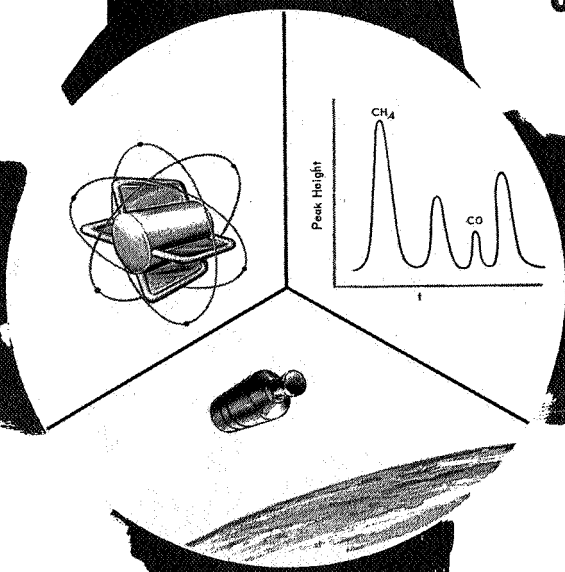


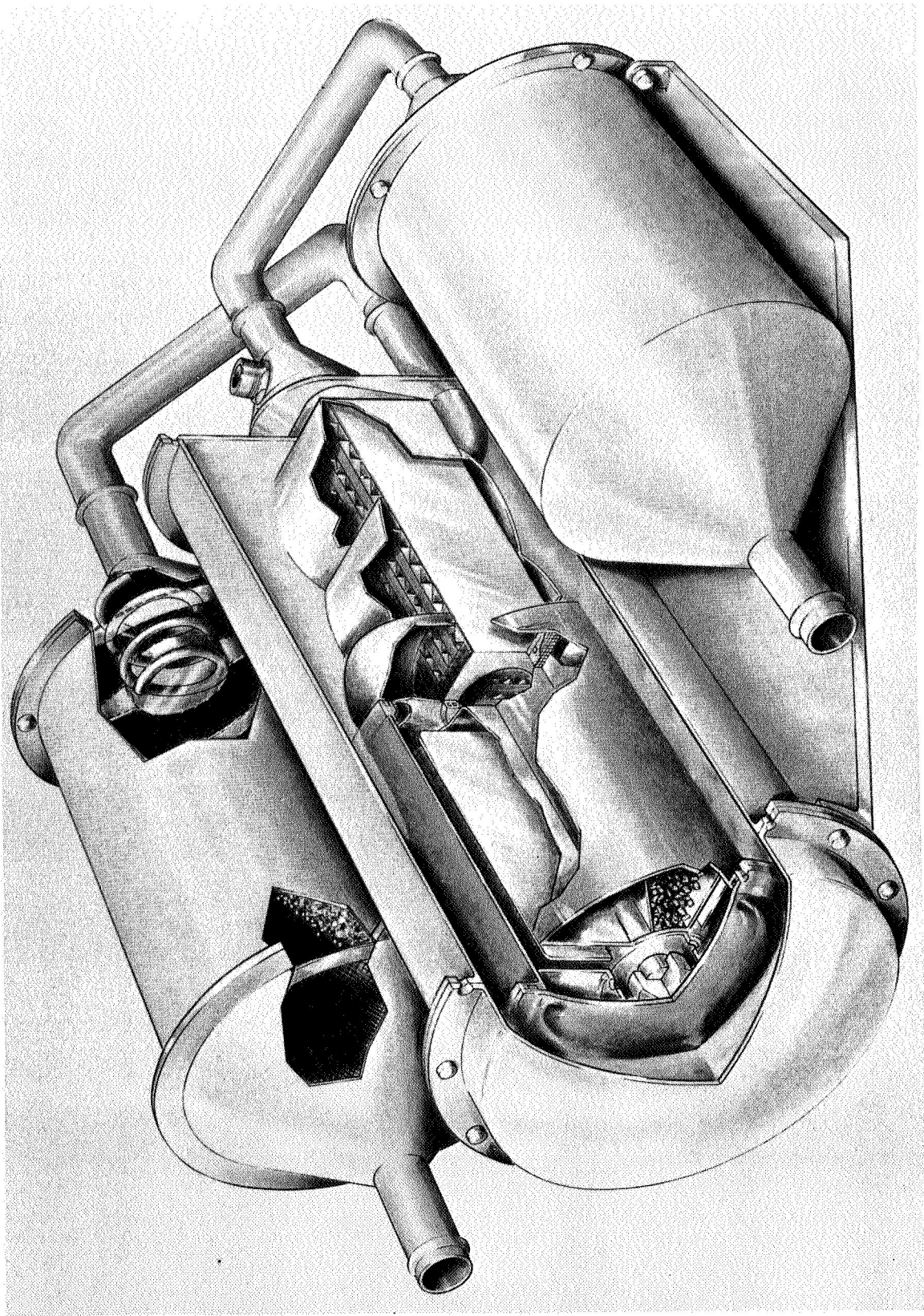
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## DEVELOPMENT AND DESIGN OF AN ISOTOPE-HEATED CATALYTIC OXIDIZER TRACE CONTAMINANT CONTROL SYSTEM (U)



PREPARED UNDER CONTRACT NAS 1-7433  
 by BIOTECHNOLOGY  
 Lockheed Missiles & Space Company  
 Sunnyvale, California

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION  
 LANGLEY RESEARCH CENTER, LANGLEY STATION • HAMPTON, VIRGINIA



Isotope Heated Catalytic Oxidizer System



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Sunnyvale, California

Thomas M. Olcott

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February 28, 1969

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DEVELOPMENT AND DESIGN OF AN  
ISOTOPE-HEATED CATALYTIC OXIDIZER  
TRACE CONTAMINANT CONTROL SYSTEM

By Thomas M. Olcott  
Biotechnology  
Lockheed Missiles & Space Company

SUMMARY

A program was conducted which resulted in the development and detailed design of pre- and post-sorbent beds to be included in the Isotope-Heated Catalytic Oxidizer System (IHCOS) and the detailed design of the catalytic oxidizer with a resistively heated simulated isotope. Preliminary design of the catalytic oxidizer had been accomplished under NAS 1-6256 and is reported in NASA CR 66346 and CR 66347.

The contaminant load developed in NAS 1-6256 and data from recent manned simulator tests and outgassing and degradation studies of materials were reviewed to develop a contaminant load for the pre- and post-sorbent beds. The contaminants selected were those contaminants known to be potentially poisonous to catalyst activity and those contaminants that might decompose and be oxidized to harmful products.

Based on the contaminants selected as potential poisons a pre-sorbent bed was designed that would protect the catalyst from poisoning. The pre-sorbent bed design effort included a survey of candidate sorbent materials to establish those most suited for use as a pre-sorbent material. Laboratory investigations were then conducted with the candidate sorbents to verify the conclusions of the literature survey and to assist in the selection of the pre-sorbent material. Lithium hydroxide was selected for the pre-sorbent material. The sizing of the pre-sorbent bed was then accomplished by establishing the stoichiometric quantity of lithium hydroxide required to remove the poisonous contaminants and by establishing that the dynamic performance of the required quantity of lithium hydroxide would be satisfactory. In establishing the configuration of this bed a trade-off was made between the weight penalty due to the bed and canister fixed weight and the weight penalty associated with the fan head rise required for flow. This resulted in selection of an optimum bed geometry.

To determine the pre-sorbent bed requirements, it was necessary to assess the affects of the charcoal main sorbent bed on the poisonous contaminant load reaching the pre-sorbent bed. To do this, a regenerative charcoal main sorbent bed system, suitable for the proposed mission, was postulated

and its affect on the contaminant load was assessed.

Following the design of the pre-sorbent bed, a long term sorbent bed evaluation test was initiated. The test was conducted with the apparatus that was used to conduct the 180 day catalyst performance test under NAS 1-6256. This apparatus was modified to include model pre- and post-sorbent beds as well as a model regenerative charcoal bed system. The test was conducted in three phases. The first phase was directed towards confirming catalyst performance and assessing the ability of the pre-sorbent bed to protect the catalyst from potential poisons. The second phase of the test investigated the production of undesirable products of oxidation. During this phase of the test the candidate undesirable products of oxidation were identified and a post-sorbent bed design was evolved. The post-sorbent bed design effort was conducted in a manner similar to the pre-sorbent bed. The selected post-sorbent material was lithium hydroxide. The final phase of the long term sorbent evaluation test was to establish the ability of the post-sorbent bed to control the undesirable products of oxidation.

Development activities related to the isotope heat source were conducted and were initiated by establishing heat source material specifications and a materials traceability program. Based on these specifications and the traceability program, materials were purchased for fabrication and joining tests and interdiffusion experiments.

Fabrication and joining techniques were evaluated for all heat source materials with specific weld and fabrication procedures developed for each joint type. Weld samples were sectioned in several different areas and examined metallographically. Photomacrographs and photomicrographs were taken to determine weld penetration and integrity. Two pyrolytic graphite shells, sized to encapsulate the noble metal clad capsule were fabricated and examined. Following the joining and fabrication tests, experiments were conducted to determine the extent of interdiffusion and compatibility between the isotope heat source materials of construction. The amount of interdiffusion occurring at all material interfaces was experimentally determined after 15 days and 60 days at 800°F and 1500°F using metallography and electron microprobe analyses. The data were extrapolated to two year durations. Short duration compatibility between pyrolytic graphite and stainless steel near and above the eutectic temperature was investigated.

A review of heater design and fabrication techniques was conducted and resulted in the selection of two heater designs for test evaluation. Two heaters of each design were fabricated and tested successfully in a simulated capsule environment for 30 days to ascertain thermal performance during operation. After testing, the composite heater specimens were sectioned and examined microscopically. Measurements were made of the width of the diffusion zone between the liner material and the heater sheath.

These studies revealed that the heater sheath, capsule and liner materials are compatible in both configurations. The sheathed helical heater unit



was selected because it allows better simulation of the isotope fuel weight and a better closure of the leads leaving the structural module.

An experimental evaluation of both the solid Johns Manville "Min K" insulation and the vacuum insulation was conducted. A simplified configuration, representing an accurate thermal model, of the Johns Manville insulation was evaluated for 180 days. During this period both normal and emergency conditions were investigated. The insulation performance was better than anticipated resulting in a reduction in the required insulation thickness from 1.5 to 1.0 inches. The vacuum insulation canister was tested for 52 days and results of this evaluation indicated that the vacuum insulation was inferior to the solid insulation. The solid insulation was selected for the final design.

The detailed design of the isotope heated catalytic oxidizer system included a stress analysis of the main structure and a review of the thermal characteristics. Following this, detailed design drawings for the entire system were prepared. The resistively heated unit is, insofar as possible, an exact duplicate of the radioisotope - fueled unit except that the thermal power is obtained from a resistively heated element located in the fuel cavity, and heater element and thermocouple leads pass through the heat exchanger core.



## INTRODUCTION

The development of a flight qualifiable isotope-heated catalytic oxidizer for control of trace contaminants was initiated under Contract NAS 1-6256. This contract between the Lockheed Missiles & Space Company (LMSC) with TRW Systems as a major subcontractor, and the NASA - Langley Research Center resulted in engineering layout drawings of the selected approach and long term testing of a model system. The results of this effort are described in NASA CR 66346, NASA CR 66347 and NASA CR 66497. The tasks accomplished under NAS 1-6256 included the following:

- o Mission Definition
- o Contaminant Load Definition
- o Isotope Selection
- o Catalyst Selection
- o Catalyst Performance Tests
- o Analysis and Optimization
- o Design Layout Drawings
- o Development Plan

Following the conclusion of this program, the NASA - Langley Research Center directed LMSC to continue this development program under Contract NAS 1-7433. TRW Systems was also a major subcontractor in this additional effort. The program conducted under NAS 1-7433 is reported herein and dealt with additional development of the isotope-heated catalytic oxidizer system including detailed design of a unit utilizing a resistively heated simulated isotope and development and detailed design of pre- and post-sorbent beds. The tasks involved in this program are shown below and are described in detail in this report.

- o Contaminant load definition for the pre- and post-sorbent beds.
- o Design and fabrication of a model pre-sorbent bed.
- o Long term sorbent bed evaluation.

- o Design and fabrication of a model post-sorbent bed.
- o Detailed design of full scale pre- and post-sorbent beds.
- o Specifications for the isotope heat source materials of construction.
- o Joining and fabrication tests on the isotope heat source materials of construction.
- o Fabrication and evaluation of the test heater to be used in the simulated isotope heat source.
- o Compatibility tests to determine the extent of interdiffusion between the graphite reentry aid and the noble metal cladding.
- o Fabrication and evaluation of the thermal insulation to be used in the isotope-heated catalytic oxidizer.
- o Detailed design of the isotope-heated catalytic oxidizer including the resistively heated simulated isotope heat source.

Information relating to the liner material and fabrication techniques and the strength member fabrication techniques are described in a classified summary of this report.

## PRE- AND POST-SORBENT CONTAMINANT LOAD DEFINITION

The contaminant load for the IHCOS pre- and post-sorbent beds is based on data obtained from the contaminant load defined in the Study and Preliminary Design of an Isotope-Heated Catalytic Oxidizer System, NAS 1-6256 (ref. 1), recent manned simulator tests, and outgassing and degradation studies of candidate materials likely to be used on space stations. The contaminants selected were those contaminants known to be potentially poisonous to catalyst activity and/or those that can be decomposed and oxidized to harmful products. A list of these contaminants is presented in Table 1, which includes: (1) contaminant origin; (2) whether contaminant is considered a potential catalyst poison; (3) whether contaminant is considered a potential producer of undesirable products; (4) design production rate; and, (5) maximum allowable concentration, values that are appropriate for a mission duration of 180 days.

### Selected Contaminants

The majority of the contaminants selected were obtained from a review of the contaminant load definition conducted during Phase I of the IHCOS Program (NAS 1-6256). The Phase I IHCOS Study resulted in a listing of 150 contaminants that might exist in a spacecraft atmosphere. The contaminants were derived from a literature search of the following sources: (1) chemical analysis of outgassing products from space cabin qualified materials reported by Lockheed Missiles & Space Company (ref. 2), North American Aviation (ref. 3), and Minneapolis Honeywell (ref. 4); (2) contaminants detected in the AF Biosatellite 30-day test (ref. 6); (4) contaminants reported by Toliver and Morris in the manned 30-day test at the AF Aerospace Medical Research Laboratory (ref. 7); (5) contaminants detected in a 27-day manned test at the AF School of Aerospace Medicine (ref. 8); (6) contaminants detected during Apollo breadboard testing (ref. 9); and, (7) candidate contaminants that might be generated from experiments onboard space stations such as MORL and AES (ref. 10).

Activities reviewed for additional candidate contaminants during this effort include: (1) recent environmental chamber tests such as the Langley ILSS tests (ref. 11) and Lockheed Missiles & Space Company Five-Day Lunar Shelter and Extravehicular Manned Test (Ref. 12); (2) space cabin material outgassing reported by Pustinger and Hodgson for the Aerospace Medical Research Laboratories (ref. 13); and (3) chlorinated hydrocarbons in closed environment atmospheres reported by R. Saunders in the 5th Annual Progress Report: The Present Status of Chemical Research in Atmosphere Purification and Control on Nuclear-Powered Submarines by Naval Research Laboratory (ref. 14).

TABLE 1

## PRE- AND POST-SORBENT CONTAMINANT LOAD DEFINITION

Contaminant	Reported In	Potential Catalyst		Undesirable Products	Potential Data Source	(1) Design Production Rate	(2) Maximum Allowable Concentration Mg/M <sup>3</sup>
		Poison	Data Source				
			Data Source				
Acetonitrile	(A)			+	(3)	Secondary	7
Ammonia	(A)			+	(3)(9)(18)	gm/day	3.5
Carbon Disulfide	(A)	+	(17)	+	(3)	Secondary	6.0
Carbon Tetrachloride	(A)	+	(5)(6)	+	(3)	Secondary	6.5
Carbonyl Sulfide	(A)	+	(17)	+	(3)	Secondary	25.0
Chlorine	(A)	+	(3)(7)(9)	+	(3)	Secondary	1.5
Chloroacetone	(A)	+	(5)(6)(17)	+	(3)	Secondary	100
Chlorobenzene	(A)	+	(5)(6)	+	(3)	Secondary	35
Chlorofluoromethane	(A)	+	(5)(6)	+	(3)(8)	Secondary	24
Chloroform	(A)	+	(5)(6)	+	(3)	Primary	24
Chloropropane	(A)	+	(5)(6)	+	(3)	Secondary	84
Cyanamide	(A)			+	(3)	Secondary	45
Dichlorobenzene	(B)	+	(5)(6)	+	(3)	Secondary	30
2,4 Dichlorobenzoic Acid	(B)	+	(5)(6)	+	(3)	Secondary	205
1,1 Dichloroethane	(A)	+	(5)(6)	+	(3)(8)	Primary	40
Dimethyl Hydrazine	(A)			+	(3)	Secondary	0.1
Dimethyl Sulfide	(A)	+	(17)	+	(3)	Secondary	15
Epichlorohydrin	(B)	+	(5)(6)	+	(3)	Secondary	1.9

Table 1 Continued

Contaminant	Reported In	Potential Catalyst		Undesirable Products	Potential Data Source	(1) Design Production Rate	(2) Maximum Allowable Concentration Mg/M <sup>3</sup>
		Poison	Data Source				
Ethyl Amine	(B)			+	(3)	Secondary	1.8
Ethylene Dichloride	(A)	+	(5)(6)	+	(3)	Secondary	40
Ethyl Mercaptan	(A)	+	(17)	+	(3)	Secondary	2.5
Ethyl Sulfide	(A)	+	(17)	+	(3)	Secondary	97
Freon 11	(A)	+	(5)(6)	+	(3)(8)(9) (14)(15)(16)	Primary	560
Freon 12	(A)	+	(5)(13)	+	(3)(8)(12) (15)(16)	Primary	500
Freon 21	(A)	+	(5)	+	(3)	Secondary	420
Freon 22	(A)	+	(5)(6)	+	(3)	Secondary	350
Freon 23	(A)	+	(5)(6)	+	(3)	Secondary	12
Freon 113	(A)	+	(5)(6)	+	(16)	Secondary	700
Freon 114	(A)	+	(5)(6)	+	(3)(4)(16)	Primary	700
Freon 114 (unsym)	(A)	+	(5)(6)	+	(3)(16)	Secondary	700
Freon 125	(A)	+	(5)(6)	+	(3)	Secondary	25
Hexamethylcyclotrisiloxane	(A)	+	(19)(20)	+	(3)	Secondary	290
Hydrogen Chloride	(A)	+	(7)(9)	+	(3)	Secondary	0.15
Hydrogen Fluoride	(A)	+	(7)(9)	+	(3)	Secondary	0.08
Hydrogen Sulfide	(A)	+	(5)(13)(17)	+	(3)(4)	gm/day	1.5
Indole	(A)			+	(3)	gm/day	126

Table 1 Continued

Contaminant	Reported In	Potential Catalyst		Undesirable Products	Potential Data Source	(1) Design Production Rate	(2) Maximum Allowable Concentration Mg/M <sup>3</sup>
		Poison	Data Source				
Methyl Amine	(B)			+	(3)	Secondary	1.2
Methyl Chloride	(A)	+	(13)	+	(3)(8)	Secondary	21
Methyl Chloroform	(A)	+	(5)(6)	+	(3)(7)(8)(10)	Primary	190
Methylene Chloride	(A)	+	(5)(6)	+	(3)	Primary	21
Methyl Mercaptan	(A)	+	(5)(17)	+	(3)	Secondary	2.0
N-Methyl Morphaline	(B)			+	(8)(9)	Secondary	7.0
mono-Methyl Hydrazine	(A)			+	(3)(4)	Secondary	0.035
Nitric Oxide	(A)	+	(3)(7)(9)	+	(3)	Secondary	32
Nitrogen Dioxide	(A)	+	(7)(9)	+	(3)	Secondary	1.8
Nitrogen Tetroxide	(A)	+	(3)(7)(9)	+	(3)	Secondary	0.9
Nitromethane	(C)	+	(3)(7)(9)	+	(3)	Secondary	25
Nitrous Oxide				+	(3)(7)(9)	Secondary	47
iso-Propyl Chloride	(A)	+	(5)(6)	+	(3)	Secondary	260
Propyl Mercaptan	(A)	+	(17)	+	(3)	Secondary	82
Skatole	(A)			+	(3)	Secondary	14.1
Sulfur Dioxide	(A)	+	(17)	+	(3)	Secondary	0.8
Tetrachloroethylene	(A)	+	(5)(6)	+	(3)	Secondary	67
Tetrafluoroethylene	(A)	+	(3)(6)	+	(3)	Secondary	205
Trichloroethylene	(A)	+	(5)(6)	+	(3)	Primary	52
Trimethyl Silanol	(B)	+	(19)(20)	+	(3)	Secondary	97



Table 1 Continued

Contaminant	Reported In	Potential Catalyst		Undesirable Products	Potential Data Source	(1) Design Production Rate	(2) Maximum Allowable Concentration Mg/M3
		Poison	Data Source				
Vinyl Chloride	(A)	+	(5)(6)	+	(3)	Primary	130
Vinyldene Chloride	(A)	+	(5)(6)	+	(3)(10)	Secondary	20
Water	(A)	+	(11)				

NOTES FOR TABLE 1

- (1) Design production rate includes 9 men metabolic production rate plus equipment production rates scaled from Apollo studies by North American Aviation. Primary contaminants are assumed to be produced at 2.5 gms/day and secondary contaminants are assumed to be produced at 0.25 grams per day.
- (2) The "maximum allowable concentration" refers to the contaminant concentrations appropriate for man allowing a continuous exposure up to 180 days. Values are obtained from reference (A). Other values not included in reference (A) are limit values taken as 0.1 of the Threshold Limit Value (TLV) for 1965, American Conference of Government Industrial Hygienists, May, 1965.
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## Potential Catalyst Poisons

The selection of potential catalyst poisons was accomplished after a review of experimental studies on catalyst deactivation. Low temperature conversion performance test studies (ref. 15) at IMSC with hydrogen sulfide were reported showing the deactivation of unprotected 0.5% palladium catalyst after 18 days of exposure. Additional tests during this same program indicated that with Freon-12, vinyl chloride, and methyl mercaptan in the gas stream, a shift in temperature as great as 180°F was required to maintain a given oxidation efficiency for 0.5% palladium catalyst.

Experiments at Atlantic Research have shown partial catalyst deactivation with Hopcalite, palladium, ruthenium, and cobalt oxide resulting from exposure to Freon 12, methyl chloride, and hydrogen sulfide (ref. 16).

The deactivation effects of cyclic silane compounds on platinum catalysts were reported both by R. H. Savage (ref. 17), and A. F. Plate and N. A. Belikova (ref. 18).

Tests by U. S. Naval Research Laboratory (NRL) on Hopcalite catalyst have indicated that water vapor present in the gas stream required an increase in catalyst bed temperature for successful performance (ref. 19). More recently, during the 180-day IHCOS long term catalyst test, it was observed that water vapor in the gas stream required an increase of 140°F in catalyst bed temperature to maintain the desired oxidation efficiency (ref. 1).

Poisoning of platinum catalysts has been demonstrated by nitrogen compounds, especially by ammonia, saturated amines, and cyanide. In a wet gas stream, the deactivation effect of the nitrogen compounds is nullified by the presence of moisture (ref. 20).

Maxted discussed the poisoning of metallic catalysts by molecules containing elements of the periodic groups Vb, such as nitrogen, phosphorous, arsenic and antimony and groups Vlb, such as oxygen, sulfur, selenium and tellurium (ref. 21). He mentioned that not all compounds of these elements are poisonous to the catalyst, but these properties depend on the electronic unshielded configuration of the element in the molecule.

## Potential Undesirable Products

Similarly, the selection of potential contaminants that will produce undesirable reactants in the catalytic oxidizer and/or the pre-sorbent material other than carbon dioxide, water, and carbon was accomplished after a review of the literature on this subject.

In general, it has been shown that catalytic oxidation of compounds containing nitrogen, sulfur, or the halogen lead to the formation of products that are much more detrimental to man and equipment than the reactants.

NRL studies on Hopcalite catalyzed oxidation of Freon-11, -12, and -114B2 show that these compounds decompose extensively to yield acid vapors (ref. 22, 23). In addition, NRL has shown this with studies on vinylidene chloride, trichloroethylene, methyl chloroform and ammonia (ref. 24). Similar reports by the Warren Spring Laboratory have indicated the catalytic decomposition of Freon-12 to toxic gaseous products (ref. 25). Church and Mayer have reported the decomposition of trichlorofluoromethane in the presence of moisture and metals at temperatures up to 1400°F. Reaction mechanisms are discussed outlining the formation of phosgene type compounds (ref. 26). Trenworth and Watson have shown that the pyrolysis of chlorofluoromethanes lead to the formation of halogenated hydrocarbons together with chlorine (ref. 27). DuPont, Freon Products Division, has reported on the thermal decomposition of Freons -11, -12, -22, -112, and -114, at elevated temperatures (ref. 28). Free halogen and acid formation at varying temperatures up to 1000°F were reported.

Work at LMSC has shown the formation of sulfur dioxide and nitrogen dioxide in the outlet stream of a Pt-Pd catalytic oxidizer system fed with a Freon-114, H<sub>2</sub>S and monomethyl hydrazine contaminated stream (ref. 29). The absence of HCl and HF was assumed to be the neutralization of these acids by the base, monomethyl hydrazine.

An estimate of possible inorganic combustion products and amounts produced from contaminants that contain elements other than hydrogen, oxygen and carbon has been reported by LMSC (ref. 20). The toxic combustion products includes HF, HCl, NO<sub>2</sub>, SO<sub>2</sub>, HCN, COCl<sub>2</sub>, Cl<sub>2</sub>, and COF<sub>2</sub>.

Saunders discussed the partial decomposition of methyl chloroform to vinylidene chloride and trichloroethylene in a Hopcalite burner (ref. 30).

#### Design Production Rate and Maximum Allowable Concentration

For those contaminants obtained from the Phase I IHCOS Program (NAS 1-6256), the production rates and maximum allowable concentrations cited were used. The major source of information on nonbiological contaminants used in NAS 1-6256 was the contaminant identification program conducted by NAA for the Apollo program in which the outgassing rate of materials within the Apollo was determined experimentally. The contaminant load estimate for the IHCOS was obtained by scaling the Apollo contaminant load to the IHCOS model spacecraft. Based on this scaling a design equipment production rate of 50 grams per day was estimated for the model spacecraft.

To determine the individual equipment contaminant production rates, the contaminant distribution (i.e., percentage of total) from the NAA Program was utilized with the exception that no primary contaminant was considered to be produced at a rate less than 5 percent of the total and no secondary contaminant was considered to be produced at a rate less than 0.5 percent of the total. Primary contaminants are those where a known large source exists, or where the contaminant has been identified in several systems. The remaining contaminants are defined as secondary contaminants.

Allowable concentrations used in NAS 1-6256 were based in the following order on Submarine Habitability Handbook values, or 0.1 of the 1965 Threshold Limit Values (TLV) or analogy to chemical compounds with established TLV or an arbitrary partial pressure limit or 0.02 torr.

For the contaminants not considered in NAS 1-6256 a production rate of 0.25 grams per day was used which corresponds with the production rate of secondary contaminants in NAS 1-6256. For these contaminants, the maximum allowable concentration was taken at 0.1 of the 1965 Threshold Limit Values (TLV) of the American Conference of Government Industrial Hygienists or if no TLV existed, an arbitrary limit of 0.02 mmHg was used. This arbitrary limit was used in NAS 9-3415 and NAS 1-6256. These levels are in reasonable agreement with the recommendations of the National Academy of Sciences, Space Science Board, Atmospheric Contaminants in Spacecrafts, June, 1968.

## PRE-SORBENT BED DESIGN

The pre-sorbent bed design effort included the following tasks:

- o Survey to screen candidate sorbents.
- o Laboratory investigations to validate literature survey conclusions.
- o Pre-sorbent bed designs optimization.
- o Establish main sorbent charcoal bed characteristics, and effects on poisonous contaminant load to the IHCOS.

The following sections describe the above tasks in detail.

### Candidate Sorbent Screening

A review of data taken by IMSC and other industrial groups as well as Government agencies was performed to identify candidate pre-sorbent materials (ref. 31-60). The selection of candidate sorbents was based on the following parameters:

- (1) Selectivity of contaminants for removal.
- (2) Sorption capacity.
- (3) Chemical stability.
- (4) Weight requirements.

The candidates identified included lithium hydroxide, charcoal, Molecular sieve 13 X silica gel, activated alumina and solid permanganate. These candidate sorbents are discussed below.

Lithium hydroxide is a strong base that is not only an effective agent for removing carbon dioxide, but as a candidate pre-sorbent material can undergo one or more of the following processes:

- (1) Acid-base neutralization reactions.
- (2) Complex formation.

- (3) Oxidation reduction reactions and
- (4) Physical adsorption - chemisorption.

Of the bases which are suitable for use in life support systems, lithium hydroxide has the greatest capacity per unit weight for the neutralization of acids. Since the solid lithium hydroxide does not remove carbon dioxide efficiently, by conversion to the non-volatile salt lithium carbonate any acid which is appreciably stronger than carbonic acid should also be removed. The effectiveness of removal however is in part determined by the rate of reaction with lithium hydroxide.

Lithium salts are soluble in many organic solvents. From these solutions crystalline complexes of the solvent and lithium salt can be isolated as stable compounds, thus illustrating the strong bonding involved in these complexes. Some of the most stable complexes of this type are the lithium salt hydrates, alcoholates and aminates (ref. 61). Thus, alcohol, and amines may be effectively removed by lithium hydroxide.

The high surface area of lithium hydroxide provides the conditions necessary to promote oxidation-reduction reactions. It is known that nitric oxide is converted to the nitrate ion when passed over solid potassium hydroxide (ref. 62). Chlorine, nitrogen dioxide and hydrogen sulphide should also be controlled in this manner. (ref. 61, 63, and 64). In addition to the above mentioned inorganic oxidation reduction reactions there are many organic contaminants such as aldehydes and ketones which are expected to be oxidized in the presence of a base and oxygen. Other reactions involve direct oxidation of organic compounds by oxygen on an activated surface and may include olefins and mercaptans (ref. 65).

Activated charcoal is one of the most effective adsorbents for the removal of a wide variety of contaminants. The affinity of a particular contaminant for charcoal has been shown to be related to its vapor pressure and molar volume. Charcoal can provide effective control of many of the potentially poisonous contaminants, however, it is not effective for compounds such as inorganic acids and certain Freon type compounds. In general, compounds with high molar volumes are well adsorbed on charcoal.

In selecting the pre-sorbent material the effect of the main sorbent bed on the poisonous contaminant load to the IHCOS should be assessed. This is discussed in more detail in the section on sorbent bed design.

Molecular sieve 13X is an effective sorbent material for contaminants



possessing a molecular diameter less than ten angstroms. Molecular sieve 13 X has a larger adsorption capacity for inorganic contaminants than activated charcoal and is chemically stable. The disadvantages of molecular sieve 13 X are its special affinity for moisture in preference to other compounds, and its high weight requirement in comparison to other sorbents for inorganic compounds such as lithium hydroxide.

Silica gel has good adsorptive capacity for water vapor and in general a fair capacity for polar compounds, however, in contrast to charcoal its capacity for most of the potential catalyst poisons is very poor.

Activated alumina, like silica gel, has good capacity for moisture. In comparison to other sorbent materials, extra precautions are required to preserve size and shape of the adsorbent. It does not possess the overall charcoal adsorbing capacity for the acid gases and halogenated compounds. Previous work by IMSC on the Fleet Ballistic Missile Toxicity Program substantiated this on experiments with Freon type contaminants tested under dynamic conditions (ref. 64 and 66).

Solid permanganate impregnated alumina removes contaminants by adsorption and chemical oxidation. Potential catalyst poisons known to be removed by "Purafil", a commercial solid oxidant include hydrogen chloride, hydrogen sulfide, nitric oxide, tetrachloroethylene, phosgene and sulfur dioxide. Other potential catalyst poisons for which theoretical considerations indicate possible removal by this method are: carbon disulfide, chloroacetone, cyanamide, ethyl mercaptan, ethyl sulfide, methylene chloride, methyl chloroform, methyl mercaptan, nitrogen dioxide, nitrous oxide, propyl mercaptan, trichloroethylene, vinyl chloride and vinylidene chloride (ref. 67). Freons, chlorine, ethylene dichloride, hydrogen fluoride, chlorobenzene, and chloropropane are unreactive with this material.

#### Laboratory Evaluation

In reviewing the candidate sorbent materials, it was determined that nearly all of the potentially poisonous contaminants appeared to be controlled by the use of either charcoal or lithium hydroxide.

These conclusions, shown in Table 2, are primarily based on the data available on sorbent capacity. However, little data is available on the dynamic performance of the candidate sorbent materials. To establish dynamic performance (removal per pass) a laboratory investigation was conducted with the two most promising sorbents and a number of potentially poisonous contaminants.

This experimental evaluation was performed on 6 x 8 and 20 x 35 mesh lithium hydroxide and on 6 x 8 and 10 x 30 mesh (Tyler) activated charcoal by passing sulfur dioxide, nitrogen dioxide, Freon-12, vinyl chloride, Genetrons 21 and, 23 nitrous oxide diluted in air at varying space velocities through the candidate pre-sorbent materials.

TABLE 2  
 ANTICIPATED REMOVAL TECHNIQUES FOR POTENTIAL CATALYST POISONS

Potential Catalyst Poisons	Anticipated Removal Technique				Charcoal
	Neutralization	Lithium Hydroxide Complex	Oxidation	Reduction	
Carbon Disulfide	+	+			
Carbon Tetrachloride					+
Carbonyl Sulfide		+			
Chlorine			+		
Chloroacetone			+		
Chlorobenzene					+
Chlorofluoromethane					+
Chloroform					+
Chloropropane					+
Dichlorobenzene					+
2,4 Dichlorobenzoic Acid	+				
1,1 Dichloroethane					+
Dimethyl Sulfide					+
Epichlorohydrin		+			
Ethylene Dichloride					+
Ethyl Mercaptan				+	
Ethyl Sulfide					+
Freon 11					+
Freon 12					+
Freon 21					+

Table 2 Continued

Potential Catalyst Poisons	Anticipated Removal Technique			Charcoal
	Neutralization	Lithium Hydroxide Complex	Oxidation Reduction	
Freon 22				+
Freon 23				+
Freon 113				+
Freon 114				+
Freon 114 (unsym)				+
Freon 125				+
Hexamethylcyclotrisiloxane				+
Hydrogen Chloride	+			
Hydrogen Fluoride	+			
Hydrogen Sulfide	+		+	
Methyl Chloride				+
Methyl Chloroform				+
Methylene Chloride				+
Methyl Mercaptan			+	
Nitric Oxide			+	
Nitrogen Dioxide	+		+	
Nitrogen Tetroxide	+		+	
Nitromethane				+
iso-Propyl Chloride				+
Propyl Mercaptan			+	
Sulfur Dioxide	+			

Table 2 Continued

Anticipated Removal Technique

Potential Catalyst Poisons	<u>Lithium Hydroxide</u>			Charcoal
	Neutralization	Complex	Oxidation Reduction	
Tetrachloroethylene				+
Tetrafluoroethylene			+	
Trichloroethylene				+
Vinyl Chloride				+
Vinylidene Chloride				+

The results of the evaluation are shown in Table 3 and Figures 1, 2 and 3. The inorganic acids were well adsorbed by the lithium hydroxide even at relatively high space velocities with a 6 x 8 mesh size. Freon-12 and vinyl chloride had relatively low removal efficiencies on lithium hydroxide, while Genetrons 21 and 23, and nitrous oxide appeared not to be removed or very poorly removed with both 6 x 8 and 20 x 35 mesh lithium hydroxide. These results are in agreement with the theoretical conclusions drawn in the previous section.

