of Hg below local ambient and a temperature of 250°F for a total period of 32 hours. After cooling down to room temperature, the unit still indicated about 22 microamps at 500 VAC. It was then discovered that the measuring instrument was in error. A small circuit breaker in the instrument was opening at 22 microamps and the instrument could not read above this point. The unit was then retested on a hi-pot type instrument. This final test shows approximately 500 microamps leakage current at 500 VAC, as shown in the Summary Data Sheet of Figure 9. This increase in leakage current indicates a very definite degradation in dielectric strength and corrective measures should be incorporated in future designs. At the end of the report period preparations were being made to disassemble the valve for failure analysis.

Filter: There was no measurable change in the flow characteristics of the filter, as shown on the Summary Data Sheet of Figure 10. Pressure drop through the filter at design flow rate remained at zero (no measurable pressure drop using a differential pressure transducer having a range of 0 to 5 psi).

Following the flow capacity tests, the unit was subjected to a bubble point check. With GN_2 pressure applied at the inlet side and the outlet wetted and covered with methanol, the bubble point was between 17.25 and 17.5 inches of H_2O. This shows a degradation from the acceptance test figures that were between 22 and 24.2 inches of H_2O. However, this is still within the acceptable range since the minimum specification bubble point for this filter weave is 15.9 inches of H_2O. Future designs should incorporate an allowance in the acceptance test to allow for some degradation.

Task II SUMMARY DATA SHEET

Component Name: Solenoid Valve
Part Number: Sterer P/N 35580; Martin P/N 6002516-009
Serial Number: 2

	Pre-Sterilization	Mid-Sterilization	Post-Sterilization
A. Leakage Rate			
1. Internal Leakage (Helium)			
Inlet Pressure (psig)	1560	1530	1544
Leakage (scc per hour)	3.3	2.0	0
2. External Leakage (Helium)			
Inlet/Outlet Pressure (psig)	2200	2200	2200
Leakage Rate (scc/sec)	Zero	Zero	Zero
B. Flow Capacity (GN_2)			
Corrected Inlet Pressure (psia)	1550	1550	1550
Corrected Inlet Temperature (°F)	70	70	70
Corrected Flow Rate (lbs/sec)	.070	.072	.071
C. Response			
Average Inlet Pressure (psig)	1545	1543	1533
Opening Time (sec) Minimum	.102	.102	.104
Maximum	.102	.108	.104
Closing Time (sec) Minimum	.082	.081	.084
Maximum	.089	.092	.084
D. Dielectric Strength			
Pin A to Case (microamps) 500 VAC	4	0	500
Pin B to Case (microamps) 500 VAC	4	0	500

Fig. 9 Performance Data, Solenoid Valve

TASK II

SUMMARY DATA SHEET

Component Name:	Filter, 5 Micron Nominal
Part Number:	Western Filter Company P/N 20477-5 Martin P/N LAB 6002513-009
Serial Number:	None

Pressure Drop (GN_2)	Pre-Sterilization	Mid-Sterilization	Post-Sterilization
1. High Pressure			
Inlet Pressure - psig	1550	1537	1552
Flow Rate - lbs/sec	.015	.015	.016
Pressure Drop - psi	0	0	0
2. Low Pressure			
Inlet Pressure - psig	375	248	280
Flow Rate - lbs/sec	.014	.016	.014
Pressure Drop - psi	0	0	0

Fig. 10 Performance Data, Filter

The unit was cut open (see Figure 11) and examined. No dirt was evident on the inlet side and no separation was visible along the welded joints or on the screen surface.

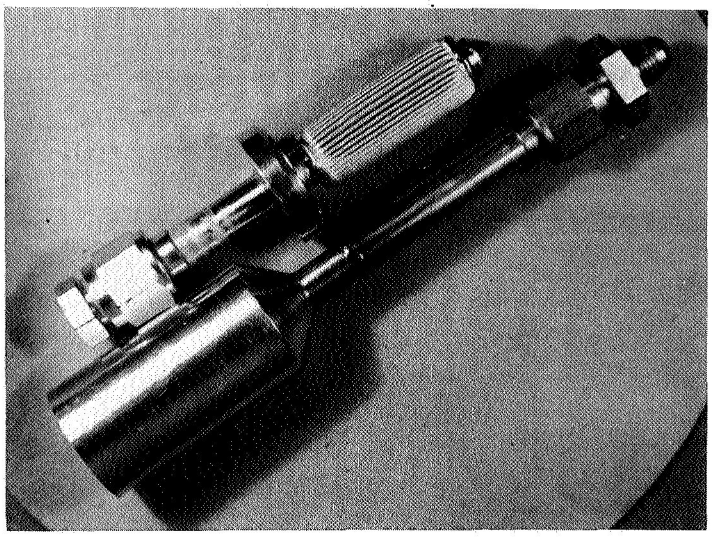


Figure 11 Component Test Filter Disassembly

Hand Shutoff Valve: The hand shutoff valve continued to exhibit internal leakage at the limiting torque value of 10 inch-pounds which was employed throughout the test; however, at the conclusion of all sterilization testing, the valve exhibited zero internal leakage (zero cc in 15 minutes) when the stem was torqued closed to 16 to 17 inch-pounds (see the Summary Data Sheet of Figure 12). The valve should, therefore, be judged satisfactory from the standpoint of shutoff capability.

The external leakage at the conclusion of the sterilization test was substantially unchanged from that observed at the mid-sterilization point, being in the neighborhood of 1×10^{-5} scc/sec of helium. The leakage was noted at the bonnet cap, indicating that both the stem packing and the bonnet cap seal were leaking. Maximum allowable leakage is specified at 1×10^{-8} scc/sec of helium at 935 psig. As detailed in Section IV-A of this report, a development program was initiated to improve the cap seal for the module valves.

Task II

SUMMARY DATA SHEET

Component Name: Hand Shutoff Valve
Part Number: VACCO NVB 32181; Martin LAB 6002512-009
Serial Number: 21385-1

	Pre-Sterilization	Mid-Sterilization	Post-Sterilization
A. Operating Torque (Helium, 248 psig)			
Shutoff Torque (inch-lbs)	10*	10*	10* 17
Leakage at Shutoff (cc/min)	1.9 to 3.8	41.0 to 44.5	16.0 to 20.0 Zero
B. Leakage (Helium, 935 psig)			
Internal (cc/min)	19	720	Zero (16 inch-lbs)
External (scc/sec)	Zero	1.12×10^{-5}	1.35×10^{-5}
C. Flow Capacity (GN_2)			
Inlet Pressure (psig)	250	250	250
Outlet Pressure (psig)	0	0	0
Flow Rate (lbs/sec)	.0765	.0720	.0725
Capacity Factor (C_v)	0.45	0.42	0.43

Remarks:

* Maximum allowable torque. Complete shutoff was not obtained, as indicated by leakage rate noted.

Fig. 12 Performance Data, Hand Shutoff Valve

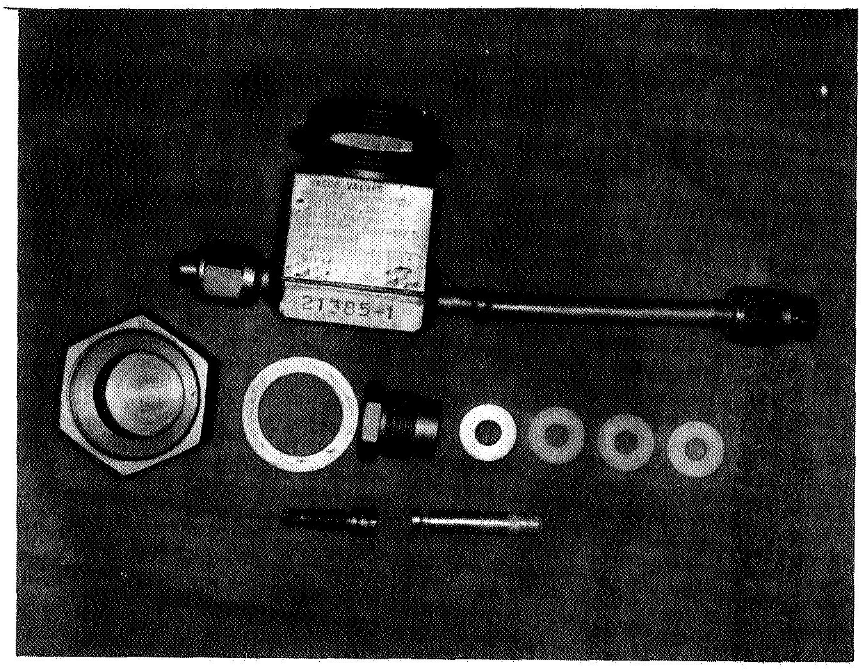


Fig. 13 Hand Valve Disassembly

Disassembly of the valve, Figure 13, disclosed a very heavy coating of white, powdery, aluminum oxide on the exposed portion of the poppet. This was most severe on the 1100-O poppet nose, see Figure 14. The oxidation of the anodized body was very slight.

The stem chevron Teflon packing showed considerable extrusion between the stem and the back-up washer, Figure 15. The stem measured 0.2476" in diameter in the region covered by the chevron packing and the back-up washer was 0.2815" at the inner diameter. This left a diametral clearance of 0.0339".

The heavy oxide coating was anticipated because material tests under Task III did show reaction between aluminum alloys and the oxidizer at sterilization temperatures. Future designs should consider all titanium construction.

The Teflon packing extrusion could have caused the external leakage reported in the post-sterilization tests results by permitting a relaxation of the sealing force. The diametral clearance between stem and washer is excessive and future tests could determine maximum clearance versus gland nut torque over extended periods at sterilization temperatures.

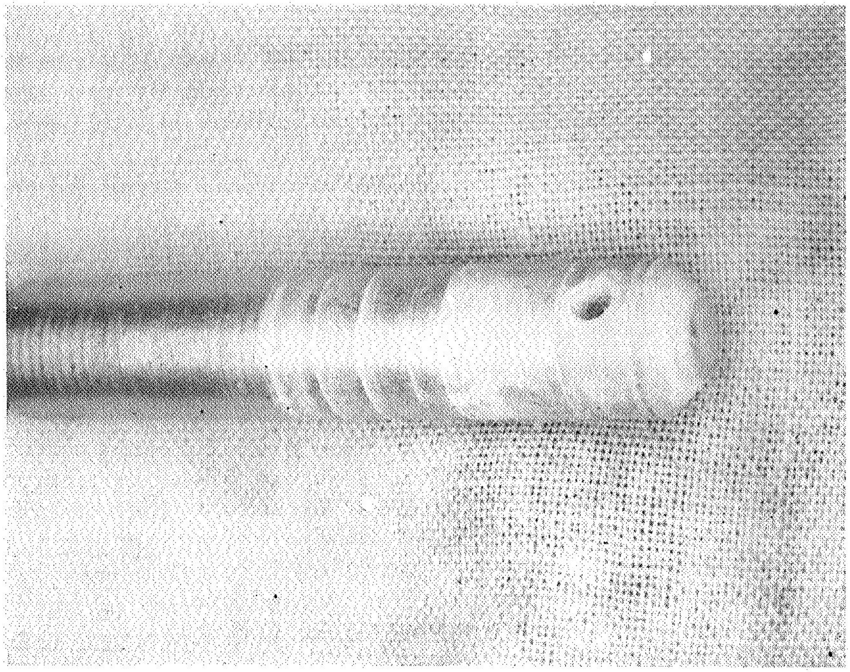


Fig. 14 Hand Valve Poppet

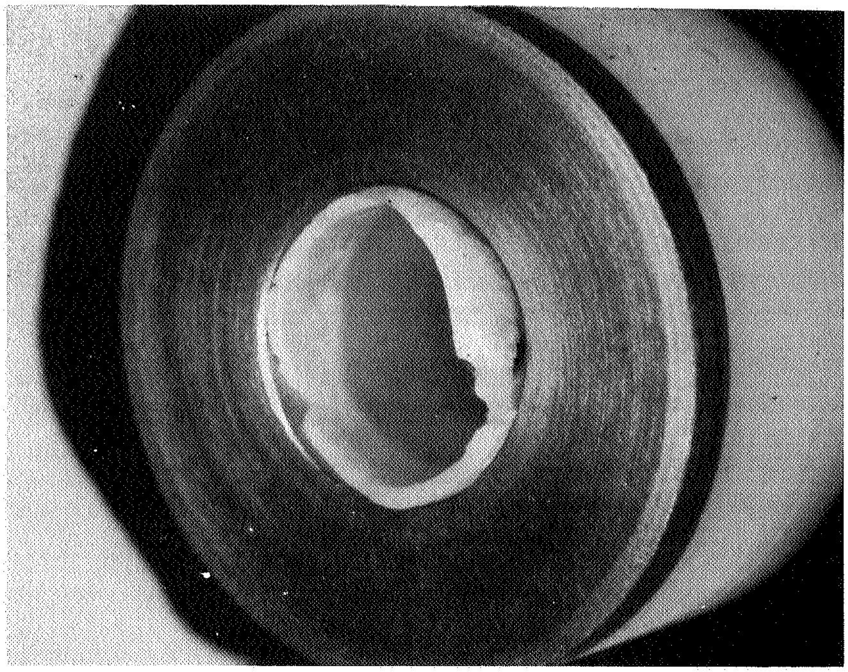


Fig. 15 Hand Valve Stem Seal Chevrons

Thrust Chamber Valves: Performance of the thrust chamber valves is shown in Figure 16 for the functional tests run before sterilization and after completion of the 12 sterilization cycles. The response data from the mid-sterilization functional test was adversely affected by interaction between the valve solenoid coils caused by the data acquisition system loading the direct coil. Loading of the direct (data pick-off) coil caused the response time of the valves to increase by approximately a factor of 3. Inasmuch as the interaction phenomenon was not discovered until after the second series of sterilization cycles was underway, accurate retrieval of the true response characteristics of the valves was not feasible. In addition, since the operating characteristics of the valves were not significantly changed after 12 sterilization cycles, it is reasonable to assume that there was no significant change in valve performance at the mid-sterilization point. The general performance trend which may be noted is that the response time of the valves increased by 1 to 2 milliseconds although there was slight decrease in the pull-in voltage which would indicate that internal friction had not increased. Internal and external leakage of the valves remained at zero throughout the sterilization program.

Pressure Regulator: The results of the post-sterilization tests on the pressure regulator (Figure 17) showed that the regulation characteristics were substantially the same as those exhibited at the mid-sterilization point, i.e., the regulation band was approximately 15 psig below the specification value. This indicated that the shift in regulation band which had occurred during the first set of six sterilization cycles did not progress measurably during the second six cycles. The internal leakage of the regulator had increased by a factor of four over the final results obtained during the mid-sterilization functional test. The leakage rate did not change after exercising the regulator.