Activated charcoal was quite effective in removing vinyl chloride and the Freon type compounds that were not controlled with lithium hydroxide. These test results support the conclusion that the charcoal main sorbent and a lithium hydroxide pre-sorbent provide effective control of nearly all of the contaminants identified as potential poisons.

#### Pre-Sorbent Bed Size

The sizing of the pre-sorbent bed was accomplished by establishing the stoichiometric quantity of lithium hydroxide required to remove the poisonous contaminants and by establishing that the dynamic performance of the required quantity of lithium hydroxide would be satisfactory. To determine the required quantity of lithium hydroxide, the production rate of the contaminants anticipated to be controlled by lithium hydroxide was determined. These contaminant production rates are listed in Table 4. The production rates listed in the table include the basic contaminant production rate established in NAS 1-6256 and the maximum quantity of the contaminant that can be removed with a flow rate of 3 cfm and an inlet concentration equal to the maximum allowable concentration. The lower of these two production rates for a given contaminant was used to establish the quantity of lithium hydroxide required for removal. The removal mechanisms for each of the contaminants are presented in Table 2. In cases where the lithium hydroxide acts as a catalyst in an oxidation reduction reaction, no lithium hydroxide is consumed. The total stoichiometric quantity of lithium hydroxide required for 180 days for all of the contaminants listed in Table 4 is 335 grams. Using a performance factor of 50% and a density of 29 lbs/ft<sup>3</sup>, the required volume of lithium hydroxide for the pre-sorbent canister is 90 in<sup>3</sup>. With a flow rate of 3 cfm this will result in a superficial space velocity of 3,400 hrs<sup>-1</sup>. Based on the data shown in Figure 2, a removal efficiency of approximately 95% can be obtained with 6 x 8 mesh lithium hydroxide (LiOH). Since the removal efficiency of the 6 x 8 mesh LiOH is essentially the same as the 20 x 35 and the pressure drop of the 20 x 35 mesh is 10 times the pressure drop of the 6 x 8, the coarser material appears best suited for the pre-sorbent bed.

In establishing the configuration of this bed, a trade off was made between the weight penalty due to the bed and canister fixed weight and the weight penalty associated with the fan head rise required for flow. To accomplish this, canister fixed weight and bed pressure loss were related to bed geometry (L/D). This relationship is shown in Figure 4 where the total equivalent weight is plotted as a function of canister diameter. This re-

TABLE 3

RESULTS OF LITHIUM HYDROXIDE AND  
CHARCOAL REMOVAL CAPABILITY TESTS

Contaminant	Sorbent	Mesh Size	Inlet Concentration Ci (Mg/M <sup>3</sup> )	Removal Efficiency R <sub>r</sub> (%)	Space Velocity ∅ (Hrs <sup>-1</sup> )
Freon 12	LiOH	6 x 8	198	.10	16,000
" "	"	"	"	.10	9,230
" "	"	"	"	.10	1,670
" "	"	"	"	.14	693
" "	"	"	"	.42	173
" "	"	20 x 35	"	.10	333
Genetron 21	LiOH	6 x 8	115	0	3,930
" "	"	"	"	0	1,680
" "	"	"	"	0	694
" "	"	"	"	0	115
" "	"	20 x 35	"	.10	3,930
" "	"	"	"	.10	1,680
" "	"	"	"	.10	694
Genetron 23	LiOH	6 x 8	115	0	6,700
" "	"	20 x 35	"	0	2,470
Nitrogen Dioxide	LiOH	6 x 8	2.2	.90	333
Sulfur Dioxide *	"	"	157	.98	11,400
Vinyl Chloride	LiOH	6 x 8	106	.10	16,000
" "	"	"	"	.10	9,230
" "	"	"	"	.10	1,670
" "	"	"	"	.10	693
" "	"	"	"	.11	173
" "	"	20 x 35	"	.12	333

Table 3 Continued

Contaminant	Sorbent	Mesh Size	Inlet Concentration Ci (Mg/M <sup>3</sup> )	Removal Efficiency $\eta_r$ (%)	Space Velocity $\phi$ (Hrs <sup>-1</sup> )
Freon 12	BD Activated Charcoal (B-C)	6 x 8	140	.85	2,850
" "	"	"	"	.86	2,100
" "	"	"	"	.88	800
" "	"	12 x 28	"	1.0	11,400
" "	"	"	"	1.0	2,675
" "	"	"	"	1.0	2,100
Freon 22	BD Activated Charcoal (B-C)	12 x 28	155	1.0	6,680
" "	"	"	"	1.0	3,000
" "	"	"	"	1.0	2,100
Genetron 21	BD Activated Charcoal (B-C)	12 x 28	100	1.0	9,700
Genetron 23	BD Activated Charcoal (B-C)	12 x 28	81	1.0	2,100
" "	"	"	"	.25	3,000
" "	"	"	"	.11	6,680
Nitrous Oxide(N <sub>2</sub> O)	BD Activated Charcoal (B-C)	12 x 28	108	1.0	866
" "	"	"	"	.34	1,330
" "	"	"	"	.23	3,000
Sulfur Dioxide *	BD Activated Charcoal (B-C)	12 x 28	66	.9	11,400
Vinyl Chloride	BD Activated Charcoal (B-C)	12 x 28	124	1.0	11,400
" "	"	"	124	1.0	2,675

Table 3 Continued

Contaminant	Sorbent	Mesh Size	Inlet Concentration Ci ( $Mg/M^3$ )	Removal Efficiency r (%)	Space Velocity $\emptyset$ ( $Hrs^{-1}$ )
Vinyl Chloride	BD Activated Charcoal (B-C)	12 x 28	124	1.0	2,100
"	"	6 x 8	"	.92	2,850
"	"	"	"	.94	2,100
"	"	"	"	.96	800

\* By Kitagawa Colormetric Tubes; minimum sensitivity  $5 Mg/M^3$



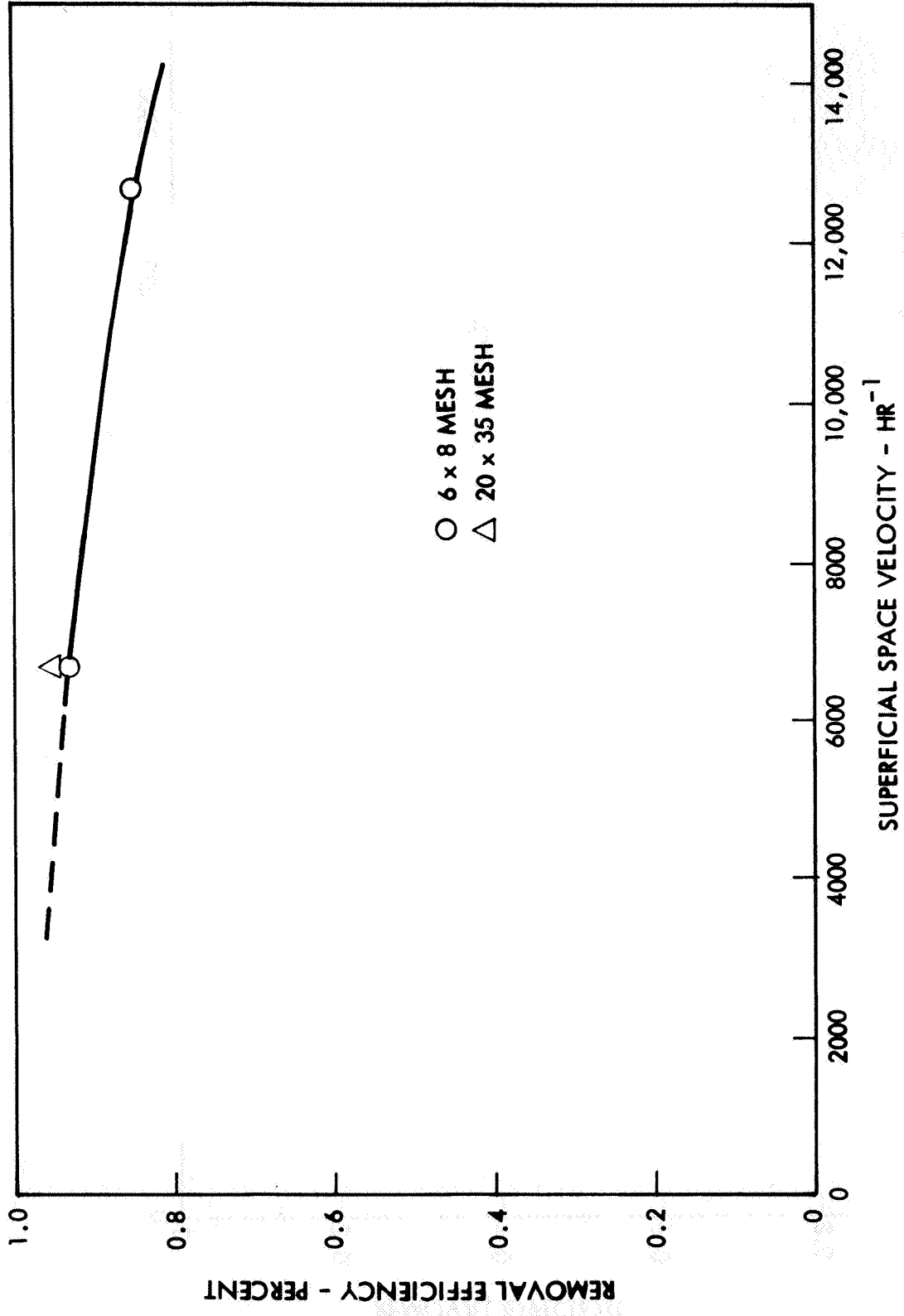


Fig. 1 Dynamic Removal Efficiency of Nitrogen Dioxide by Lithium Hydroxide

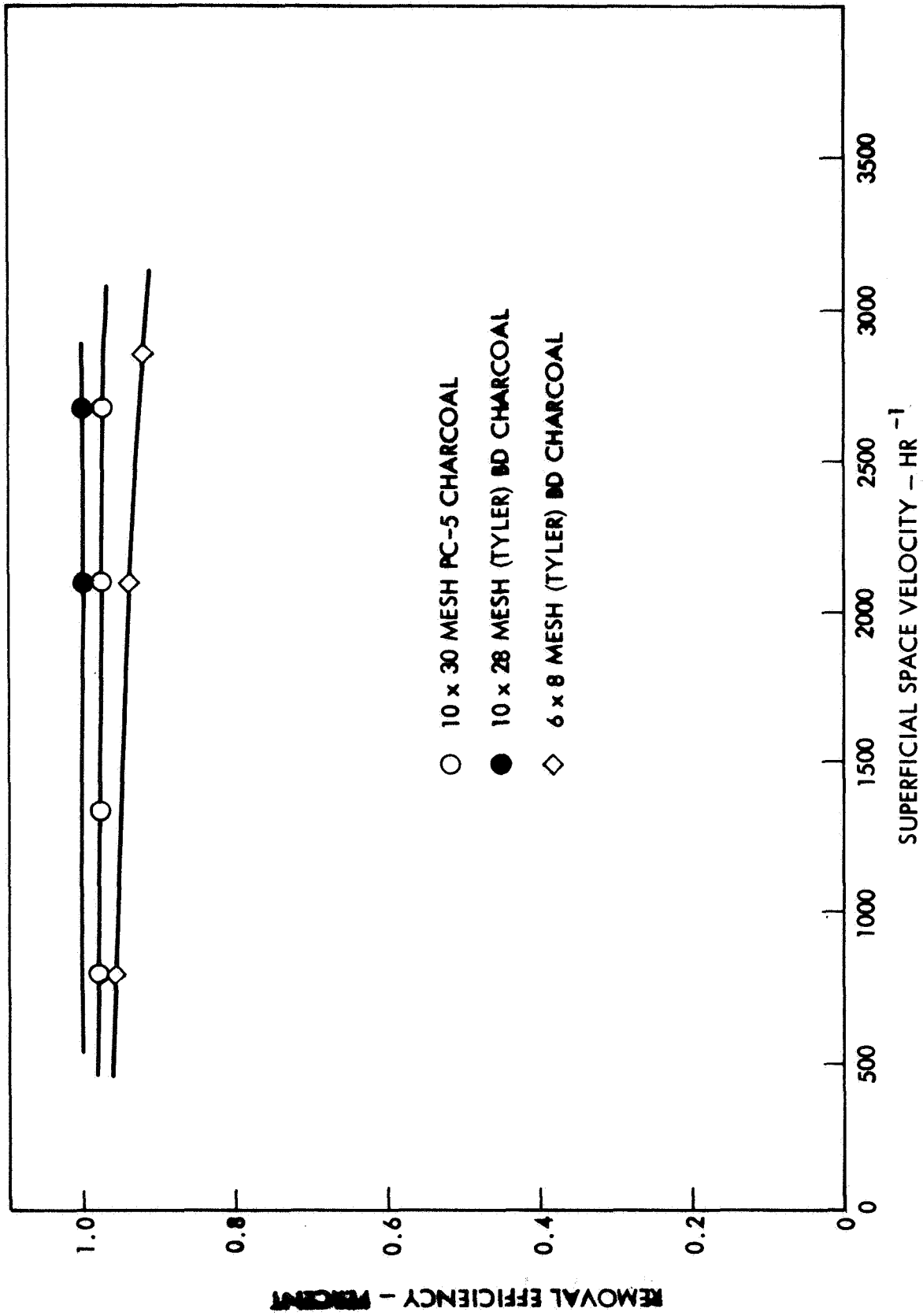


Fig. 2 Removal Efficiency of Vinyl Chloride with Charcoal

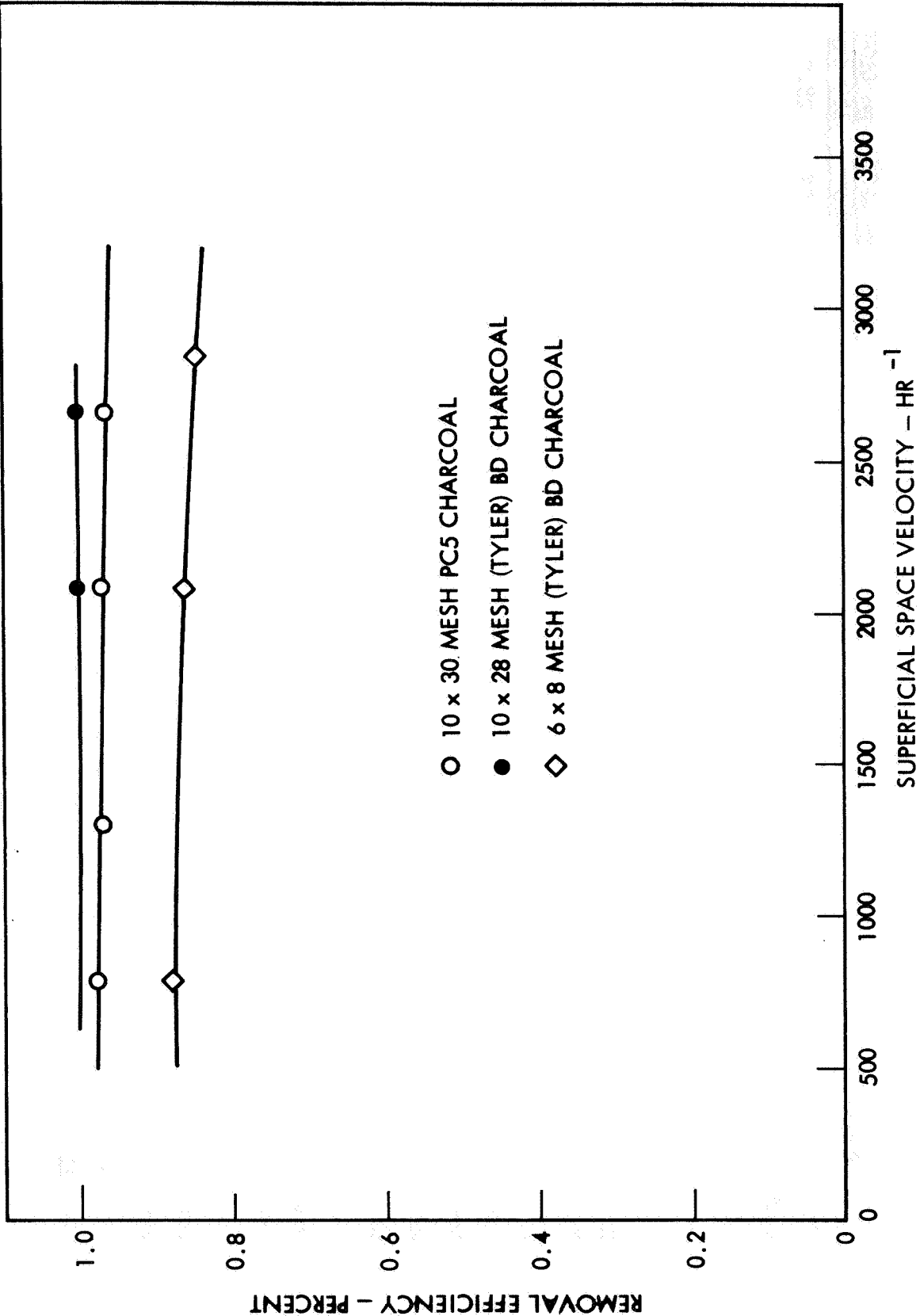


Fig. 3 Removal Efficiency of Freon-12 with Charcoal

TABLE 4

## STOICHIOMETRIC LITHIUM HYDROXIDE REQUIRED FOR CONTAMINANT REMOVAL

Contaminant	IHCOS Production Rate gm/day	Maximum Allowable Concentration Mg/M <sup>3</sup>	Maximum* Removal Rate With 3 cfm & η r=0.9	gm LiOH gm cont	gm LiOH day	gm LiOH 180 day
Carbon Disulphide	0.25	6		0.630	0.157	28.2
Carbon Sulphide	0.25	25		0.704	0.176	31.6
Chlorine	0.25	1.5	0.17	0.676	0.115	20.7
Chloroacetone	0.25	100		0.259	0.065	11.7
2,4 Dichlorobenzene Acid	0.25	205		0.125	0.031	56.2
Epichlorohydrin	0.25	1.9	0.21	-0-	-0-	-0-
Ethyl Mercaptan	0.25	2.5		-0-	-0-	-0-
Hydrogen Chloride	0.25	0.15	0.0165	0.676	0.011	20.2
Hydrogen Fluoride	0.25	0.08	0.0088	0.120	0.001	0.2
Hydrogen Sulfide	0.0007	1.5		0.704	0.0004	0.1
Methyl Mercaptan	0.25	2	0.22	-0-	-0-	-0-
Nitric Oxide	0.25	32		0.800	0.200	36.0
Nitrogen Dioxide	0.25	0.9	0.099	0.520	0.052	9.3
Nitrogen Tetroxide	0.25	1.8	0.198	0.520	0.103	18.6
Propyl Mercaptan	0.25	82		-0-	-0-	-0-
Sulfur Dioxide	0.25	0.8	0.088	0.584	0.051	9.3
Tetrafluorethylene	0.25	205		-0-	-0-	-0-
Vinylidene Chloride	0.25	20		0.298	0.061	11.0
Trichloethylene	2.5	52		0.181	0.453	81.5

\*Not listed when greater than IHCOS production rate.

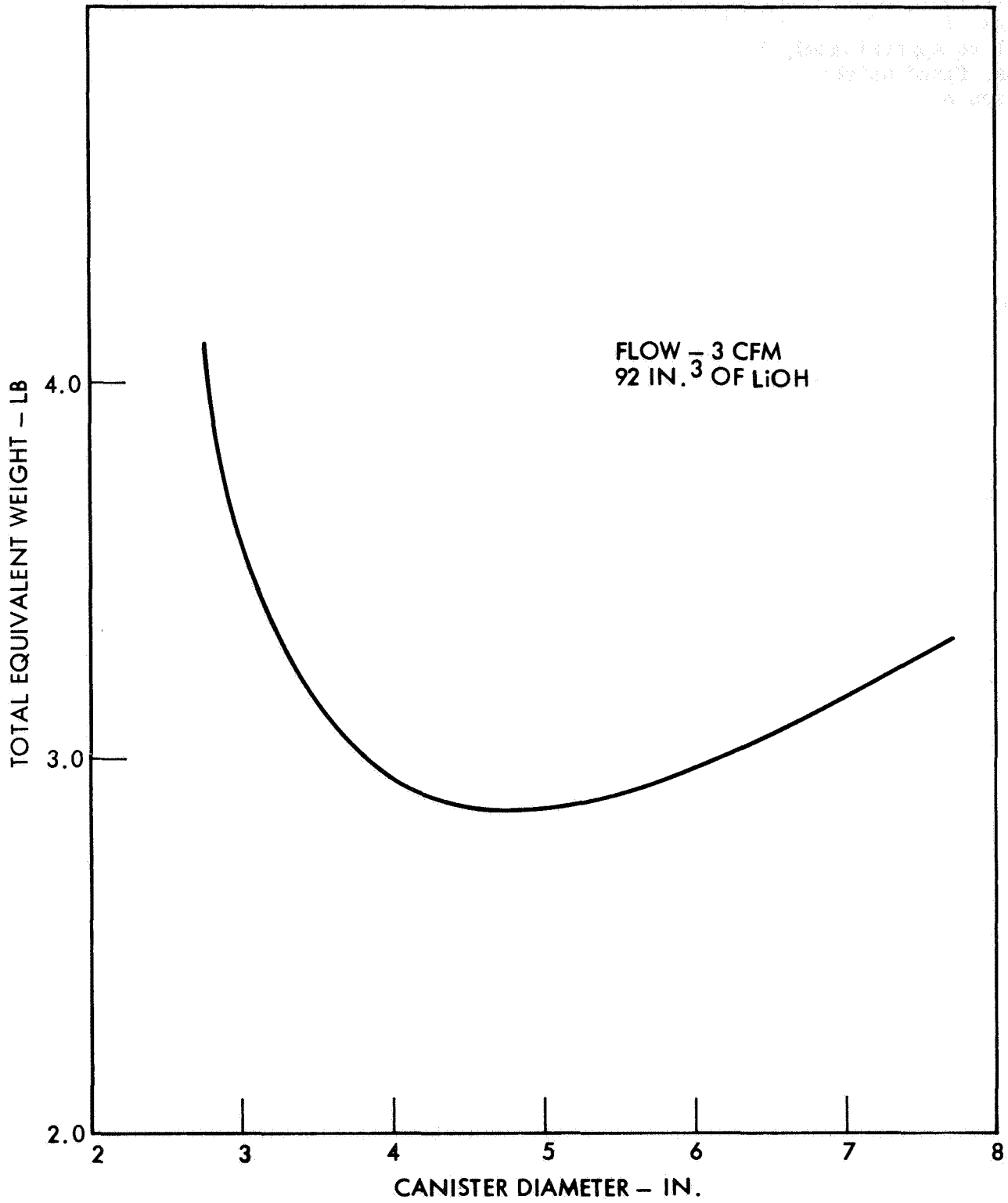


Fig. 4 Pre-sorbent Bed Optimization

veals that the optimum canister diameter is 4.75 inches and the optimum bed length is 5.20 inches. The overall canister length including conical ends will be approximately 11.9 inches and will weigh approximately 1.20 lbs. The total fixed weight with lithium hydroxide will be approximately 2.75 lbs. The pressure drop at 3 cfm and 10 psia will be 0.35 inches of water.

### Main Sorbent Bed Characteristics

In establishing the IHCOS pre-sorbent bed requirements, it was necessary to assess the effects of other components in the life support system on the poisonous contaminant load to the IHCOS. The first step in the assessment was to establish the location of IHCOS in the life support system. A review of possible locations for IHCOS has revealed that the most probable location is down stream of the trace contaminant removal system main sorbent bed and in parallel with the carbon dioxide removal system as shown in Figure 5.

The advantages of this location are that the pressure drop of the CO<sub>2</sub> removal system and the IHCOS are about equal and that the flow requirements of IHCOS plus the CO<sub>2</sub> removal system are equal to the flow requirements of the charcoal main sorbent bed. With this arrangement, the poisonous contaminants removed by charcoal will not reach IHCOS.

To establish the contaminants controlled by charcoal, the equilibrium capacity of the charcoal was estimated for each of the contaminants assumed to be controlled by charcoal. This was accomplished by the use of a correlation termed the "potential plot" which is based on ideas originally proposed by M. Polanyi (ref. 68). This theory relates adsorption capacity to molar volume and vapor pressure of a contaminant through an adsorption potential parameter termed "A".

$$A = T/V_m \log (P_s/P) \text{ where}$$

T = temperature, degrees Kelvin

V<sub>m</sub> = liquid molar volume, at boiling point at  
standard pressure,  $\frac{\text{ml}}{\text{mole}}$

P<sub>s</sub> = saturation pressure

P = vapor pressure

A plot of adsorption capacity vs "A", Figure 6, has been made based on experimental studies (ref. 59, 69). All of the contaminants of interest can be placed on the line by a determination of their "A" values. This generally can be done from the literature, although sophisticated interpretation or extrapolation of the available data may be necessary including estimation of vapor pressure and molar volume from critical constants and molecular weights.

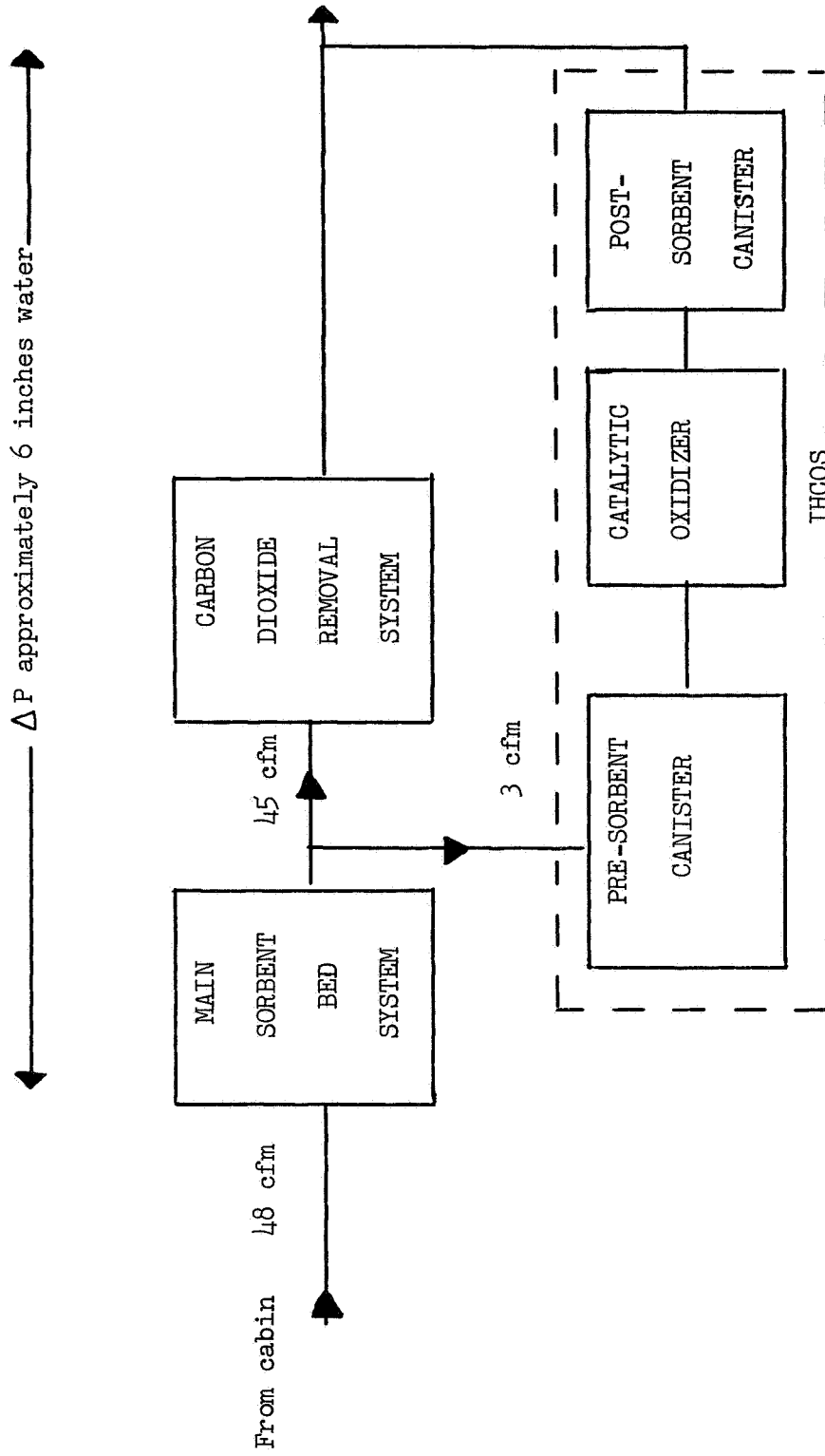


Fig. 5 Proposed IHCOS Location in Space Craft Environmental Control System

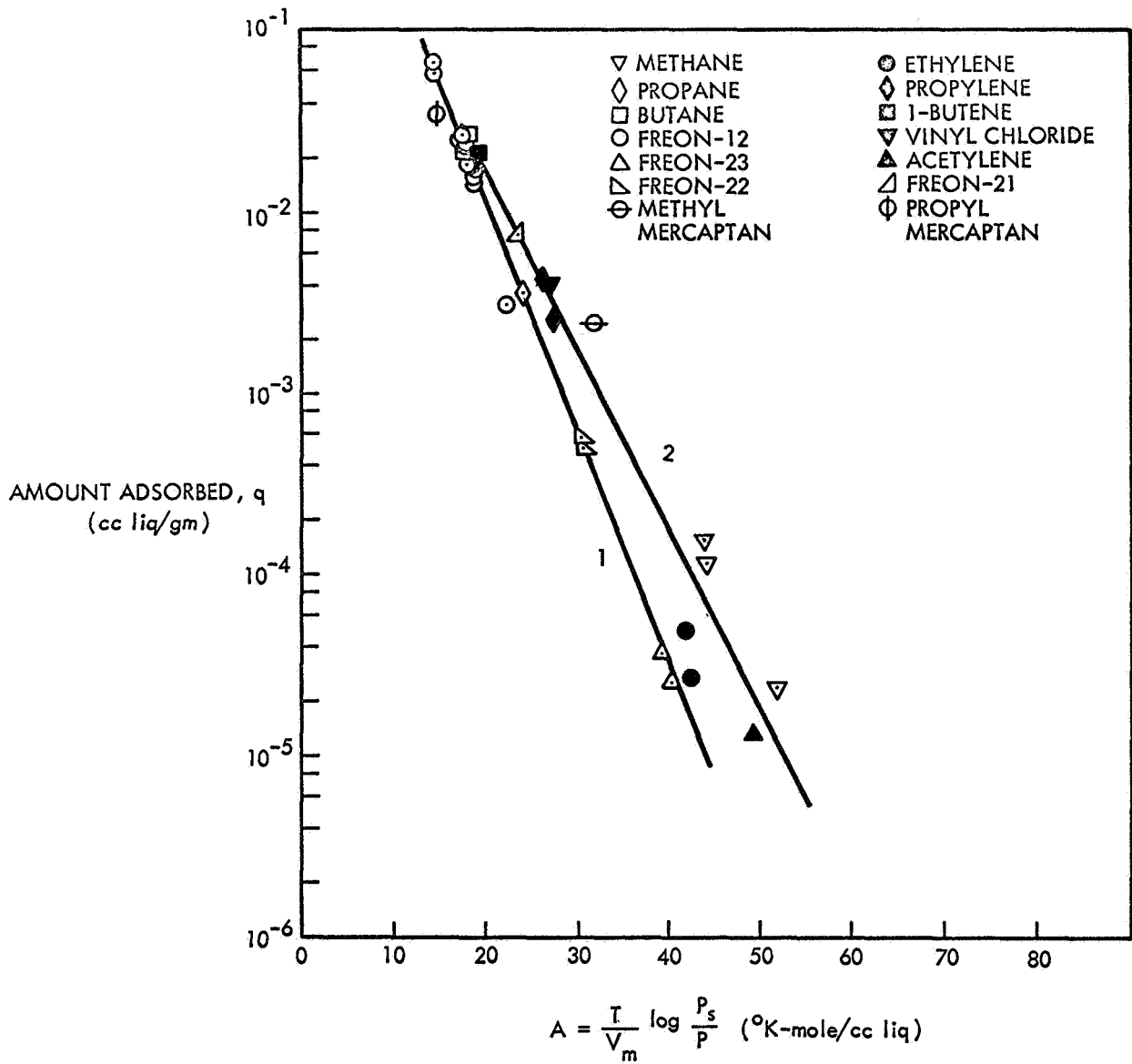


Fig. 6 Potential Plot for BD Charcoal



TABLE 5

## POTENTIAL POISONOUS CONTAMINANTS REMOVED BY CHARCOAL

<u>Contaminant</u>	<u>"A"</u> <u>Value</u>	<u>Charcoal</u> <u>Requirements</u>	
		<u>#/180 Day</u>	<u>#/Day</u>
Carbon Tetrachloride	19.1	3.3	0.0184
Chloroacetone	22.6	3.4	0.0190
Chlorobenzene	14.7	3.0	0.0165
Chlorofluoromethane	40.0	1750.	9.7
Chloroform	24.1	115.*	0.64
Chloropropane	22.6	5.5	0.035
Dichlorobenzene	13.5	2.5	0.014
Dichloroethane	19.3	40.*	0.22
Dimethyl Sulfide	27.0	29.	0.161
Ethylene Dichloride	21.8	3.0	0.017
Ethyl Mercaptan	27.9	29.5	0.165
Ethyl Sulfide	15.8	2.4	0.013
Freon 11	22.0	83.*	0.460
Freon 12	26.8	330.*	1.83
Freon 21	32.0	175.	0.97
Freon 22	40.	770.	4.3
Freon 23	52.	160,000.	890.
Freon 113	18.0	3.3	0.018
Freon 114	18.0	3.3	0.018
Freon 114 (unsym)	22.	8.2	0.045
Freon 125	33.	132.	0.730
Hexamethylcyclotrisiloxane	8.7	0.3	0.002
Methyl Chloride	44.5	880.	4.7
Methyl Chloroform	16.	15.	0.083
Methylene Chloride	32.5	774.*	4.3
Nitromethane	39.	450.	2.5
Iso-Propyl Chloride	22.6	11.	0.056

Table 5 Continued

<u>Contaminant</u>	<u>"A"</u> <u>Value</u>	<u>Charcoal</u> <u>Requirements</u>	
		<u>#/180 Day</u>	<u>#/Day</u>
Propyl Mercaptan	19.8	6.1	0.034
Tetrachloroethylene	17.8	4.7	0.026
Trichloroethylene	16.9	17.5*	0.093
Vinyl Chloride	32.6	1000.*	5.50
Vinylidene Chloride	27.3	21.	0.110

\*Primary Contaminants

Table 5 presents the "A" value for each of the contaminants identified as being controlled with charcoal, including the quantity of charcoal required for removal expressed in lbs/day and lbs for the 180 day mission. It can be seen from examination of this table that the charcoal requirements for 180 days are quite excessive. It is anticipated, however, that for a 180-day mission, a regenerative charcoal system would be utilized. This is the type of system proposed for the Basic Subsystem Module (BSM) (ref. 70).