Tear down of the unit started at the square flange holding the inlet tube and filter to the main body. The four screws required very low torque to loosen (approximately 5 in-lbs).

Task II

SUMMARY DATA SHEET

Component: Thrust Chamber Valves

	Pre-Sterilization	Post-Sterilization
A. Oxidizer Valve, S/N 575		
1. Pull-in Voltage (VDC)		
Maximum	14.0	13.2
Minimum	14.0	13.0
2. Opening Response (sec)		
Maximum	.0118	.0125
Minimum	.0112	.0123
3. Closing Response (sec)		
Maximum	.0084	.0090
Minimum	.0079	.0090
4. Leakage: External (bubbles GN_2)	Zero	Zero
Internal (cc GN_2/hr)	Zero	Zero
5. Pressure Drop, Design flow (psi)	27.5	29.2
6. Insulation Resistance (megohms)	500+	500+
B. Fuel Valve, S/N 576		
1. Pull-in Voltage (VDC)		
Maximum	11.5	11.3
Minimum	11.5	11.3
2. Opening Response (sec)		
Maximum	.0089	.0118
Minimum	.0087	.0120
3. Closing Response (sec)		
Maximum	.0094	.0096
Minimum	.0091	.0087
4. Leakage: External (bubbles GN_2)	Zero	Zero
Internal (cc GN_2/hr)	Zero	Zero
5. Pressure Drop, Design Flow (psi)	13.8	14.2
6. Insulation Resistance (megohms)	500+	500+

Fig. 16 Performance Data, Thrust Chamber Valves

Task II SUMMARY DATA SHEET

Component Name: Pressure Regulator
Part Number: Sterer P/N 35570; Martin P/N LAB 6002515-009
Serial Number: 1

	Pre-Sterilization	Mid-Sterilization Initial	Mid-Sterilization Final	Post-Sterilization
A. Leakage Rate				
External (Bubbles GN_2)	Zero	Zero	Zero	*
Internal (GN_2 scc/hr)	4.2	56,000	1200	4900
B. Hysteresis				
Initial Outlet Lock-up Pressure (psig)	269	250	264	256
Minimum Outlet Pressure (psig)	259	243	248	246
Maximum Outlet Pressure (psig)	263	247	253	254
C. Regulation				
Inlet Pressure, Initial (psig)	1560	1513	1500	1519
Inlet Pressure, Final (psig)	408	320	342	351
Average Flow Rate (lbs/sec)	.015	.014	.015	.015
Outlet Pressure (psig) Minimum	247	231	234	231
Maximum	250	235	234	235
D. Response				
Inlet Pressure, Average (psig)	1650	1500	--	1500
Outlet Pressure, Lock-up (psig)	260	252	--	244
Overshoot (psig)	0	0	--	0

* One 1/4" diameter bubble every five minutes.

Fig. 17 Performance Data, Pressure Regulator

The filter, Figure 18, showed some accumulation of dirt on the upstream side but this was not excessive.

The area inside the unit in the vicinity of the poppet was heavily coated with moisture.

The ball poppet, Figure 19, was coated with oxide. The contact area of the poppet seat was bright as well as the sliding areas along the poppet guide. Several other bright areas (see arrows) around the seat contact indicated a probable contaminant rubbing of the oxide coat. No contaminants could be seen in the seat area at this time.

The low torque required to loosen the square flange holding screws indicates a relaxation of the holding force during sterilization cycling. This was probably the cause of the external leak at this point on post-sterilization testing. It is recommended that future designs consider increasing the number of holding screws to six or eight and/or change the hard seat cone seal to a flat flange with spring loaded type of metallic seal ("K" type seal).

The presence of moisture inside the unit and the bright spots along the poppet seat contact indicates that contaminants could have damaged the body seat and are responsible for the higher internal leak. Future designs may consider a downstream filter to trap back flow contaminants and/or exercise care in system blowdown procedure to assure complete pressure relief on the downstream side.

Propellant Tanks: The fuel tank post-sterilization functional test consisted of an expulsion test at minus 1g conditions and an external leakage test. Inasmuch as the fuel tank had been subjected to sterilization testing in the inverted position, a partial expulsion was first made in the plus 1g orientation in order to fill the propellant trap. Subsequent attempts to perform minus 1g expulsions resulted in expulsion of only a portion of the capacity of the trap. Furthermore, the effluent was a mixture of gas and liquid throughout the expulsion sequence. The quantity of fuel expelled during the various expulsion attempts responded to the method used to fill the trap. Since the intent of the post-sterilization functional test was to assess heat-induced

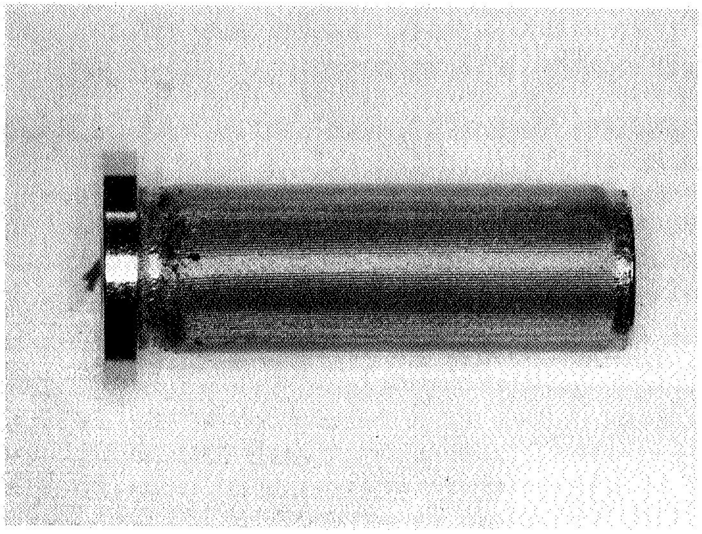


Fig. 18 Regulator Internal Filter

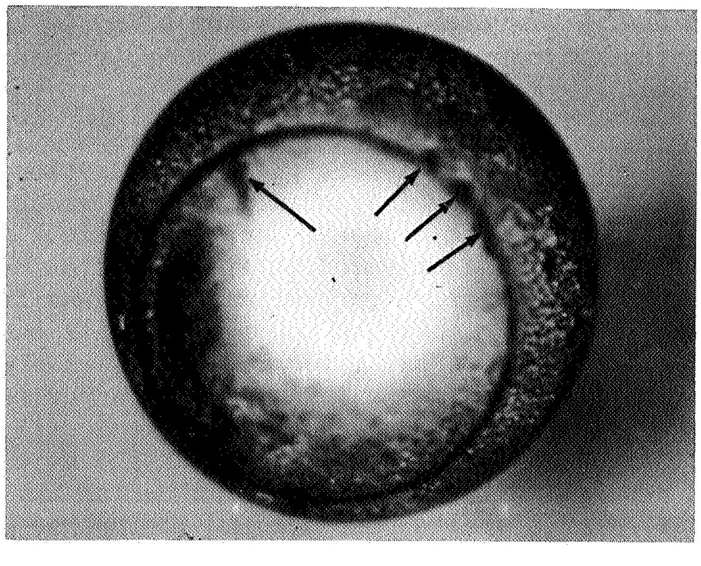


Fig. 19 Regulator Poppet Ball

degradation, the results shown in Figure 20 represent the expulsions associated with the most effective trap filling technique. The 0.96 pound quantity expelled at minus 1g conditions was demonstrated after the tank had been in the minus 1g attitude for about 16 hours. Completely gas-free flow was not attained in any of the several minus 1g expulsion tests.

A sample of the MMH fuel was taken from the fuel tank test item after about half of the 50 pound load had been expelled. The laboratory analysis, shown in Table 1, indicated that no significant degradation of the fuel had taken place. The fuel was still water-white at the end of the Phase II sterilization testing, indicating that no oxidation had taken place.

After the initial attempts to obtain single phase liquid flow under minus 1g conditions, a short test program was initiated to accomplish two objectives. The first objective was to establish a tank fill technique which would assure filling of the trap so that minus 1g operation of the trap could be determined exclusive of sterilization effects. The second objective was to demonstrate initiation of positive 1g outflow without gas entrainment to assure proper engine operation during the module firing.

The initial tank fill had been accomplished by evacuating the tank in the upright position and allowing fuel to fill through the outlet port. This resulted in some initial fuel vapor caused by fuel flashing into the vacuum. Load was determined by weight checking the source vessel during tank fill. At the completion of fill only propellant liquid and vapor were in the tank resulting in a tank pressure of approximately 2 psia under ambient conditions. It is felt that this technique could result in fuel vapor being held in the trap with consequent trap performance degradation. The second technique used for filling involved overfill of the tank and subsequent drain back to the proper load. This sequence was accomplished by filling the tank under a 1 atmosphere blanket of nitrogen in the upright position until the tank was completely filled, i.e., liquid fuel flowed from the gas inlet port. At this time the tank was inverted and liquid was flowed into the gas inlet port until all bubbles from the liquid outlet port were removed. The tank was then rotated to the upright position and nitrogen was introduced at the gas inlet port to drain back to the correct load. The

Task II SUMMARY DATA SHEET

Component Name:	Propellant Tank
Part Numbers:	
Oxidizer:	Martin LAB 6002514-009 Pressure Systems, Inc. 80092
Fuel:	Martin LAB 6002514-019 Pressure Systems, Inc. 80092
Serial Numbers:	
Oxidizer:	S/N 0001
Fuel:	S/N 0001

	Pre-Sterilization	Mid-Sterilization	Post-Sterilization
I. Fuel Tank			
External Leakage			
Helium at 400 psig (scc/sec)	Zero	--	Zero
Hydrion Paper Indication (pH)	--	No basic indication	--
Expulsion, -1g			
Quantity Loaded - lbs	50.5	--	--
Quantity Expelled - lbs	--	--	0.96
II. Oxidizer Tank			
External Leakage			
Helium at 930 psig (scc/sec)	Zero	--	
Hydrion Paper Indication (pH)	--	Leakage at test fitting	Not tested
Internal Leakage, GN_2, 1 psid (cc/hr)	Zero	210 cc/min (He)	
Expulsion, -1g			
Quantity Loaded - lbs	80.7	--	--
Quantity Expelled - lbs	--	--	

Fig. 20 Performance Data, Propellant Tanks

Table 1

MMH Gas Chromatograph Analysis Results

MMH From the Task II Fuel Tank After Sterilization

LAB Report 67B2123

Component	Volume Fraction - %
Monomethyl Hydrazine	98.95
Nitrogen	0.12
Ammonia	0.09
Water	0.70
Unknown (methylamine?)	0.14

quantity drained was collected in a receiver vessel on a scale to determine proper drain back. As a check the propellant tank had been weighed empty and was again weighed after fill. Load quantity desired was 51.03 pounds and the amount loaded was 50.70 pounds which is well within the loading accuracy required.

Under this load condition the tank was outflowed in the upright position and flow was observed in a sight glass. No gas flow was noted and after a flow of approximately 10 pounds of fuel the tank was inverted and minus 1g outflow was attempted. Again a two-phase mixture was expelled with almost the same total liquid weight which had been expelled using the vacuum loading technique.

As a result of the special testing accomplished on the fuel tank during the completion of Phase II, it was decided that the overfill loading technique would be used on the module fuel tank. In this way single phase liquid outflow to the engine will be assured even though minus 1g outflow will not be attempted as a part of the firing sequence.

Upon completion of the expulsion tests, the fuel tank was decontaminated and then cut in two at the girth weld. After cleaning, the lower half containing the screen trap, see Figure 21, was subjected to a leak and bubble test. The weld joint proved to be intact and the first leak appeared at the outer row of rivets, where the titanium is riveted to the stainless steel trap, see arrows Figure 22. This leak started at 5 inches of H_2O pressure. The screen started bubbling at 8 inches of H_2O pressure. The indication here was both at the sandwich connection edge and through the screen.

The screen trap was then separated from the tank half and a hole drilled in the upper plate to permit gas pressure injection. The trap unit was then subjected to a bubble check. Leaks started at the closure plate riveted connection, see Figure 23, at 1½ inches of H_2O pressure.

The above test indicates that the screen trap was functioning properly although the bubble point was lower than when installed; therefore, some other factor was responsible for the two-phase flow indicated earlier.

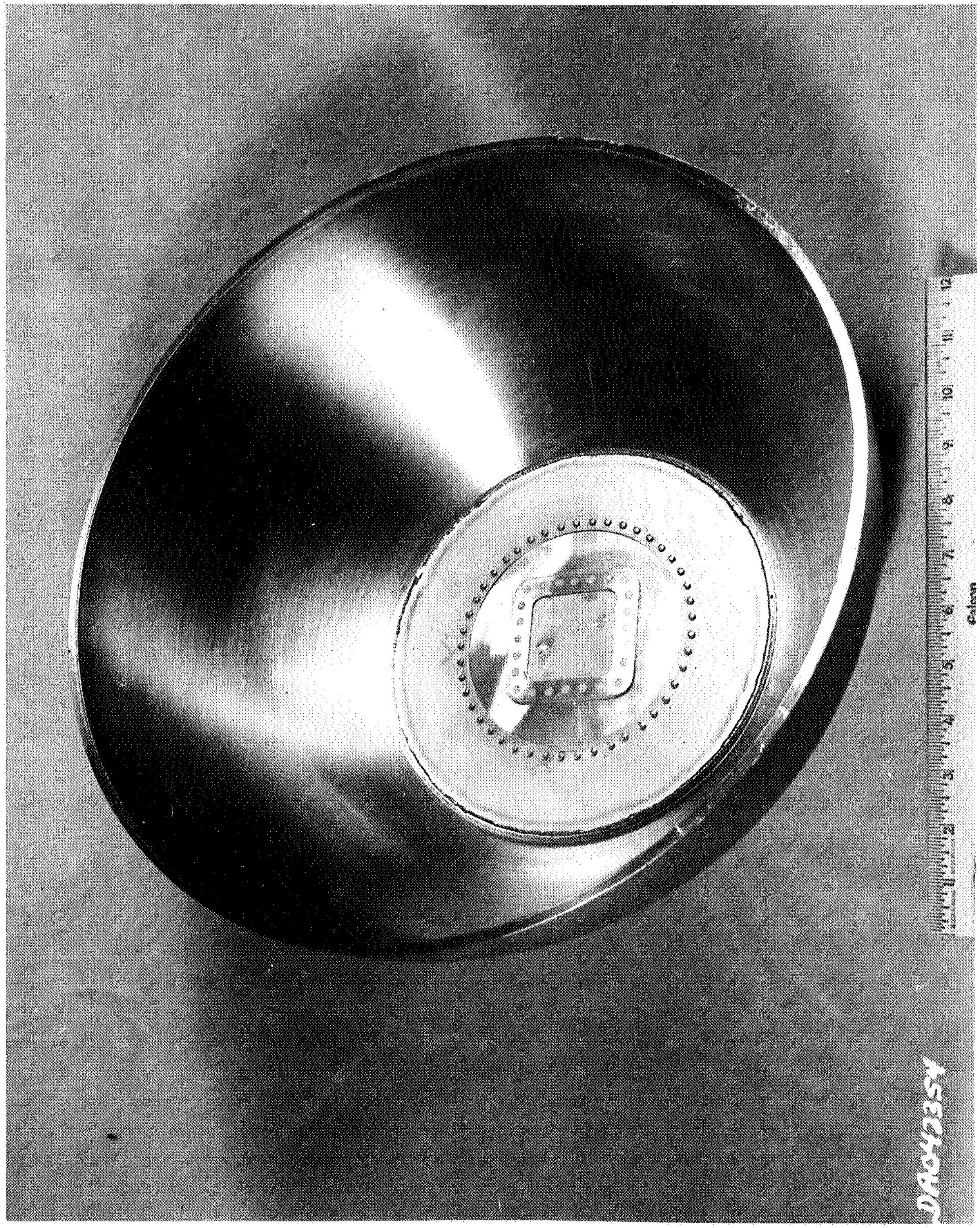


Figure 21 Fuel Tank Screen Trap Assembly

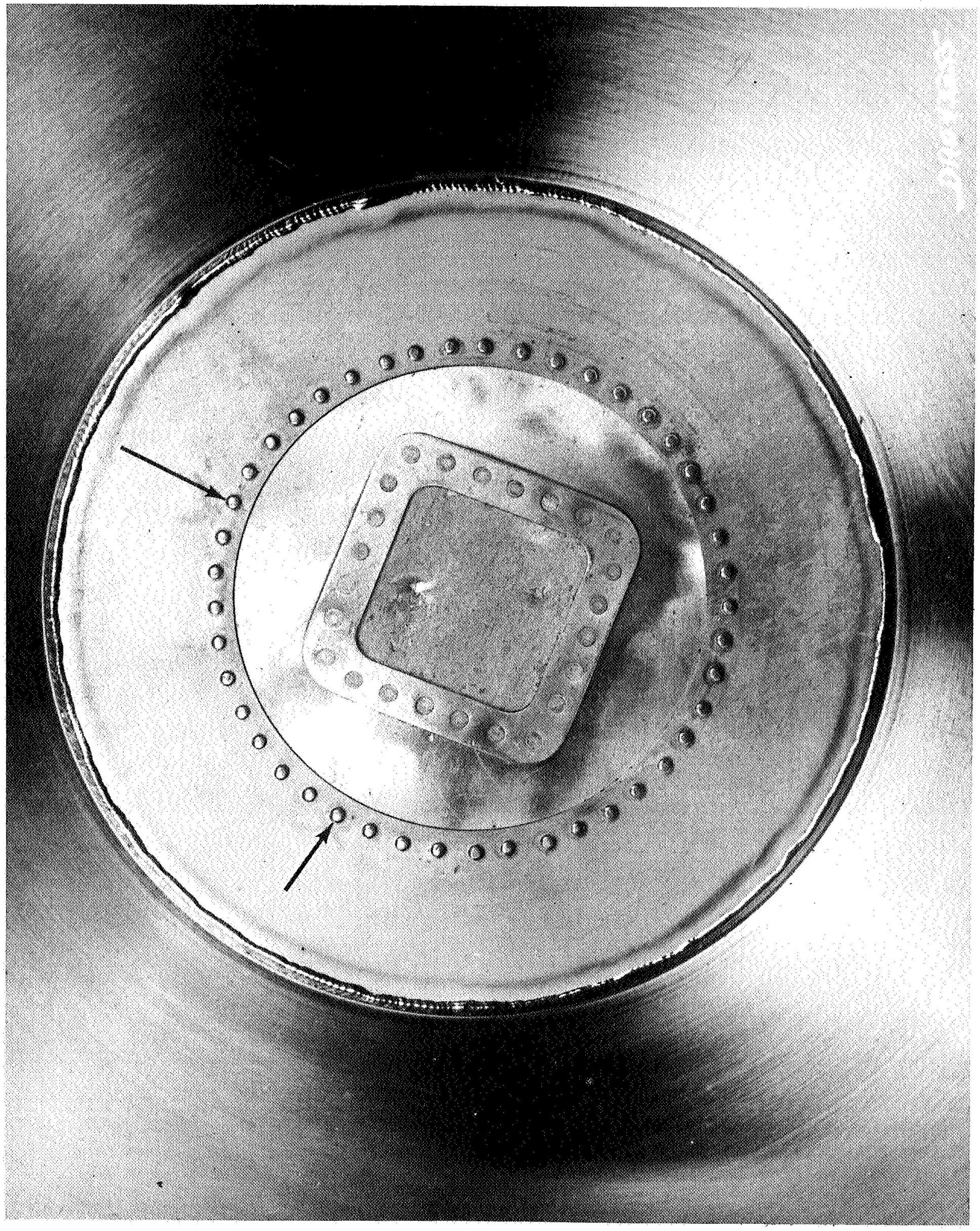


Figure 22 Fuel Tank Screen Trap Leakage Points

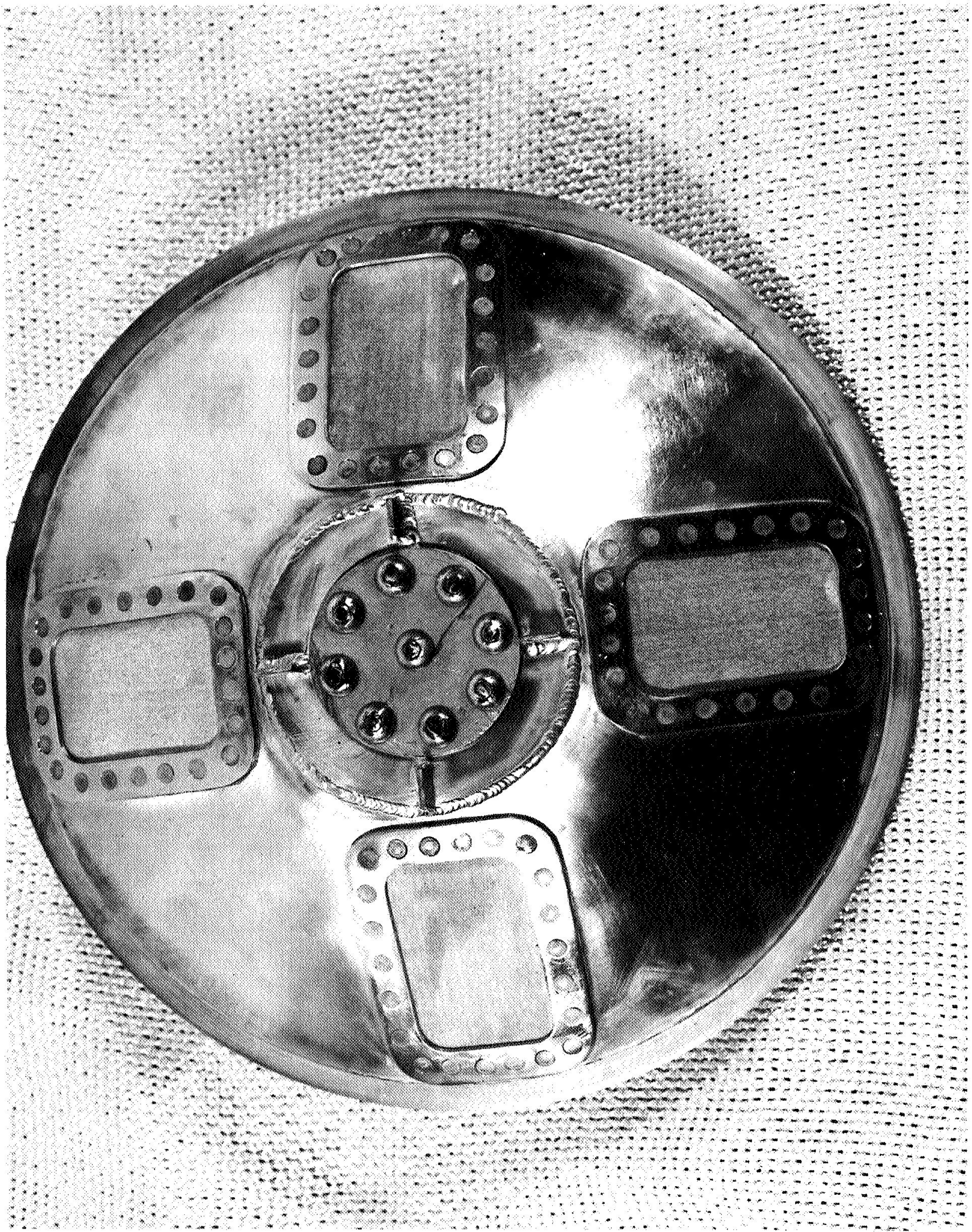


Figure 23 Screen Trap Assembly

An examination of the trap and tank assembly indicated that there was an area in suspect, namely, the flow area between the smaller diameter base of the cone frustrum and the tank wall. It appears that on fabrication, this area was smaller than had been anticipated due to shop tolerances. If this area should provide a restriction in excess of the bubble point of the trap closure plate rivets (1½" H_2O) then the two-phase flow would indeed occur.

To test this theory, accurate measurements were made of the tank and trap (see Appendix A) and calculation showed that the pressure drop at rated flow was equivalent to 2.663" H_2O pressure. Therefore, two-phase flow would occur due to a breakdown of the trap at the closure plate rivets.

C. PROPULSION MODULE ASSEMBLY AND TESTING

Assembly of the propulsion module was completed and leak checks performed to assure compliance with the design criteria. Following completion of leakage tests, the baseline functional tests were performed on the module solenoid (GN_2 loading) valve, the two thrust chamber valves and the pressure regulator as stipulated in the Test Plan. The results, Table 2, showed that the solenoid valve and the thrust chamber valves were performing satisfactorily. The pressure regulator exhibited excessive internal leakage and low pressure regulation. The leakage value was 68 scc/hr of GN_2 compared to the allowable value of 10 scc/hr. Further, the regulation pressure was 241-245 psig compared to the allowable of 248 ± 5 psig. The regulator was returned to the supplier for repair and adjustment so the sterilization exposure would be initiated with the regulation band in the required limits. The supplier's investigation revealed a scratch mark in the regulator valve seat, presumably the result of passage of a foreign particle. The regulator seat was repaired, and the regulation spring was heat-treated at 325°F for 24 hours at its working stress level to obviate the set-point degradation which had been exhibited by the component test regulator.

Upon receipt of the repaired regulator, it was found that the inlet tube had been indexed incorrectly; therefore, the inlet flange and tube assembly was removed in the Class 100 clean room and reinstalled in the correct position. Following installation in the module, the pre-test functional tests were conducted. The results of the functional test, shown in Table 2, showed that the regulation band was within specification limits; however, the internal leakage was again somewhat in excess of the allowable leakage (14.5 scc/hour, compared to the allowable 10 scc/hour).

Prior to the above-described installation and functional test, the facility GN_2 supply and exhaust interfaces at the module service panel were equipped with 5 micron nominal filters to protect the regulator from contaminant particles in the supply gas and also from possible back-flow of effluent GN_2.

The performance of the pressure regulator was judged adequate for the desired performance of the module; therefore, no further testing was done.

Table 2

Baseline Performance of Propulsion Module Components

	Maximum	Minimum
I. Thrust Chamber Valves		
A. Oxidizer Valve		
1. Opening Response (sec)	.0092	.0089
2. Closing Response (sec)	.0062	.0060
3. Leakage - External (bubbles GN_2)	Zero	
Internal (cc GN_2/hr)	Zero	
B. Fuel Valve		
1. Opening Response (sec)	.0073	.0070
2. Closing Response (sec)	.0070	.0062
3. Leakage at 250 psig:		
External (bubbles GN_2)	Zero	
Internal (cc GN_2/hr)	12	
II. GN_2 Loading Solenoid Valve		
A. Leakage, External (scc/sec Helium):	Zero	
B. Dielectric Strength (milliamps leakage)	0.006	
III. Regulator		
A. Internal Leakage (scc/hr GN_2)	14.5	
B. External Leakage (bubbles GN_2)	Zero	
C. Regulation:		
Inlet Pressure, Initial (psig)	1498	
Inlet Pressure, Final (psig)	350	
Average Flow Rate, GN_2 (lbs/sec)	0.015	
Outlet Pressure Variation (psig)	250	247
D. Hysteresis:		
Initial Outlet Lock-Up Pressure (psig)	263	
Outlet Pressure Range (psig)	250	247
E. Response:		
Inlet Pressure, Average (psig)	1520	
Outlet Pressure, Lock-up (psig)	261	
Overshoot (psig)	0	

Propulsion Module Preparation

Following the installation of the pressure regulator and completion of functional tests, the propulsion module propellant tanks were loaded. The oxidizer tank was loaded by the evacuation method used in Phase II. The fuel tank was loaded to 51.9 pounds of MMH by the overfill technique developed during the Phase II expulsion tests. The fuel tank loading was performed first since it involved rotating the module. The oxidizer tank was then loaded to 84.5 pounds of N_2O_4 with the module in the upright position.

Upon completion of propellant tank loading, the propulsion module was installed in the decontamination chamber lid as shown in the photographs of Figures 24 and 25. The module nitrogen tank was then loaded with GN_2 to a stabilized pressure of 1550 psig. X-ray photographs of the propellant tanks were then taken to verify propellant levels.

During the final inspection of the module prior to placing it in the decontamination chamber, a slight leakage of oxidizer vapor was detected at the inlet flange of the oxidizer tank propellant outlet ordnance valve. A torque check of the three 8-32 capscrews in the inlet flange revealed that the torque had relaxed from the original assembly value of 30 inch-pounds. An inspection was made of the other four valve inlet flanges. It was found that although the rest of the inlet flanges were leak free, and the capscrews were still tight, the flange faces were not butted together. In order to achieve the desired condition of butted flanges, a higher torque value of 40 inch-pounds was established; however, the stress so imposed on the threads in the aluminum valve flange were calculated to be near the yield region. In order to provide an adequate safety margin, the 1/2-inch long machine screws were replaced with 3/4-inch screws in order to permit installation of a back-up nut.