In this system two charcoal beds are required. The beds are identical, however, one is being regenerated while the other is in the adsorption cycle. The regenerative bed is heated to approximately 300°C and exposed to vacuum. Based on the total contaminant load developed in Phase I of NAS 1-6256, it was concluded that a regenerative charcoal main sorbent system would have the following characteristics.

The system would have a flow rate of 48 cfm (45 cfm to the CO<sub>2</sub> removal and 3 cfm for IHCOS) which is sufficient to control all of the contaminants listed that are absorbed on charcoal, with the exception of dimethylhydrazine, monomethyl hydrazine and pyruvic acid. Ammonia and hydrogen fluoride are also flow limited at 48 cfm, but are not adsorbed on charcoal. However, ammonia can be controlled with an acid impregnation on charcoal. For the purpose of sizing the main sorbent beds, it was assumed that the charcoal requirements reflected in the potential poison list Table 4, are typical of the requirements which would result from an evaluation of all contaminants listed in the Phase I IHCOS contaminant load. In establishing the performance of a bed of this type, the potential plot theory assumes that with low loading the contaminants act independently. Therefore, a bed sized for the contaminant requiring the greatest amount of charcoal (highest "A" value) will satisfactorily adsorb all other contaminants. This theory has been modified slightly to account for the displacement of high "A" value contaminants by low "A" contaminants (ref. 69).

One of the conclusions of this program was that contaminants with an "A" value greater than 25 would not displace other contaminants, for contaminants with an "A" value less than 25 and with a difference in "A" value greater than 20 the lower "A" value contaminant will block the surface to or displace the higher "A" contaminant.

Inspection of the contaminants in Table 5 reveals that Freon 23 is the most difficult contaminant to control followed by chlorofluoromethane.

Freon 23 with an assumed design production rate of 0.25 grams per day would require 890 lbs per day of charcoal, which is prohibitive. Sizing the bed for chlorofluoromethane reduces the required charcoal quantity to 9.7 lbs per day. With this quantity of charcoal, the capability for removal of Freon 23 would be 0.0027 grams per day. Specific sorbents for Freon 23 have been investigated and no sorbent superior to charcoal was found. Experimental results would be desirable to verify the capacity of charcoal for Freon 23. The use of Freon 23 as a spacecraft refrigerant or solvent should be restricted.

Based on a 9.7-lb per day charcoal requirement and a cycle time of 3.5 days, each bed would contain 34 lbs of charcoal. To account for displacement effects of contaminants with an "A" value below 25 interfering with contaminants of higher "A" value additional charcoal must be added. Taking a contaminant with an "A" value of 25 and a production rate of 2.5 grams per day, the required quantity of charcoal for removal is approximately 1.0 lb per day. Adding this increment of charcoal should provide removal for all contaminants with "A" values less than 25. This would leave the remainder of the bed free for higher "A" value contaminants.

Thus each of the beds sized for a 3.5 day cycle would require 38 lbs of charcoal.

## LONG TERM SORBENT BED EVALUATION

The long term evaluation of the pre- and post-sorbent beds and catalytic oxidizer for the Isotope Heated Catalytic Oxidizer System was performed for a period of 180 days, beginning on January 22, 1968, and ending on July 19, 1968. This section presents the objectives, apparatus, and procedures used, the results obtained, and a discussion of the results.

### Objective

The primary objective of this test effort was to determine the long-term performance characteristics of the lithium hydroxide pre- and post-sorbent beds, and the catalytic oxidizer system. Additional objectives included evaluating the effects of the regenerative main sorbent system and the silica gel portion of a carbon dioxide removal system. This effort was accomplished by monitoring the removal efficiency and removal mechanism of various selected contaminants at points throughout the system during a 180-day test period.

### Apparatus

The test apparatus is presented schematically in Figure 7 and is illustrated in Figures 8 and 9. Listed below are the major items used in the test.

- o Cylinders for gaseous contaminant supply and for portions of the background gas.
- o Pressure gauges and regulator to measure and control system pressure.
- o Inlet and exit sampling septa for obtaining gas samples.
- o Preheater for heating incoming gas to the catalyst bed.
- o Catalytic oxidizer tube to contain catalyst (catalyst volume = 57 cc).
- o Furnace and temperature controller to control catalyst bed temperature.
- o Air cooled heat exchanger for cooling exit gas from catalyst bed.

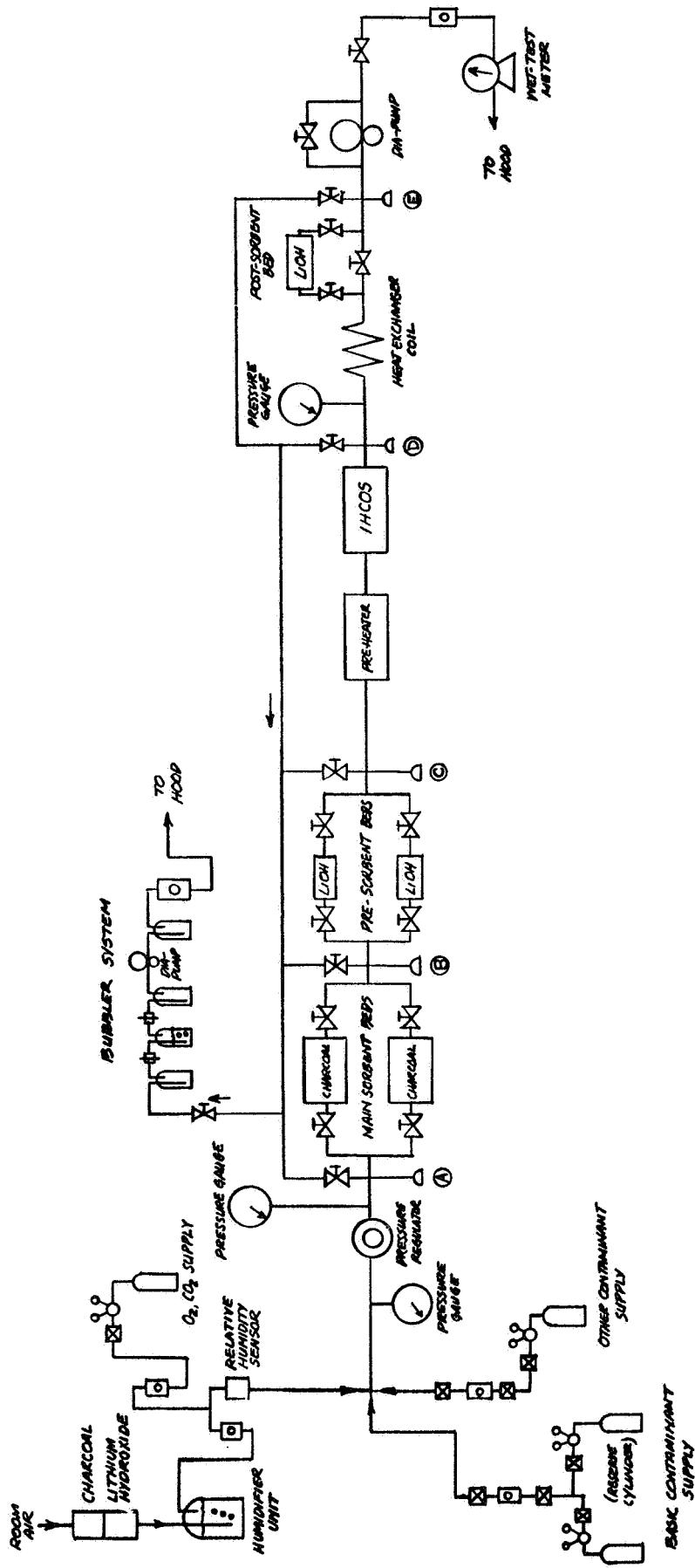


Fig. 7 Long Term Sorbent Bed Evaluation Test Apparatus

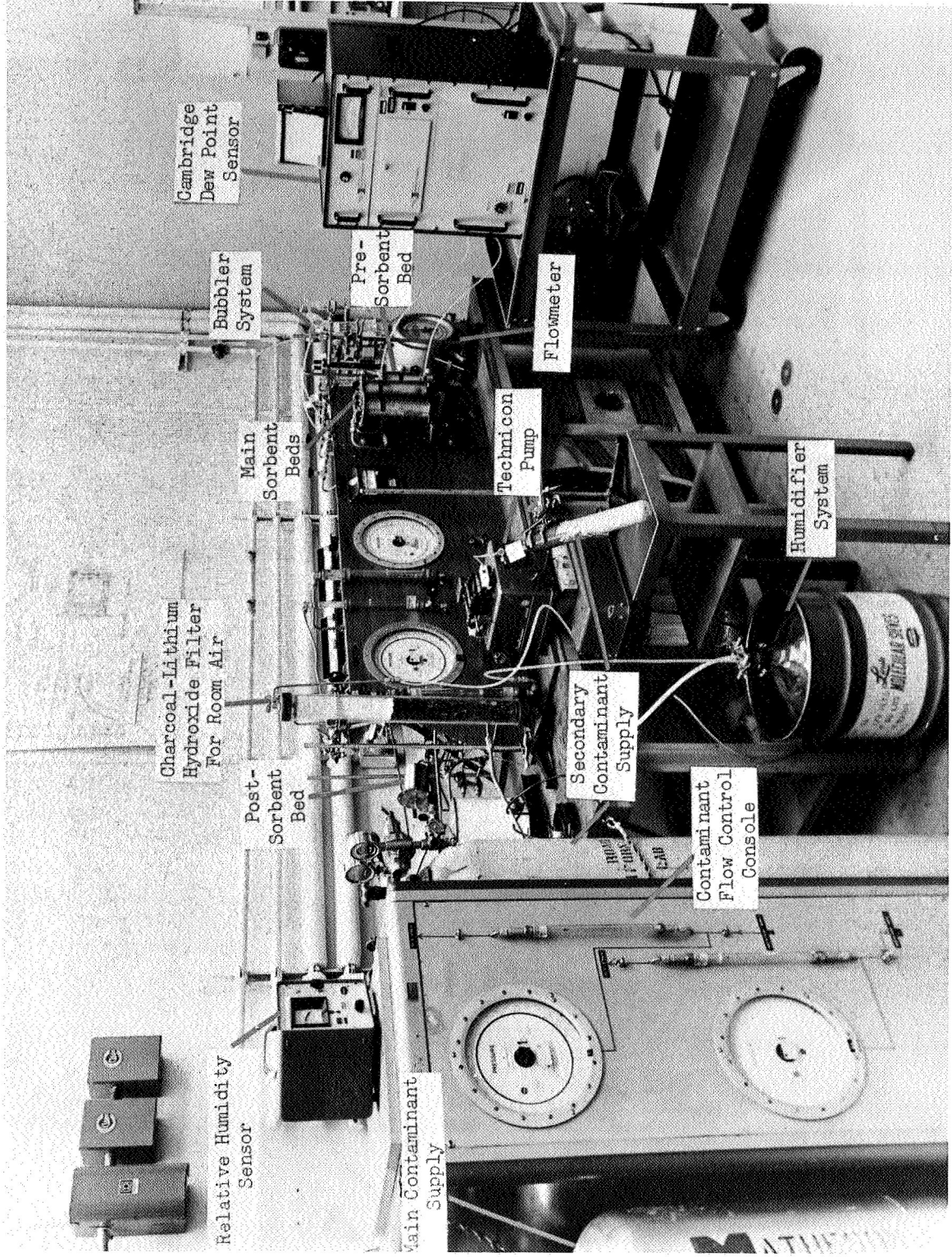


Fig. 8 Long Term Sorbent Bed Test Apparatus

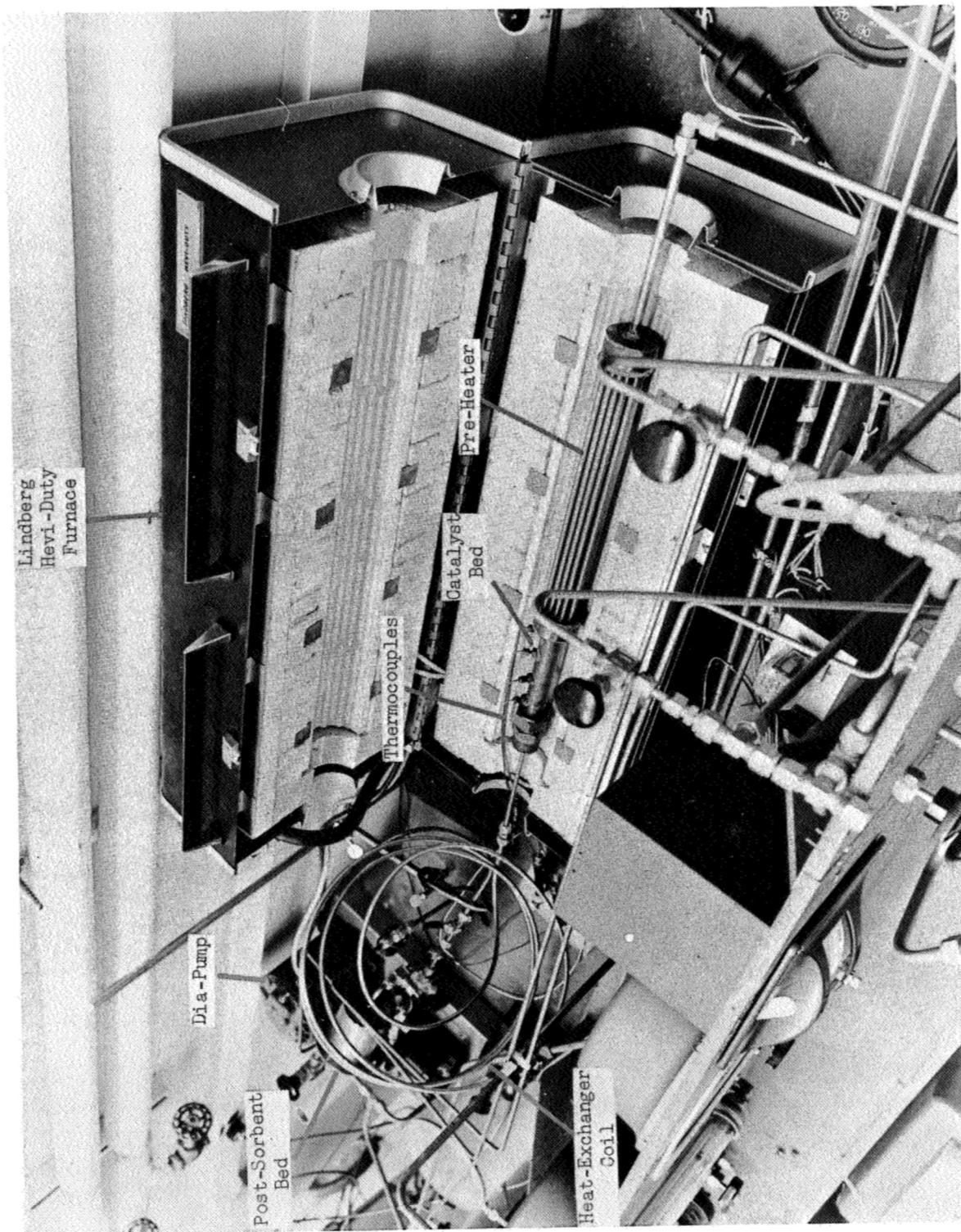


Fig. 9 Furnace, Pre-heater, Catalyst Bed, and Post-Sorbent Bed



- o Diaphragm pump and flow control valves for maintaining pressure and for varying system flow rate.
- o Flowmeter and wet test meter to determine system flow rates.
- o F & M gas chromatographs Model 720, 1609, 810, 700A, and 700B, equipped with flame ionization, electron capture and thermal conductivity detectors for contaminant analysis.
- o Water humidifier system for moist-inlet gas stream conditions.
- o HygroDynamics, Inc., electronic hygrometer-indicator to determine humidity of inlet gas stream.
- o Lithium hydroxide and activated charcoal adsorbent beds to remove contaminants present in room air.
- o Main sorbent beds containing Barnebey-Cheney 8 x 10 mesh BD charcoal.
- o Pre-sorbent and post-sorbent beds containing 6 x 8 mesh Foote Mineral Co. environmental grade lithium hydroxide.
- o Diaphragm pump, flow meter and pyrex glass bubblers for colorimetric analysis.
- o Perkin-Elmer Model 202 Spectrophotometer for colorimetric analysis.
- o Technicon proportioning pump for introducing gaseous contaminants into the system.
- o Cambridge Hygrometer for measuring the effluent dew point from the silica gel beds.

#### Procedure

The long term "sorbent" and catalyst bed performance test data were obtained with the system operating at a space velocity of  $21,000 \text{ hr}^{-1}$ , with an average catalyst bed temperature of  $680 \pm 20^\circ\text{F}$ . The total system pressure was held constant at 10 psia. The background gas consisted of the contaminant mixture used during the previous 180 day catalyst test conducted during NAS 1-6256 (acetylene, n-butane, carbon monoxide, ethane, propylene and methane), 160 mmHg oxygen, 4 mmHg carbon dioxide, water vapor, and balance nitrogen. Gas supplies were introduced via distribution consoles from pressurized gas cylinders, with the exception of the filtered air drawn from the

room. The system inlet dew point was maintained at approximately 55°F with a water bubbler humidifying system. Other contaminants, potential poisons and producers of undesirable products, were added to the system at different time intervals over the 180-day period. The contaminants introduced and the analysis techniques are indicated in Table 6 and described in the following sections.

Contaminant introduction.-The following contaminants: hydrogen sulfide, methyl chloride, nitrogen dioxide, sulfur dioxide, and ammonia, were introduced directly into the system using a Technicon Proportioning Pump which has a minimum delivery rate of 0.015 cc per minute. Also, sulfur dioxide and nitrogen dioxide were introduced through the distribution console, using calibrated gas cylinders containing 0.48% and 0.75 (by volume) of SO<sub>2</sub> and NO<sub>2</sub> respectively. Similar techniques were applied to Freon-12, vinyl chloride, and Freon-114. Known concentration mixtures were obtained in pressurized gas cylinders and were introduced at the desired concentration levels into the system through distribution consoles.

The periods during which these contaminants were introduced are shown in Figure 10. Freon-12 and vinyl chloride were introduced into the system continually except during the following periods: 82nd to 98th days, 103rd through 108th days, and 116th through 142nd days. Methyl chloride was introduced into the system on the 37th day and on the following day the flow was terminated. Ammonia was introduced on the 44th through the 64th day. Hydrogen sulfide was introduced during the 108th through the 141st days and 177th through the 180th days while Freon-114 was introduced during the 115th through the 142nd days and the period between the 173rd and 180th days.

A change in the NO<sub>2</sub> and SO<sub>2</sub> introduction point was made on the 64th day. The contaminants were introduced at the outlet of the main charcoal bed at point B. NO<sub>2</sub> was turned off on the 79th day and SO<sub>2</sub> on the 92nd day. NO<sub>2</sub> and SO<sub>2</sub> were again introduced into the system ahead of the main charcoal bed on the 109th day through the 143rd day.

Sample location.-Samples for chemical analysis were obtained at points A, B, C, D, and E (see Figure 7). Methane and carbon monoxide were analyzed daily at points C and D. Freon-12 and vinyl chloride were analyzed at points A, C and D. Acetylene, n-butane, ethane and propylene were analyzed once weekly at points C and D. Nitrogen dioxide and sulfur dioxide were analyzed at points A, B, C and D; methyl chloride and Freon-114 at points A, C, and D; HCl and HF at points D and E; ammonia at points C and D.

Chemical analysis techniques.-All hydrocarbons and carbon monoxide were analyzed by gas chromatographic techniques using the F & M Models 1609, 700, and 810 gas chromatographs. The Model 700 gas chromatograph units were operated with Model 810 electrometers. All of these contaminants were analyzed by flame ionization detection. The halogenated compounds such as Freon-12 and Freon-114 were also analyzed by gas chromatography using electron capture detection.

TABLE 6

## CONTAMINANT INTRODUCTION AND ANALYSIS

Introduction Periods Days	Contaminants Introduced	Desired Inlet Concentration (Mg/M <sup>3</sup> at 10psia)	Sample Analysis Location (3)	Chemical Analysis Method
1 through 180	Methane	1,720	C,D	Gas Chromatography
1 through 180	Carbon Monoxide	29	C,D	Gas Chromatography
1 through 180	Acetylene	180	C,D	Gas Chromatography
1 through 180	Propylene	180	C,D	Gas Chromatography
1 through 180	n-Butane	180	C,D	Gas Chromatography
1 through 180	Ethane	180	C,D	Gas Chromatography
37 through 38	Methyl Chloride	25	A, C,D	Gas Chromatography
44 through 64	Ammonia	3.5	C,D	Colorimetry
61 through 80 (1)	HCl (1)	(1)	D	Colorimetry
1-79, 109-143, and 172-180	NO <sub>2</sub>	0.9	A,B,C,D	Colorimetry
1-92, 109-143, and 172-180	SO <sub>2</sub>	0.8	A,B,C,D	Colorimetry
108 through 141	H <sub>2</sub> S	1.5	A, C,D	Colorimetry
115-142, and 173-180	Freon-114	5.6	A, C,D	Gas Chromatography
114 through 172 (1)	HCl (1)	(1)	D,E	Colorimetry
114 through 172 (1)	HF (1)	(1)	D,E	Colorimetry
(2)	Freon-12	0.46	A, C,D	Gas Chromatography
(2)	Vinyl Chloride	1.5	A, C,D	Gas Chromatography

(1) Contaminants were products of oxidation.

(2) Introduced continuously except 82-98th, 103-108th, and 116-142nd days.

(3) Reference Figure 7

Contaminants Introduced

CH<sub>4</sub>, CO, Acetylene,  
n-Butane, Ethane,  
Propylene

Methyl Chloride

Ammonia

Freon-12

Vinyl Chloride

H<sub>2</sub>S

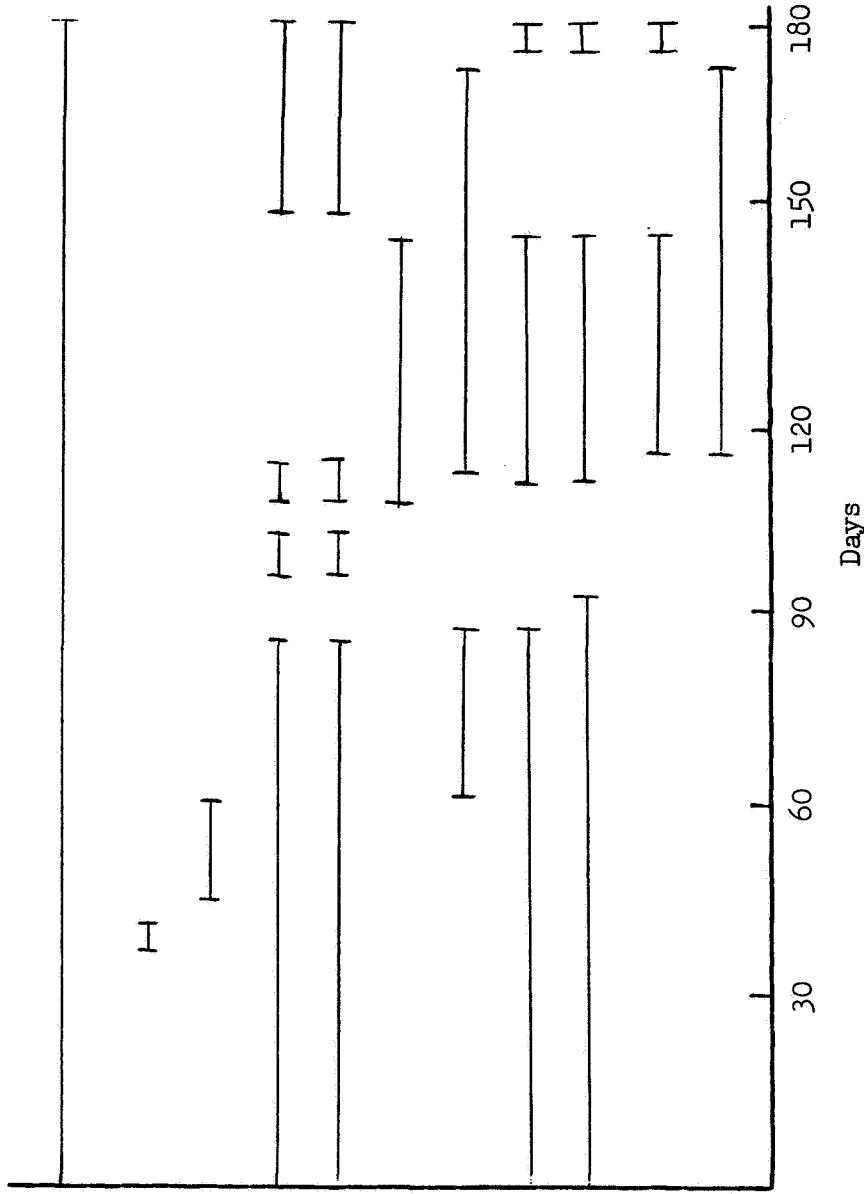
HCl (1)

NO<sub>2</sub>

SO<sub>2</sub>

Freon-114

HF (1)



(1) Contaminants were analyzed as products of oxidation.

Fig. 10 Contaminant Introduction Periods

The analytical technique used for  $\text{SO}_2$  is described by Jacobs (ref. 72). This technique used a 0.04M solution of sodium tetrachloro-mercurate as an absorbing reagent and a hydrochloric acid-bleached solution containing para-rosaniline as a dye reagent. Standardization was performed with known solutions of sodium bisulfite and absorption measured at 560 $\mu$ . Samples for colorimetric analyses were collected by using the bubbler system shown in Figure 8.

The bubbler system consisted of three 100 cc pyrex glass bubblers arranged in series and connected by clear tygon tubing. The first and third bubblers acted as protective traps and the middle bubbler contained the absorbing reagent. This bubbler was filled with 20 cc of reagent and system atmosphere was passed through at a rate of 200 cc per minute. The length of bubbling varied from 5 to 120 minutes, depending on the concentration and kind of contaminant being analyzed. The technique used for the determination of  $\text{NO}_2$  was the direct absorption method described by Saltzman (ref. 71). This technique used a solution containing sulfanilic acid, N-(1-naphthyl)-ethylene diamine dihydrochloride, acetic acid, and Kodak Photoflo which acted both as the absorbing and dye reagent. Color development was almost instantaneous and the light absorption was read at 550 $\mu$  (millimicrons). Standardization was performed with known solutions of potassium nitrite ( $\text{KNO}_2$ ). Hydrogen sulfide analysis was performed using the colorimetric technique described by Jacobs (ref. 72). This technique uses a dilute alkaline solution of cadmium sulfate as an absorbing reagent and a dye reagent containing para-aminodimethylaniline in sulfuric acid solution, which reacts with ferric and chloride ions to form methylene blue. Standardization was performed with known solutions of methylene blue and the absorption was measured at 670  $\mu$ .

Ammonia analysis was performed daily using the colorimetric technique described in the Technicon Methodology (ref. 73). This technique used 0.1 N sodium hydroxide as an absorbing reagent and a combination of a solution of alkaline phenol and a solution of alkaline hypochlorite as a dye reagent. Standardization was performed with known solutions of ammonium sulfate, and absorption was measured at 610  $\mu$ . HCl analysis was performed by using the turbidimetric method described in Jacobs (ref. 72). This technique used 0.1 N silver nitrate solution with 0.1 N sodium hydroxide used as the absorbing reagent. Standardization was performed with known solutions of sodium chloride. HF was colormetrically analyzed by the SPADNS method as described in Standard Methods (ref. 74). This technique used a dye reagent containing sodium 2 - (para-sulfophynylazo) - 1,8 dihydroxy-3, 6-napthalene disulfonate and zirconyl chloride octahydrate in dilute hydrochloric acid. The absorbing reagent was 0.1 N NaOH solution and standardization was performed by using known solutions of sodium fluoride. Absorption was measured at 560  $\mu$ .

Main sorbent charcoal beds.-The main sorbent beds, represented by canisters A and B, were filled with Barnebey-Cheney 8 x 10 mesh (Tyler) BD charcoal which was prepared by screening 4 x 10 mesh charcoal. The beds were initially planned to be on line for one week after which time a fresh bed was to be switched on line. However, during the course of the experiment, it was deter-

mined that the beds should be switched more frequently. The beds were then changed three times weekly: Monday at 8:30 a.m., Wednesday at 5:00 p.m., and Friday at 5:00 p.m. While one charcoal bed was on line, the contents of the other was removed, weighed, desorbed by applying vacuum and heat (200°C for 10 hours) cooled to room temperature, re-weighed and re-packed. Also, other charcoal adsorbents were tested such as Barnebey-Cheney 8 x 12 mesh GI-7883 charcoal which was used on the 54th--56th days and Barnebey-Cheney 8 x 12 mesh KE which was used on the 59th and 60th days.

Pre-sorbent lithium hydroxide bed.-The lithium hydroxide pre-sorbent canister was loaded with 78.5 grams of lithium hydroxide and placed on line at the outset of the test. No changes were made to this bed during the 180 day test.

Catalyst bed.-The catalyst bed was loaded with 57 cc of 0.5% Pd on alumina catalyst (Englhard, Lot No. 11-707). The catalyst bed was operated at a temperature of 680°F until the 86th day. Some modifications were made in the test conditions between the 87th and 94th days in an attempt to restore catalyst activity. On the 87th through 92nd days, the catalyst bed temperature was raised to 800°F and the total system outflow was reduced to 4600 cc per minute. On the 89th day, the water bubbler was stopped and a canister with 7 pounds of silica gel was placed on line to reduce the moisture in the inlet system flow. On the 92nd day, the silica gel canister was removed, the water bubbler re-installed, the catalyst bed temperature lowered to 683°F and the outlet flow increased to 6700 cc per minute. Since the catalyst did not recover its initial activity as a result of these changes, the test was halted. The catalyst bed was removed and re-packed with new catalyst. The test was then continued with the new catalyst operating at the original temperature of 680°F. The old catalyst was re-weighed and analyzed by infrared spectroscopy.

Prior to the last thirty-seven days of the 180 day test period, system modifications were made for post-sorbent evaluation. A post-sorbent canister loaded with 181 grams of lithium hydroxide was placed on line at the outlet of the catalyst bed on the 143rd day. The outlets of the catalyst bed (D) and the post-sorbent bed (E) were analyzed daily for oxidation products, hydrogen chloride and hydrogen fluoride during the period between the 143rd and 172nd days. Analysis for intermediate oxidation products was performed by gas chromatography, specifically using the flame ionization and electron capture detectors.

Silica gel bed.-The main sorbent charcoal beds were removed and each filled with 600 grams of Davison 6 x 8 mesh (Grade 40, Type 2) silica gel, and placed back on line on the 172nd day bypassing the humidifier system. Freon-12, vinyl chloride, and NO<sub>2</sub> were introduced into the system along with the basic background contaminants. Contaminant removal performance was determined before water saturation by sampling the contaminant inlet and outlet bed concentrations. The dew point of the silica gel bed outlet was monitored with a Cambridge Hygrometer system that was connected in parallel with the LiOH bed.

Post-test chemical and physical analysis.-After the long term test was completed, the following chemical and physical tests were performed. The lithium hydroxide pre-sorbent and post-sorbent beds were examined visually and for acidity. Also, both beds were re-weighed, and their weight gains calculated. The 0.5% palladium catalyst was removed, examined visually, and re-weighed. The tubing used for cooling the outlet gas stream from the catalyst bed was removed, examined visually for corrosion, and analyzed chemically. Chemical analysis was performed by washing the tubing with 100 cc of distilled water (10 times), evaporating the wash water to 45 cc, and analyzing specifically for NO<sub>2</sub>, SO<sub>2</sub>, HCl and HF.

### Results

The system inlet dew point data is presented in Figure 11. Methane inlet concentration data, catalyst bed conversion efficiency, and catalyst bed temperature throughout the 180-day test period are presented in Figure 12. Carbon monoxide inlet concentration and catalyst bed conversion efficiency data are presented in Figure 13. Catalyst bed conversion efficiency data and inlet concentrations for propylene, ethane, n-Butane and acetylene are presented in Figure 14. Vinyl chloride and Freon-12 concentration data at the inlet and outlet of the charcoal bed and at the outlet of the catalyst bed is presented in Figure 15. Freon-114 and hydrogen sulfide concentration data at the inlet and outlet of the charcoal canister, the lithium hydroxide canister outlet and the catalyst bed outlet are presented in Figure 16. Hydrogen chloride and hydrogen fluoride concentration data at the inlet and outlet of the post sorbent canister is presented in Figure 17. Nitrogen dioxide concentration data at the inlet and outlet of the charcoal canister, outlet of the lithium hydroxide canister and outlet of the silica gel canister is presented in Figure 18. Sulfur dioxide concentration data at the inlet and outlet of the charcoal canister and outlet of the lithium hydroxide canister is presented in Figure 19. Ammonia concentration data at the inlet and outlet of the catalyst bed is presented in Figure 20.

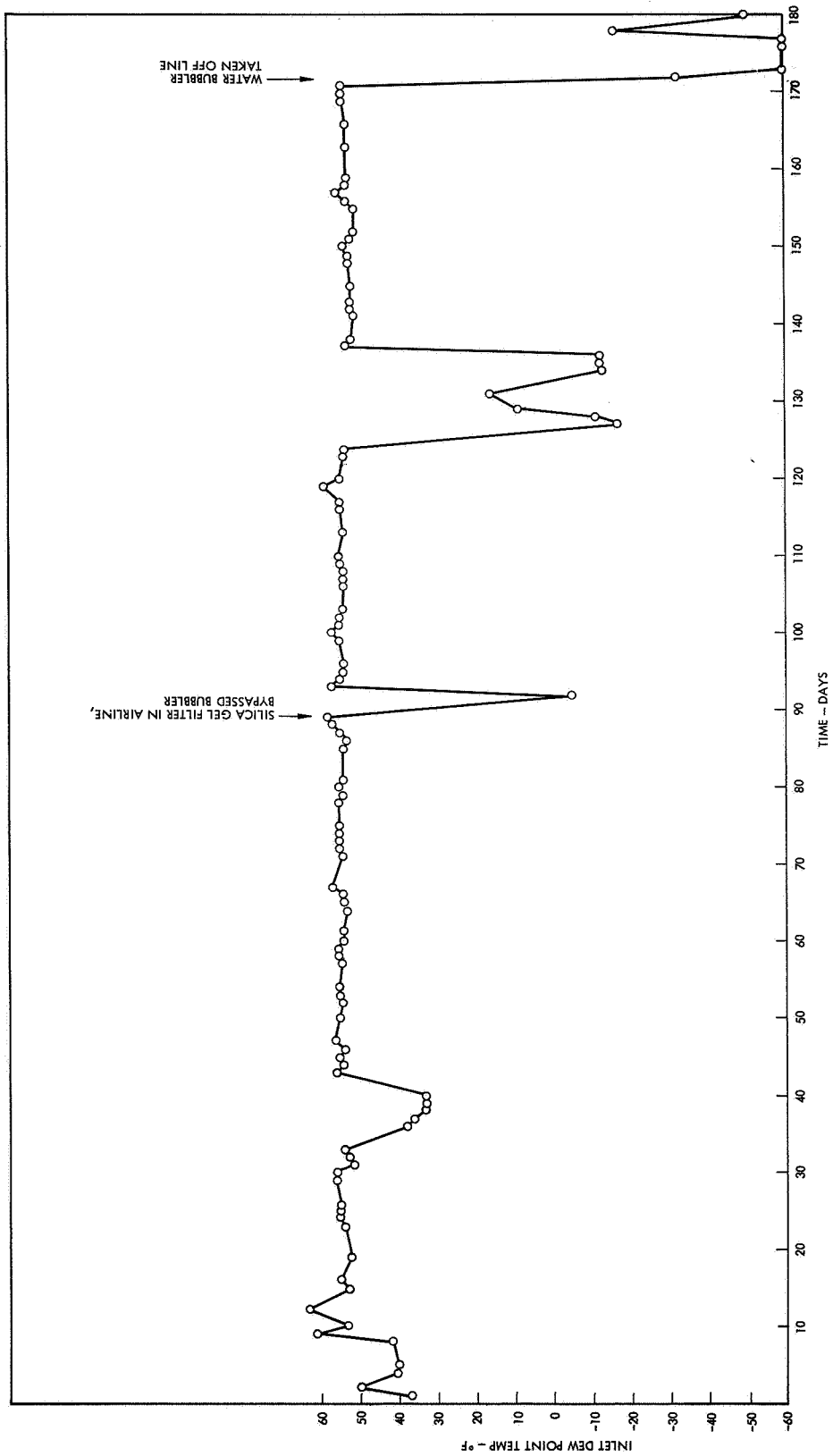


Fig. 11 System Inlet Dew Point Temperature



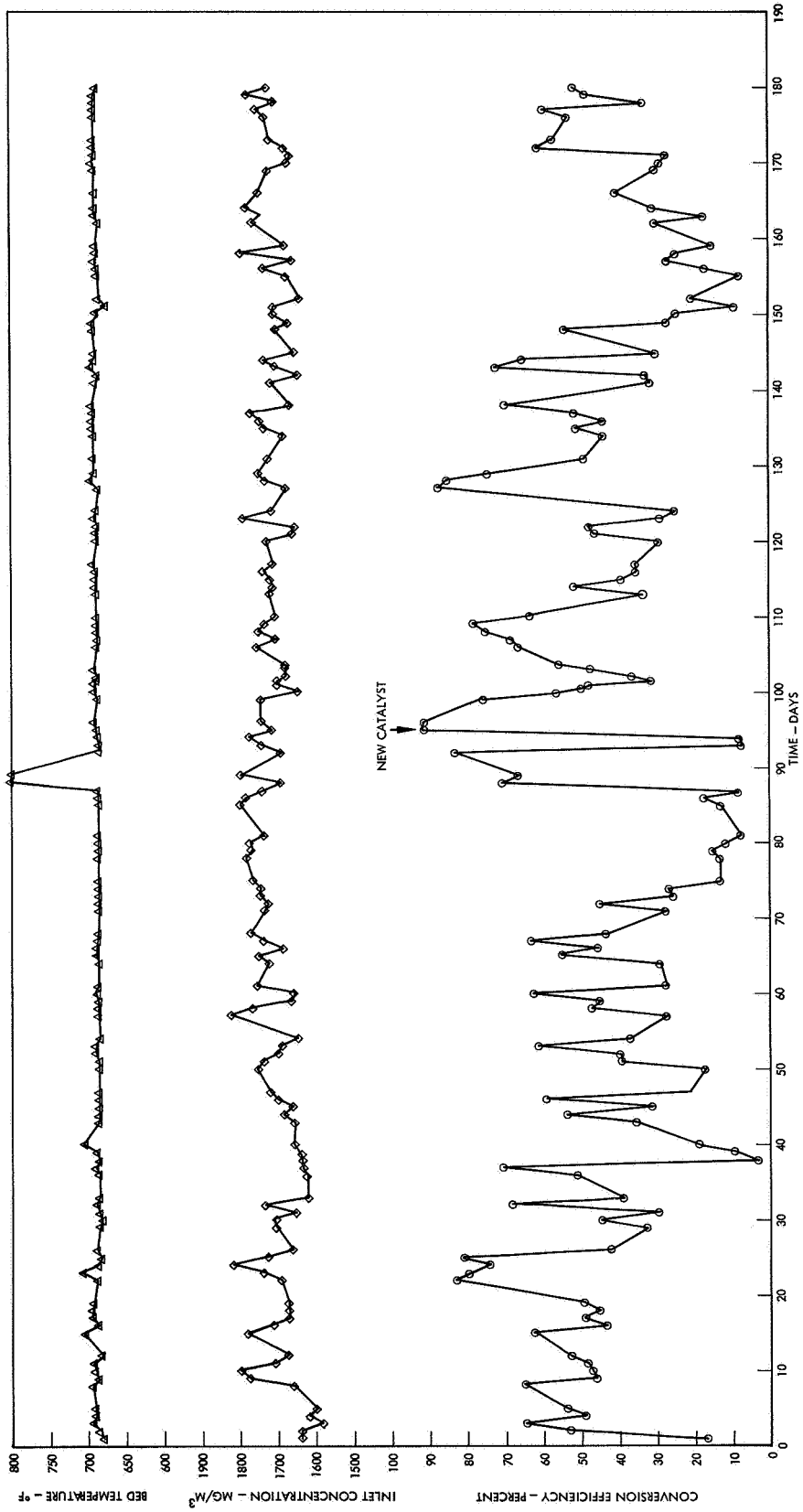


Fig. 12 Methane Performance

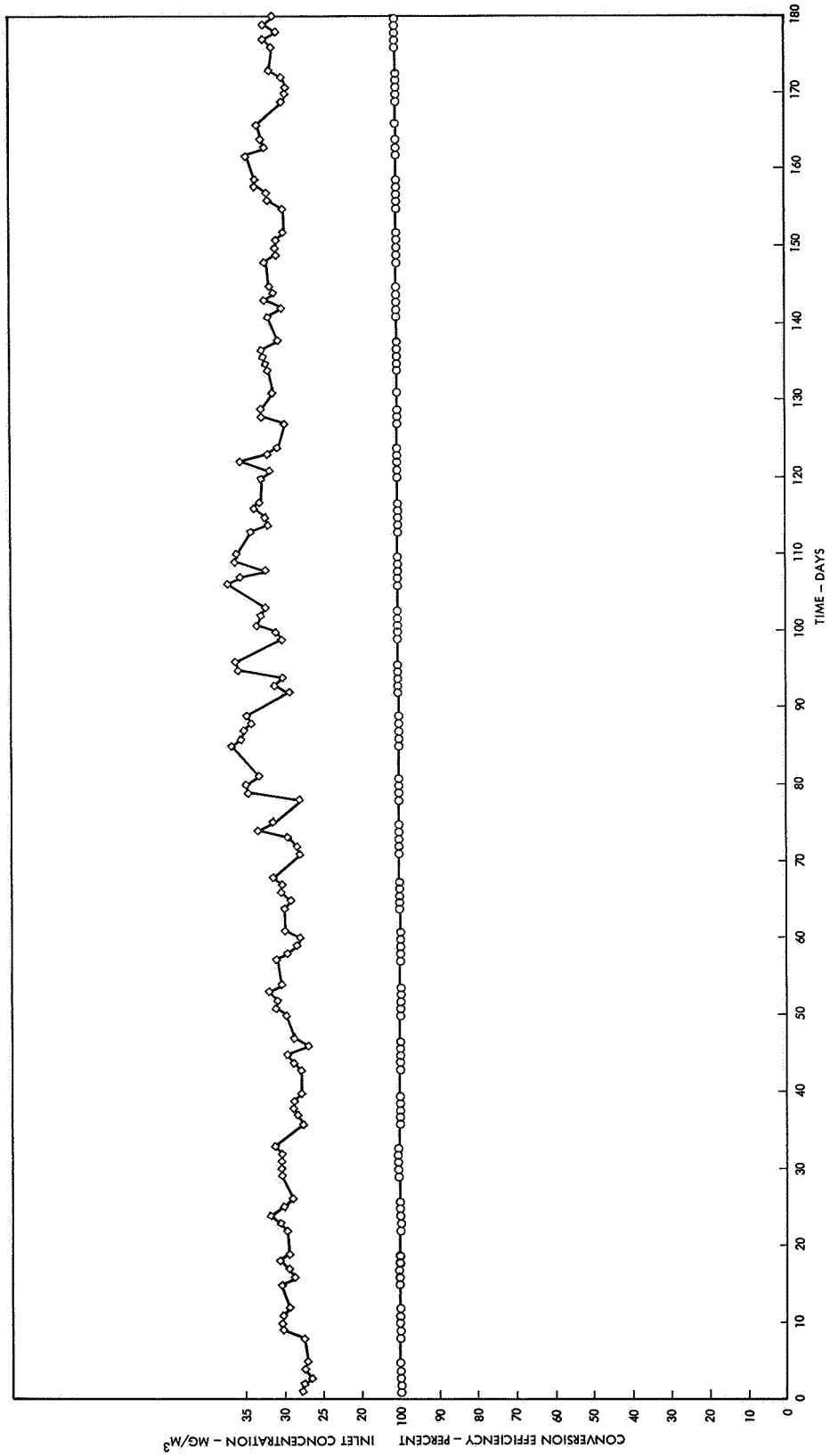


Fig. 13 Carbon Monoxide Performance

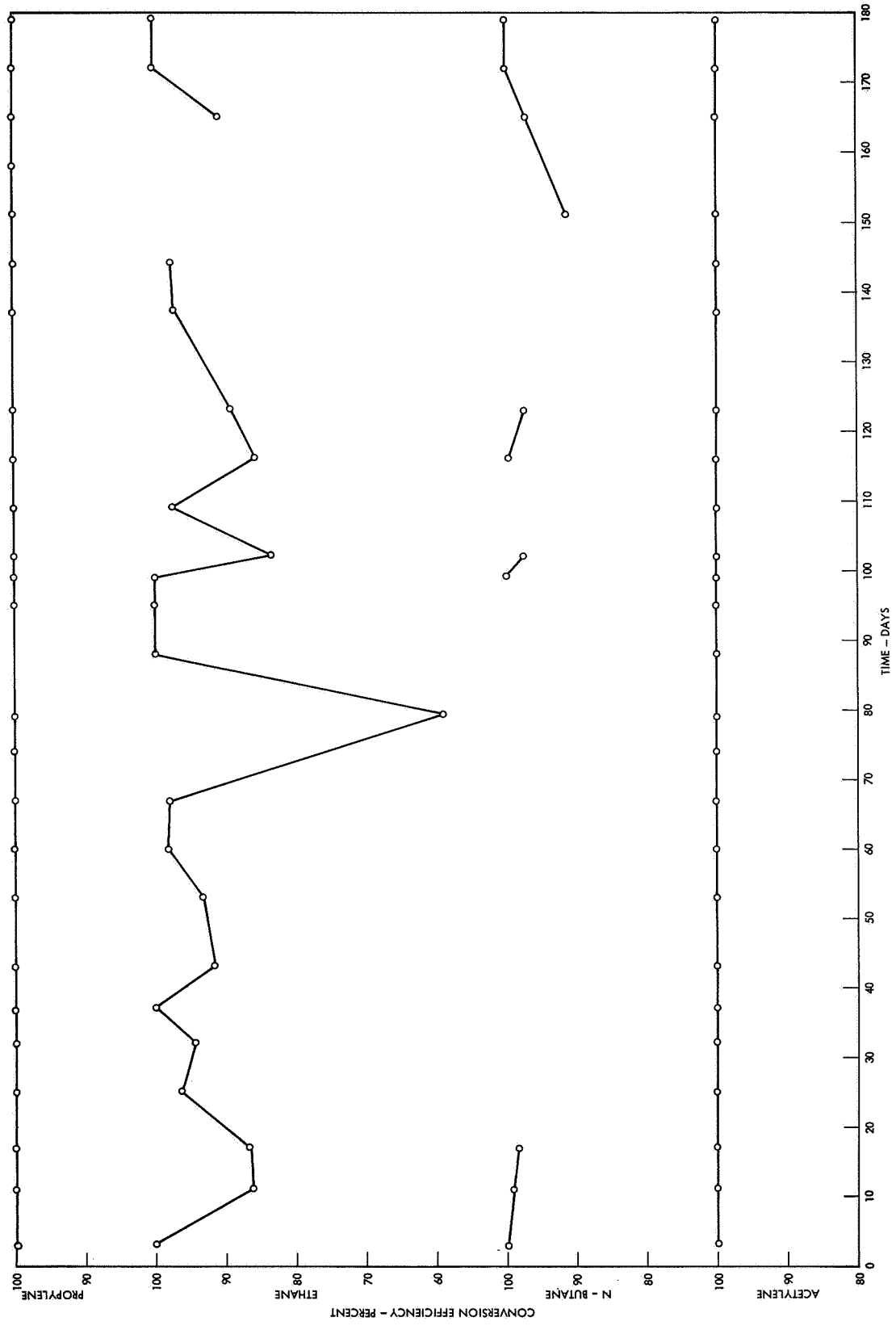


Fig. 14 Acetylene, n-Butane, Ethylene, Ethane, Propylene - Performance

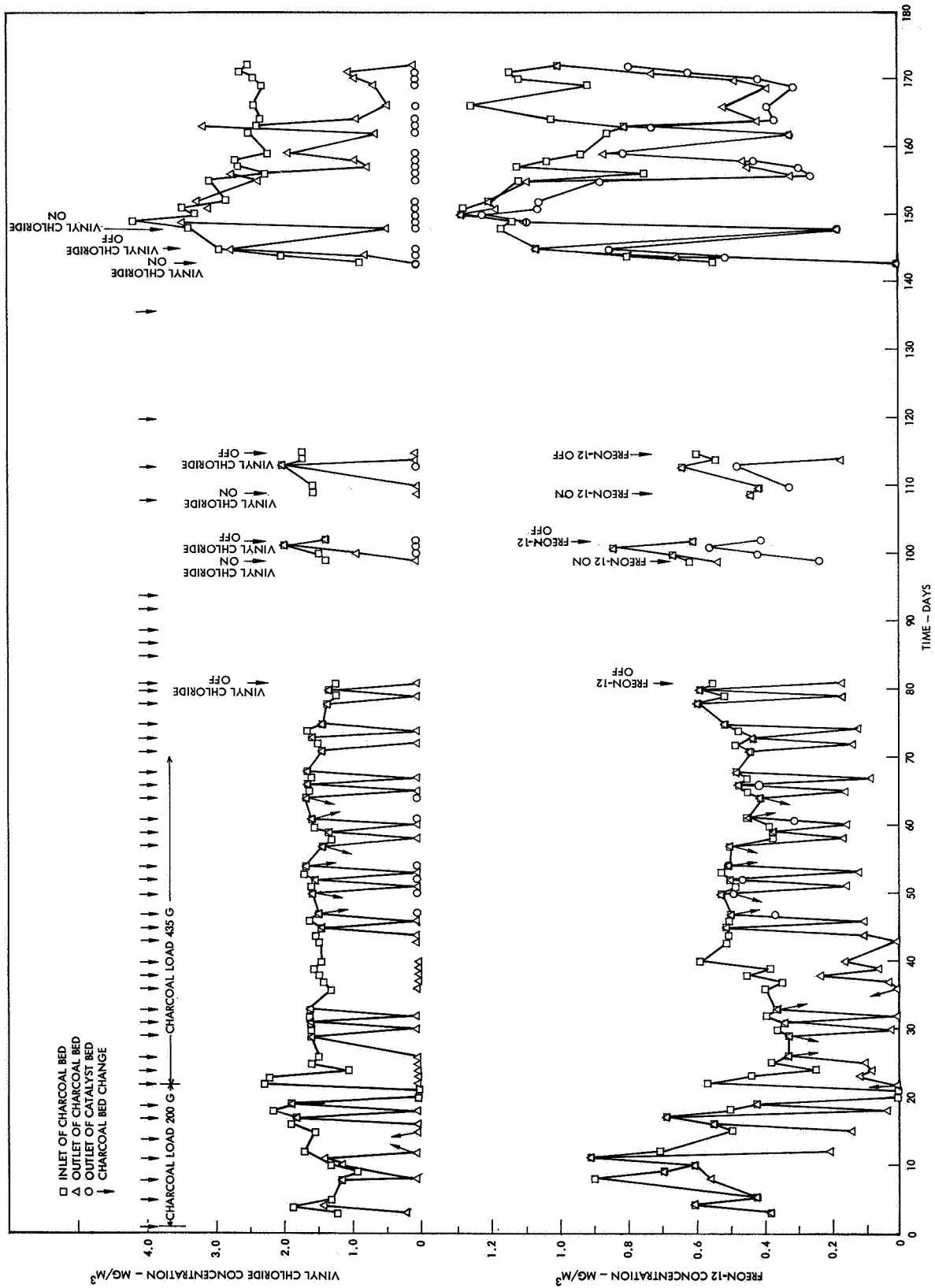


Fig. 15 Vinyl Chloride and Freon-12 Performance

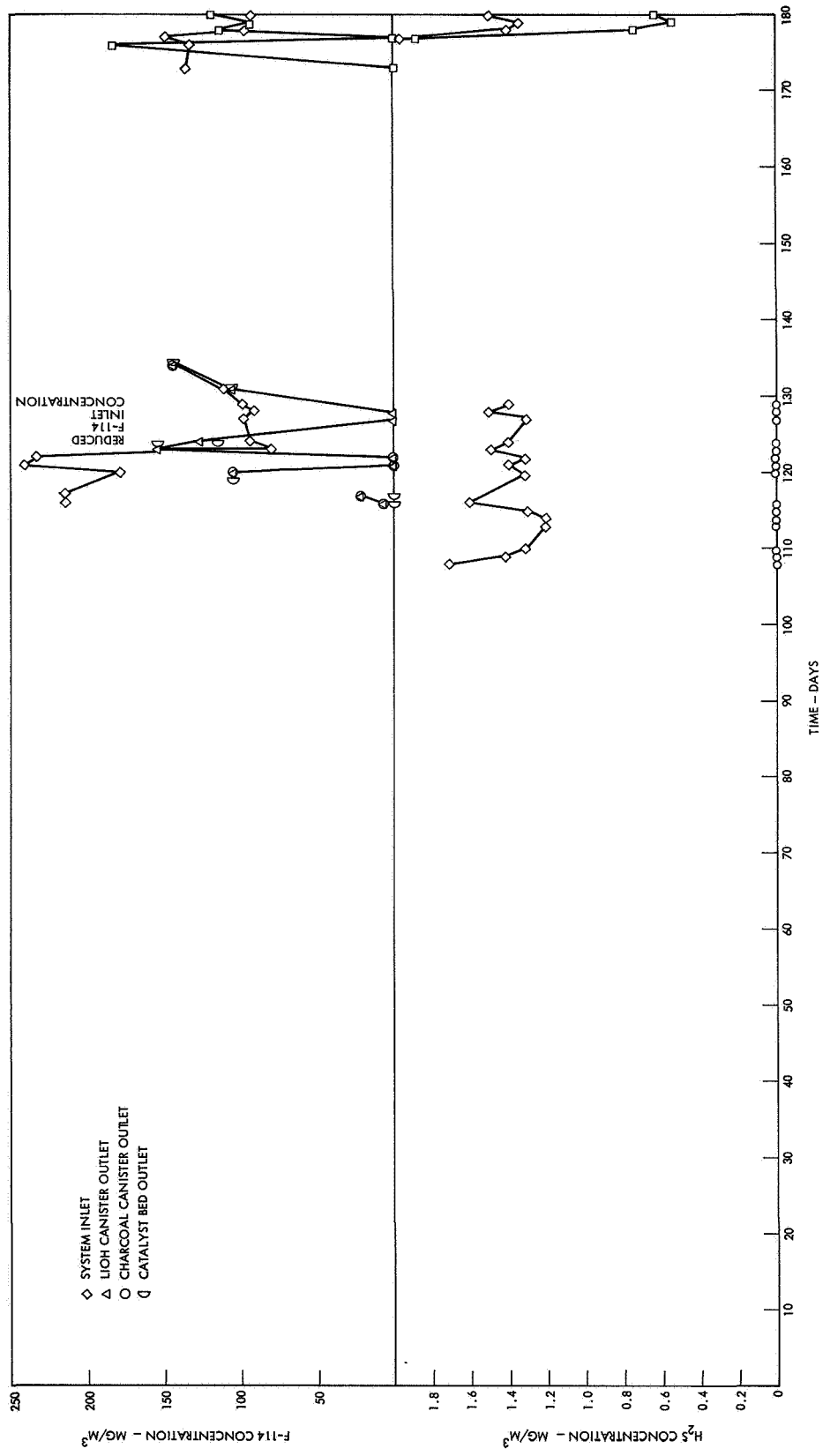


Fig. 16 Freon-114 and Hydrogen Sulphide Performance

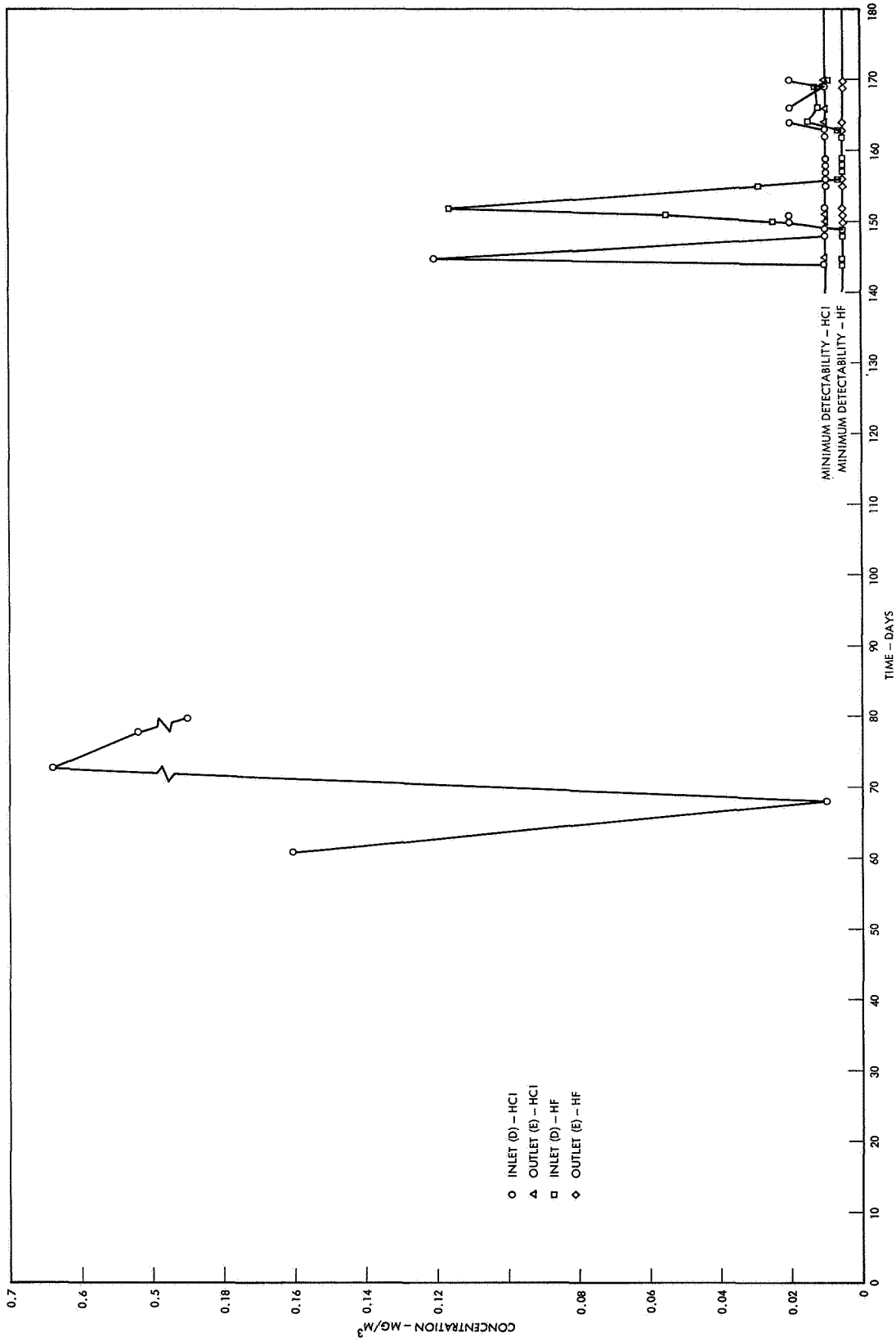


Fig. 17 Hydrogen Chloride and Hydrogen Fluoride Performance

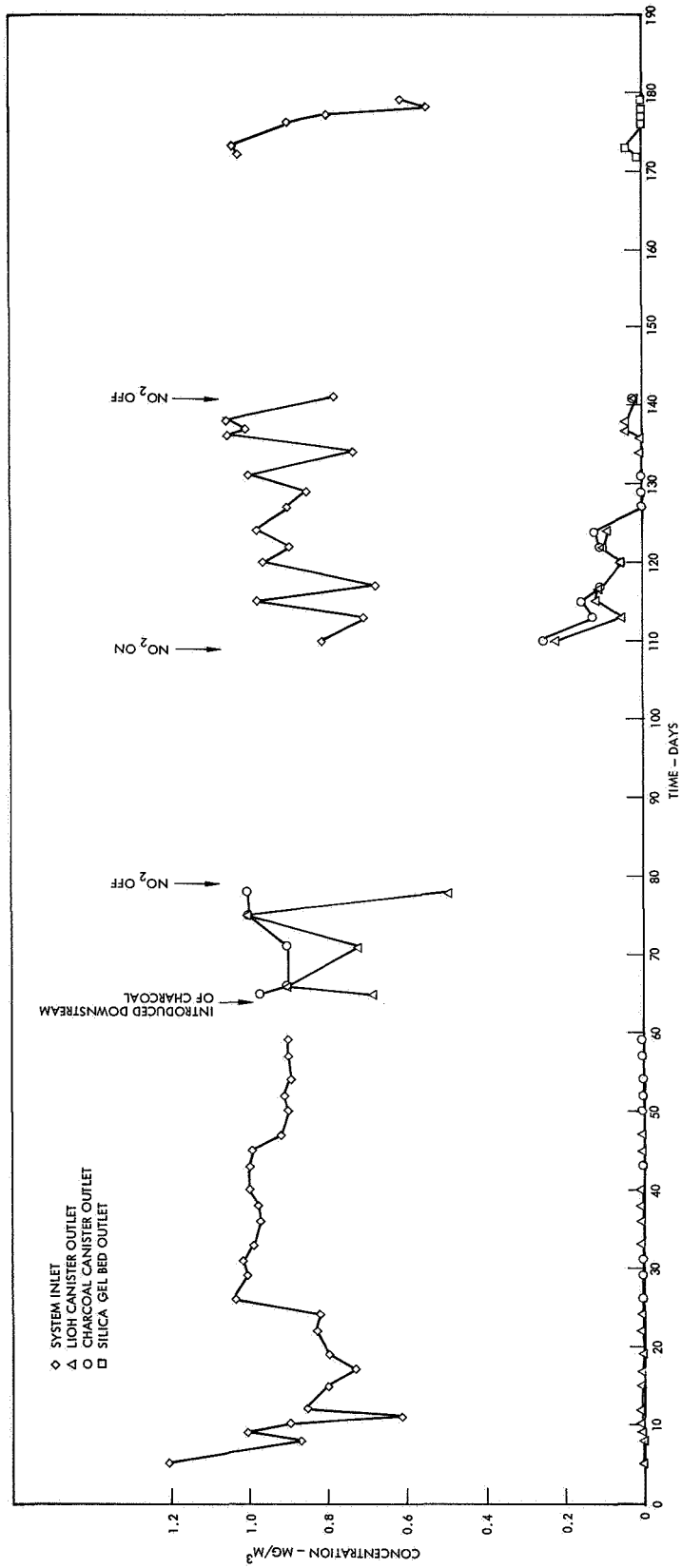


Fig. 18 Nitrogen Dioxide Performance

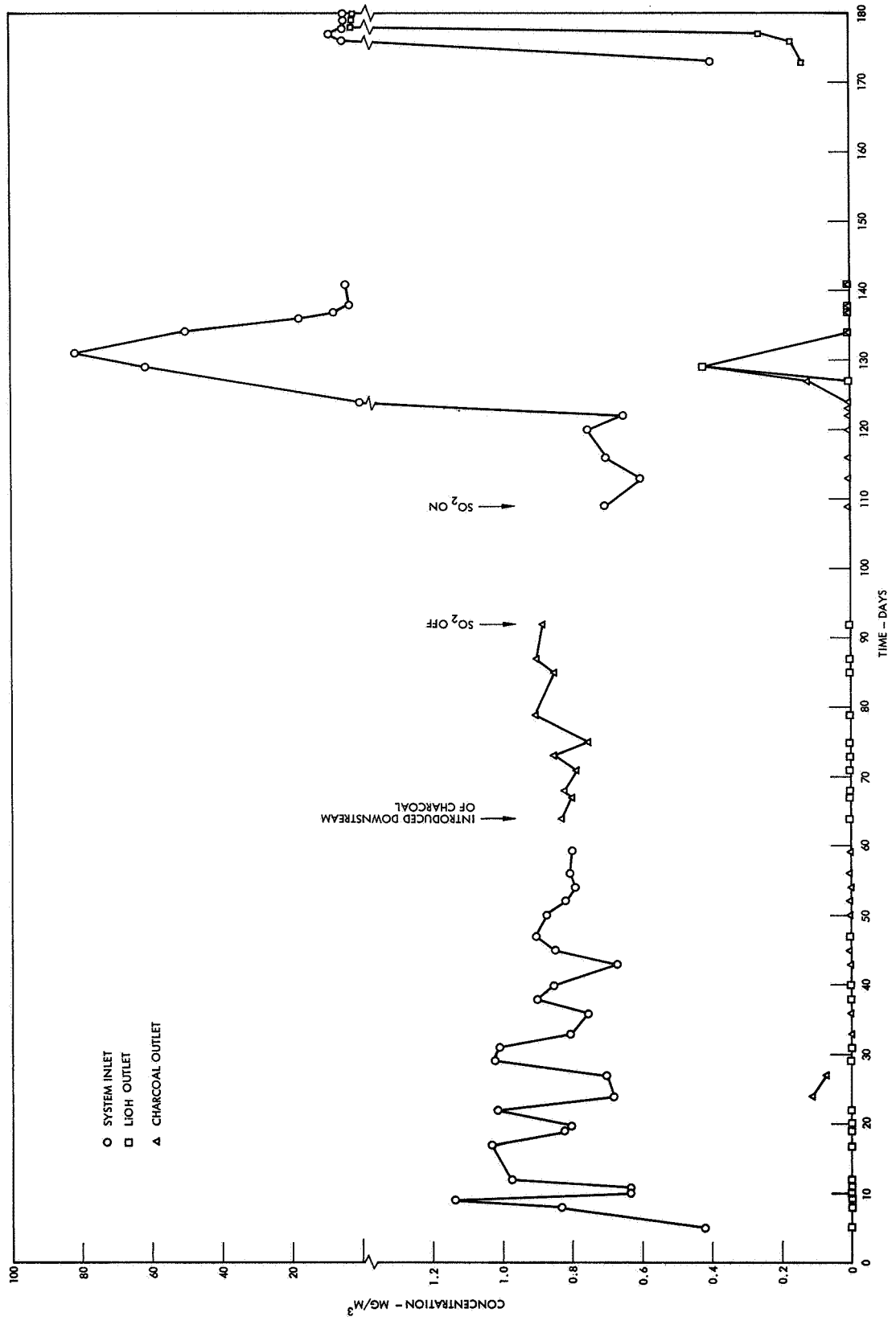


Fig. 19 Sulfur Dioxide Performance



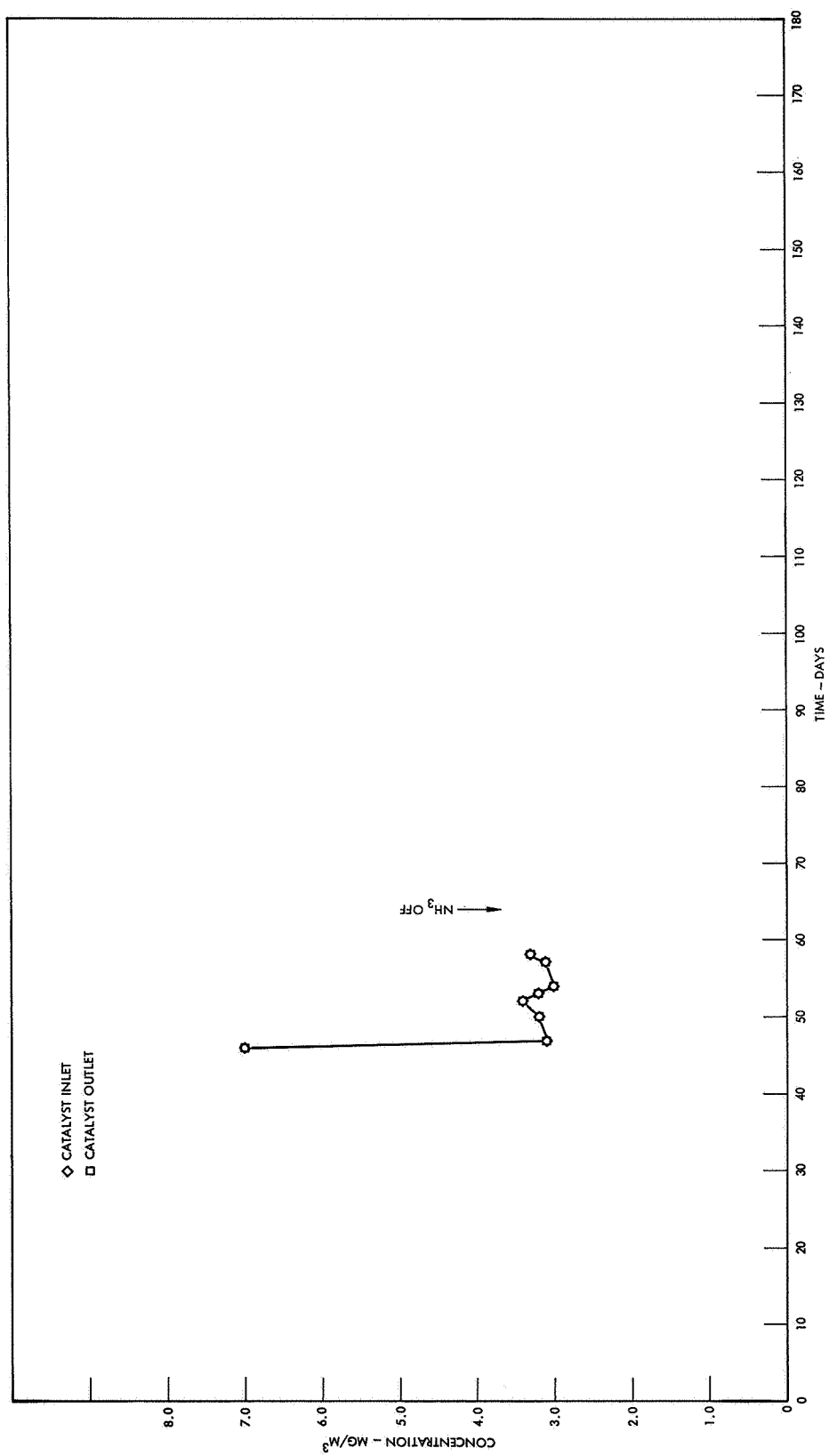


Fig. 20 Ammonia Performance

## Discussion

During the test, investigations were made relative to (1) catalyst poisoning and the need for a pre-sorbent bed, (2) the formation of undesirable products of oxidation and (3) the control of undesirable products of oxidation by a post-sorbent bed. The following sections discuss the results of the tests pertinent to these investigations.