The above-described changes to the ordnance valve inlet flange fasteners were accomplished without perturbing the propellant tanks. For safety reasons, the nitrogen tank pressure was reduced to zero prior to changing the valve flange fasteners.

Concurrent with installation of the new fastener configuration, the Phase II components test valve was proof pressure tested and leak checked with the same flange fastener configuration using the new torque value of 40 inch-pounds. These tests confirmed the integrity of the configuration. As an additional check, the propulsion module fuel tank was pressurized to operating pressure, and the ordnance valve flanges were checked for

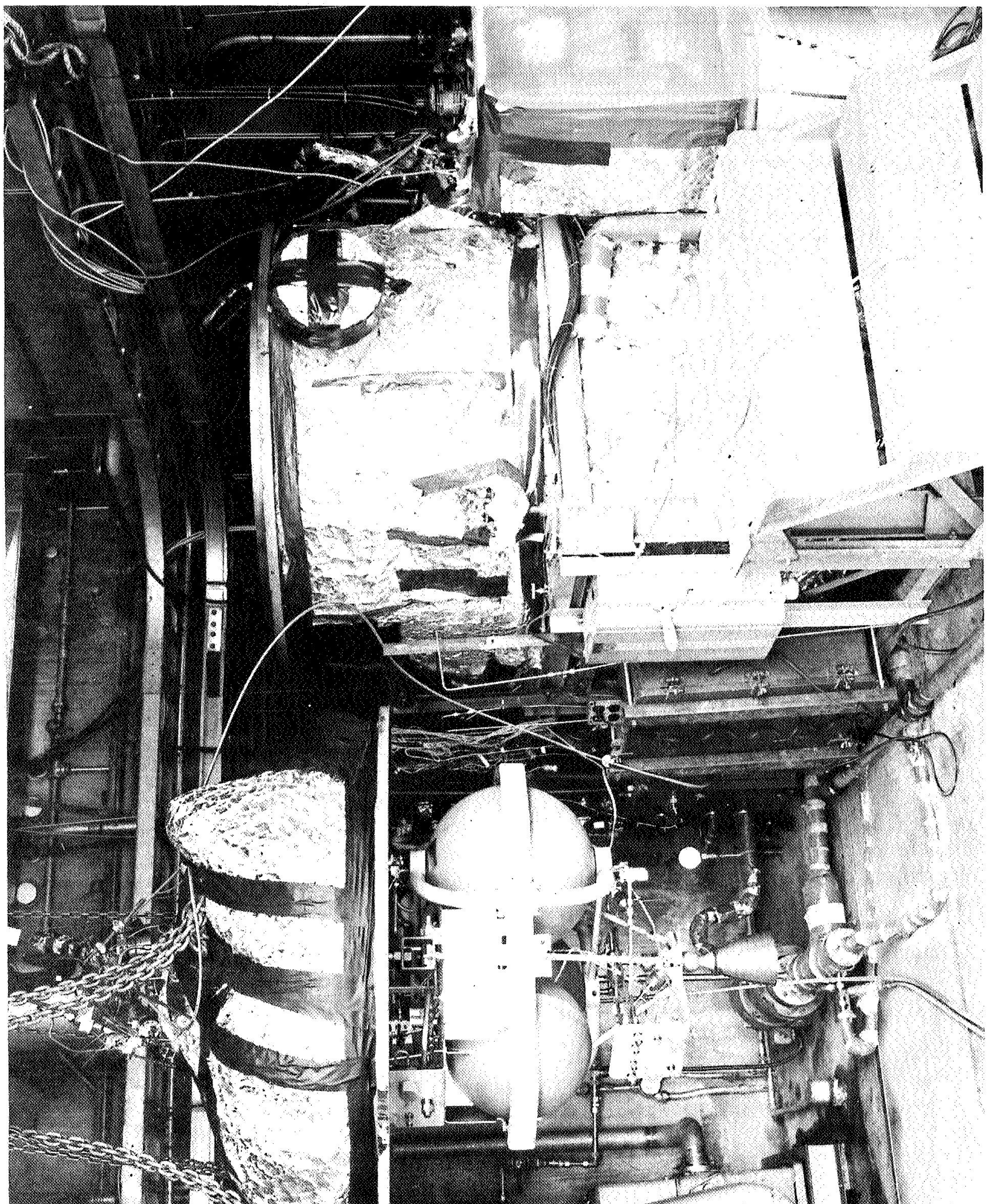


Figure 24 Assembled Module and Chamber

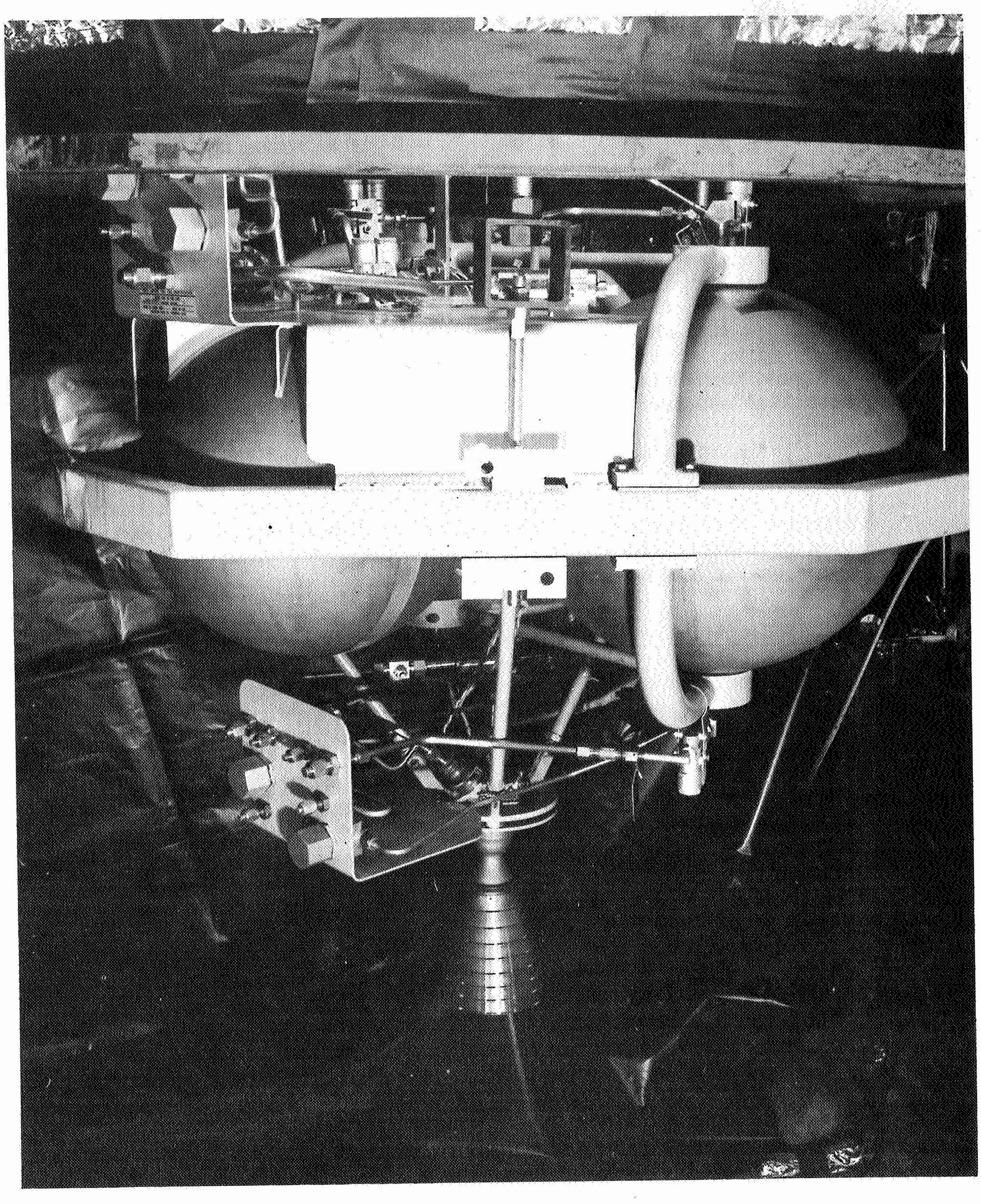


Figure 25 Module Attached to Chamber Lid

leakage with a fuel vapor detector. The oxidizer system was also checked after heating the tank to produce an oxidizer pressure of 100 psig. The latter method of pressurization was used in order to avoid flexing the expulsion bladder. No leaks were detected in the oxidizer system.

As the final item of preparation of the module for decontamination and sterilization testing, the module hand shutoff valves were equipped with the stem cap seals previously described, and the stem lockscrew holes were filled on the outside of the lockscrew with the Devcon WR epoxy resin. After opening the required valves to admit the propellant tank pressures to the pressure transducers and safety package, the stem caps were installed and torqued to the 90 inch-pound specification value.

The installation of the propulsion module in the decontamination/sterilization chamber is shown schematically in Figure 26. It will be noted that the module fuel tank was connected in such a manner that the tank could be pressure drained through operation of remotely-controlled valves in the event of uncontrolled decomposition of the MMH.

Module Decontamination Tests

The required six ETO decontamination tests were completed in this report period. Each of the tests was run according to the procedure described in the Test Plan, consisting of 1.5-hour heating and humidifying phase; chamber evacuation and pressurization to 22 psia with sterilant gas (Oxyfume-12); re-humidification to 55 ± 5% relative humidity and commencement of the 26-hour constant exposure period of 122°F chamber temperature. Following the completion of the 26-hour exposure period, the 1.5-hour cooling phase was accomplished as described in the Test Plan. A brief description of each of the six tests completed to date is given below:

Decontamination Cycle No. 1: This test was started on 16 November, but was aborted shortly after commencement of the 26-hour exposure period, due to an inability to control chamber temperature. After a series of check runs, the problem of uncontrolled heating of the chamber was traced to Joule heating of the sterilant gas by the blower. In addition, problems were encountered in the relative humidity control and monitoring systems. The resolution of the above-mentioned problems is detailed in the Test Facilities section of this report.

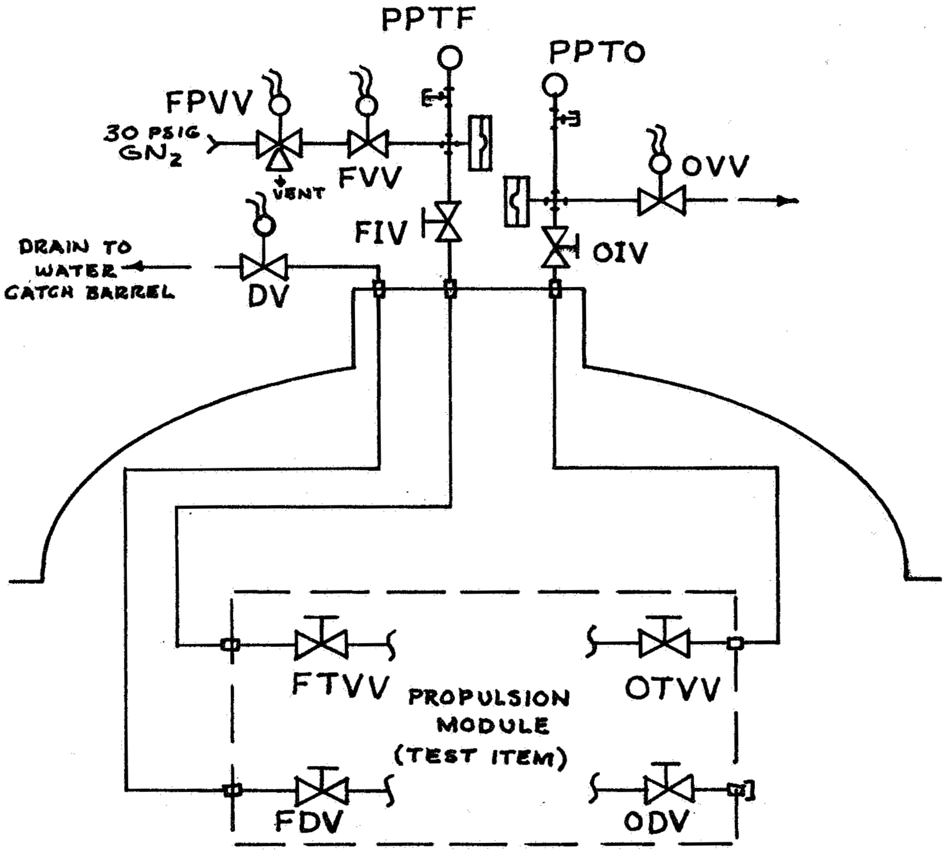


Figure 26 Decontamination and Sterilization Chamber Schematic

During the initial heating phase of the several attempts at Cycle Number 1, it became apparent that the 132°F water supply temperature to the chamber heat exchanger was not hot enough to maintain the required temperature ascent schedule. This was attributed to the thermal load created by the propulsion module, since the chamber heating system had performed satisfactorily with 132°F water during the qualification tests (no test item in chamber). In order to accommodate the thermal load of the module, the operating procedure was changed to permit operation of the chamber water supply system at 150°F during the initial 1.5-hour heating and humidity conditioning phase. Since that phase of the test does not involve the use of sterilant gas, the specification constraint of 132°F maximum heater temperature for heating a sterilant gas environment was not violated.

With the above changes, decontamination Test Number 1 proceeded without significant incident to a conclusion on 21 November. Some initial adjustment of the humidity control system was required to maintain the desired range of relative humidity (Alnor dew point). A data history of Cycle Number 1 is shown in Figure 27.

<u>Decontamination Cycle No. 2</u>: This test was started on 21 November, approximately 3 hours after the completion of Cycle Number 1. Due to a procedural oversight, the heating water temperature was maintained at 132°F instead of 150°F; therefore, the ascent to the desired chamber temperature of 122°F required 2.3 hours. The balance of the test was completed without significant incident. As noted in the data display of Figure 28, the chamber temperature rose slowly to a maximum temperature of 130°F during the latter part of the test. This temperature rise was due to having set the cold water bleed flow at too low a rate just prior to the period of unattended chamber operation from T + 17 hours to T + 25 hours (night shift).

Sterilant gas consumption due to chamber leakage remained in the range of 4 to 5 pounds per hour. Automatic control of humidity was maintained throughout the test, although the control set point was slightly higher than desired, as may be seen from the relative humidity history. A chamber gas sample was taken during the test; however, the sample container capacity proved to be inadequate for effective purging of the Orsat analyzer. A larger container was acquired after Cycle Number 2 had been completed.