Catalyst poisoning.-In the investigations relative to catalyst poisoning, the performance of the charcoal main sorbent system, a lithium hydroxide basic pre-sorbent and the silica gel desiccant bed portion of a carbon dioxide removal system were considered.

Main Sorbent System: The complete test apparatus was first operated for three days with the hydrocarbon contaminants used in the previous 180-day long term test to check out the apparatus and to verify catalyst performance. The performance of the catalyst was found to be the same as experienced in the previous test. On the fourth day of testing, the first candidate catalyst poisons to be controlled by the charcoal bed, Freon-12 and vinyl chloride, were introduced upstream of the charcoal main sorbent bed. These contaminants were selected on the basis of candidates for removal by the charcoal bed, being among those less well adsorbed. The charcoal bed was sized, based on potential plot data taken in a relatively dry gas stream, for breakthrough to occur after 7 days. The beds were to be cycled on a 7-day period with desorbed charcoal being used in each new canister. Both the Freon-12 and vinyl chloride broke through the bed prior to cycling indicating that the performance of the charcoal bed was less than anticipated. This test was repeated several times with the beds being cycled twice a week. The Freon-12 and vinyl chloride were still breaking through before the beds were cycled. The poisoning effect on the catalyst brought about by Freon-12 and vinyl chloride being allowed to enter the catalyst bed was evident from the methane removal characteristics. Methane removal efficiency would start out at a fairly high value with a fresh bed and would then drop after the Freon-12 and vinyl chloride broke through. The methane removal efficiency would, however, recover soon after a fresh charcoal canister was placed on line. The test was continued in this mode until the sixteenth day.

Since the 6 x 8 mesh charcoal showed poor removal efficiency for the Freon-12, and vinyl chloride, a finer 8 x 10 mesh charcoal was placed into the system on the sixteenth day and again on the eighteenth day after pre-treatment by degassing and heating. No improvements in Freon-12 and vinyl chloride removal were noted. Freon-12 and vinyl chloride flow into the main flow stream was turned off for the weekend (days 20 and 21) as a means of aiding in increasing the catalyst bed performance.

On the twenty-second day, the charcoal bed volume was increased to hold 435 grams of 8 x 10 charcoal that was prepared by heating above 200°C and degassing under one micron pressure for ten hours. This was done as an attempt to improve performance of the main sorbent system since it was still allowing Freon-12 and vinyl chloride to enter the catalytic oxidizer. The Freon-12 and vinyl chloride contaminants were reintroduced into the system. Analysis at the inlet and outlet of the charcoal bed showed 100% removal of both Freon-12 and vinyl chloride immediately after the new canister was put on line. After approximately 3 days, vinyl chloride had not broken through and Freon-12 had partially broken through.

A routine was then established for periodically changing the charcoal beds on Monday mornings and on Wednesday and Friday afternoons which resulted in approximately a 2.5 day cycle. As anticipated catalyst performance was always higher after changing the charcoal beds. The initial removal of Freon-12 and vinyl chloride by the charcoal resulted in higher catalyst performance but as the charcoal removal efficiency for the contaminants decreased, so did the catalyst bed performance. This change in cycle time did effect one of the other contaminants being introduced, n-butane, in that it was now being removed by the charcoal beds and was not reaching the catalyst. The capacity of the charcoal for the contaminants in question was at least 1/3 less than had been anticipated owing to the use of potential plot data taken in a dry gas stream.

To verify that moisture effects was the problem, the test was modified to reduce the inlet dew point to the charcoal. On the thirty-sixth day, the humidifier was by-passed creating an inlet dew point in the range of 32-38°F. The effect of the lowered dew point was an increase in the charcoal performance for Freon-12 and vinyl chloride confirming that water vapor had an adverse affect on charcoal capacity.

In a final attempt to increase the performance of the main sorbent system, different charcoals were investigated. On the 54th day of testing, an 8 x 12 mesh Barneby Cheney charcoal type G-1 was employed. No improvement in the Freon-12 and vinyl chloride removal characteristics was noticed as compared with the type BD charcoal previously used. On the 59th day, an 8 x 12 mesh Barneby Cheney charcoal type KE was used. Again no improvement in the Freon-12 and vinyl chloride removal characteristics was observed. Type BD charcoal was then used for the remainder of the test.

The final investigation conducted with Freon-12 and vinyl chloride was to reconfirm that the poisoning caused by these contaminants was reversible in nature. This was done starting on the 99th day when the charcoal bed cycle time was increased to 4 days. This test was repeated again on the 109th day. In both cases, the Freon-12 and vinyl chloride broke through the charcoal bed and caused the catalyst performance to degrade considerably; removal efficiency dropped from better than 80% to less than 10%. In both cases, the removal efficiency recovered to its initial value immediately following elimination of the Freon-12 and vinyl chloride from the flow stream.

Lithium hydroxide pre-sorbent bed: The first candidate catalyst poisons to be controlled by the lithium hydroxide pre-sorbent bed, introduced into the system were sulfur dioxide and nitrogen dioxide.

Sulfur dioxide and nitrogen dioxide were introduced into the flow stream on the fifth day with the aid of the Technicon proportioning pump. Samples for contaminant concentrations were taken at the inlet to the charcoal bed, exit to the charcoal and inlet to the lithium hydroxide bed, and the exit to the lithium hydroxide bed.

The  $\text{NO}_2$  inlet concentration varied from 0.61 to 1.2  $\text{mg}/\text{m}^3$ . No detectable levels were identified at the exit to the charcoal bed showing that the nitrogen dioxide was being removed by the charcoal bed.

Sulfur dioxide inlet concentration to the charcoal beds varied from 0.42 to 1.14  $\text{mg}/\text{m}^3$ . Samples were taken at the same locations as those taken for nitrogen dioxide. Colorimetric analysis for sulfur dioxide showed that it was also removed by the charcoal bed except on the 25th and 29th days of the test.

The removal of  $\text{NO}_2$  and  $\text{SO}_2$  by the charcoal was attributed to these contaminants combining with water to form acids and then being chemisorbed. A post-removal examination of the BD charcoal beds verified the presence of these acids. It was considered that  $\text{SO}_2$  and  $\text{NO}_2$  removal was related to the inlet dew point and that for lower dew points,  $\text{NO}_2$  and  $\text{SO}_2$  may not be controlled in the main sorbent charcoal bed. The charcoal adsorption capacity for these contaminants is quite low. At  $20^\circ\text{C}$  and 760 mm under dry gas conditions, the approximate retentivity for these acid contaminants is 10% as compared for example to a value of 20% for methyl mercaptan (ref. 75). Thus, for this reason, and because the lithium hydroxide pre-sorbent bed was placed in the system to control the contaminants such as  $\text{NO}_2$  and  $\text{SO}_2$ , it was considered necessary to introduce the  $\text{NO}_2$  and  $\text{SO}_2$  downstream of the main sorbent charcoal to verify the lithium hydroxide removal capacity.

Introduction of the acid gases downstream of the charcoal bed was initiated on the 64th day of the test. Methane conversion efficiency dropped from 55% on the 65th day to approximately 12% on the 75th day. During this period,  $\text{SO}_2$  was removed completely from the flow stream by the  $\text{LiOH}$  at inlet concentrations approximating 0.80  $\text{mg}/\text{m}^3$  while  $\text{NO}_2$  was partially removed. Removal efficiency for  $\text{NO}_2$  at inlet concentrations approximating 1.0  $\text{mg}/\text{m}^3$  varied from no removal to 30%. The remaining  $\text{NO}_2$  that flowed into the catalytic oxidizer was completely reacted to other products. It was not determined what products were being formed.

On the 79th day of the test,  $\text{NO}_2$  was turned off since it was suspected to be the cause of the catalyst poisoning due to it being only partially removed by the lithium hydroxide and reacting in the catalytic oxidizer. The catalyst did not recover and Freon-12 and vinyl chloride introduction was stopped on the 81st day of the test. The last potential poison sulphur dioxide was turned off on the 92nd day.

Steps to reactivate the catalyst were then carried out following procedures that might be employed in a spacecraft life support system. This included reducing the gas flow rate, allowing the catalyst bed temperature to increase and supplying reduced humidity air into the system.

On the 87th day, the catalyst bed temperature was raised to 800°F where methane conversion efficiency jumped to 71%. During this period, the humidifier system was by-passed and a silica gel bed was placed in the line ahead of the charcoal bed to minimize the amount of moisture entering the flow system.

Due to the increase in methane conversion efficiency to 83% after 5 days, the catalyst bed temperature was lowered to 687°F, the silica gel bed was removed from the line, and the humidifier system was placed in the system. Methane analysis showed that the conversion efficiency was less than 10% and that the catalyst had not been reactivated. Fresh catalyst was re-packed in the catalyst bed on the 94th day and the test was continued on the same day. Down time was estimated at three hours. This included the time for the furnace to reach 687°F operating temperature from room temperature. Methane conversion efficiency was at 91% on the 95th day and 96th day showing that the new catalyst was performing as well as the previous catalyst before poisoning.

It was felt at this time that the cause of the catalyst poisoning was due to poisoning by the  $\text{NO}_2$ ; however, during the test some  $\text{SO}_2$  had reached the catalyst bed due to a by-pass flow around the lithium hydroxide bed. This occurred while the colorometric analyses were being carried out.

The poisoned catalyst was analyzed by infrared spectrophotometric techniques by extracting the products formed on the catalyst pellets and by analyzing the concentrated extracts which were incorporated into a KBr pellet. The unused catalyst pellets were analyzed by similar methods for use as standards for comparison. The poisoned catalyst showed that the major portion of the extracted material was a sulfate type compound while the unused or standard catalyst showed the extract to be an oxide type. These results indicated strongly that  $\text{SO}_2$  deactivated the catalyst.

Sulfur compounds, particular in the lowest valence state, are potent catalyst poisons due to the electron octet vacancies (ref. 76, 77). Unshared electron pairs such as  $\text{SO}_2$  ( $\text{O}::\overset{\cdot\cdot}{\text{S}}::\text{O}$ ) result in chemisorptive bonding between the sulfur atom and the active catalyst sites. Unlike physical bonding, considerable energy is required to disrupt the chemisorptive bond. In this particular case the sulfur compound reacted and/or accumulated on the active catalyst sites causing the catalyst to be less active.

At this point in the testing, it had been demonstrated that  $\text{LiOH}$  would completely remove  $\text{SO}_2$  and partially control  $\text{NO}_2$ ; however, these contaminants were also controlled in the charcoal in the present of moisture.

On the 108th day further attempts were initiated to introduce contami-

nants that would pass through the charcoal bed but would be removed by the lithium hydroxide. Hydrogen sulfide was added to the contaminant flow along with the hydrocarbon contaminants, carbon monoxide, Freon-12, vinyl chloride, sulfur dioxide and nitrogen dioxide. The hydrogen sulfide was introduced upstream of the charcoal bed. The charcoal removed the H<sub>2</sub>S beyond the breakthrough for both Freon-12 and vinyl chloride.

The same experiment was repeated except Freon-114 was introduced in place of the Freon-12 and vinyl chloride. Freon-114 was substituted on the basis that the charcoal has a higher capacity for retaining Freon-114 over H<sub>2</sub>S and thus the H<sub>2</sub>S breakthrough would probably occur before the breakthrough of the Freon-114. Results of the experiment showed, however, that Freon-114 penetrated the charcoal prior to H<sub>2</sub>S.

At this point in the testing, the requirement for pre-sorbent lithium hydroxide bed had not been experimentally defined, because contaminants for which a lithium hydroxide pre-sorbent is required were being removed by the charcoal in a humidified atmosphere.

A review of the past charcoal bed performance showed that at least three distinct removal mechanisms existed for the contaminants in question. These mechanisms included adsorption which was accounted for in the sorbent bed design, oxidation, and combining with the water retained by the charcoal. It was felt that oxidation accounted for the removal of H<sub>2</sub>S and combining with water accounts for the control of SO<sub>2</sub> and NO<sub>2</sub>. The control of contaminants by combining with water in the charcoal is dependent upon the presence of water in the charcoal and therefore dependent on the location of the charcoal in the spacecraft environmental control system. If the charcoal was located in a low dew point stream, such as integration with a molecular sieve-type CO<sub>2</sub> removal system, the water would not be present in the charcoal bed. These conditions were of interest and would relate to the possible need of a basic pre-sorbent in a dry gas version of IHCOS. For this reason, the test with Freon-114, H<sub>2</sub>S, SO<sub>2</sub> and other contaminants was repeated with a low dew point gas stream.

On the 124th day, a silica gel canister was placed ahead of the incoming atmosphere and the humidifier system was by-passed. Immediately following this, the sulfur dioxide broke through the charcoal bed. The sulfur dioxide also penetrated the lithium hydroxide bed during this test. Hydrogen sulfide, however, did not break through the bed.

As anticipated, these results confirmed that these acid gases (SO<sub>2</sub> and NO<sub>2</sub>) are poorly retained by charcoal in a dry gas inlet stream condition but will combine with water adsorbed on the charcoal under humidified atmospheric conditions. Hydrogen sulfide, however, is controlled by the charcoal under wet or dry gas conditions confirming that its removal mechanism is not related to combining with water but instead probably due to oxidation. The performance of the lithium hydroxide in controlling the SO<sub>2</sub> under dry gas conditions was also poor. Sulfur dioxide had been completely removed by LiOH in previous tests with a humidified gas stream. Examination of these results and

a review of the information available on the performance of lithium hydroxide in other reactions indicated that in general the presence of water vapor is required for maximum reactivity. Thus, in the dry gas condition, the acid gases do pass through the charcoal indicating the need for a pre-sorbent, however, the selected basic pre-sorbent is not of value under these conditions. The test results showed that in a dry gas design, the acid gases should be controlled at a point in a system where moisture is present. If the dry gas unit were integrated with a molecular sieve type carbon dioxide removal system, the acid gases might be controlled to some degree in the silica gel bed.

**Silica gel bed:** The charcoal canisters were filled with silica gel (Davidson Type 2, Grade 40) and Freon-12, vinyl chloride, and NO<sub>2</sub> were introduced into the system along with the other hydrocarbon background contaminants. The objective of this test was to determine if the NO<sub>2</sub> would be retained by the silica gel up to the point of water break through. The dew point from the silica gel bed was monitored with a Cambridge Hygrometer System by sampling the flow in parallel with the pre-sorbent LiOH bed.

Freon-12 penetrated the bed within one hour and vinyl chloride within three hours time. Nitrogen dioxide did not break through the bed. After saturation of the bed, NO<sub>2</sub> outlet concentration showed 98.5% removal. The following day the humidifier system was by-passed and another fresh silica gel bed was placed in line. A longer breakthrough time from this setup was anticipated. Dew point outlet dropped to -68°F. Sulfur dioxide, nitrogen dioxide, Freon-114, and hydrogen sulfide were introduced into the flow system.

Freon-114 penetrated the silica gel bed after twelve hours. The outlet concentration reached a level where it was higher than the inlet concentration, due to the outgassing of the Freon-114 from the silica gel bed. Nitrogen dioxide was 96.0% removed from the flow stream showing slightly less removal capability than previously. However, sulfur dioxide showed only 65% removal by the silica gel bed at inlet concentration of 0.40 mg/m<sup>3</sup>. The water vapor had not broken through at the time. Further testing with higher SO<sub>2</sub> inlet concentrations showed that initial exposure of SO<sub>2</sub> resulted in nearly complete removal by the silica gel bed but after 20 hours of continuous exposure, the removal efficiency dropped with increasing bed saturation to approximately 17%. At this time the silica gel bed had reached water breakthrough. A repeat experiment with a higher inlet humidity showed that at silica gel water breakthrough after 10 hours of continuous exposure, SO<sub>2</sub> was removed approximately 60%. Thus, it appears that NO<sub>2</sub> is well removed by silica gel but that only partial removal of SO<sub>2</sub> can be anticipated.

Undesirable products of oxidation.-The second phase of the test was to establish if undesirable products of oxidation were being formed. After the test had proceeded for 37 days, the investigation relative to the formation of undesirable products of oxidation were initiated. To this end, methyl chloride was introduced into the flow stream at 21 mg/m<sup>3</sup> inlet concentration as a potential producer of undesirable products of oxidation. The methyl chloride passed readily through the charcoal and lithium hydroxide presorbent beds as

anticipated, however, it poisoned the catalyst bed causing methane conversion to drop to approximately 4%. No undesirable products were detected during this period. Methyl chloride flow into the system was terminated the next day (38th day).

The catalyst slowly recovered and by the 44th day of the test, methane conversion efficiency had increased to approximately 54%.

On the 44th day, ammonia was added to the system as a potential producer of undesirable oxidation products. The results from the inlet and outlet analysis of the catalytic oxidizer showed that the ammonia was not oxidized but remained unchanged. Even with a high inlet concentration of 11.4 mg/m<sup>3</sup> no potential undesirable oxidation products were noted. Ammonia was introduced from the 44th to the 64th day. During this period, the inlet concentration was approximately 3.2 mg/m<sup>3</sup>. Since no undesirable products were found in the outlet flow of the catalytic oxidizer no further ammonia was introduced into the system.

When the charcoal bed became saturated with Freon-12 and vinyl chloride, the analysis of the outlet flow from the catalytic oxidizer showed that Freon-12 and vinyl chloride were oxidized by the catalyst. The outlet analysis on the 47th day showed that vinyl chloride was 100% oxidized and Freon-12 was oxidized approximately 27%.

Since the Freon-12 and vinyl chloride were penetrating the charcoal beds, it was decided to allow these contaminants to pass into the catalytic oxidizer system and determine what undesirable oxidation products they were producing and possibly use them as a source of undesirable products. The outlet from the catalytic oxidizer system was analyzed for Freon-12 and vinyl chloride during the period from the 47th to the 80th day. The results showed that vinyl chloride was oxidized 100% and Freon-12 varied in conversion efficiency from no conversion to 41%. Intermittent analysis for undesirable products from oxidation was accomplished during this period by concentrating the outlet flow from the catalytic oxidizer with a refrigerated dry ice bath and by analyzing the concentrated sample through gas chromatographic and mass spectrometry analysis techniques.

The Freon-12 and vinyl chloride decomposition products identified were HCl and HF. HCl outlet concentrations as high as 0.68 mg/m<sup>3</sup> were determined during this period. In addition, infrared spectrophotometric scans of catalyst outflow samples (10 meter path gas cell) showed no undesirable products besides the inorganic acids mentioned.

Post-sorbent bed.-The final portion of the 180 day test was devoted to evaluation of the effectiveness of the post-sorbent bed. On the 143rd day of the test, Freon-12 and vinyl chloride were added to the flow stream to create undesirable products of oxidation in order to determine the performance of the lithium hydroxide post-sorbent bed. The Freon-12 and vinyl chloride were oxidized in the catalyst bed and generated HCl and HF as oxidation products.



During this experimental period, the Freon-12 and vinyl chloride inlet concentrations were increased to allow for more product formation. On the weekends and after the analyses were completed, the inlet concentrations were lowered to allow the catalyst bed to recover from the poisoning effects of these contaminants. The charcoal pre-sorbent bed was not changed since it required a few days before Freon-12 or vinyl chloride would break through.

Analysis of the catalyst bed outlet showed 100% reactivity of vinyl chloride. Freon-12 conversion efficiencies varied from zero to thirty-three percent. These results showed that products were being formed in the catalyst. The HCl concentrations at the post-sorbent bed inlet were at the lower limit of detectability ( $0.01 \text{ mg/m}^3$ ). Only during the 145th day did the HCl inlet concentration rise to  $0.12 \text{ mg/m}^3$ . At no time was HCl detected at the outlet of the post-sorbent bed.

The low HCl inlet concentration into the post-sorbent bed was due to the reactivity of HCl with the cooling coil tube located at the outlet of the catalyst burner. An analysis and inspection of the heat exchanger tube after the completion of the test showed a high residual chloride content. Also, the lower HCl production may be accounted for by the incomplete oxidation of vinyl chloride to other products since the percent conversion was determined by the analysis for vinyl chloride inlet and outlet concentrations and not the possible other intermediates that could be formed from incomplete oxidation.

Similar to the results obtained for HCl, the HF produced by the catalytic oxidizer was removed by the post-sorbent lithium hydroxide bed. The lower limit of detectability for HF was  $0.005 \text{ mg/m}^3$ . HF inlet concentration to the post-sorbent bed varied from 0.116 to  $0.005 \text{ mg/m}^3$ , but mostly at the lower limit of detectability. Also, it was assumed that the HF produced by the catalytic degradation of Freon-12 was partly removed by reactivity with the catalyst impregnated  $\text{Al}_2\text{O}_3$  base material and/or the stainless steel cooling coil placed in the outlet of the catalytic oxidizer as indicated by the chemical analysis of the heat exchanger tube after the completion of the test.

Thus, the post-sorbent bed was effective in controlling the products of oxidation from the vinyl chloride and Freon-12.

## Conclusions

Based on the results of the long term testing, the following conclusions were reached relative to the pre- and post-sorbent beds.

Pre-sorbent bed.-The initial concept was for the basic pre-sorbent bed to remove those contaminants not effectively controlled by the charcoal main sorbent. A review of the main sorbent bed performance based on the potential plot theory for prediction of adsorption capability indicated that a number of acidic contaminants were poorly adsorbed on charcoal. This led to the selection of a basic pre-sorbent bed. In conducting the long term test, it became apparent that the charcoal was controlling to a great extent the contaminants anticipated to require a basic sorbent. This was attributed in part to the moisture in the charcoal. These results indicated that a basic pre-sorbent would not be required. In a dry gas situation the acid gasses pass through the charcoal, indicating the need for a pre-sorbent, however, the selected basic pre-sorbent is not effective in this situation.

The catalyst poisoning experienced during the long term test included poisoning by Freon-12 and vinyl chloride. It was demonstrated on several occasions that poisoning from these compounds and also from methyl chloride was reversible in nature. When the contaminant source was removed, the catalyst performance would recover to its initial value. The poisoning that resulted from introduction of  $\text{SO}_2$  and  $\text{NO}_2$  into the catalyst bed was irreversible in nature. It was concluded from these experiences that protecting the catalyst from poisoning by the acid gases is far more important than protection from the compounds that produce only temporary poisoning effects. Since the acid gases caused permanent poisoning in the long term test and since they are well adsorbed by  $\text{LiOH}$  in a moist stream, it seems advisable to retain the pre-sorbent canister as insurance against premature breakthrough of these contaminants from the charcoal or main sorbent bed.

It is therefore concluded that the proposed pre-sorbent bed (4.75" I.D. and 5.20" bed length) will be employed in the IHCOS design.

Post-sorbent bed.-The initial design of the basic post-sorbent bed that was modeled in the long term test, is described in the following section of this report. This bed was effective in controlling the undesirable products of oxidation that were produced during the test. It is therefore concluded that the proposed post-sorbent bed (5.5" I.D. and 7.9" bed length) will be employed in the IHCOS design.

## POST-SORBENT BED DESIGN

The post-sorbent bed design effort was conducted in a manner similar to the pre-sorbent bed design and consisted of the following tasks:

- Establishing the potential undesirable products of oxidation
- Post-sorbent selection, bed sizing and optimization

### Contaminant Load

In establishing the potential undesirable products of oxidation, the complete contaminant list was reviewed and an estimate was made of those contaminants that would probably be introduced into the catalytic oxidizer. This was done for all contaminants that were considered a source of undesirable products of oxidation, and is shown in Table 7. The first step in establishing which contaminants would reach the catalytic oxidizer was to review the effectiveness of the upstream removal techniques. This was accomplished by considering the results of the long term test results to date and the known characteristics of the upstream sorbents. The upstream sorbents that were considered included the charcoal main sorbent and a lithium hydroxide pre-sorbent.

Charcoal main sorbent.-Experience to date with the charcoal main sorbent has indicated that at least three distinct removal mechanisms exist for the contaminants in question. These mechanisms include adsorption, oxidation and reactions with the water present in the charcoal.

The relative effectiveness of adsorption for removing a mixture of contaminants is best correlated with the potential plot which is described in detail in the section on the main sorbent bed design. In assessing the effectiveness of adsorption, it was decided that contaminants with an "A" value less than 20 would be well adsorbed and that none of this contaminant would reach the catalytic oxidizer. This is in agreement with the results of the long term test where Freon-114 with an "A" value of 18 was well adsorbed and premature breakthrough from the charcoal did not occur. For "A" values between 20 and 30, it was assumed that the contaminant would be moderately well adsorbed and that a portion of the contaminant load would break through the charcoal bed and could pass into the catalytic oxidizer. It was assumed that 50% of the contaminant passed through the bed in this situation. The performance of Freon-12 with an "A" value of 26.8 during the long term test indicates that a significant portion of this contaminant would pass through the charcoal canister. For "A" values greater than 30, it was assumed that the contaminant was poorly adsorbed and that the majority of the contaminant

TABLE 7

## POST-SORBENT BED CONTAMINANT LOAD

Contaminant Potential Source Undesirable Products	Anticipated Removal Technique							Degree of Removal	Anticipated Delivery Rate gm/day
	Charcoal-Main Sorbent		Lithium Hydroxide-Pre-Sorbent	Complex	Oxidation Reduction	Neut.	Comb. With Water		
	Adsorp.	Oxid.							
Acetonitrile	+							Low	0.25
Ammonia	+	(1)						High	0
Carbon Disulfide	+		+					Medium	0.125
Carbon Tetrachloride	+			+				High	0
Carbonyl Sulfide								Medium	0.125
Chlorine							+	High	0
Chloroacetone	+						+	Medium	0.125
Chlorobenzene	+							High	0
Chlorofluoromethane	+							Low	0.25
Chloroform	+							Medium	1.25
Chloropropane	+							Medium	0
Cyanamide	+						+	Medium	0.125
Dichlorobenzene	+							High	0
2,4 Dichlorobenzoic Acid								High	0
1,1 Dichloroethane	+						+	High	0
Dimethyl Hydrazine	+						+	High	0
Dimethyl Sulfide	+							Medium	0.125

Table 7 Continued

Contaminant Potential Source Undesirable Products	Anticipated Removal Techniques							Degree of Removal	Anticipated Delivery Rate gm/day
	Charcoal-Main Sorbent		Lithium Hydroxide-Pre-Sorbent	Complex	Oxidation Reduction	Neut.	Comb. With Water		
	Adsorp.	Oxid.							
Epichlorohydrin	+			+				Medium	0.10
Ethyl Amine	+			+				Medium	0.10
Ethylene Dichloride	+							Medium	0.125
Ethyl Mercaptan				+			+	High	0
Ethyl Sulfide	+							High	0
Freon-11	+							Medium	1.25
Freon-12	+							Medium	1.25
Freon-21	+							Low	0.25
Freon-22	+							Low	0.25
Freon-23	+							Low	0.25
Freon-113	+							High	0
Freon-114	+							High	0
Freon-114 (unsym)	+							High	0
Freon-125	+							Low	0.25
Hexamethylcyclotrisilox	+							High	0
Hydrogen Chloride						+		High	0
Hydrogen Fluoride						+		High	0
Hydrogen Sulfide				+		+		High	0
Indole	+							High	0

Table 7 Continued  
Anticipated Removal Techniques

Contaminant Potential Source Undesirable Products	Charcoal-Main Sorbent				Lithium Hydroxide-Pre-Sorbent		Degree of Removal	Anticipated Delivery Rate gm/day
	Adsorp.	Oxid.	Comb. With Water	Neut.	Complex	Oxidation Reduction		
Methyl Amine							Low	0.25
Methyl Chloride	+						Low	0.25
Methyl Chloroform	+						High	0
Methylene Chloride	+						Low	2.50
Methyl Mercaptan		+				+	High	0
N-Methyl Morphaline							High	0
Mono-Methyl Hydrazine	+		+				Medium	0.125
Nitric Oxide	+	+	+	+		+	High	0
Nitrogen Dioxide	+		+	+		+	High	0
Nitrogen Tetroxide	+		+			+	High	0
Nitromethane	+						Low	0.25
Nitrous Oxide	+						Low	0.25
Iso-Propyl Chloride	+						Medium	0.125
Propyl Mercaptan	+	+				+	High	0
Skatole	+						High	0
Sulfur Dioxide	+		+	+			High	0
Tetrachloroethylene	+						High	0
Tetrafluoroethylene	+	+				+	High	0
Trichloroethylene	+						High	0
Trimethyl Silanol	+						High	0

Table 7 Continued

Contaminant Potential Source Undesirable Products	Anticipated Removal Techniques							Degree of Removal	Anticipated Delivery Rate gm/day
	Charcoal-Main Sorbent		Lithium Hydroxide-Pre-Sorbent		Oxidation Reduction	Complex	Degree of Removal		
	Adsorp. Oxid.	Comb. With Water	Neut.	Complex					
Vinyl Chloride	+	+					Medium	1.25	
Vinylidene Chloride	+						Medium	0.125	

(1) Assumes reaction with impregnated acid.

would pass through the charcoal. The performance of methyl chloride with an "A" value of 44.5, during the long term test was indicative of this type of performance. For these situations, it was conservatively assumed that 100% of the contaminant load would pass through the charcoal.

A number of contaminants appear to oxidize or decompose on the charcoal and their products of oxidation subsequently removed by the charcoal. This type of removal has been observed in the long term test with hydrogen sulfide and in other tests with methyl mercaptan, ethyl mercaptan, propyl mercaptan and nitric oxide. It is anticipated that this mechanism will occur with tetrafluorethylene and to some extent with vinyl chloride. This explains why vinyl chloride was controlled better than Freon-12 in the long term catalyst test even though vinyl chloride has the higher "A" value.

Examination of the charcoal beds during the long term test indicated that a number of the acidic contaminants reacted with the water retained in the charcoal bed. This was observed in the case of both sulfur dioxide and nitrogen dioxide. It is assumed that this would also occur for the other acidic contaminants.

Lithium hydroxide pre-sorbent.-In reviewing the complete contaminant list, charcoal offers some degree of removal for all contaminants when the mechanisms of adsorption, oxidation and reacting with the water are considered. However, for situations where water vapor is not present or at a low concentration, the acidic contaminants would not be retained in the charcoal bed and would require a downstream basic sorbent for control. In this case, a lithium hydroxide pre-sorbent might be used to prevent these acid gases from entering the catalyst bed since they act as catalyst poisons. Testing has revealed that lithium hydroxide is only effective in a dry gas stream when impregnated with a desiccant compound such as lithium chloride.

Lithium hydroxide is a strong base that can remove many contaminants by either acid-base neutralization reactions, complex formations or oxidation reduction reactions. For those situations where charcoal does not control the acidic gases and a basic pre-sorbent bed is required, the degree of removal of acidic gases is quite high. The complex reaction is a weak reaction which provides only moderate to low removal capability. The oxidation-reduction is also a weak reaction compared with neutralization and also provides only a moderately low removal capability.

Anticipated delivery rate.-In establishing the anticipated delivery rate of potential producers of bad products to the catalytic oxidizer the overall degree of removal was assessed and applied to the contaminant production rate as previously described. The factor of 0%, 50%, or 100% was applied to the production rate of the contaminant in question except in the case where the allowable concentration is low enough that a 3 CFM flow rate and the indicated removal results in a reduced production rate. This occurred for epichlorohydrin and ethyl amine.

#### Post-Sorbent Selection

After establishing which contaminants would reach the catalytic oxidizer



and determining what their most probable oxidation products would be, the choice of post-sorbent material was made. Lithium hydroxide was chosen on the basis that most of the oxidation products were acidic, namely hydrogen chloride, hydrogen fluoride, nitrogen dioxide and sulfur dioxide. This was substantiated experimentally with the oxidation of Freon-12 and the detection of the resultant hydrogen chloride at the catalytic oxidizer outlet.

#### Post-Sorbent Bed Configuration

The sizing of the post-sorbent bed was accomplished by establishing the acidic products produced in the catalytic oxidizer and the quantity of post-sorbent material required to control these products. These results are presented in Table 7 and described in detail below.

Stoichiometric Production of Acidic Products.-The stoichiometric amounts and acidic products; e.g., HF, HCl, SO<sub>2</sub> and NO<sub>2</sub> were determined in terms of moles of acidic product produced per mole of contaminant entering the catalytic oxidizer. These are shown in Table 8 and were derived by considering the individual oxidation reactions of the various contaminants.

Anticipated Amounts of Acidic Products.-The anticipated amounts of acidic products produced in grams per 180 days in Table 8 were determined from the stoichiometric amounts of acidic products and by considering the removal efficiency of the catalytic oxidizer for individual contaminants. For example, during the long term test it was found that Freon-12 was oxidized partially with a maximum conversion of 40%. On the other hand, vinyl chloride was found to be oxidized completely. By using these experimental guidelines and by considering the individual contaminants and their ability to undergo oxidation, it was decided to use 40% conversion efficiency for all the Freons which entered the catalytic oxidizer, 100% for the less stable halogenated hydrocarbons such as vinyl chloride, vinylidene chloride, and methyl chloride and 100% for sulfur or nitrogen containing compounds.

Quantity of Lithium Hydroxide Required.-The amount of lithium hydroxide required for each acidic product was determined in Table 8 in grams per 180 days. This was calculated from the anticipated amounts of acidic products produced and the neutralization reaction which each undergoes with lithium hydroxide. For example, based on the stoichiometric reactions, 1 gram each of hydrogen fluoride, hydrogen chloride, sulfur dioxide, and nitrogen dioxide require 1.20, 0.66, 0.38 and 0.52 grams of lithium hydrogen, respectively.

The total stoichiometric quantity of lithium hydroxide required for 180 days is 715 grams. Using a performance factor of 50%, the required volume of lithium hydroxide for the post-sorbent canister is 187 in<sup>3</sup> with a 3 cfm flow rate. This yields a superficial space velocity of 1600 hrs<sup>-1</sup> which should produce a high removal efficiency per pass for the contaminants in question.

In establishing the configuration of this bed a trade-off was made between the weight penalty to the bed and canister fixed weight and the weight

TABLE 8

## REQUIRES QUANTITY OF POST-SORBENT

	Stoichiometric Moles of Acidic Products/Day			Anticipated Amount of Acidic Products Grams/180 Days			Amount of Lithium Hydroxide Required Grams/180 Days		
	HF	HCl	NO <sub>2</sub>	HF	HCl	NO <sub>2</sub>	HF	HCl	NO <sub>2</sub>
Acetonitrile			1			50			26.1
Ammonia									
Carbon Disulfide		2				37.8		14.2	
Carbon Tetrachloride									
Carbonyl Sulfide		1				24.0		9.0	
Chlorine									
Chloroacetone		1			8.8			5.8	
Chlorobenzene									
Chlorofluoromethane	1	1		5.4	9.9		6.5	6.5	
Chloroform		3			82.4			54.0	
Chloropropane									
Cyanamide									
Dichlorobenzene			1			23			12.0
2,4 Dichlorobenzoic Acid									
1,1 Dichloroethane									
Dimethyl Hydrazine									
Dimethyl Sulfide			1			23.2		9.7	
Epichlorohydrin		1			7.0			4.6	
Ethyl Amine			1			18			9.4

TABLE 8 Continued

	Stoichiometric Moles of Acidic Products/Day			Anticipated Amount of Acidic Products Grams/180 Days			Amount of Lithium Hydroxide Required Grams/180 Days		
	HF	HCl	NO <sub>2</sub>	HF	HCl	NO <sub>2</sub>	HF	HCl	NO <sub>2</sub>
Ethylene Dichloride		2			16.9			11.1	
Ethyl Mercaptan									
Ethyl Sulfide		3		13.1	72.0		15.7	47.3	
Freon-11	1			29.7	54.2		35.6	35.6	
Freon-12	2			3.4	12.8		4.1	8.4	
Freon-21	1			8.37	7.56		10.0	5.0	
Freon-22	2			15.4			18.5		
Freon-23	3								
Freon-113									
Freon-114									
Freon-114 (unsym)									
Freon-125	5			15.0			18.0		
Hexamethylcyclotrisilox									
Hydrogen Chloride									
Hydrogen Fluoride									
Hydrogen Sulfide									
Indole									
Methyl Amine			1						34.7
Methyl Chloride	1				32.6			21.4	
Methyl Chloroform									

TABLE 8 Continued

	Stoichiometric Moles of Acidic Products/Day				Anticipated Amount of Acidic Products Grams/180 Days				Amount of Lithium Hydroxide Required Grams/180 Days			
	HF	HCl	SO <sub>2</sub>	NO <sub>2</sub>	HF	HCl	SO <sub>2</sub>	NO <sub>2</sub>	HF	HCl	SO <sub>2</sub>	NO <sub>2</sub>
Methylene Chloride		1				189.0				124.0		
Methyl Mercaptan												
N-Methyl Morphaline				1				22.5				11.8
mono-Methyl Hydrazine												
Nitric Oxide												
Nitrogen Dioxide												
Nitrogen Tetroxide				1				34.0				17.8
Nitromethane				1				68.4				35.7
Nitrous Oxide												
iso-Propyl Chloride		1				10.2			6.7			
Propyl Mercaptan												
Skatole												
Sulfur Dioxide												
Tetrachloroethylene												
Tetrafluoroethylene												
Trichloroethylene												
Trimethyl Silanol												
Vinyl Chloride		1				131				86		
Vinylidene Chloride		2				17				11		

penalty associated with the fan head rise required for flow. This optimization was performed in the same manner as the pre-sorbent optimization. The resulting plot of total equivalent weight as a function of canister diameter is shown in Figure 21. This reveals that the optimum canister diameter is 5.5 inches and the optimum bed length is 7.9 inches. The overall canister length including conical ends will be 15.5 inches and the canister will weigh approximately 1.8 pounds. The total weight with lithium hydroxide will be approximately 4.9 pounds. The pressure drop at 3 cfm and 10 psia will be 0.35 inches of water.

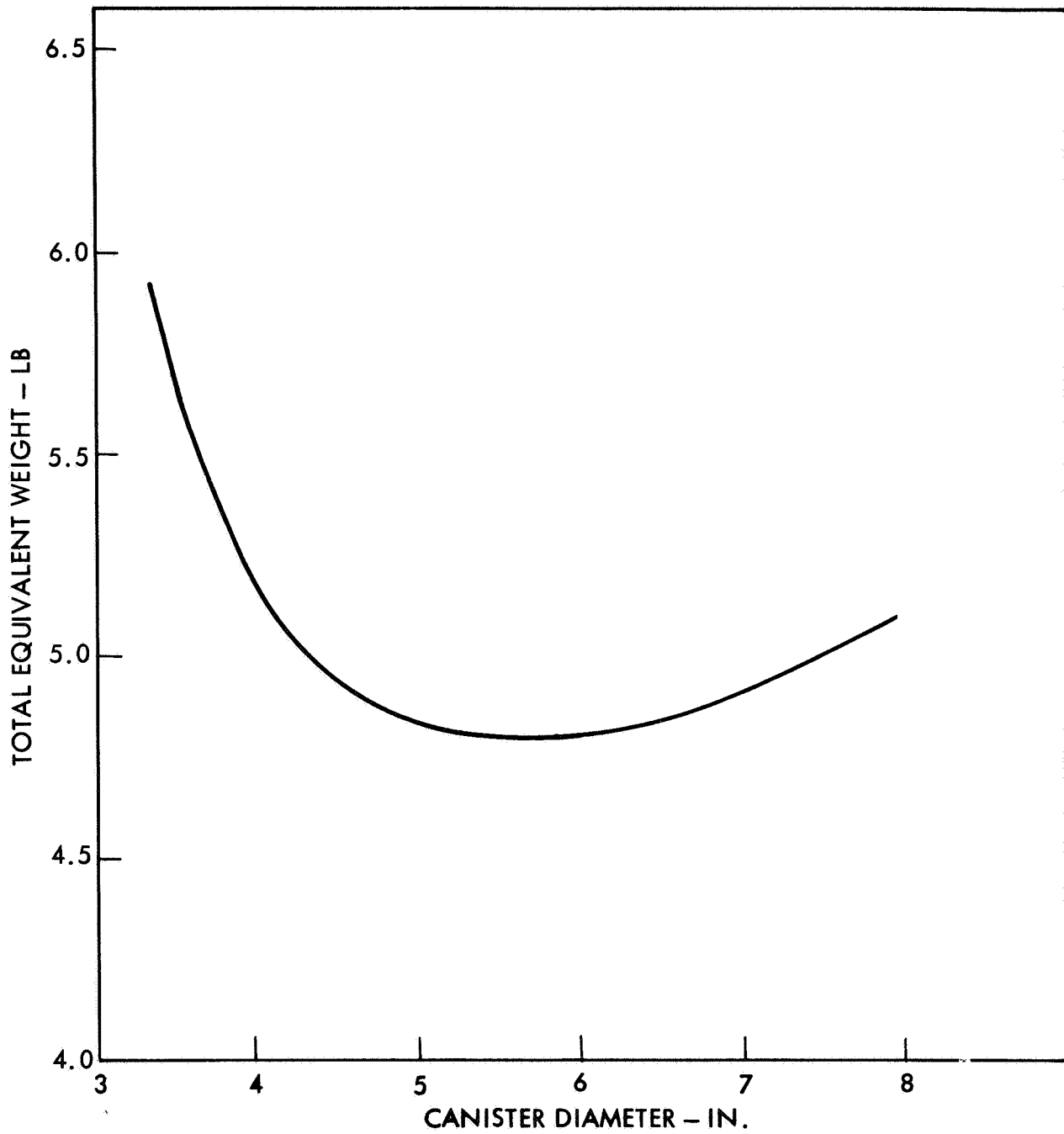


Fig. 21 Post-Sorbent Bed Optimization

## HEAT SOURCE MATERIAL SPECIFICATIONS

To ensure that the materials of construction, for the isotope heat source, have reproducible physical and chemical properties and to establish material selection guidelines, a materials traceability program was developed. This program included the preparation of material specifications which are presented in Appendix A. These specifications were used for the procurement of materials required for the fabrication and joining tests and inter-diffusion experiments. Materials traceability was performed by TRW design engineering liaison during fabrication and joining evaluation and on purchased materials by vendor certification and spot check evaluation. A typical materials traceability program used for end item flight hardware is presented in Appendix B.





## JOINING AND FABRICATION TESTS

A number of fabrication processes were evaluated for each of the parts comprising the isotope heat source assembly. The selected processes, which are described in detail reflect the experience gained by TRW in past isotope heat source programs, adapted to meet the IHCOS requirements.

### Fabrication Methods

Table 9 summarizes the IHCOS assembly materials, part description, and the method of fabrication. Fabrication, inspection and assembly of all the piece parts were conducted at TRW except for fabrication of the two pyrolytic graphite shells, which was performed by the Super-Temp Corporation. As a preface to the discussion of the selected manufacturing techniques, a short description of the recommended processes is given below.

TABLE 9 IHCOS HEAT SOURCE PIECE PART FABRICATION METHODS

<u>Part Description</u>	<u>Material</u>	<u>Fabrication Techniques</u>
Liner Shell	*	*
Liner End-Caps	*	*
Strength Member	TZM	*
Cladding Shell	Pt-20Rh	Seamless tubing
Clad End-Caps	Pt-20Rh	Spinning and finish machine
Reentry Body	Pyrolytic Graphite	Chemical vapor deposition
Transition Member	ATJ Graphite	Machine from bar stock
Outer Member Shell	321 SS	Machine from tubing
Fins	321 SS	Machine from plate
Outer Member End-Caps	321 SS	Machine from plate

\*Refer to Classified Supplement

Spinning.-Spinning is a method of flowing metal by pressure of a roller against a rotating piece of metal. The final shape is determined by the mandrel over which the metal is flowed. By continuous rotation of the metal and pressure of the roller, the metal is gradually forced to conform to the shape of the mandrel. For the Pt-20Rh alloy clad end-caps, spinning can be done at room temperature since the material is extremely ductile. A finish machining operation is normally required after spinning to trim off excess material. Tooling expense is relatively low and overall cost moderate.

Roll forming.-Forming of sheet, plate, and bar by rolling into a circular, conical, or cylindrical shape is an established process and lends itself to almost any material. It is limited to parts of fairly simple shape, but where applicable requires little tooling and is an economical method of producing such parts. There is little material waste, machining is eliminated or minimized, the process is simple, and chance of contamination is minimal.

After roll forming, a welding operation can be performed to complete the part. The Pt-20Rh cladding shell can be fabricated in this manner.

Machining.-Machining, employing conventional lathe and milling machines, is an established manufacturing method for shaping almost any material and can be used over a wide size range. It is suitable for either simple or complex parts requiring very close tolerances. Contamination effects are negligible, tooling is easily adaptable to conventional machines, and machining techniques require a minimum of development time. The ATJ graphite, 321 SS, TZM, and liner parts fall in this category.

Chemical vapor deposition.-In this process the material is produced by the thermal decomposition of a gaseous compound of the desired material on a hot surface. The production of pyrolytic graphite is carried out by passing a hydrocarbon gas over a hot surface held at approximately 4000°F. The carbon atoms are removed from the gas by a thermal decomposition process and are deposited in a manner similar to a vacuum plating operation. A mandrel of the desired shape serves as the decomposition surface.

### Selected Joining Techniques

It is imperative that isotopic heat sources be carefully designed, fabricated, and assembled.

The importance of good welding procedures cannot be over-emphasized. Tungsten-inert gas (TIG) welding and electron-beam (EB) welding are currently used for effecting heat source closures. With both methods, welding parameters such as welding current, speed of rotation, type of inert gas used, size of electrode, gap length, gas flow rate, welding voltage, number of welds, and time of weld must be determined for each type of closure to yield the desired, reproducible penetration. To determine these parameters, sample assemblies are welded, sectioned, and examined metallographically.

A discussion of the various weld joints and selected joining techniques

for all parts, except the liner and strength member, follows.

Liner.-The discussion of the liner fabrication techniques is presented in a classified supplement to this report.

Strength member.-The discussion of the strength member fabrication techniques is presented in a classified supplement to this report.

Cladding.-The Pt-20Rh cladding consists of one center shell and two end-caps. Because of the high cost of this material, fabrication was limited to the two end-caps. Since Pt-20Rh is a relatively ductile material, it was decided to fabricate the end-caps utilizing a spinning operation. The stainless steel master form used to form these parts, and the finished end-caps, are shown in Figure 22.

There are two general methods for fabricating the Pt-20Rh cylindrical shell: welded seam and seamless. The welded seam method involves rolling or die-shaping sheet stock in a cylinder and fusion welding the axial seam to form a solid tube. This method can be accomplished using sheets two to three times the required thickness, welding, and then redrawing the tube with appropriate extrusion dies. Alternately, the tube can be formed from a sheet of the required wall thickness and welded. The former method of producing seam tubing is generally preferred due to improved grain structure in the weld region, and eliminates problems concerned with weld drop-through and distortion.

In order to more thoroughly investigate the fabricability of tubes by the seam welding process, TRW has formed Pt-Rh tubing to SNAP-29 diametric tolerances, and investigated EB and TIG welding processes for making the seam weld. Figure 23 shows one of the Pt-Rh tubes after forming and EB welding. A press brake, using appropriately configured dies, was used to form 0.020 inch sheet. After the sheet was formed, fixtures were used to support the tube during tacking and final welding operations. Experience gained during initial efforts indicated that 0.003 inch diametric tolerances could be obtained by this process, and that the working properties of the material were adaptable to die press forming techniques. EB and TIG welding both resulted in high quality welds. When TIG welding was employed, using a copper cooling mandrel and Pt-Rh filler material, weld concavity and drop-through were essentially eliminated. Metallographic inspections were also used to verify weld penetration. The typical structure of a Pt-Rh weld appears in Figure 24, showing a butt weld in 0.020 inch Pt-10Rh sheet performed by the electron-beam process.

Seam welded tubing has the advantage of minimum material waste, relatively low tooling cost, and nominal development effort. The principal disadvantage of seam welded tubing is associated with the integrity of a full axial weld. Since strength, purity, and integrity of the tube are extremely critical, possible discontinuities in the weld region are of considerable concern. The complications involved in analyzing the potential long-term

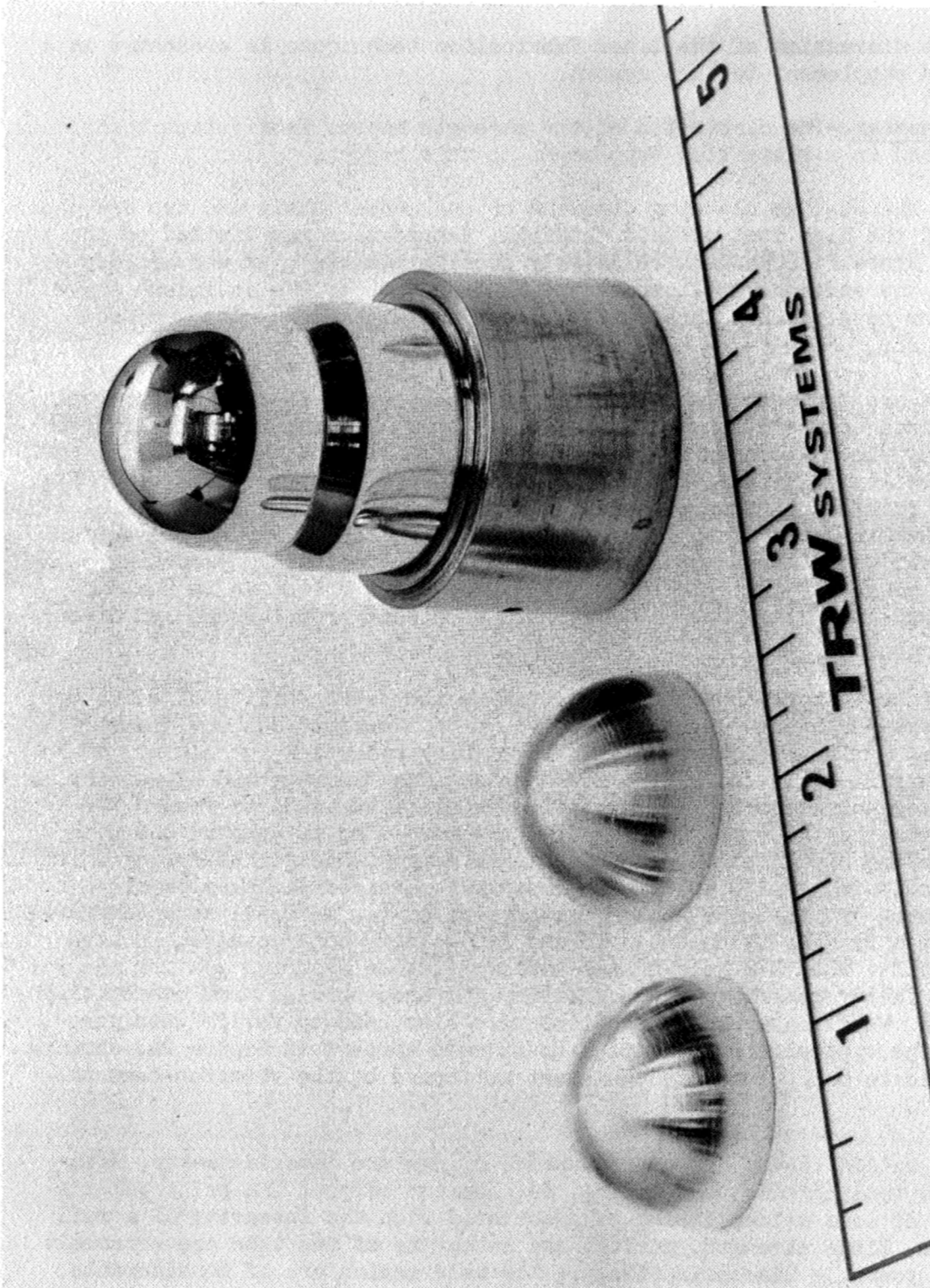


Fig. 22 Pt-10Rh Cladding End-Caps and Spinning Form

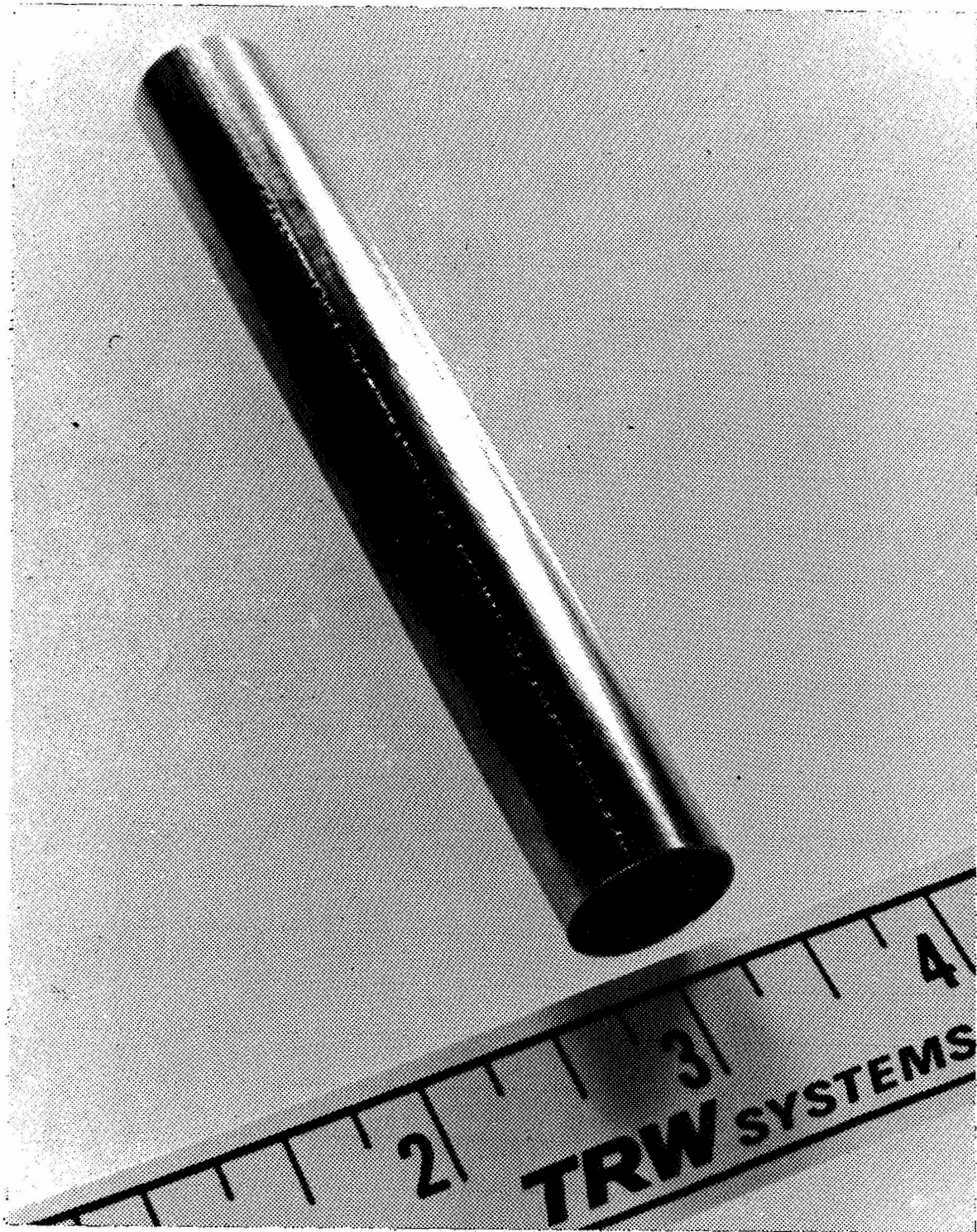


Fig. 23 Seam Welded Pt-Rh Tubing

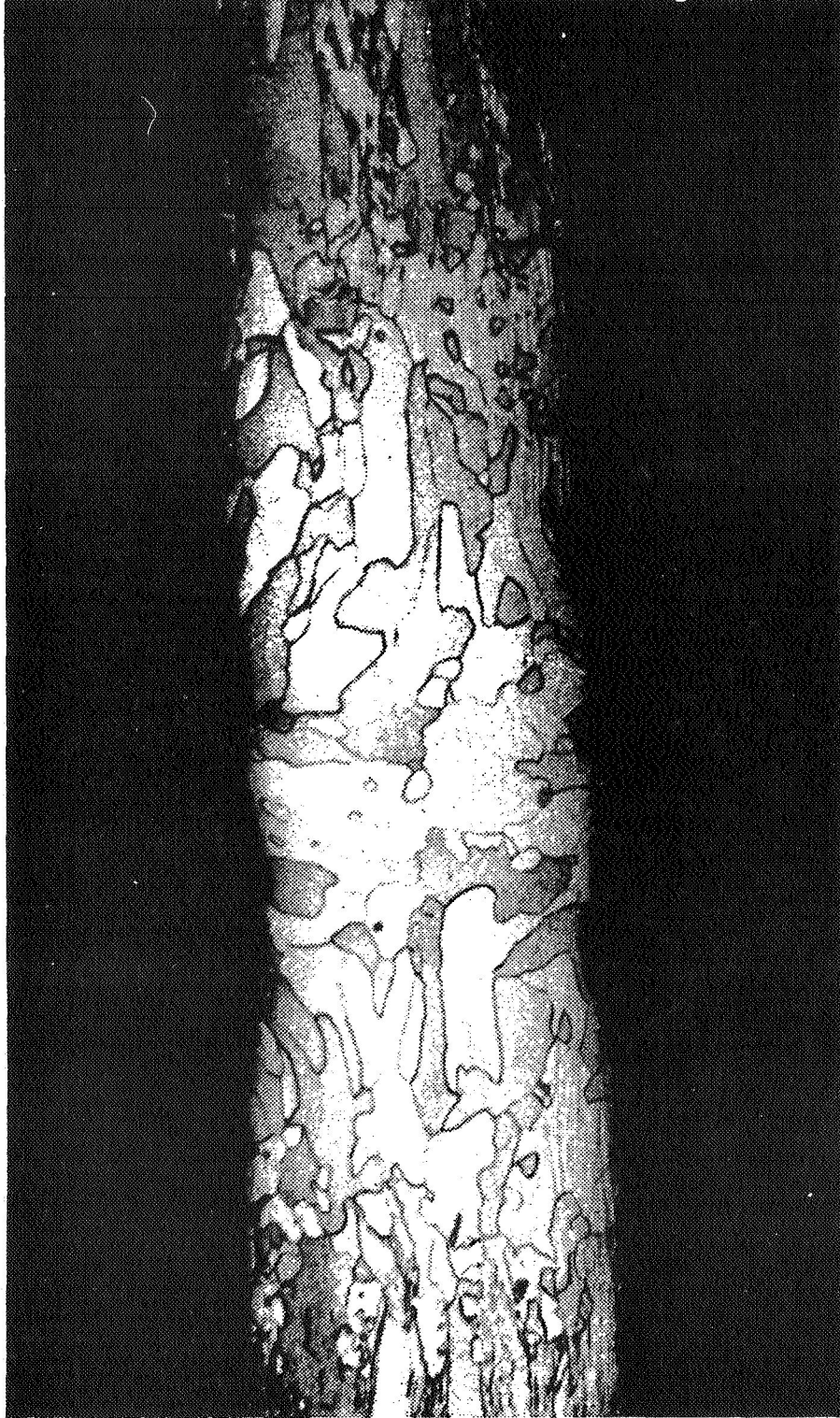


Fig. 24 Photomicrograph of the Weld Region in 0.020 Inch Pt-10Rh Sheet-50X  
(No Filler Material Used)

effects caused by the fusion process and in performing sophisticated non-destructive examination tests suggest that alternate fabrication techniques, such as seamless tubing, should be considered.

Seamless tubing requires the application of specialized manufacturing techniques and tooling. However, such techniques exist for producing seamless Pt-20Rh tubing in the size required for the IHCOS program. TRW believes they can be applied to yield high quality structures of tight diametric and straightness tolerances with negligible material impurity content and a high-strength grain structure.

Seamless Pt-20Rh tubing can be formed from a disc of the alloy which is drawn through dies of various sizes until the desired dimensions are achieved. Generally, mandrels are used for shaping seamless tubing and to maintain the internal diameter. However, to achieve close tolerances and high surface quality on the IHCOS components, a plug-type die rather than a mandrel may be preferred for final drawing operations; difficulties in removing the tube from a mandrel can thus be avoided. This process results in close control of wall thicknesses and high surface quality. Due to the excellent ductility of Pt-20Rh, complete intermediate anneals may not be required. Although some stress relieving of the tubes may be desirable, complete anneals will be undesirable due to large reduction in strength of annealed Pt-Rh alloys compared to cold worked material.

Favorable TRW experience with seamless Pt-Rh tubing makes this fabrication method the primary choice for the liner shell.

Reentry member.-Two pyrolytic graphite assemblies, shown in Figures 25 and 26 were fabricated by Super-Temp Corporation. The hemispherical ends were considered a potential problem area because of the anisotropy of pyrolytic graphite. In the direction perpendicular to the surface, the material contraction is approximately 20 times greater on cooling than in the parallel direction. This means that closed shapes of pyrolytic graphite may develop residual stresses on cooling from the deposition temperature. Super-Temp Corporation precluded this effect by cooling at a slow rate.

One fabrication problem did occur in machining the threads used to join the two members. Microscopic examination of the parts revealed a partially laminated structure, with the propagation of laminations occurring during threading. The relatively low interlaminar shear strength of this material, combined with the laminar structure, caused portions of the threads to break off.

One of the pyrolytic graphite assemblies was re-machined to determine the effect of using a coarser thread (16 threads per inch versus the original 28 threads per inch). An improved, but still imperfect, thread resulted.

The pyrolytic graphite parts were vapor deposited onto a male mandrel (external deposition). This method of deposition results in higher residual stresses than those observed in material deposition on female mandrels (in-

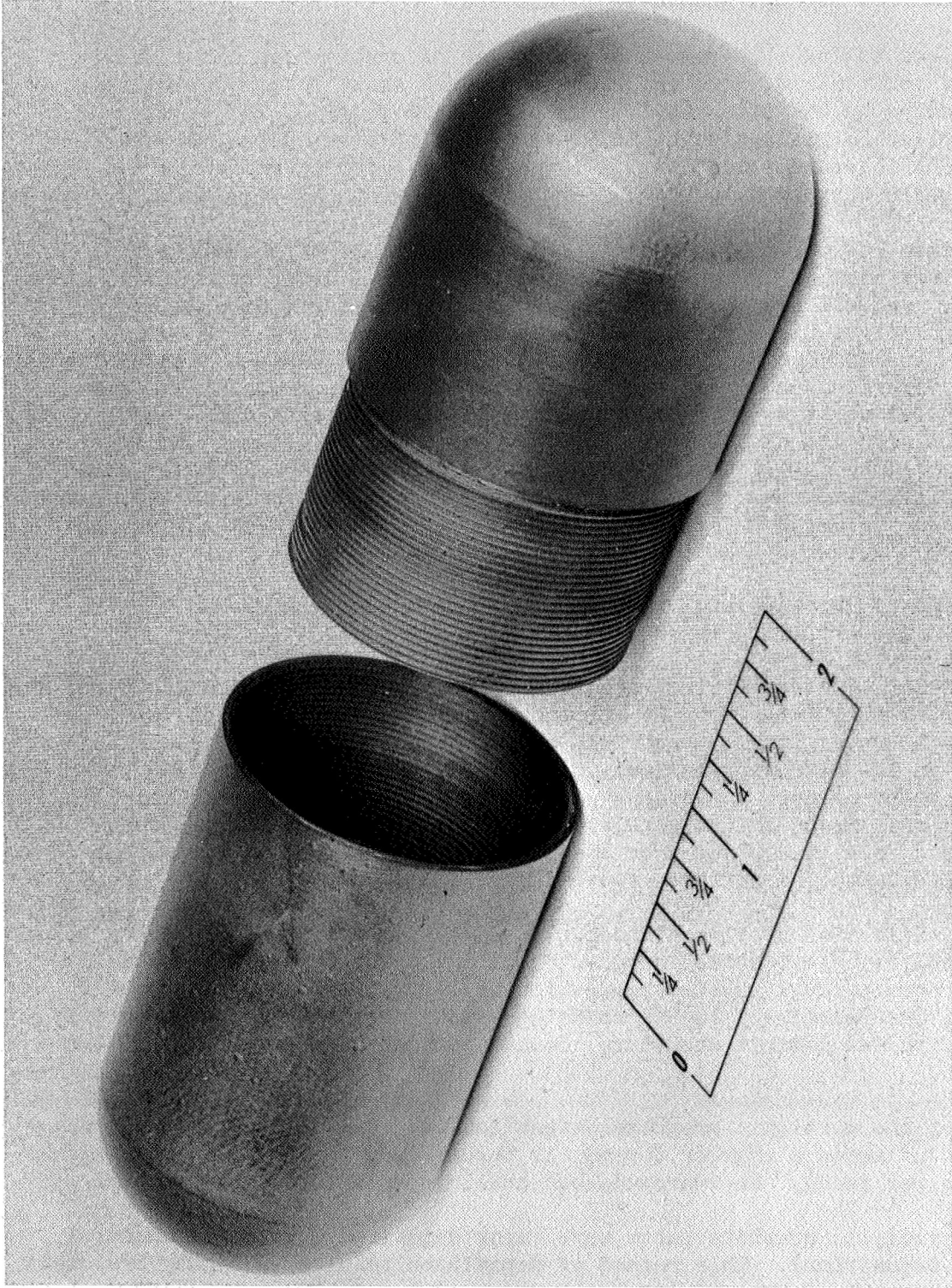


Fig. 25 Pyrolytic Graphite Reentry Member Shells



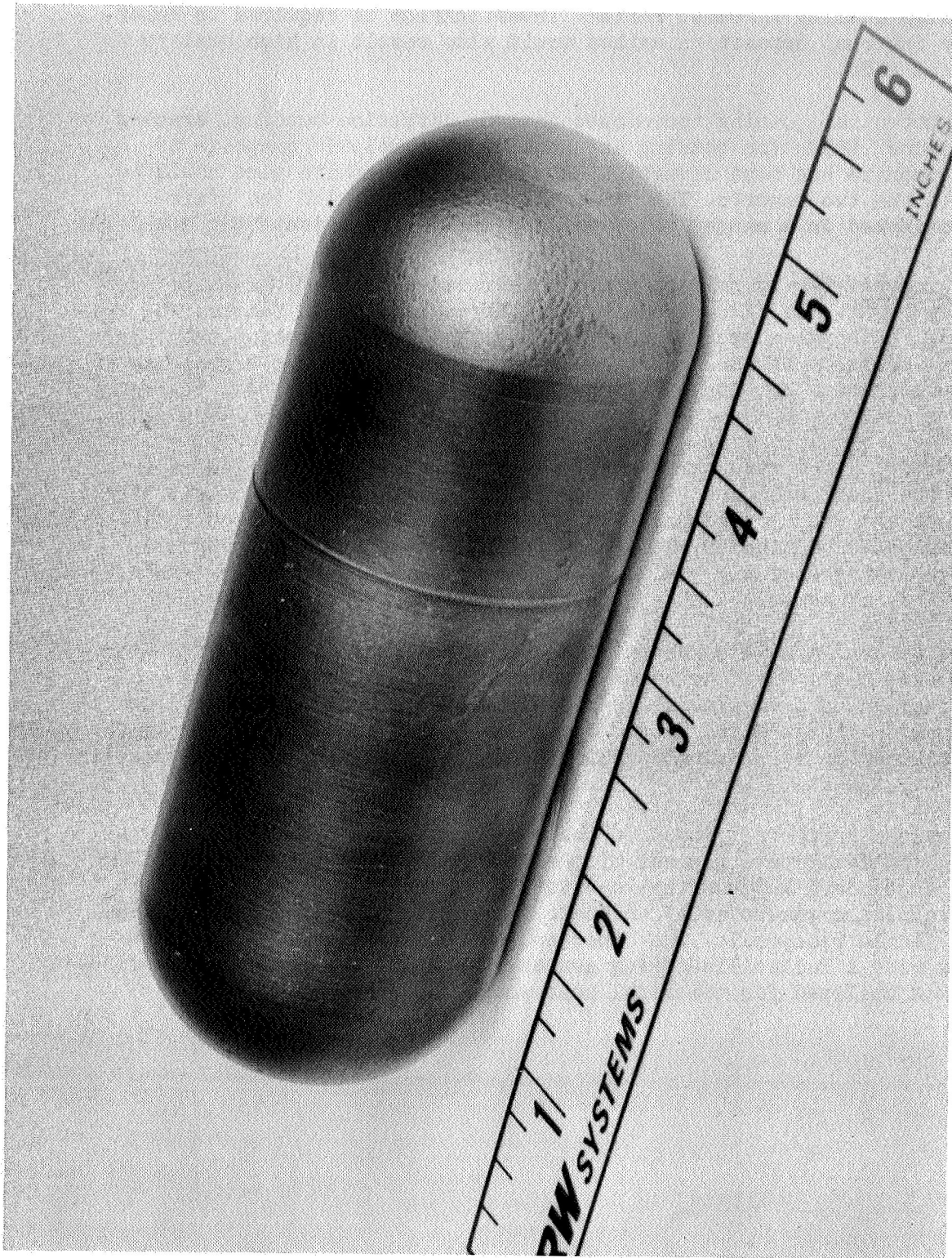


Fig. 26 Pyrolytic Graphite Reentry Member Assembly

ternal deposition). Two sample right circular cylinders shown in Figure 27 were fabricated by Super-Temp Corporation for TRW during an earlier capability evaluation. These parts were machined with little difficulty and resulted in high quality threads. Further investigation is required to determine if the internal deposition method would also result in high quality hemispherical ends.