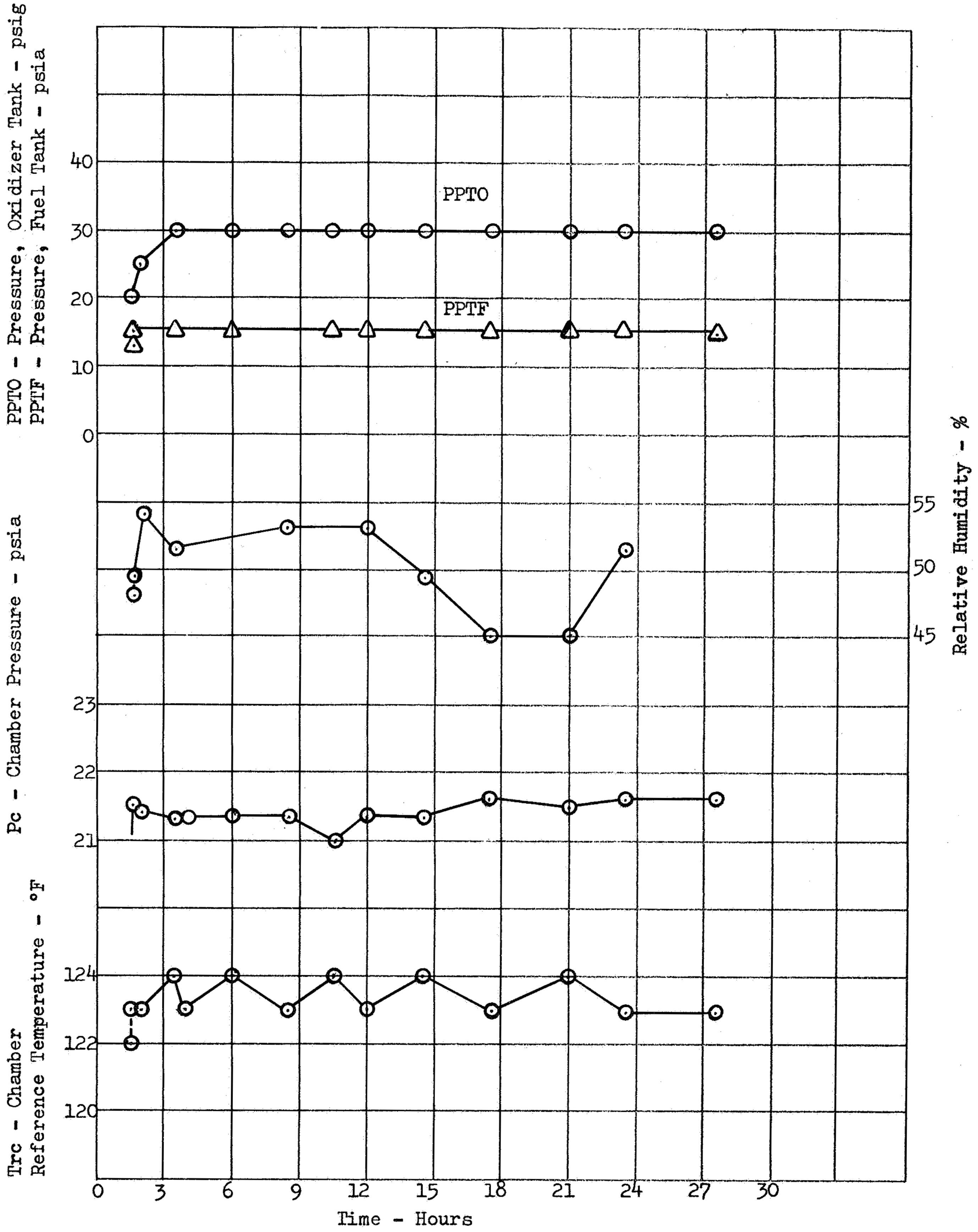
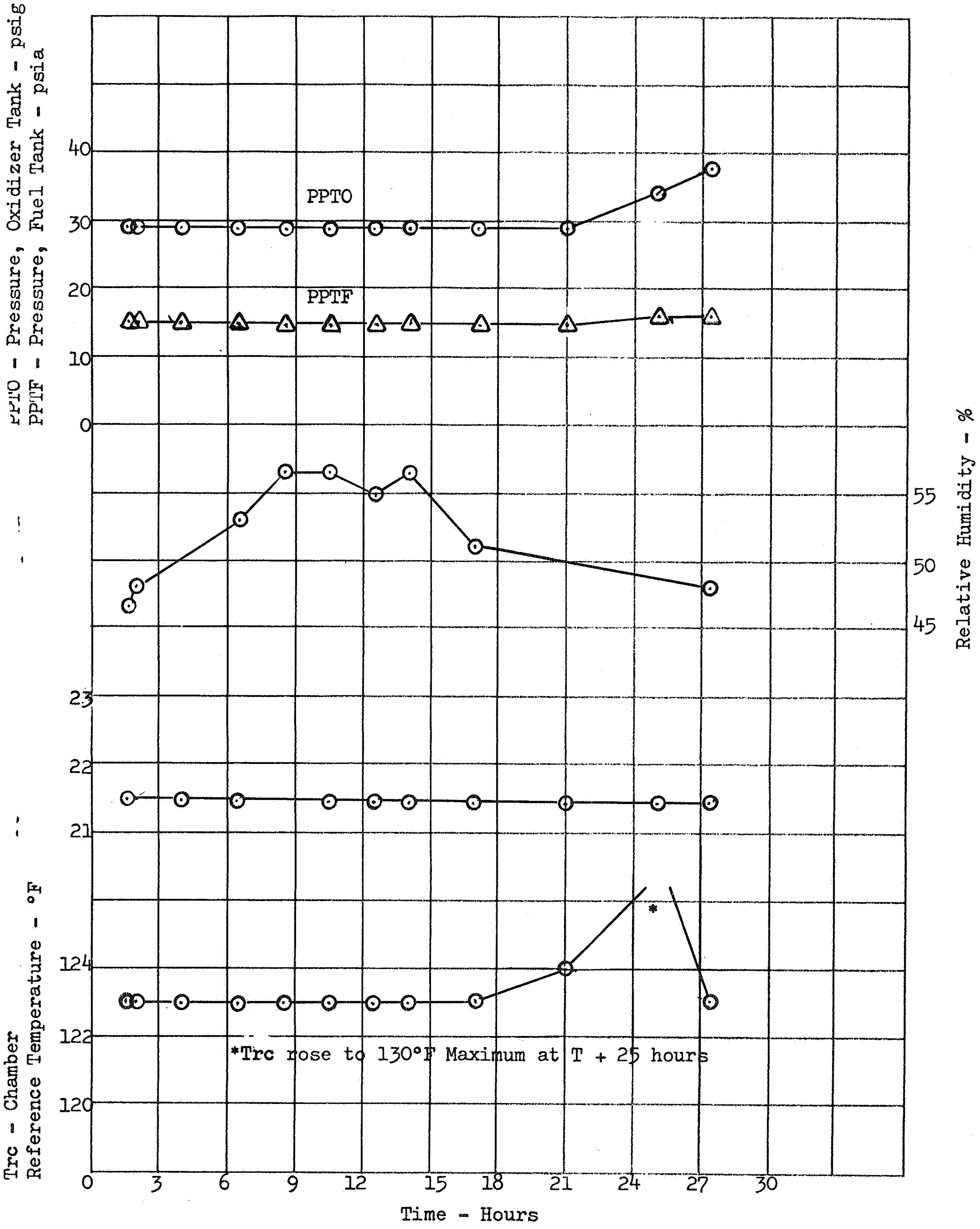


Figure 27 ETO Cycle No. 1 Test History



*Trc rose to 130°F Maximum at T + 25 hours

Figure 28 - ETO Cycle No. 2 Test History

Decontamination Cycle No. 3: This test was started on 27 November and completed on 28 November without incident. A sample of chamber gas was taken during the test. The sample assayed at only 18% by volume ethylene oxide (ETO); however, investigation showed that the sample temperature was such that the vapor pressure of ETO was very close to the 22 psia pressure of the sample. In order to obtain assurance that the ETO fraction was in the vapor state, it was decided to heat subsequent samples to 120°F prior to conducting the Orsat analysis. The data from Cycle Number 3 are shown in Figure 29.

Decontamination Cycle No. 4: This test was started on 29 November and completed without incident on 30 November. A chamber gas sample was taken and assayed at 26.5% ETO content by volume, compared to the specification value of 27.3%. Data from this run are shown in Figure 30.

Decontamination Cycle No. 5: This test was started on 4 December and completed without incident on 5 December. A chamber gas sample was taken and assayed at 26% ETO content by volume, compared to the specification value of 27.3%. Data from this test are shown on Figure 31.

Decontamination Cycle No. 6: This final test of the ETO decontamination series was started on 6 December and completed without incident on 7 December. Data from this test are shown in Figure 32.

Module Sterilization Tests

Upon completion of the decontamination test series, the propulsion module was removed from the chamber to permit changeover of the chamber to the heat sterilization configuration. The module remained mounted in the chamber lid and all connections from the module to the pressure transducers and safety packages remained undisturbed.

Prior to starting the required series of six heat sterilization tests, the pressure in the module GN_2 storage tank was checked since the pressure is not monitored during decontamination or sterilization testing. The pressure in the GN_2 tank was found to have decayed from the original 1550 psig to 1120 psig over the 23-day decontamination test period. The GN_2 leakage was traced to internal leakage through the transducer isolation