Other potential joining techniques include diffusion bonding, brazing or the use of pins. Diffusion bonding and brazing of pyrolytic graphite to pyrolytic graphite has been investigated. Pyrolytic graphite pins could be used to join the two members. The pin would be machined with its grain structure oriented in a manner which would preserve its anisotropic qualities.

Transition member.-Two transition members fabricated from ATJ graphite are shown in Figure 28. The free machining of this material made fabrication relatively easy, although a special template was required to machine the internal contour. Addition of the transition members to the pyrolytic graphite member completes this portion of the heat source assembly as shown in Figure 29.

Structural module.-The structural module is shown in Figure 30. The material chosen for the components was 321 (austenitic) series stainless steel. Steels in this group are widely used and have the highest resistance to corrosion in the stainless steel family. They also possess the greatest strength at elevated temperatures of any stainless and heat-resisting steel, yet retain their ductility at temperatures approaching absolute zero.

The structural module consists of a cylindrical shell, fins and end-caps. The cylindrical shell was fabricated from tubular stock. The fins and end-caps were fabricated from plate stock. Conventional machining was utilized for these parts without difficulty. Both sides of the fin were then welded to the shell section to provide maximum structural strength and heat transfer area.

The conical supports located on the end-cap will be cylindrical in the final design to facilitate removal of the isotope heat source from the IHCOS assembly. A standard Woodruff key slot will be machined in one support to eliminate angular movement after the heat source assembly is mated to a complementary female receptacle. The other end-cap has a cylindrical support which mates with a Belleville-spring assembly to allow for thermal expansion. A TIG weld is utilized for the final heat source closure.

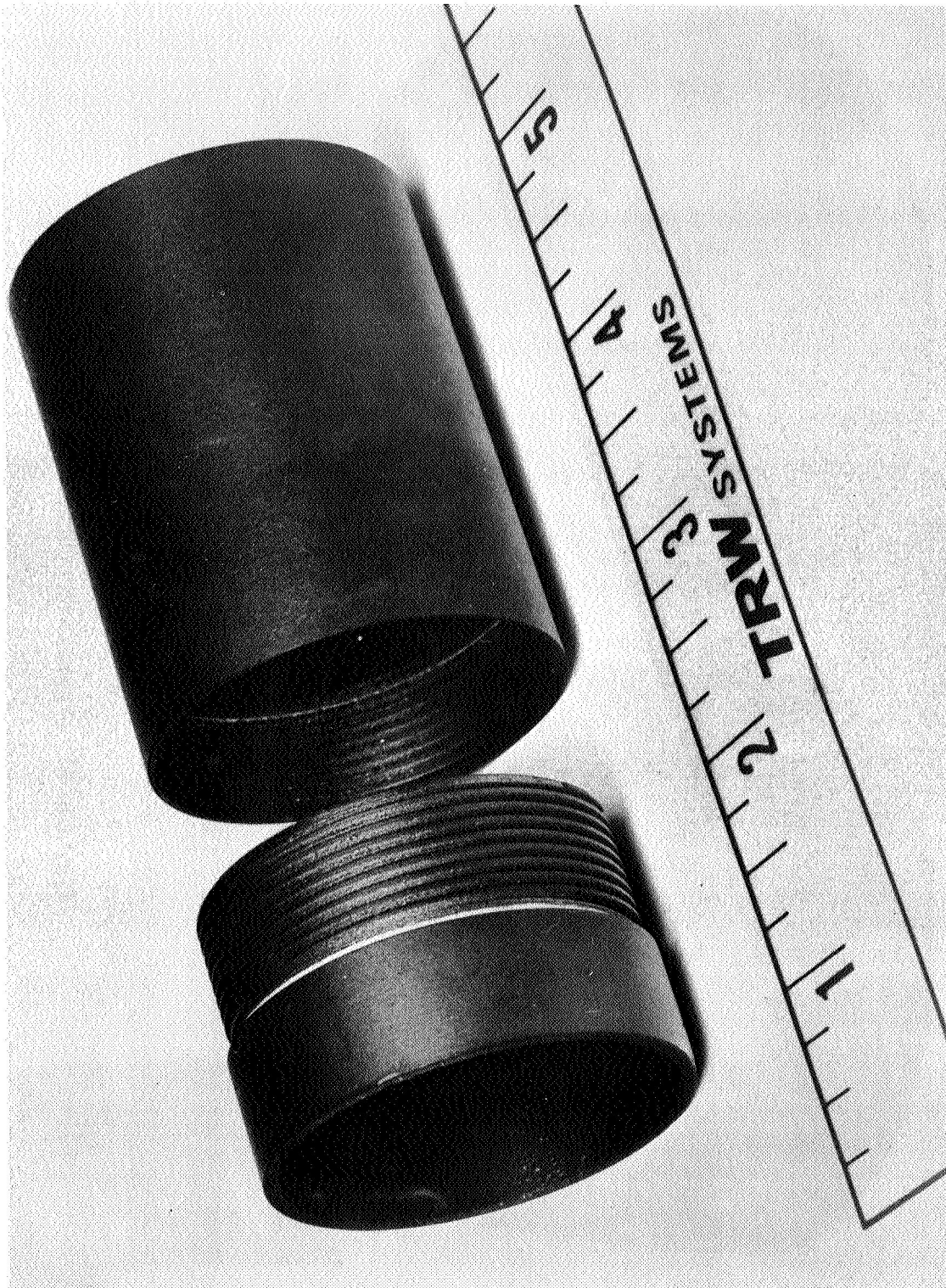


Fig. 27 Pyrolytic Graphite Sample Shells

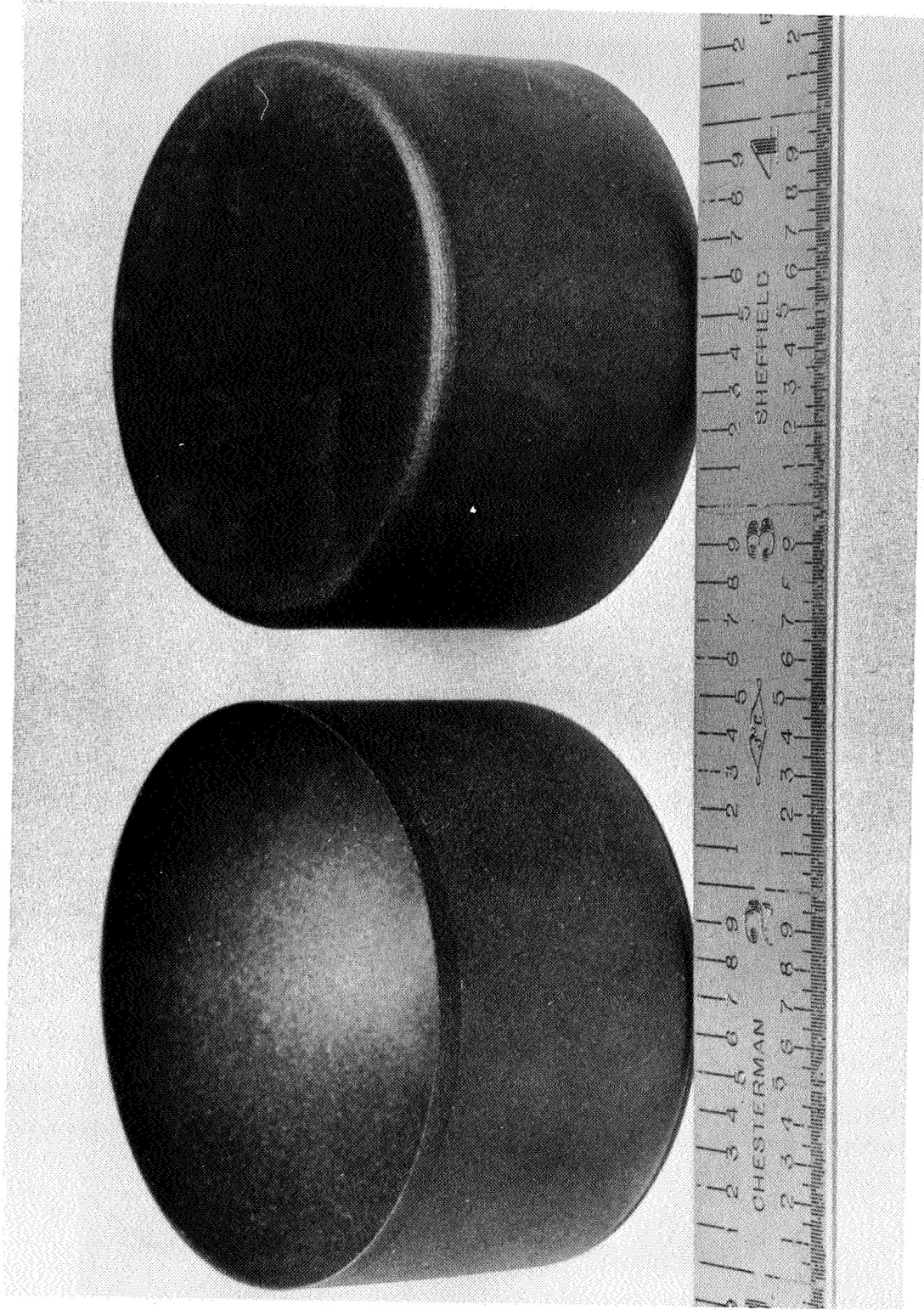


Fig. 28 ATJ Graphite Transition Members

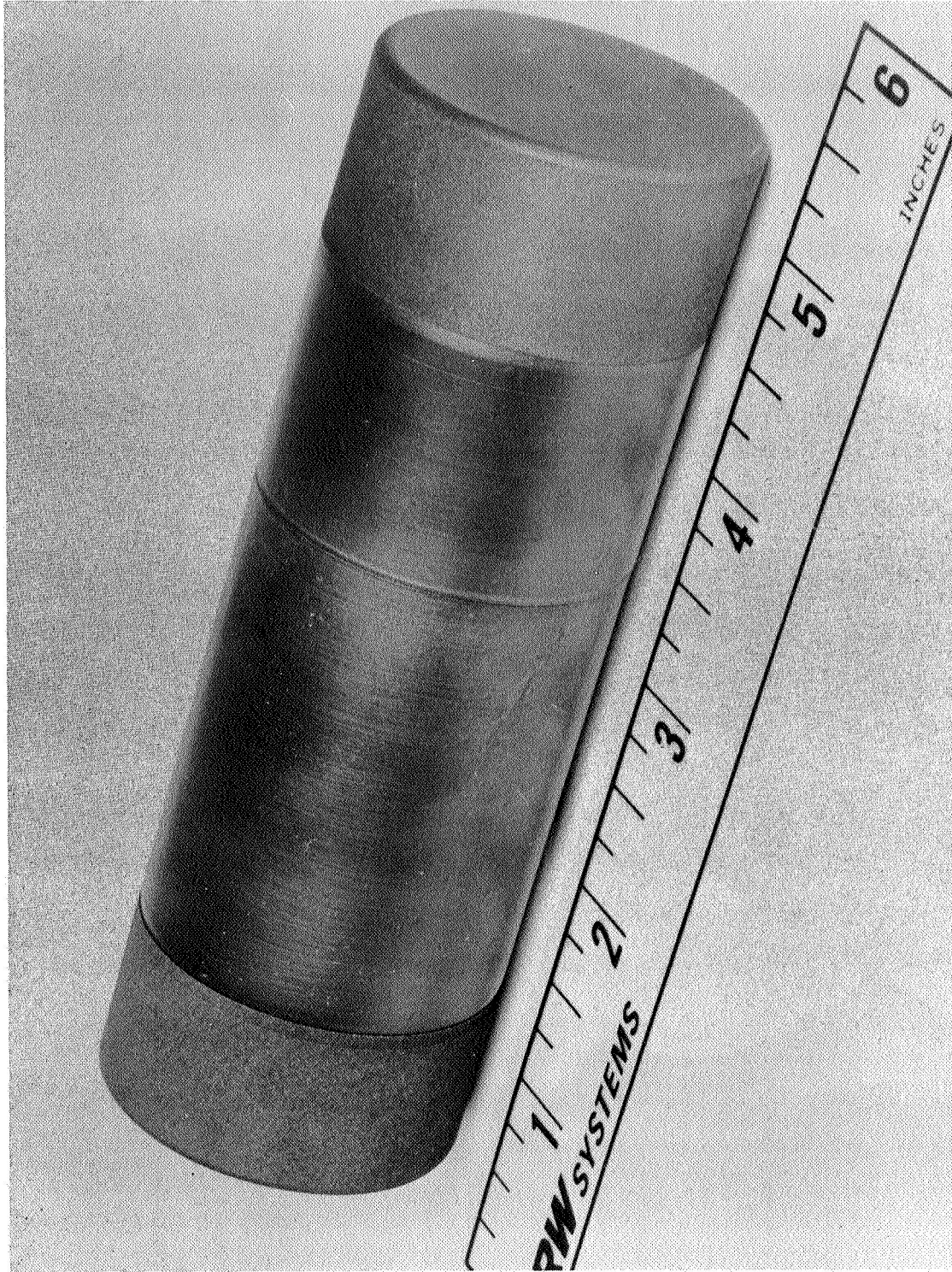


Fig. 29 Pyrolytic and ATJ Graphite Assembly

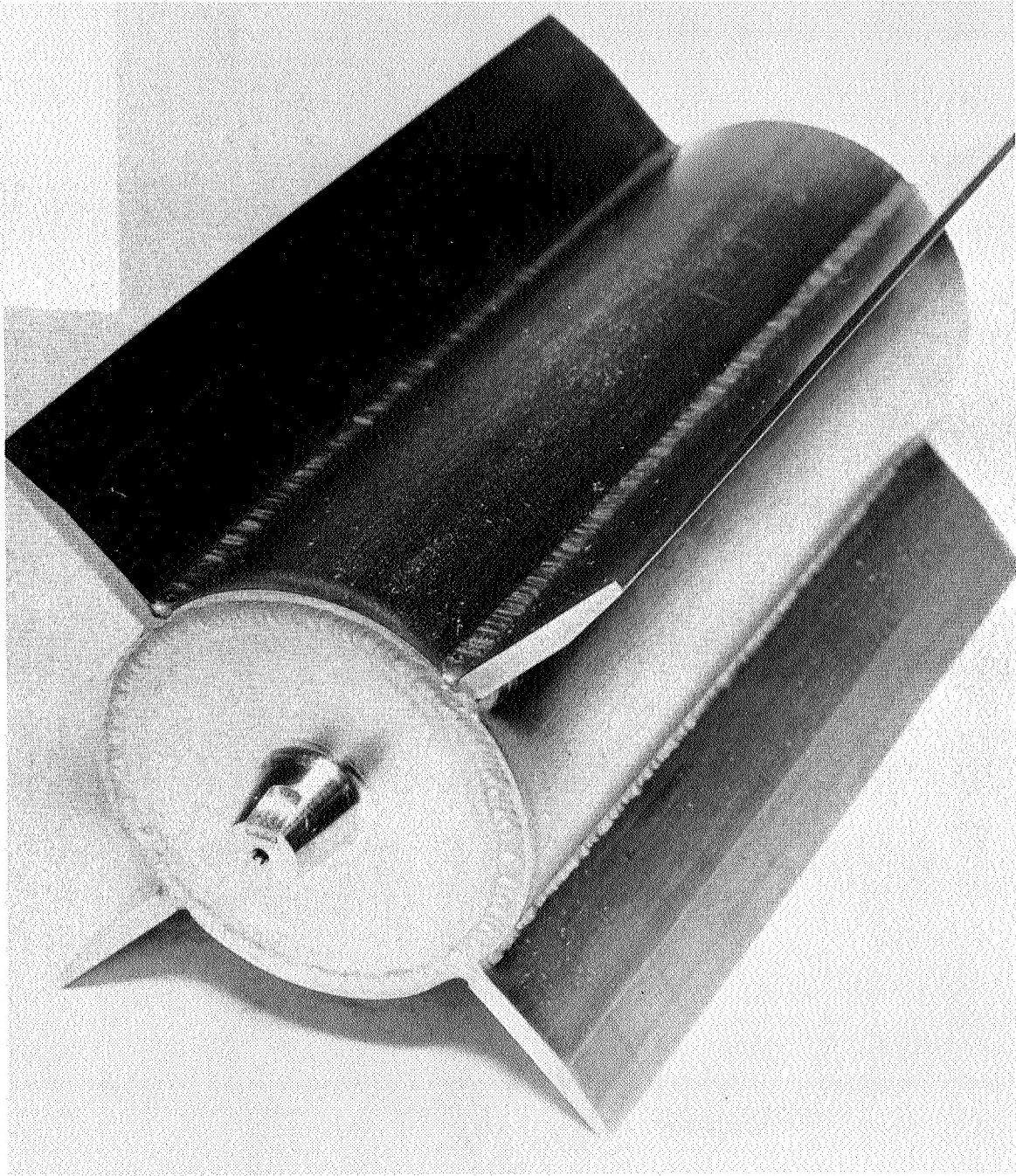


Fig. 30 Structural Module Assembly

## MATERIALS COMPATABILITY STUDIES

The experimental studies described herein were conducted to (1) predict the extent of interdiffusion that would occur between the various materials at temperatures of 800°F and 1500°F and (2) to assess the effects of exposing the pyrolytic graphite-stainless steel interface to temperatures near and at the melting point of stainless steel. Under normal operation conditions, the isotope heat source liner surface temperature will not exceed 800°F. The potential failure mode of the system will result in a liner surface temperature of 1500°F. During reentry, the structural module will approach or exceed its melting temperature for 2 to 5 minutes.

### Interdiffusion Studies

Interdiffusion between materials of the various layers would be detrimental if, during the life of the mission, the extent were sufficient to:

- modify the composition of the liner material, destroying its compatibility with the fuel form,
- alter the composition of the structural alloy to the point of degrading its mechanical properties, thus risking failure by excessive creep or stress rupture,
- impair the oxidation protection characteristics of the cladding, thereby permitting the structural alloy to oxidize upon exposure to air at elevated temperature, or,
- modify the chemical properties of the reentry body, accelerating its oxidation rate during reentry.

Specimen preparation and thermal exposure.-Specimens used in the interdiffusion study consisted of 3/8-inch diameter, 0.030 to 0.050 inch thick discs which were cut from sheet stock by electrical discharge machining. Flat sample surfaces were attained by hand polishing the machined discs with 600 grit silicone carbide paper. Each test sample was rinsed in acetone and air dried before being placed in a molybdenum sample holder. The sample holder is shown schematically in Figure 31 along with the sample arrangement. The number and positions of samples shown were based on providing duplicate interfaces of each material combination to be examined. Four such specimen holder assemblies were prepared.

Since thermal expansion of the specimens was greater than that of the holder, axial pressure developed during heating served to maintain contact

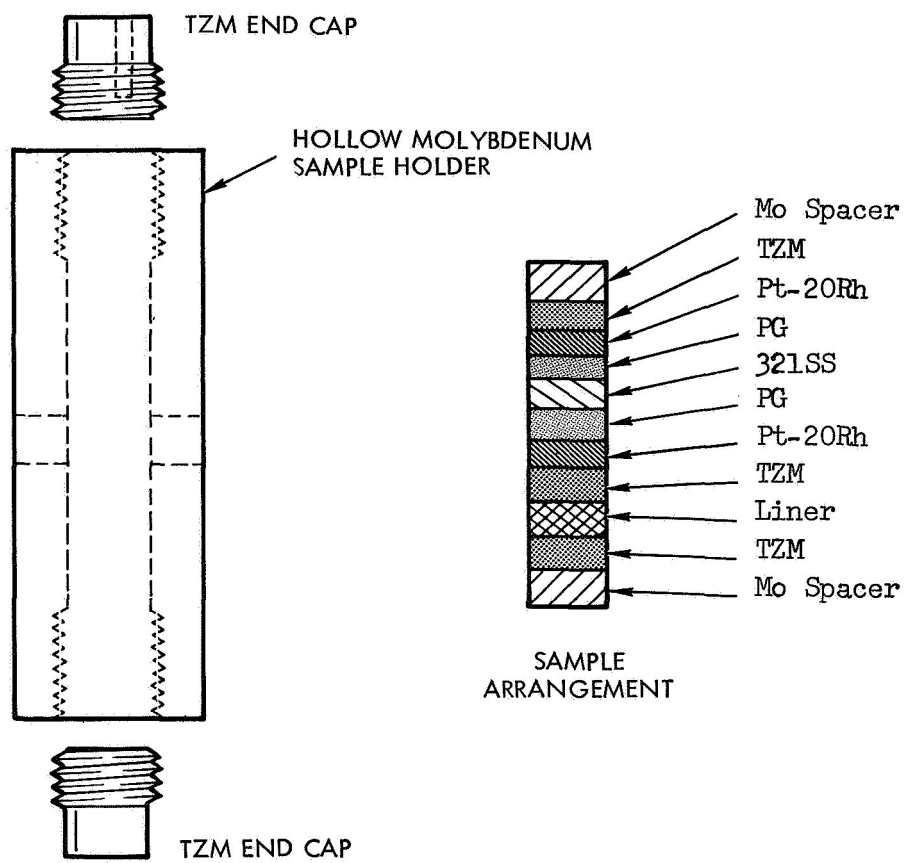


Fig. 31 Sample Holder for Diffusion Studies



at the interfaces. Assuming a tensile yield strength for molybdenum of 15,000 psi at 1500°F and 25,000 psi at 800°F, the axial compressive stress on the discs could not exceed 45,000 psi at 1500°F or 75,000 psi at 800°F. According to reported data, these pressure levels are insufficient to appreciably affect diffusion rates (ref. 78).

The assemblies were surrounded by tantalum foil getter material and contained in mullite tubular chambers in which a vacuum of  $10^{-5}$  torr was maintained. Each chamber held two capsule assemblies. The two chambers were heated in silicone carbide resistance furnaces. A photograph of one of the furnace systems is shown in Figure 32. One furnace operated at 1500°F, the other at 800°F. After 15 days, one capsule was removed from each furnace. The others were allowed to remain at temperature for 60 days.

Methods of interface evaluation.-The thickness of the diffusion modified material zone at each interface was determined by metallography and electron microprobe analysis.

Each assembly was sectioned longitudinally through the stack of discs, mounted and polished to observe the structure at each interface. Although metallographic techniques reveal gross phase changes or modifications in the microstructure induced by compositional changes due to interdiffusion, small variations in composition are often not sufficient to cause a visible change in microstructure. The metallographic examinations were, therefore, supplemented by electron microprobe analyses, which directly show variations in composition.

Basically, the principle of operation of the microprobe is as follows: A highly collimated beam of electrons impinges upon the surface of the sample to be analyzed, activating the bombarded material, causing it to emit X-rays. Emitted X-rays pass through a crystal where they are diffracted and into a detector where wavelengths and intensity at each wavelength are determined. The X-rays are characteristic of the material from which they are radiated. The intensity at a characteristic wavelength is indicative of the quantity of that element present. The diameter of the impinging beam is less than 1 micron (0.0004 inch) making possible the analysis of a volume of material as small as 2 microns on a side ( $5 \times 10^{-13}$  in<sup>3</sup>). The exact volume is dependent on the molecular properties of the material. Moving the sample under a stationary beam yields a plot of X-ray intensity at a selected wavelength versus position in the sample. Neglecting corrections due to absorption and fluorescence, this intensity is approximately equivalent to concentration of the element of interest emitting the selected characteristic wavelength. Such scans were made across selected areas of interfaces between each combination of materials being evaluated in an attempt to define the precise limits of diffusion modified material zones. A photograph of the apparatus used is shown in Figure 33. Microprobe operating parameters are shown in Table 10.

Interdiffusion results.-Photomicrographs at 400X magnification of sections through the liner material TZM interface are shown after 15 days at 800°F in Figure 34, after 60 days at 800°F in Figure 35, after 15 days at 1500°F in

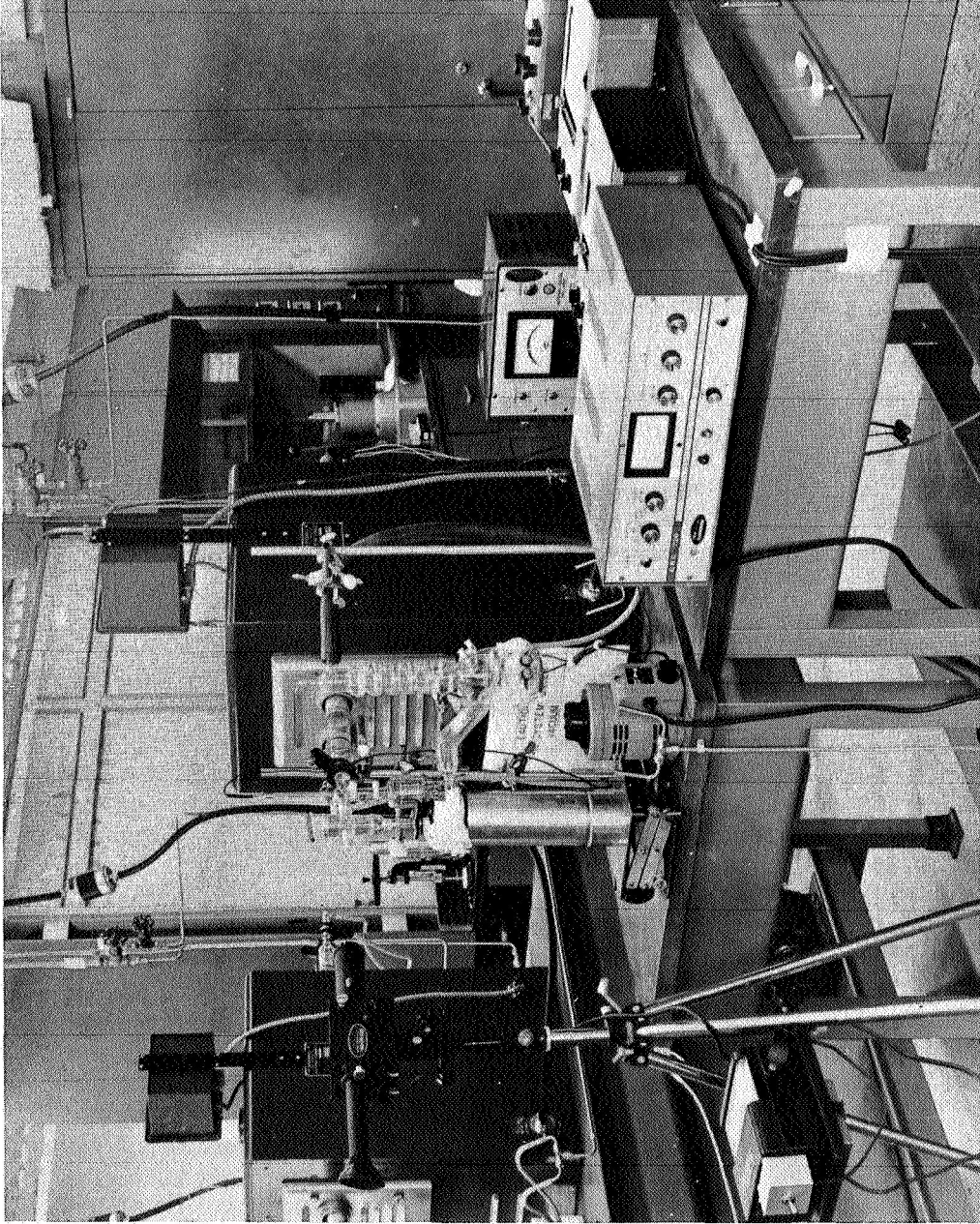


Fig. 32 Furnace System Used for Thermal Exposure of Interdiffusion Specimens

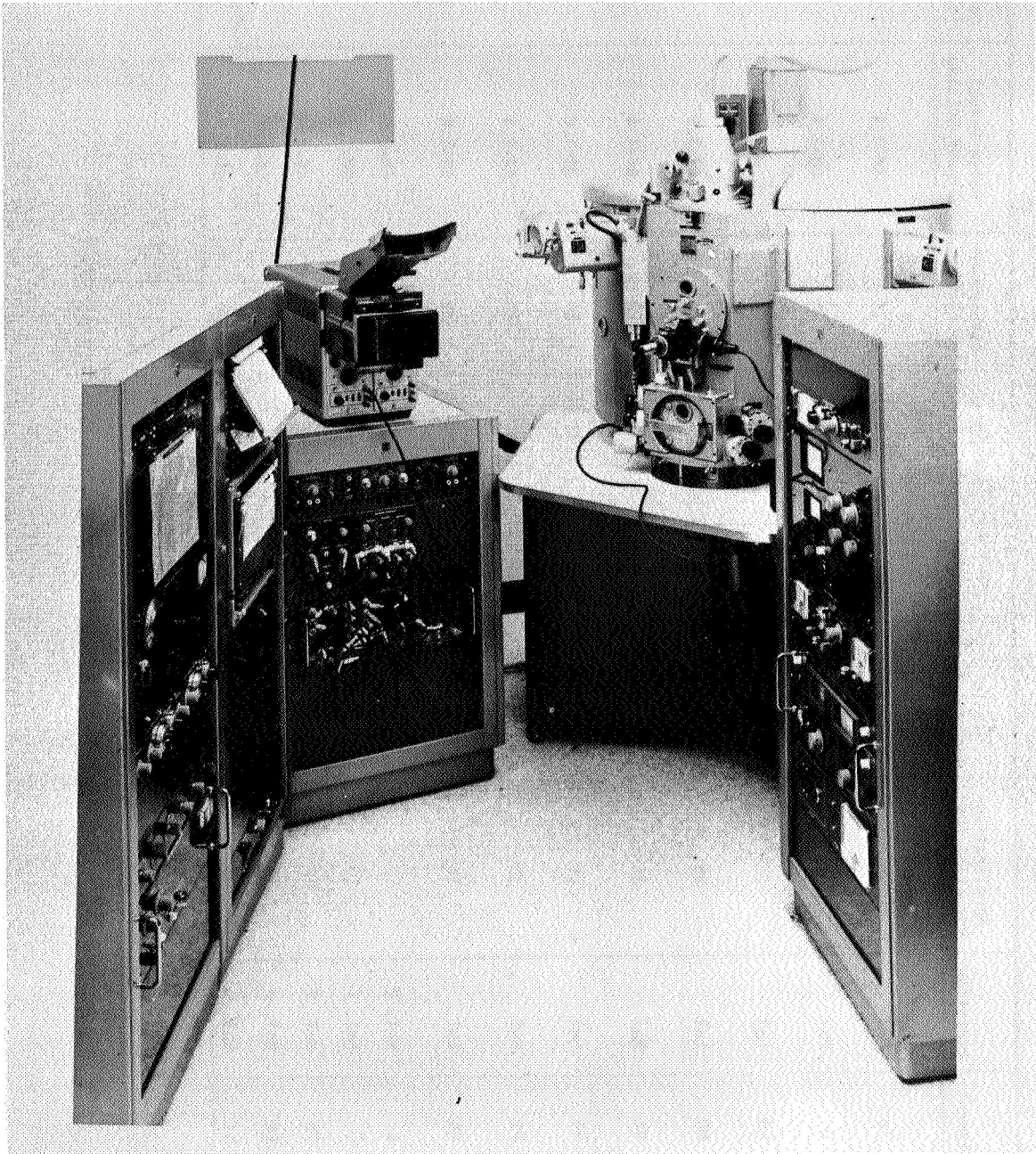


Fig. 33 Electron Microprobe Analyzer

Interface	Element	Radiation	Accelerating Potential (Kv)	Sample Current ( $\mu$ a)	Crystal	Spectrometer Setting	Attenuator Setting	Pulse Height Analyzer Setting	Time Constant (seconds)	Sensitivity (%)	Scale Factor
liner - TZM	Ta	M $\alpha$ 1	25	.007	ADP	2.7415	26	64-1.4	.5	100	100
liner - TZM	W	M $\alpha$ 1	25	.007	ADP	2.6387	26	64-1.4	.5	100	100
liner - TZM	MO	L $\alpha$ 1	25	.007	KAP	1.6340	15	8-1.0	1	70	100
TZM - (Pt-20Rh)	MO	L $\alpha$ 1	25	.01	KAP	1.6264	15	64-1.5	1	58	100
TZM - (Pt-20Rh)	Pt	M $\alpha$ 1	25	.01	ADP	2.2891	28	64-1.4	.5	64	100
TZM - (Pt-20Rh)	Rh	L $\alpha$ 1	25	.01	ADP	1.7280	25	32-1.8	.5	100	100
(Pt-20Rh) - PG	Pt	M $\alpha$ 1	10	.1	ADP	2.2891	28	64-1.4	.5	10	100
(Pt-20Rh) - PG	Rh	L $\alpha$ 1	10	.1	ADP	1.7364	26	64-1.4	.5	53	100
(Pt-20Rh) - PG	C	K $\alpha$ 1, 2	10	.1	LSD	1.7694	28	64-2.0	1	29	10
PG - SS	C	K $\alpha$ 1, 2	10	.08	LSD	1.7760	15	64-1.0	1	60	10
PG - SS	Fe	K $\alpha$ 1, 2	10	.08	ADP	1.4481	20	64-2.0	.5	100	100
PG - SS	Cr	K $\alpha$ 1, 2	10	.08	ADP	1.7216	24	64-1.7	.5	100	100
PG - SS	Ni	K $\alpha$ 1, 2	10	.08	LiF	1.6580	20	64-1.5	.5	100	100

TABLE 10 MICROPROBE OPERATING PARAMETERS

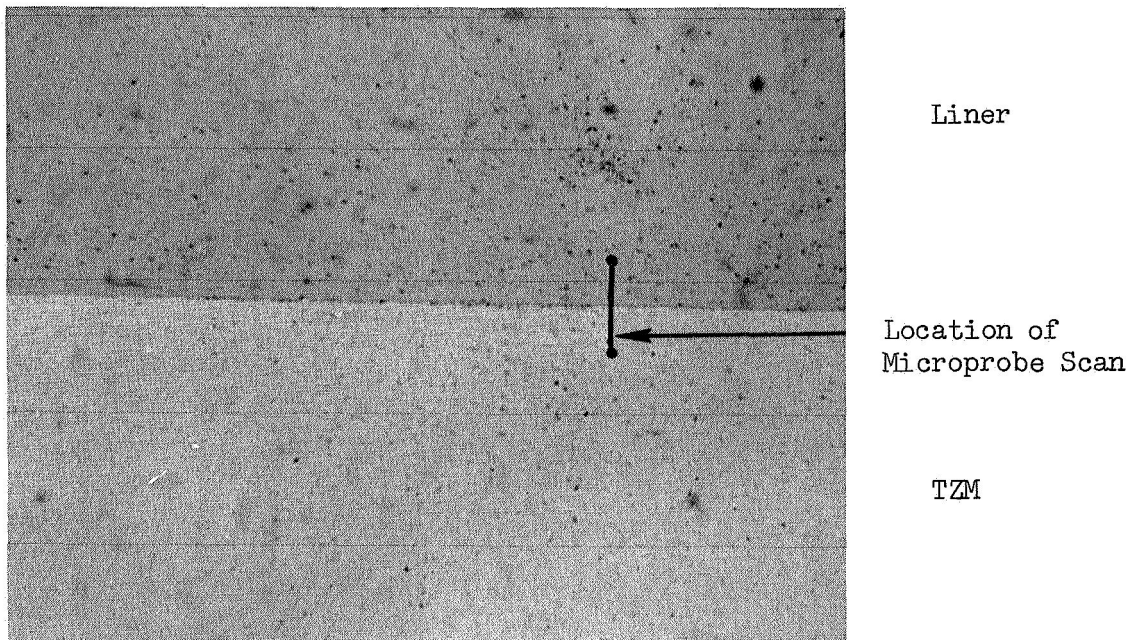


Fig. 34 Interface Between Liner and TZM After  
15 Days at 800°F (400X magnification)

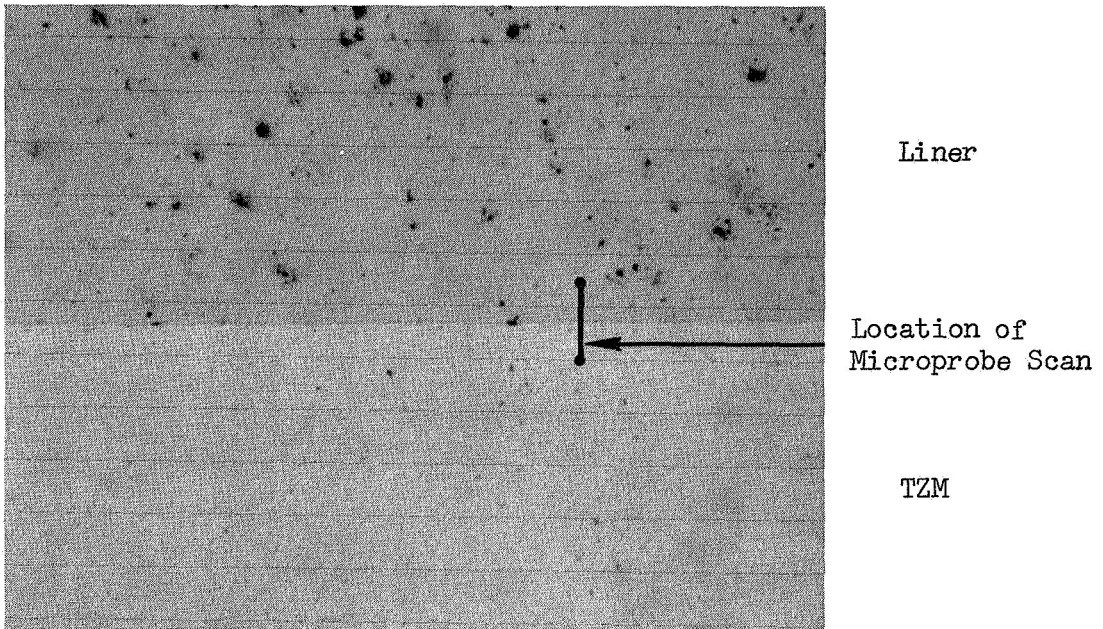


Fig. 35 Interface Between Liner and TZM After  
60 Days at 800°F (400X magnification)

Figure 36, and after 60 days at 1500°F in Figure 37. Corresponding photomicrographs of the TZM-(Pt-20Rh) interface are shown in Figures 38 and 41; of the (Pt-20Rh)-PG interface in Figures 42 through 45, and of the PG stainless steel interface in Figures 46 through 49.