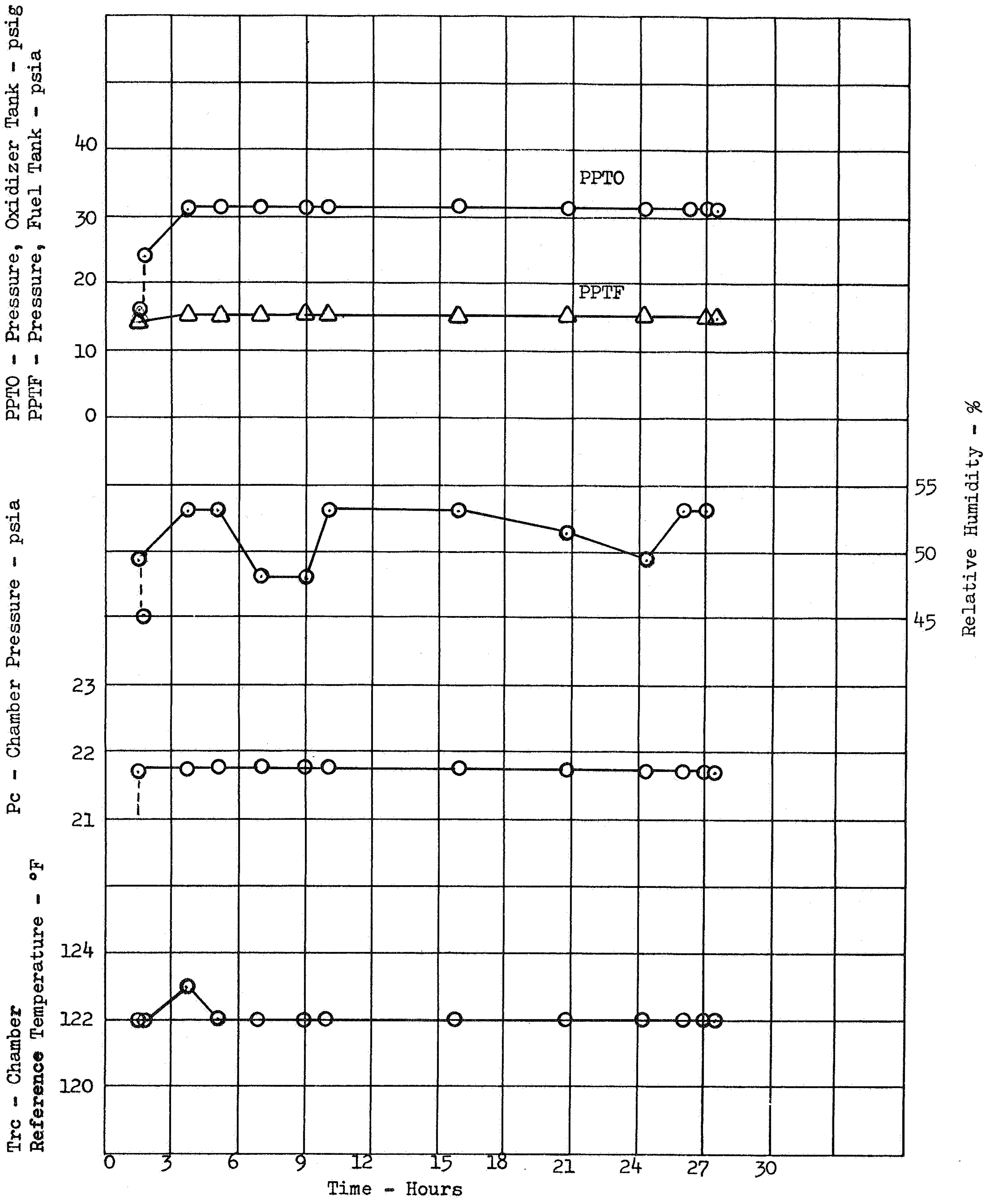


Figure 29 - ETO Cycle No. 3 Test History

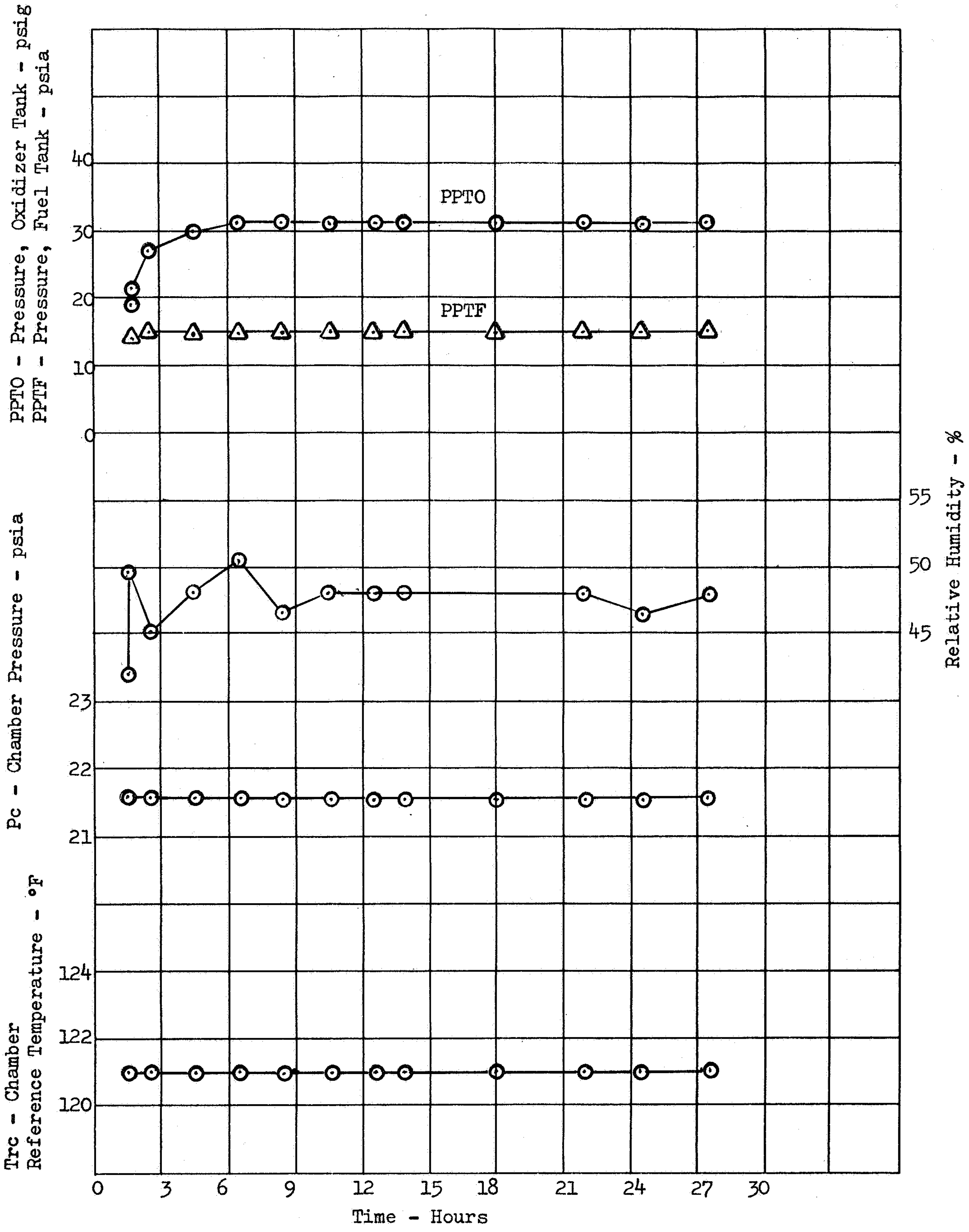


Figure 30 - ETO Cycle No. 4 Test History

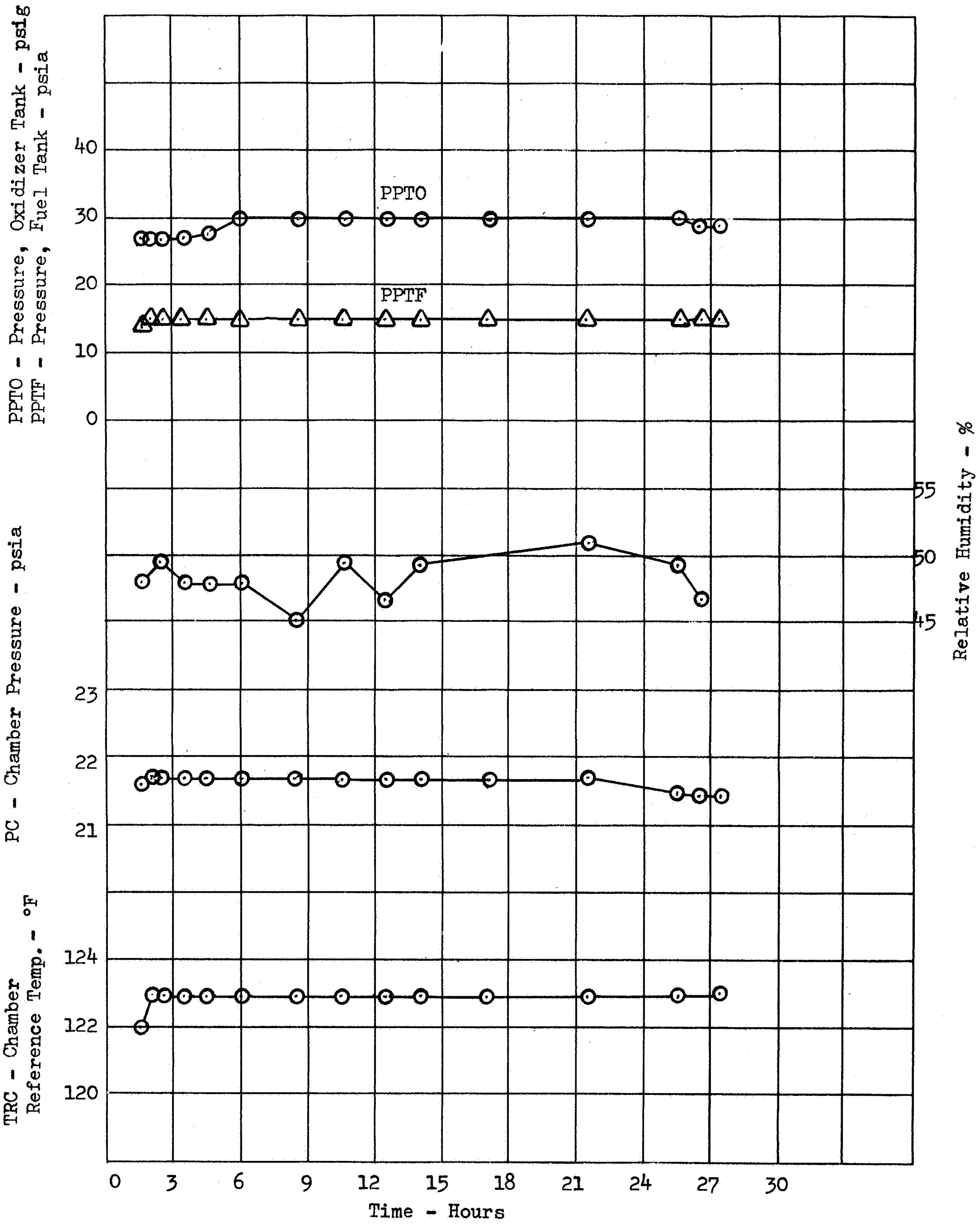


Fig. 31 ETO Cycle No. 5 Test History

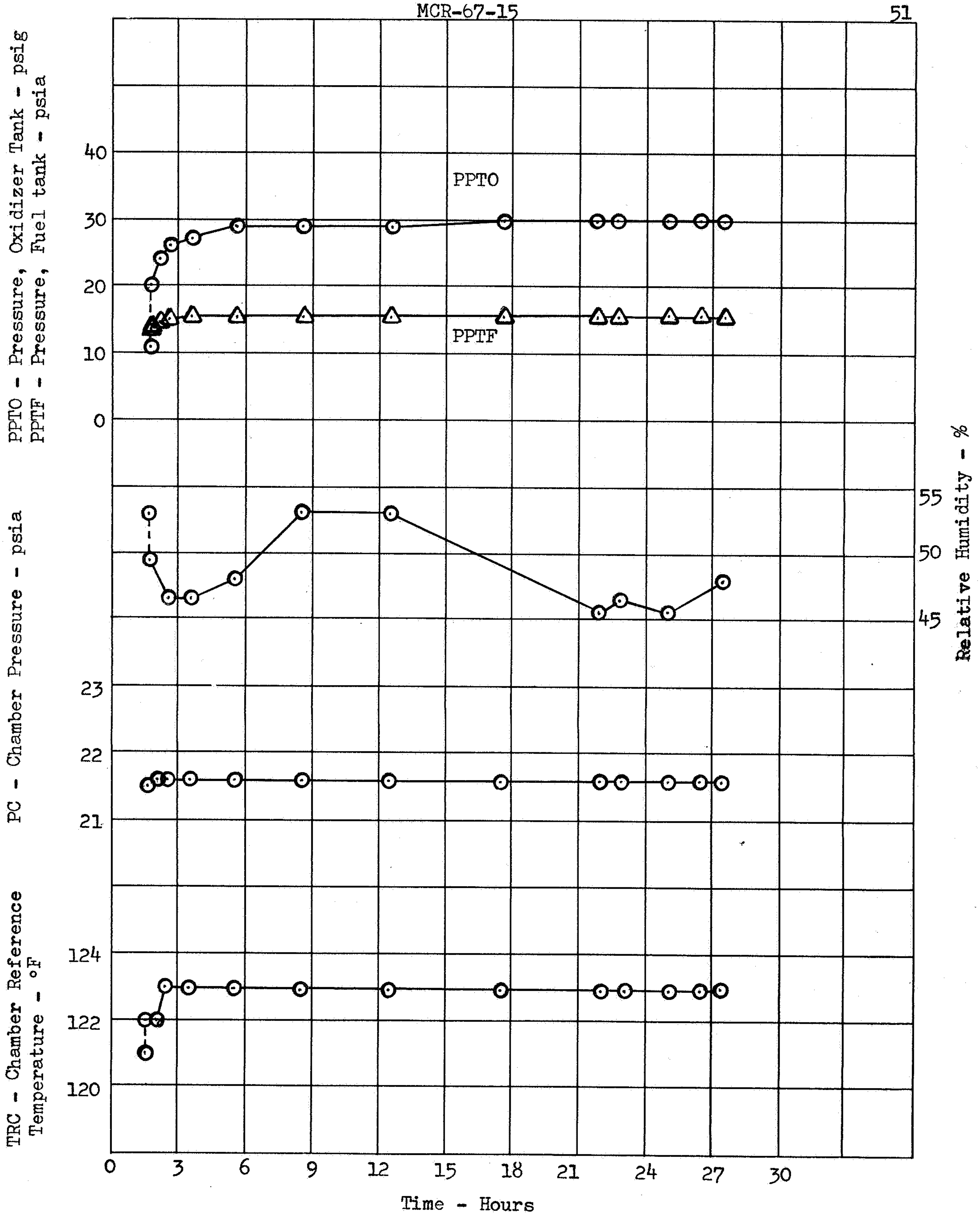


Fig. 32 ETO Cycle No. 6 Test History

valve (test equipment) which had been installed to permit installation of a pressure transducer for the firing test after the decontamination and sterilization tests. It was decided that the module GN_2 loading (solenoid) valve inlet port would be a suitable point to use for pressure measurement during the firing test; therefore, the transducer isolation valve and its connecting flared tubing were removed from the module. The module GN_2 tank was recharged to 1500 psig with GN_2, then topped off to 1550 psig with helium as an aid in locating the source of any future leakage.

Four of the required six heat sterilization tests on the propulsion module were completed in this reporting period. Histories of the chamber temperature and propellant tank pressures for sterilization Cycles 1 through 4 are shown in Figures 33 through 36, for the constant-temperature exposure period. All cycles were completed without significant incident with the exception of an automatic chamber shutdown during the latter part of Cycle No. 2 which was caused by a spurious signal from the chamber fuel vapor detector. Cycle time was resumed at T + 67.5 hours after reheating the chamber and permitting the propellant vapor pressures to attain pre-shutdown values.

Pressure in the module GN_2 tank was checked at 1540 psig after completion of sterilization Cycle No. 3 with the tank at room temperature, thus indicating that the GN_2 system was leak free.

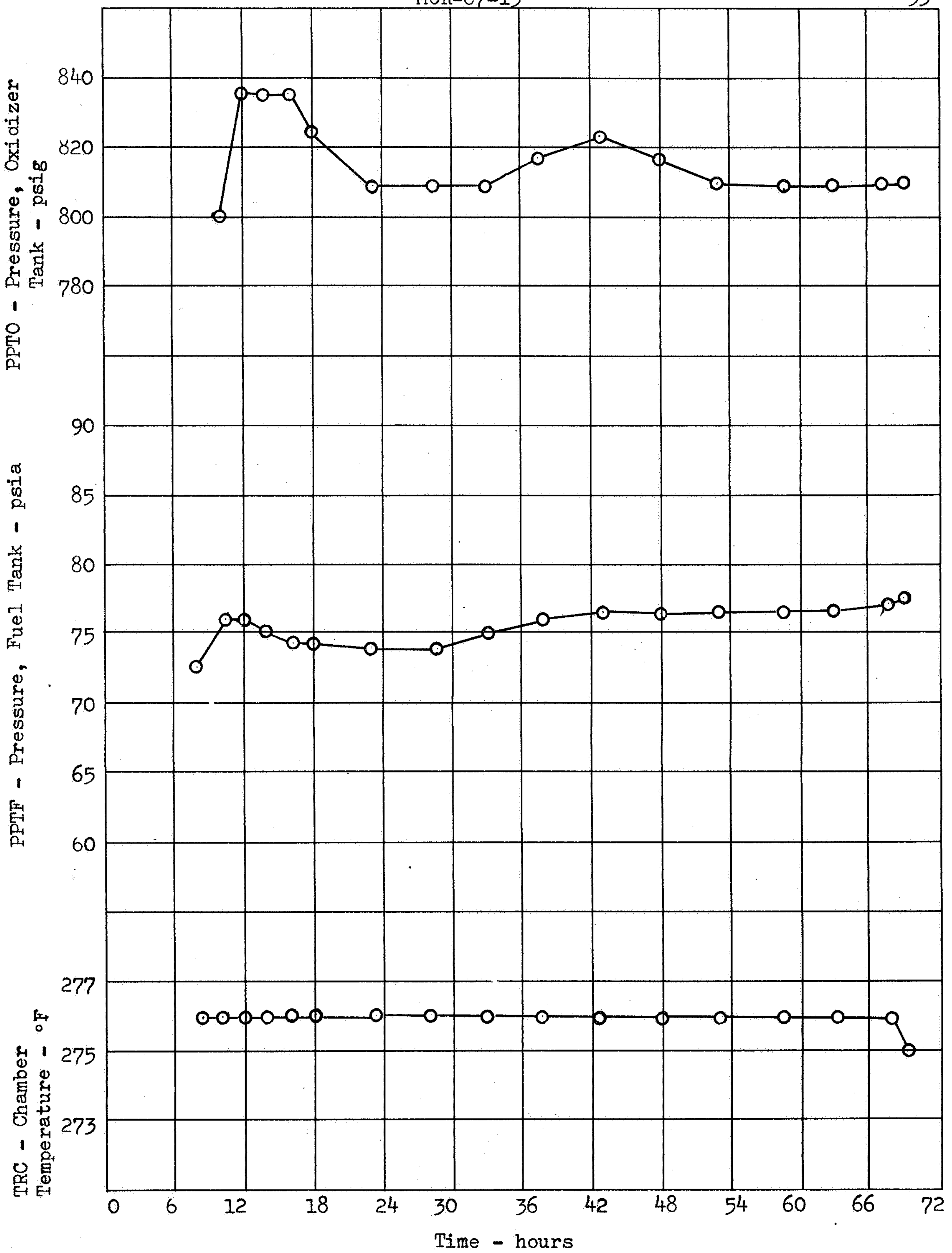


Fig. 33 Module Sterilization Cycle No. 1

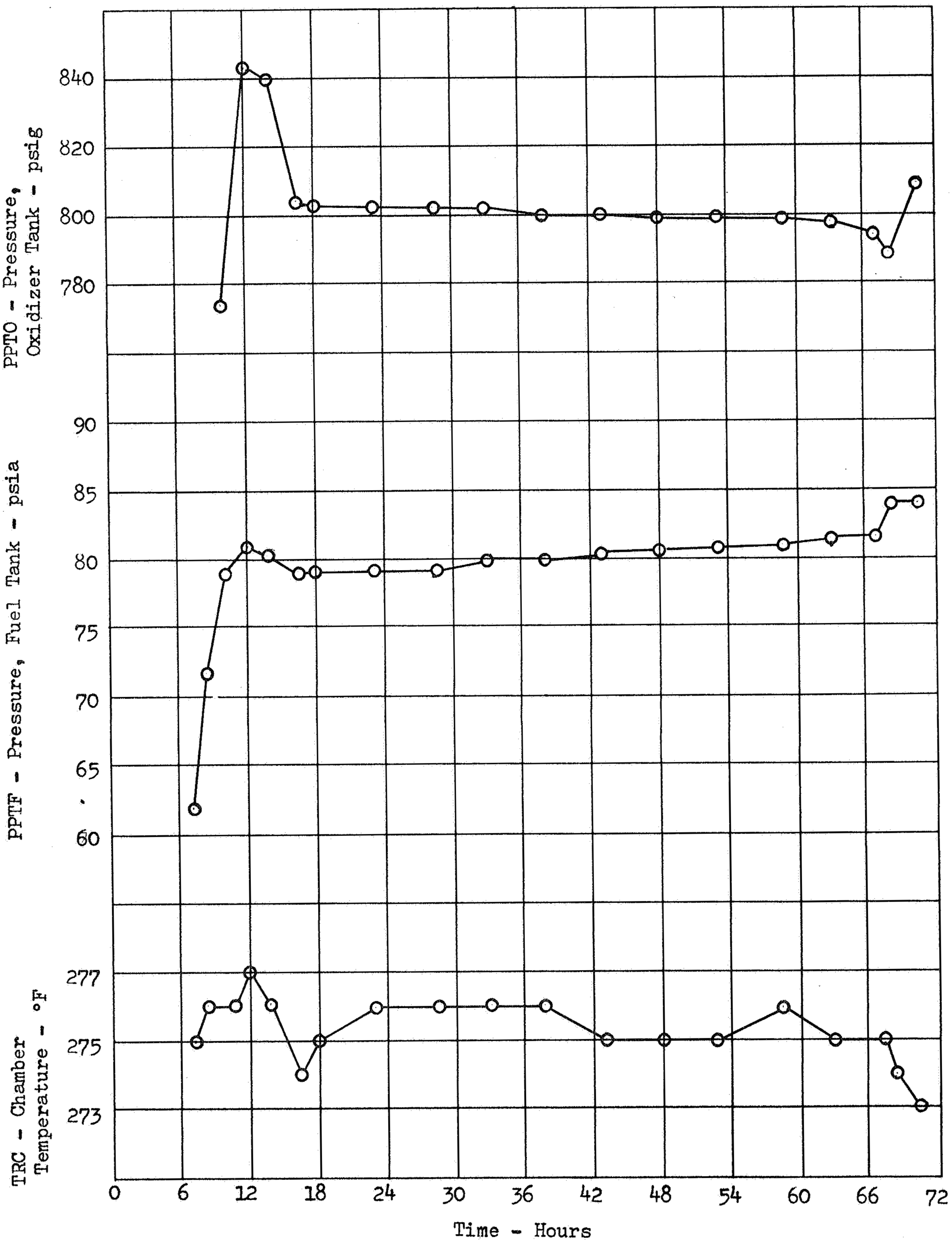


Fig. 34 Module Sterilization Cycle No. 2

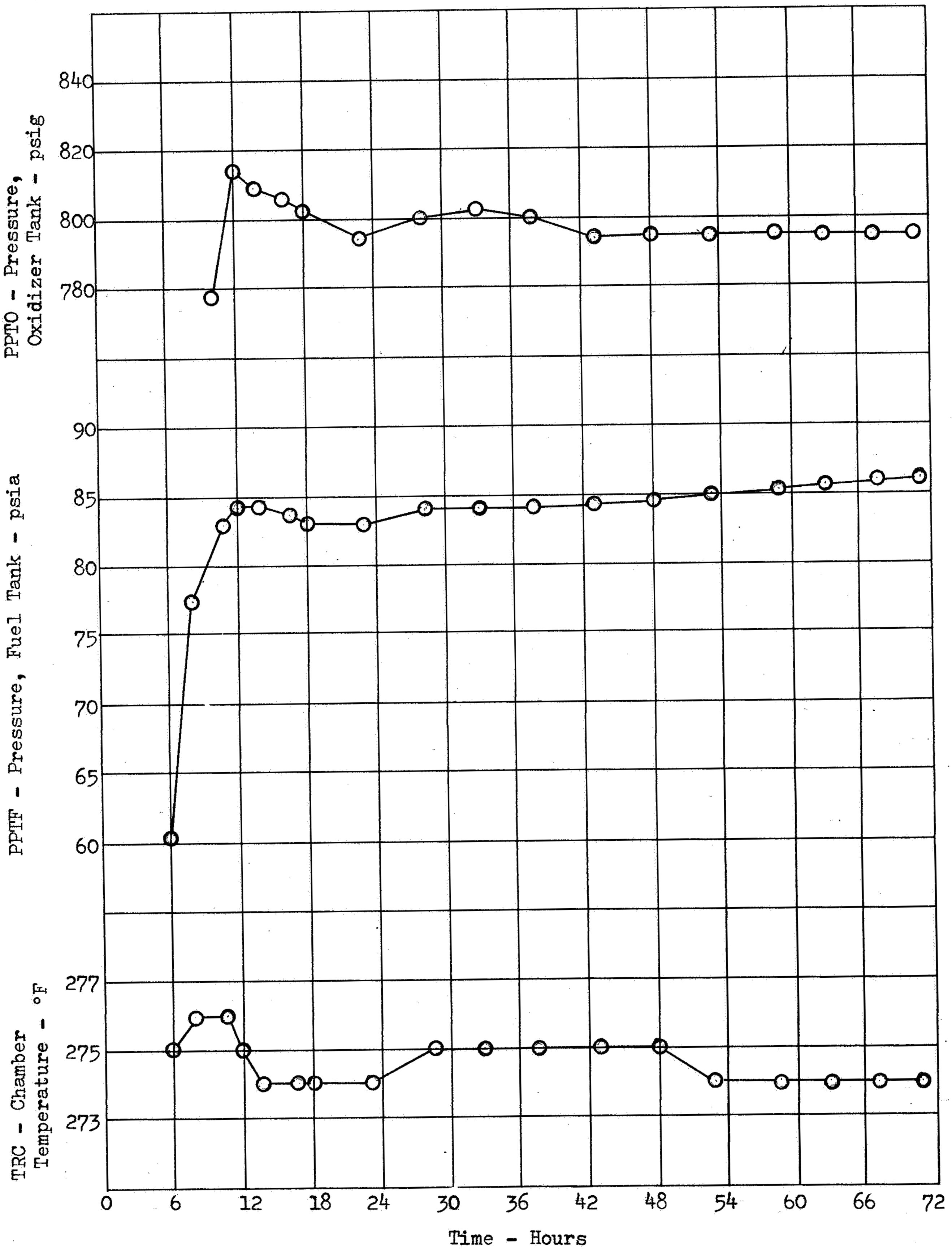


Fig. 35 Module Sterilization Cycle No. 3

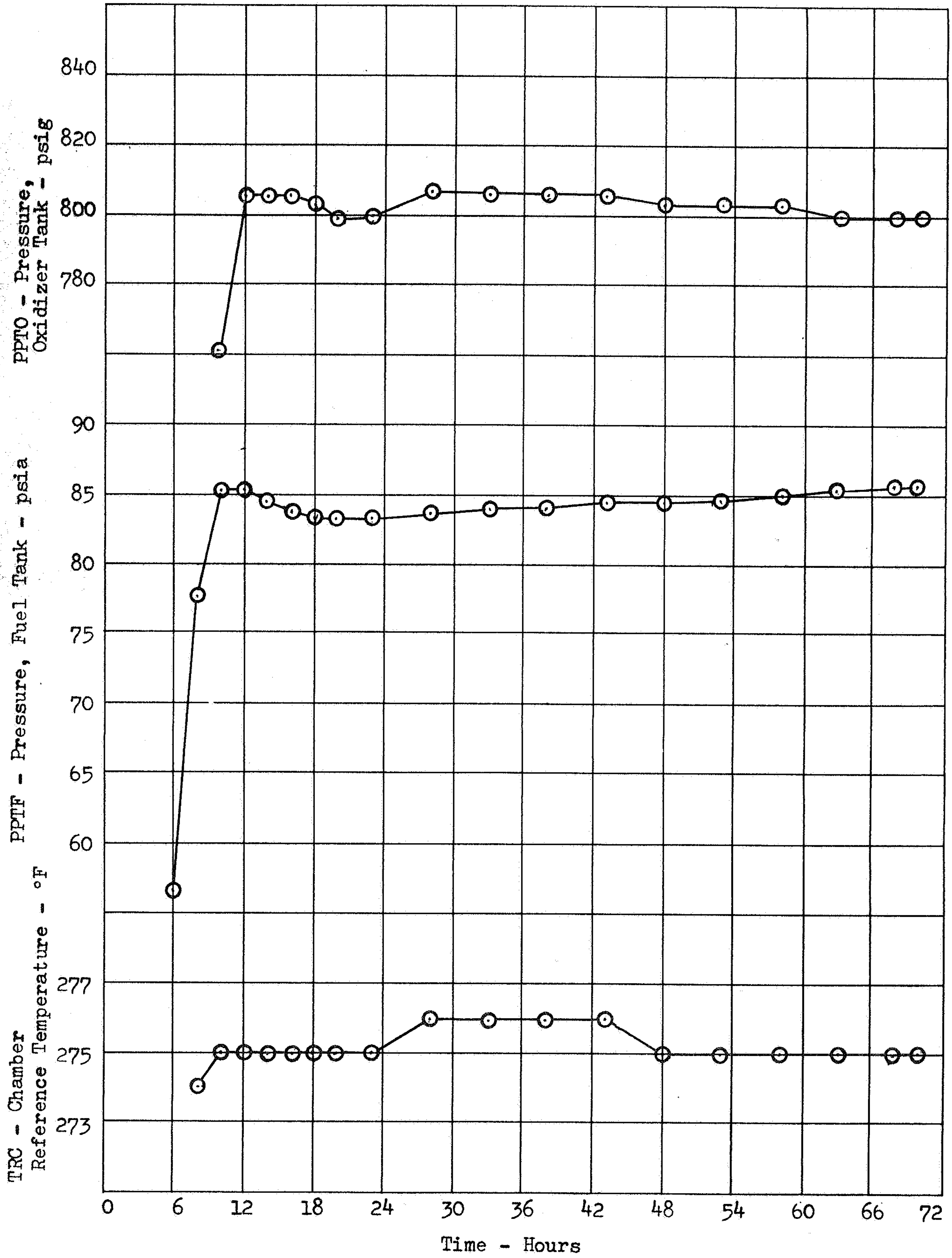


Fig. 36 Module Sterilization Cycle No. 4

D. TEST FACILITIES

Build-up of the test fixtures and electrical controls in the firing test cell has been in progress, with the major portion of the work having been completed at the end of the quarter. Installation of the propulsion module is scheduled for early January, 1968, immediately following completion of the remaining two heat sterilization cycles.

During this reporting period, several problems arose relative to the operation of the ETO decontamination chamber, particularly in the area of relative humidity control. The nature of these problems, and the resolution of the same, are detailed in the sections below.

Decontamination Chamber Temperature Control

A problem involving temperature control of the chamber was encountered at the beginning of the first decontamination test. The problem evidenced itself in a slow increase in chamber temperature at a time when no heating water was being admitted to the chamber. The possible causes of the phenomenon were initially thought to be an exothermic reaction of the ETO with the water vapor, a reaction between the ETO and possible undetected propellant vapor leaking from the module, the heat load introduced with the 132°F sterilant gas make-up, or the Joule heating by the chamber blower.

A systematic series of tests were made to obtain an explanation of the uncontrolled temperature increase. The series culminated in a test in which all chamber conditions were representative of a decontamination test, except the blower was turned off for an extended period. The chamber temperature decayed slowly during this test, indicating that the dissipation of the blower energy was responsible for the temperature increase.

Although no rigorous analysis has been conducted at this time, the explanation of this phenomenon is credited to the significant increase in blower horsepower when circulating the relatively dense sterilant gas and the relatively low heat capacity of the sterilant gas. The phenomenon was not observed when operating with nitrogen gas in the chamber.

The above-described problem was resolved by providing a continuous low flow rate of tap water (approximately 60°F temperature) into the chamber heat exchanger to extract heat at a slightly greater rate than that added by the blower in order to provide positive cooling to the extent that the hot water control valve would be required to open periodically. This system has the adverse characteristic of cooling a fraction of the chamber heat exchanger below the dew point causing cyclic condensation and re-evaporation of water. This cyclic phenomenon was detected by observation of an abrupt increase in the output of the sensitive electro-hygrometer cell upon opening of the hot water admission valve; however, Alnor dew point readings taken during the occurrences of this phenomenon proved that the relative humidity excursions did not exceed the desired band width of 45% to 55%.

Future control systems for the water supply system to the heat exchanger should incorporate a mixing control valve which would provide water temperatures at the heat exchanger which would range between the maximum of 132°F (55°C) and a minimum which is a safe margin above the dew point of the chamber gas.

Chamber Humidity Control

The humidity control system initially was conceived as an essentially open-loop system consisting of a super-heated steam generator and two visually-monitored humidity sensing systems. The prime measurement system for humidity is an Alnor dew pointer which is installed next to the decontamination chamber in an enclosure which maintains the Alnor instrument at a temperature approximately equal to that of the chamber to prevent condensation of the water vapor in the chamber gas. Since the Alnor instrument is not a continuous readout device and has no output signal to use for recording and control, a secondary humidity sensing system is installed in the chamber. The secondary system is an electrical hygrometer manufactured by Hygrodynamics, Inc., and consists of a lithium chloride cell, the resistance of which responds to temperature and water vapor content and an electrical control box which has an output which is used to drive a strip-chart recorder.

The feasibility of utilizing the electro-hygrometer for more facile monitoring of relative humidity was established in Phase II through the results of pilot tests. The test results showed that, although the output of the hygrometer cell was affected by the presence of sterilant gas, the output could be correlated with the true relative humidity as determined with the Alnor dew pointer. Furthermore, the test results indicated that the effect of sterilant gas on the sensing cell was not cumulative, i.e., there was no change with exposure time. It was, therefore, concluded that the electrohygrometer could be used as a relative measure of humidity conditions in the chamber after establishing the required conditions on the basis of Alnor dew point data.

Difficulties encountered during the attempts at the first decontamination test demonstrated that, due to normal chamber leakage and possible hydrolysis of the ethylene oxide, frequent water vapor injection was required. It became apparent that the open-loop system was feasible only if an operator was constantly monitoring the hygrometer output since attempts at steady-state injection of water vapor were unsuccessful. During these attempts, however, sufficient correlation between the recorded output of the electro-hygrometer and the Alnor dew point readings was obtained to permit installation of a control microswitch on the hygrometer output recorder. The function of the microswitch was to open the superheated steam injection valve whenever the hygrometer output dropped to mid-scale on the recorder and to reclose the valve as soon as the hygrometer output signal responded with an increase in signal on the recorder. A metering valve was installed in the steam injection line to control the time constant of the system to prevent excessive excursions in relative humidity.

The automatic control of relative humidity which was evolved in the above manner was proven to be entirely satisfactory through tests two through six. Humidity control is completely automatic; however, Alnor dew point readings have been taken at 1 hour to 2 hour intervals during the day and evening work shifts. Alnor checks made on the mornings following unattended night operation have verified that the hygrometer recorder/controller has been effective in maintaining the relative humidity within specification limits with no operator attention.

The existing electro-hygrometer system is being operated considerably off its design point due to the significant change in the resistance of the cell caused by the sterilant gas (resistance on the order of 20,000 ohms, as opposed to the normal resistance of approximately 2 megohms. This condition caused the output of the system to be more sensitive than would be desired; however, future systems could be biased electrically to provide optimum operation with the known, specified chamber environment. At present the recorded output of the electro-hygrometer is meaningful only as a relative indicator of the constancy at which the chamber relative humidity is being maintained, and its primary function is to control the steam admission valve. It is strongly suspected that the electro-hygrometer output also responds to ethylene oxide concentration; however, future investigations with an optimized system will be required to fully establish the characteristics.

E. LONG TERM STORAGE TESTING

During the report period the first set of long term storage tanks were opened for inspection.

It has been observed that titanium exposed to N_2O_4 for extended periods of time shows evidence of discoloration very similar to heat treat scale in color. One titanium assembly, which consisted of a welded Ti-6Al-4V tube approximately one inch diameter and 12" long with welded end caps, and a specimen stressed to 50,000 psi was inspected after exposure to N_2O_4 (inhibited with NO) at 275°F for 600 hours with a continued uninterrupted exposure to the same N_2O_4 for 128 days at ambient temperature.

Figure 37 shows the sectioned tube assembly and the stressed specimen. The spiral shaped specimen shown in Figure 37 is teflon which was also present in the titanium assembly during test. Figures 38 and 39 show the face and the edge of stressed specimen, respectively, after exposure.

Examination of the titanium assembly and stressed specimen showed the following results:

1. Discoloration was somewhat random on all exposed surfaces of the titanium assembly and stressed specimens with more apparent discoloration on and near welds.

2. Examination of the titanium assembly and stressed specimen showed no evidence of cracking in any discolored area.

3. The material in the discolored areas tenaciously adhered to the base metal.

A discussion of these results is as follows:

1. The discoloration of the titanium is believed to be the result of increased oxide thickness on the surface of the titanium. The increased oxide is probably the result of the exposure to 275°F where it would not be unreasonable to expect additional oxidation of the titanium to occur. Although no attempt was made to determine the increase in oxide thickness, it is believed to be in the order of angstroms. As expected, examination of polished cross-sections using light microscopy techniques up to 2000X revealed no discernable differences between oxide thicknesses on unexposed titanium and the most discolored areas of the exposed titanium.

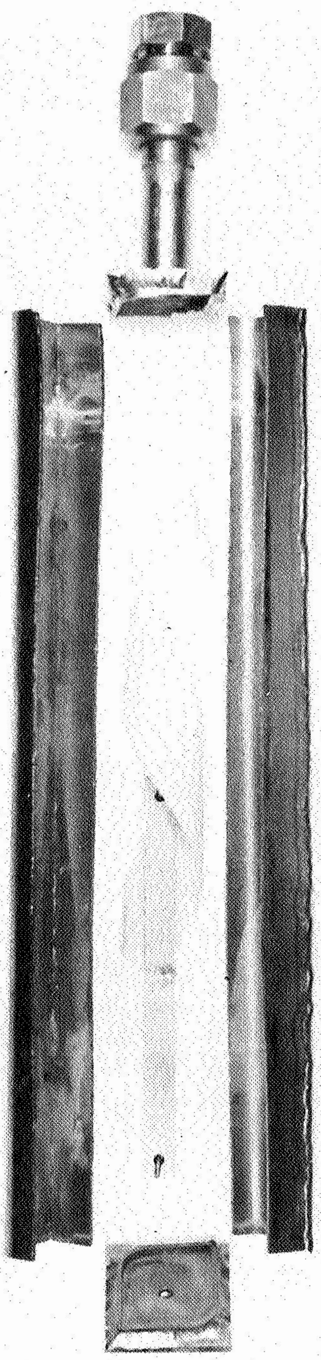


Fig. 37 Sectioned Titanium Assembly with Stressed Specimen of Titanium and Teflon Specimen

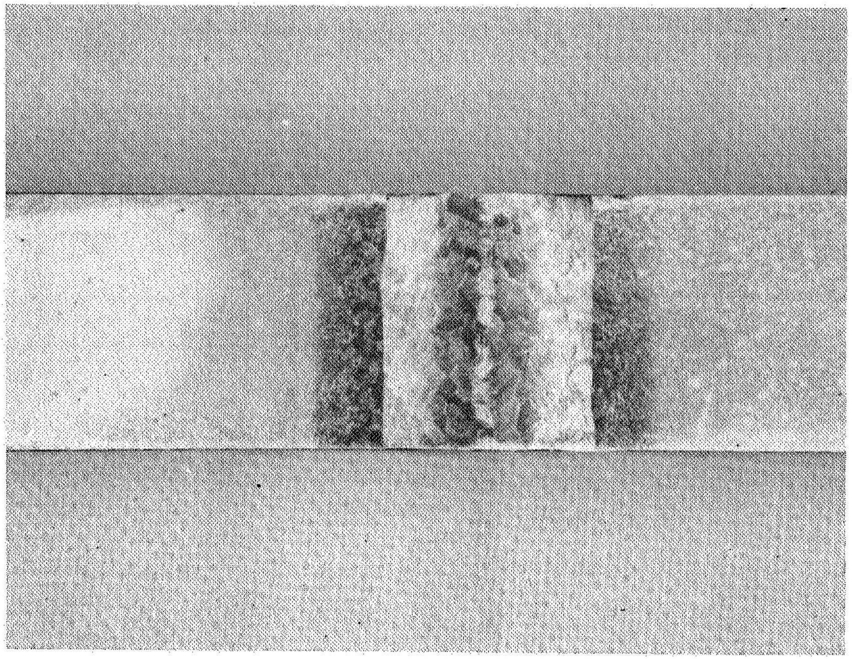


Fig. 38 Face of Stressed Titanium Specimen

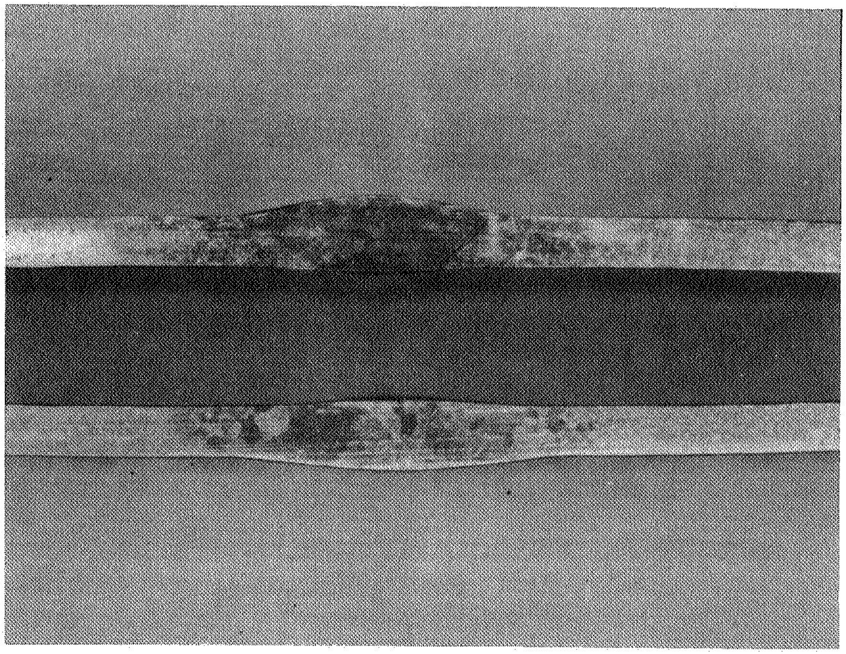


Fig. 39 Edge of Stressed Titanium Specimen

2. The presence of the teflon in the titanium assembly was not believed to have influenced the formation of the discolored surfaces on the titanium.

3. The presence of the discoloration on the titanium has no detrimental affect on the properties of the titanium.

4. The presumed formation of titanium oxide in increasing thicknesses from exposure to N_2O_4 at sterilization temperatures would logically progress at a decreasing rate with an increase in oxide thickness because of the protective nature of titanium oxide.

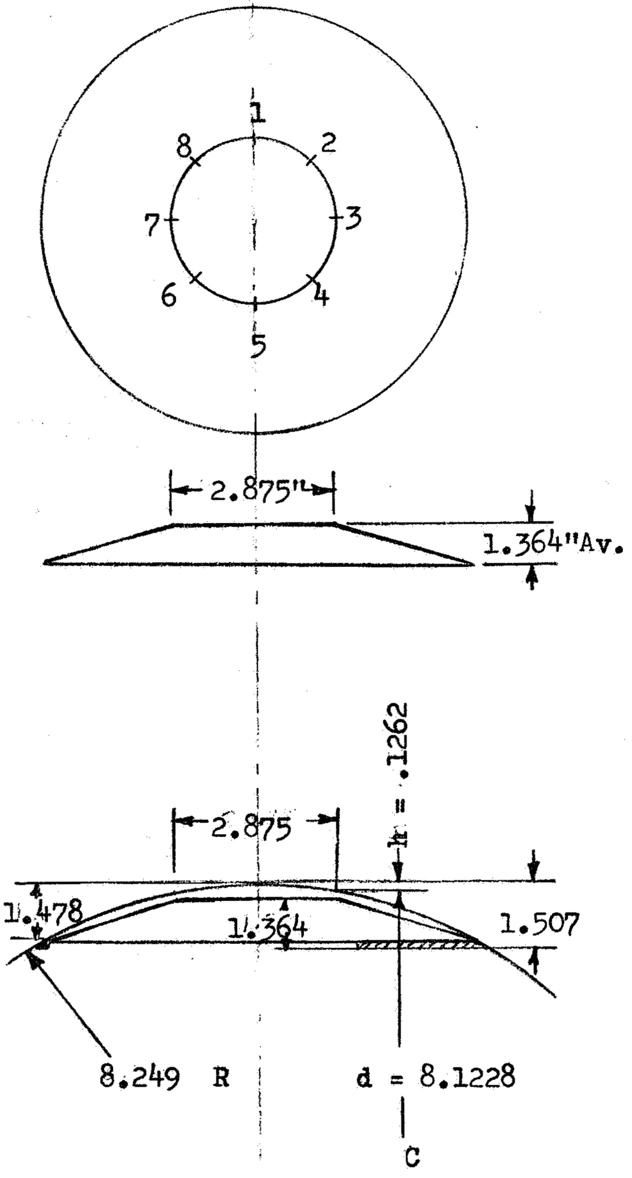
Visual examination of the teflon sample from the tube assembly gave no indication of degradation.

The tube assembly containing MMH was also opened for examination. The propellant was water clear and the screen sample and tube wall gave no indication of degradation.

APPENDIX A

CALCULATION OF FLOW ANNULUS

Position	Height -	Base	Actual Height
1	2.553	1.200	1.353
2	2.565	1.200	1.365
3	2.568	1.200	1.368
4	2.573	1.200	1.373
5	2.571	1.200	1.371
6	2.553	1.200	1.353
7	2.571	1.200	1.371
8	2.558	1.200	1.358
		Average	1.364



From Burington Handbook - page 12

$$\ell = 2\sqrt{R^2 - d^2}$$

$$2.875 = 2\sqrt{(8.249)^2 - d^2}$$

$$8.2656 = 4(68.046 - d^2)$$

$$8.2656 = 272.184 - 4d^2$$

$$4d^2 = 263.9184$$

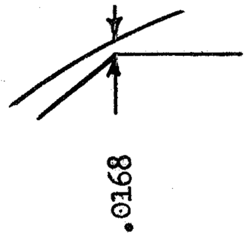
$$d^2 = 65.9796$$

$$d = 8.1228$$

$$h = R - d$$

$$h = 8.249 - 8.1228$$

$$h = .1262''$$



1.478"
.029
1.507
-1.364
.1430
- .1262
.0168

Annulus

$2.875\pi \times .0168 = A \qquad A = .152\ \text{in}^2$

Annulus = $.152\ in^2$

Equivalent orifice diameter $d_{eo} = \sqrt{\frac{A}{.7854}} = \sqrt{\frac{.152}{.7854}} = \sqrt{.19353}$

$$\underline{d_{eo} = .4399\ \text{in.}}$$

$$W = 0.525\ C_f\ d_{eo}^2 \sqrt{\rho\ \Delta P}$$

$$.14 = 0.525\ (0.6)(.19353)\ \sqrt{54.86\ \Delta P}$$

$$2.2965 = \sqrt{54.86\ \Delta P}$$

$$5.2739 = 54.86\ \Delta P$$

$\underline{\Delta P = .09613\ \text{psi}}$ 1 psi = 27.7 in. H_2O

then $.09613 \times 27.7 = \underline{\underline{2.663\ \text{in. } H_2O\ (\Delta P)}}$