A summary of the distances over which changes in intensity occurred for characteristic X-ray wavelengths of each element analyzed in the microprobe scans is given in Table 11. A portion of the distance over which the intensity parameter changes is due to excitation of a finite volume of material on both sides of the interface as the beam crosses the interface. It is necessary to discriminate between this effect and actual interdiffusion when interpreting the microprobe data. For an accelerating potential of 25 KV, the thickness of the activated material volume is generally not more than 2 to 4 microns for the heavier elements such as the metals under study in this program (ref. 79). The thickness of activated material volume for lighter elements such as carbon may be 4 to 6 microns. The values in Table 11 for capsule 1 (exposure to 800°F for 15 days) appear to represent the distances over which intensity parameters are changing due to the above described effect. It can thus be assumed that the amount of interdiffusion occurring under these conditions is negligible.

Two of the data points in this tabulation are inconsistent with the other data and are, therefore, regarded as invalid. These are the TZM-(Pt-20Rh) interface in capsule 4 and the (Pt-20Rh)-PG interface in capsule 3. Although the location selected for scanning the TZM-(Pt-20Rh) interface indicates good contact even when examined under high magnification light or electron optics, it is possible that an extremely small gap does exist which prevented interdiffusion. The irregularity of the (Pt-20Rh)-PG interface could account for the unexpectedly large distance of changing intensity parameter for this interface in capsule 3. The results of a scan at a location parallel to a mechanical protrusion of Pt-20Rh into pores of the PG does not reflect true interdiffusion.

In the absence of microprobe scans on a set of un-assembled discs for control samples, the values shown for capsule 1 were subtracted from longer time and higher temperature exposures for corresponding interfaces. The resulting diffusion modified material zone thicknesses are shown in Table 12. The amount of interdiffusion at each interface is extremely small and approaches the minimum distance that can be resolved by this type of analysis.

Extension to longer times.-The width of diffusion modified material zones after longer periods of thermal exposure can be predicted from the relationship  $x = (k t)^{1/n}$ , in which  $x$  is the diffusion zone width,  $t$  is time and  $k$  and  $n$  constants. Although  $n$  is dependent on concentration and will change as diffusion proceeds, values between 2 and 5 include nearly all observed diffusion behavior. The predicted two-year diffusion zone thicknesses based on  $n = 2$ , the value yielding results most sensitive to increments in time, are shown in Table 13. In cases where valid data points were obtained for both 15 and 60-day thermal exposures, the extrapolated value was determined using the more severe case. Values for (Pt-20Rh)-PG and PG-stainless steel inter-

TABLE 11

DISTANCE OVER WHICH X-RAY INTENSITY CHANGED  
IN MICROPROBE SCANS

Interface	Distance (microns)*			
	800°F		1500°F	
	15 Days	60 Days	15 Days	60 Days
Liner - TZM	2	2	2	2
TZM - (Pt-20Rh)	2	3	4	2
(Pt-20Rh) - PG	5	22	6	14
PG - SS	5	--	11	15

\* 25.4 microns = .001 inch



TABLE 12

THICKNESS OF DIFFUSION MODIFIED MATERIAL ZONES  
AS INDICATED BY MICROPROBE ANALYSES

Interface	Thickness (microns)*			
	800°F		1500°F	
	15 Days	60 Days	15 Days	60 Days
Liner - TZM	-0-	-0-	-0-	-0-
TZM - (Pt-20Rh)	-0-	1	2	-
(Pt-20Rh) - PG	-0-	-	1	9
PG - SS	-0-	-	6	10

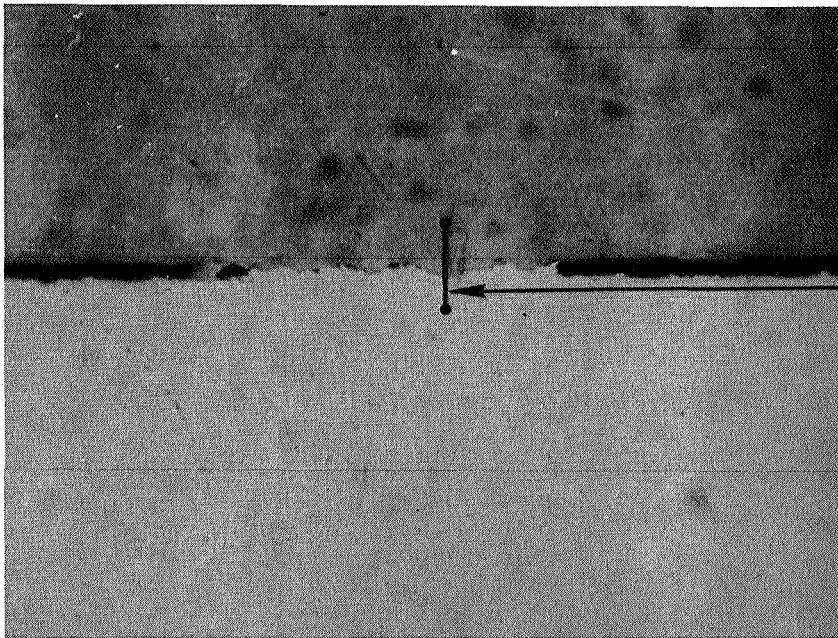
\* 25.4 microns = .001 inch

TABLE 13

EXTRAPOLATED THICKNESS OF DIFFUSION MODIFIED MATERIAL  
 ZONES AFTER TWO YEARS OPERATION

Interface	Thickness (microns)*	
	800°F	1500°F
Liner - TZM	-0-	-0-
TZM - (Pt-20Rh)	4	14
(Pt-20Rh) - PG	-	32
PG-SS	-	42

\* 25.4 microns = .001 inch

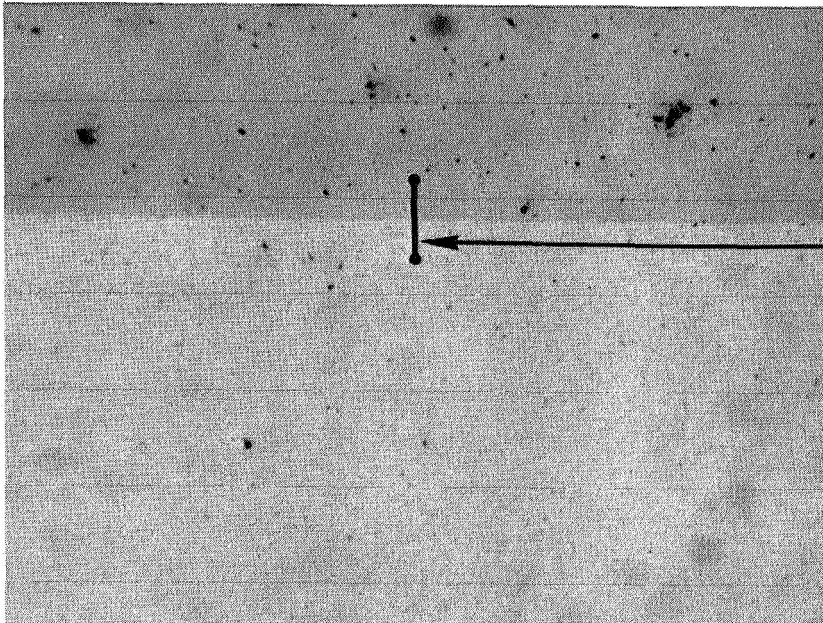


Liner

Location of  
Microprobe Scan

TZM

Fig. 36 Interface Between Liner and TZM After  
15 Days at 1500°F (400X magnification)

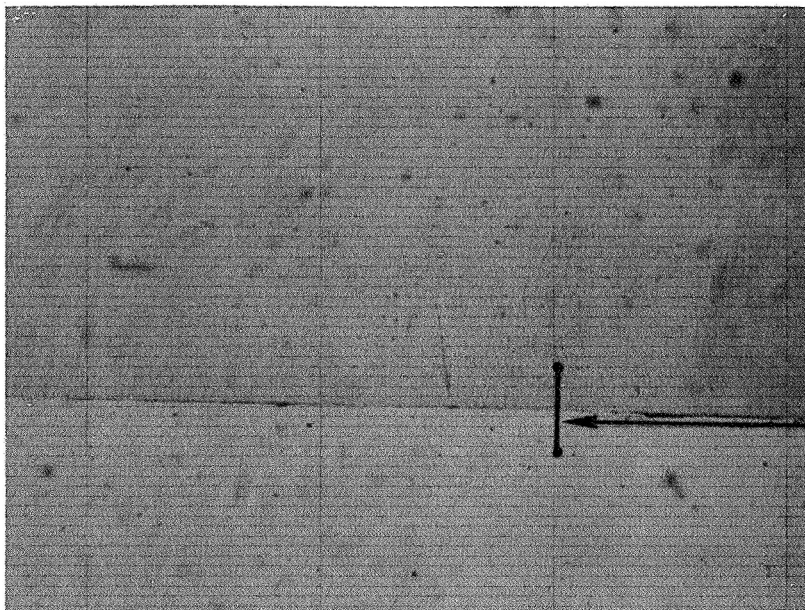


Liner

Location of  
Microprobe Scan

TZM

Fig. 37 Interface Between Liner and TZM After  
60 Days at 1500°F (400X magnification)

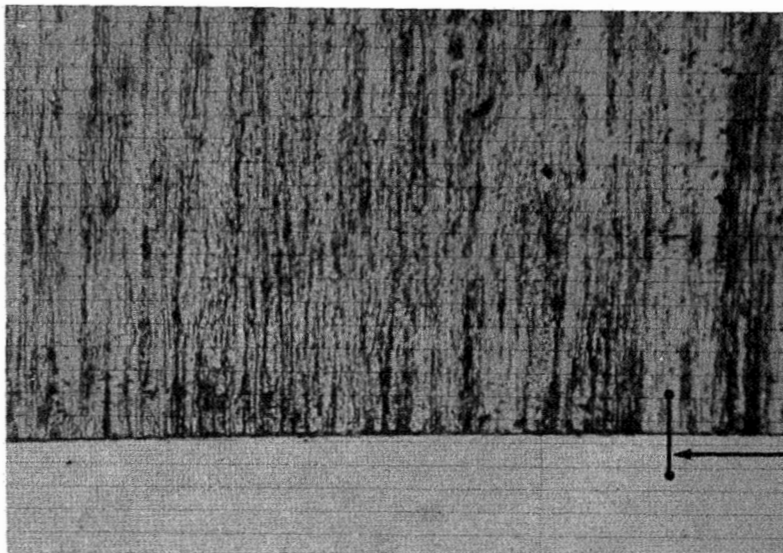


TZM

Location of  
Microprobe Scan

Pt-20Rh

Fig. 38 Interface Between TZM and Pt-20Rh After  
15 Days at 800°F (400X magnification)



TZM

(etched to show  
microstructure)

Location of  
Microprobe Scan

Pt-20Rh

Fig. 39 Interface Between TZM and Pt-20Rh After  
60 Days at 800°F (400X magnification)

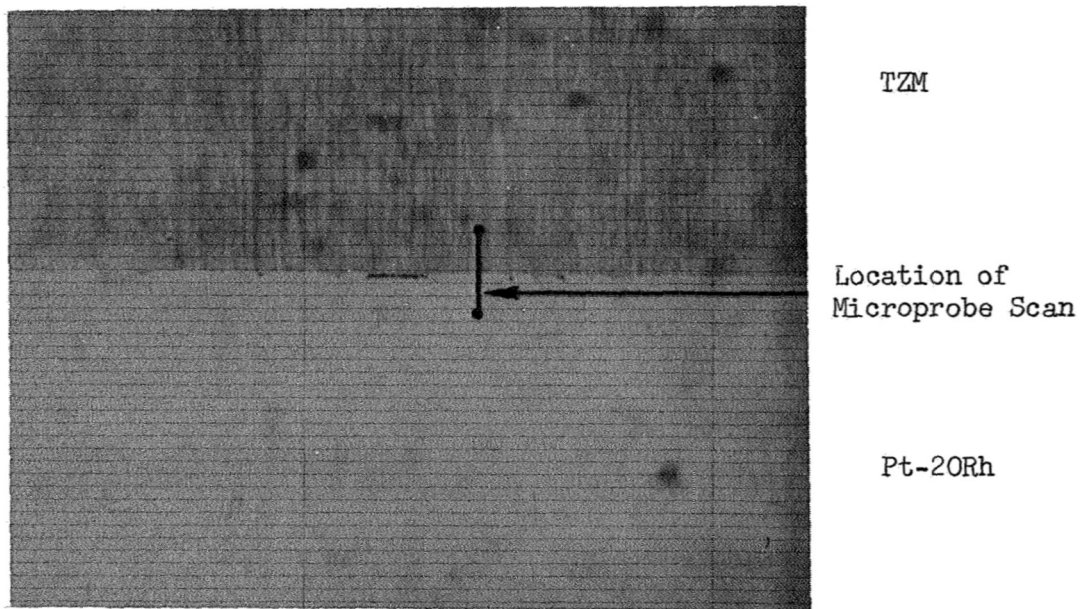
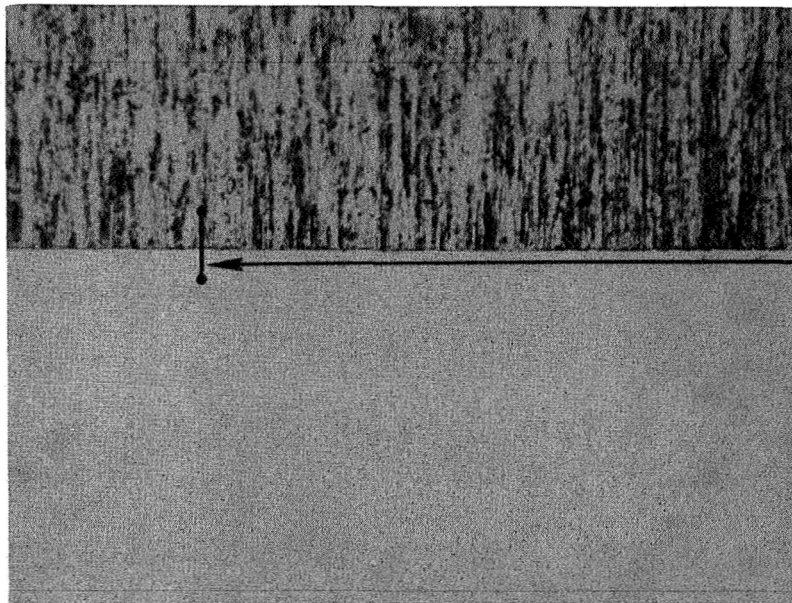


Fig. 40 Interface Between TZM and Pt-20Rh After  
15 Days at 1500°F (400X magnification)



TZM

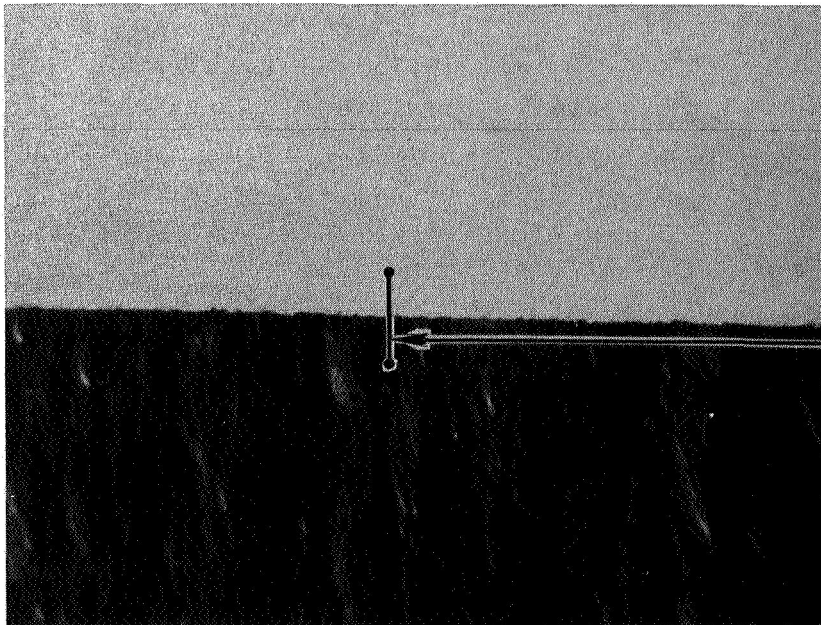
(etched to show  
microstructure)

Location of  
Microprobe Scans

Pt-20Rh

Fig. 41 Interface Between TZM and Pt-20Rh After  
60 Days at 1500°F (400X magnification)





Pt-20Rh

Location of  
Microprobe Scan

PG

Fig. 42 Interface Between Pt-20Rh and PG After  
15 Days at 800°F (400X magnification)

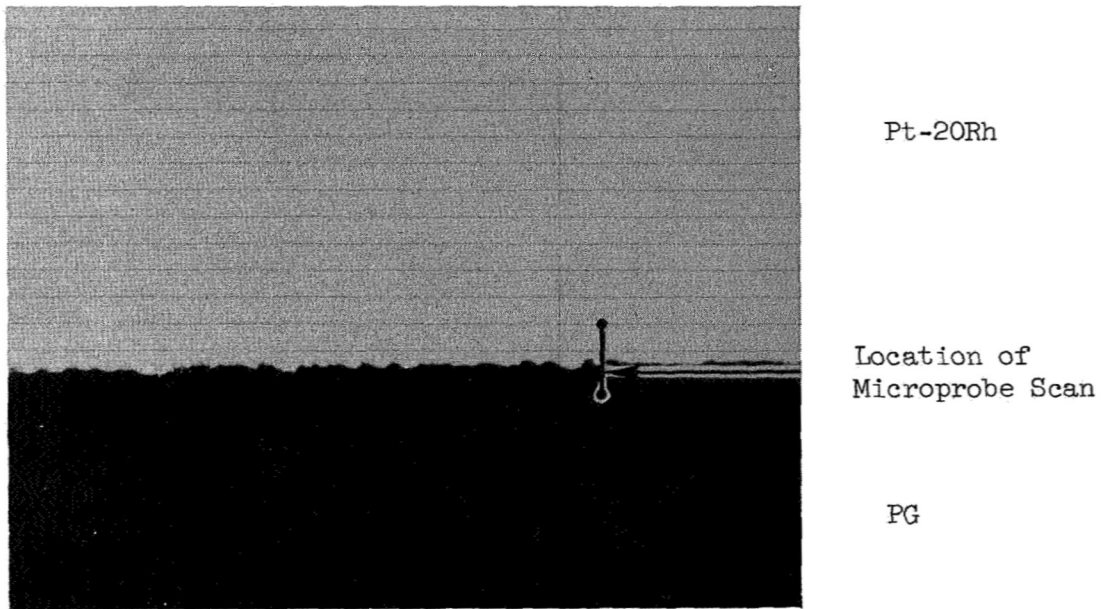
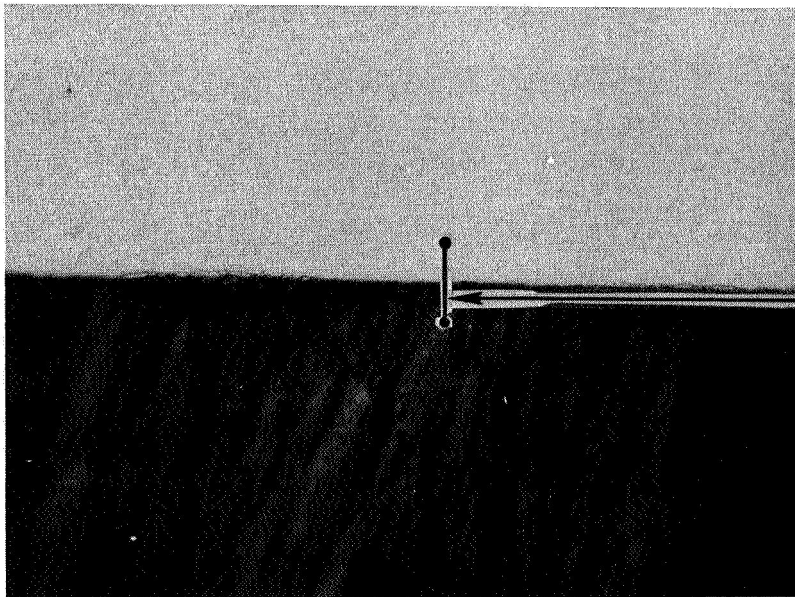


Fig. 43 Interface Between Pt-20Rh and PG After  
60 Days at 800°F (400X magnification)

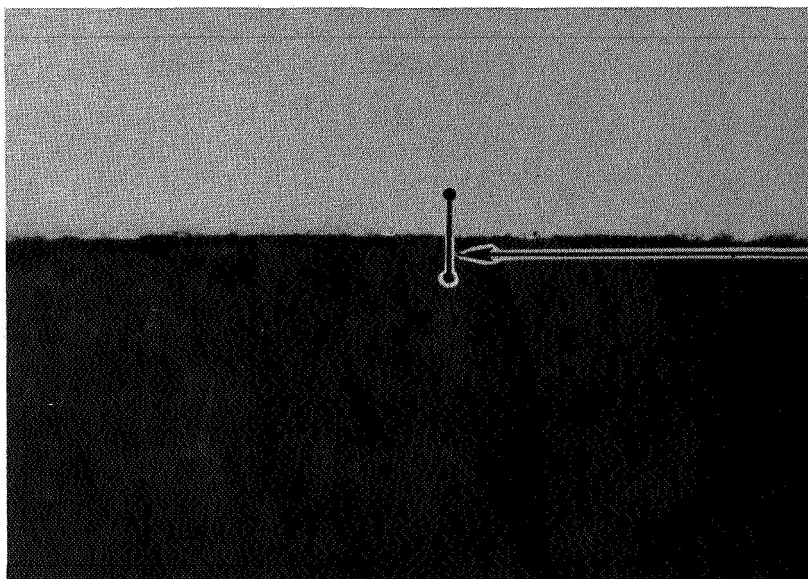


Pt-20Rh

Location of  
Microprobe Scan

PG

Fig. 44 Interface Between Pt-20Rh and PG After  
15 Days at 1500°F (400X magnification)

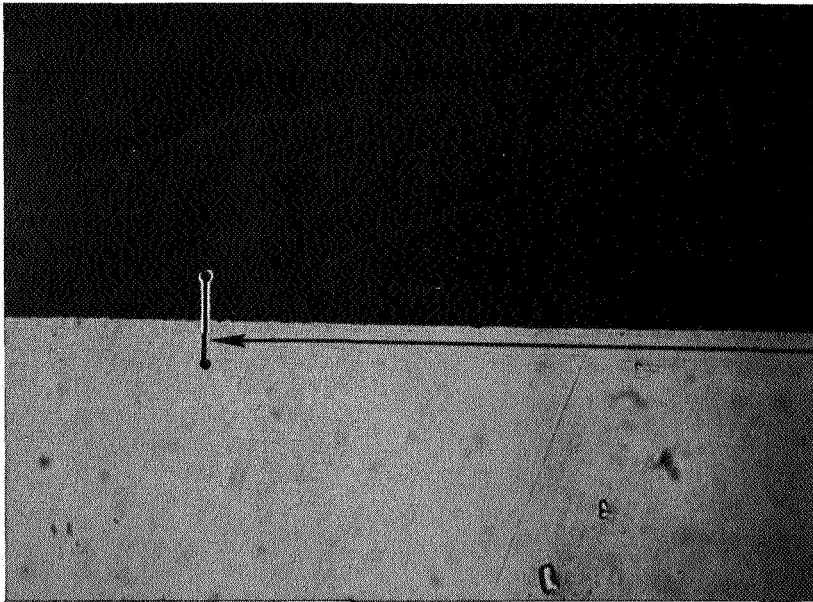


Pt-20Rh

Location of  
Microprobe Scan

PG

Fig. 45 Interface Between Pt-20Rh and PG After  
60 Days at 1500°F (400X magnification)

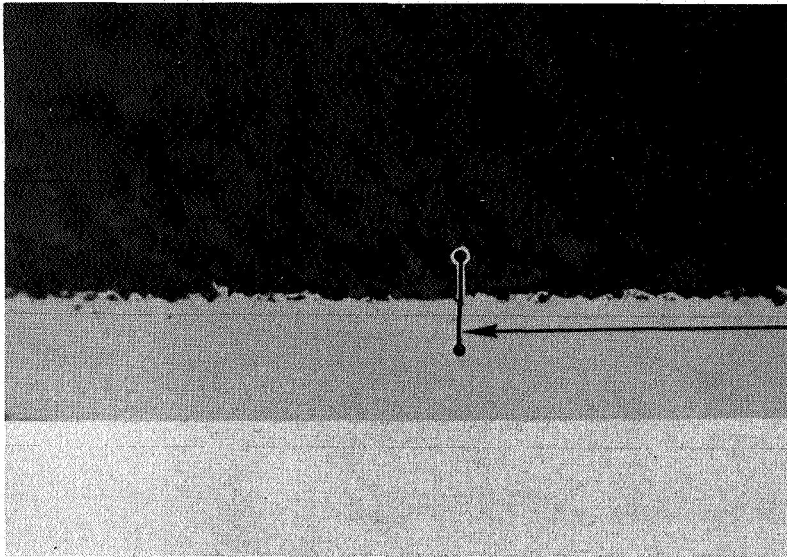


PG

Location of  
Microprobe Scan

Stainless Steel

Fig. 46 Interface Between PG and Stainless Steel  
After 15 Days at 800°F (400X magnification)

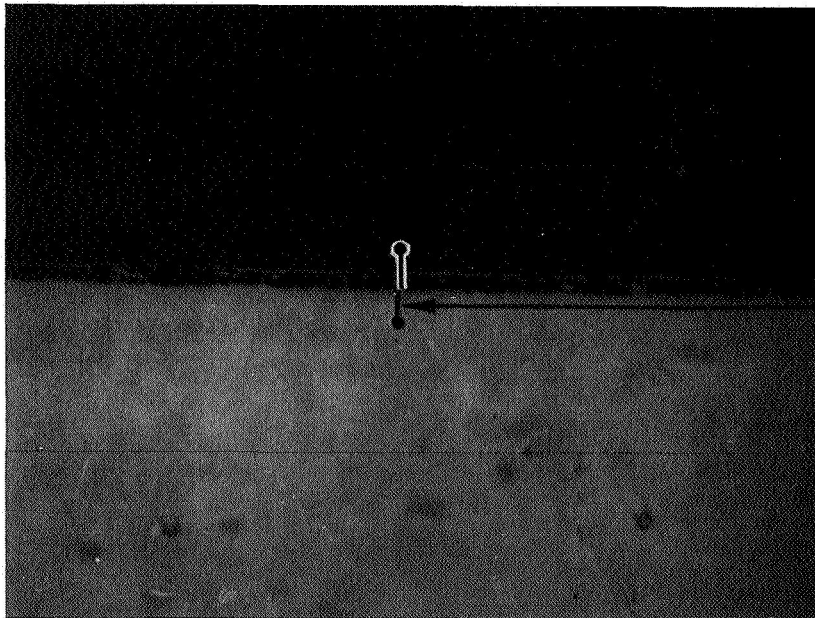


PG

Location of  
Microprobe Scan

Stainless Steel

Fig. 47 Interface Between PG and Stainless Steel  
After 60 Days at 800°F (400X magnification)

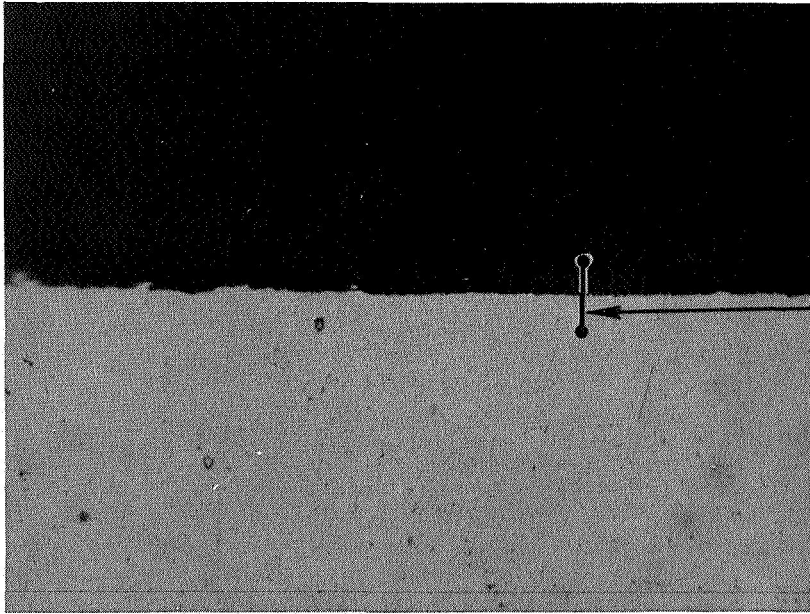


PG

Location of  
Microprobe Scan

Stainless Steel

Fig. 48 Interface Between PG and Stainless Steel  
After 15 Days at 1500°F (400X magnification)



PG

Location of  
Microprobe Scan

Stainless Steel

Fig. 49 Interface Between PG and Stainless Steel  
After 60 Days at 1500°F (400X magnification)



faces at 800°F could not be determined due to the lack of valid experimental data at this temperature. The expected total thickness of diffusion modified material at 800°F is in every case, however, considerably less than the corresponding predicted thickness at 1500°F.

The maximum predicted amount of interdiffusion at interfaces within the IHCOS capsule after 2 years operation at 1500°F occurs at the PG-stainless steel interface. The expected thickness of diffusion modified material is 42 microns. The major portion of the zone would be within the stainless steel and have little or no effect on thermal properties of the PG reentry member.

The (Pt-20Rh)-PG interface operating under the same conditions is expected to exhibit an interdiffusion zone 32 microns thick. This zone would form principally on the (Pt-20Rh) side of the interface and should be subtracted from the total cladding thickness in estimating the remaining useful thickness.

The expected interdiffusion zone thickness at the TZM-(Pt-20Rh) interface after operation under the above conditions is 14 microns. This would probably occur to approximately the same extent on either side of the interface, consuming about 7 microns each of the cladding and structural alloy.

No appreciable interdiffusion is expected to occur at the liner TZM interface. Furthermore, any diffusion that does take place will not alter the properties of either material to any significant extent.

Since predicted interdiffusion at 1500°F is not extensive and interdiffusion rates at 800°F are far less than those at the higher temperature, only the 1500°F data should be considered in specifying wall thickness of each material.

#### Pyrolytic Graphite - Stainless Steel Compatibility

The pyrolytic graphite, which provides reentry protection for the capsule assembly, will oxidize at a uniform predictable rate during reentry. A compositional or mechanical change in this material resulting from exposure to stainless steel at the reentry temperature could change its oxidation characteristics.

An experiment was conducted in order to investigate the chemical and mechanical effects of exposing pyrolytic graphite to stainless steel near or slightly above the eutectic temperature ( 2500°F) for short time periods.

A 1/2 inch diameter, 0.030 inch thick disc of type 321 stainless steel was placed on a 1/2 inch diameter, 0.050 inch thick disc of pyrolytic graphite and heated by induction in vacuum until evidence of liquid phase formation appeared. The sample was then held at constant temperature for 5 minutes and cooled. A second assembly was prepared and similarly heated to a temperature 10°F less than that which caused melting in the initial sample. This assembly was maintained for 5 minutes at temperatures and cooled. Both

samples were sectioned longitudinally and metallographically examined.

A photomicrograph of the sample that had melted is shown in Figure 50. Dimensional measurements before and after testing show that a layer of PG about 50 microns thick (not apparent in the photomicrograph) was consumed at the surface that had come in contact with the molten metal. In addition, several cracks developed in both the AB and C planes of the PG. This is presumed to be due to stresses induced upon cooling by differences in thermal expansion characteristics between the stainless steel and PG.

Should this condition occur, heat source integrity is maintained since the maximum capsule temperature will be about 2600°F, the melting point of type 321 stainless steel. If temperatures remain high enough during reentry to effect removal of the stainless steel, this condition is not detrimental to PG. The PG is not affected if the eutectic temperature is not attained during reentry and no liquid is formed.

A photomicrograph of a section through the PG disc that had been in contact with stainless steel below the eutectic temperature is shown in Figure 51 at 50X and in Figure 52 at 400X. The material shows no evidence of chemical interaction or mechanical degradation as a result of contact with the stainless steel.

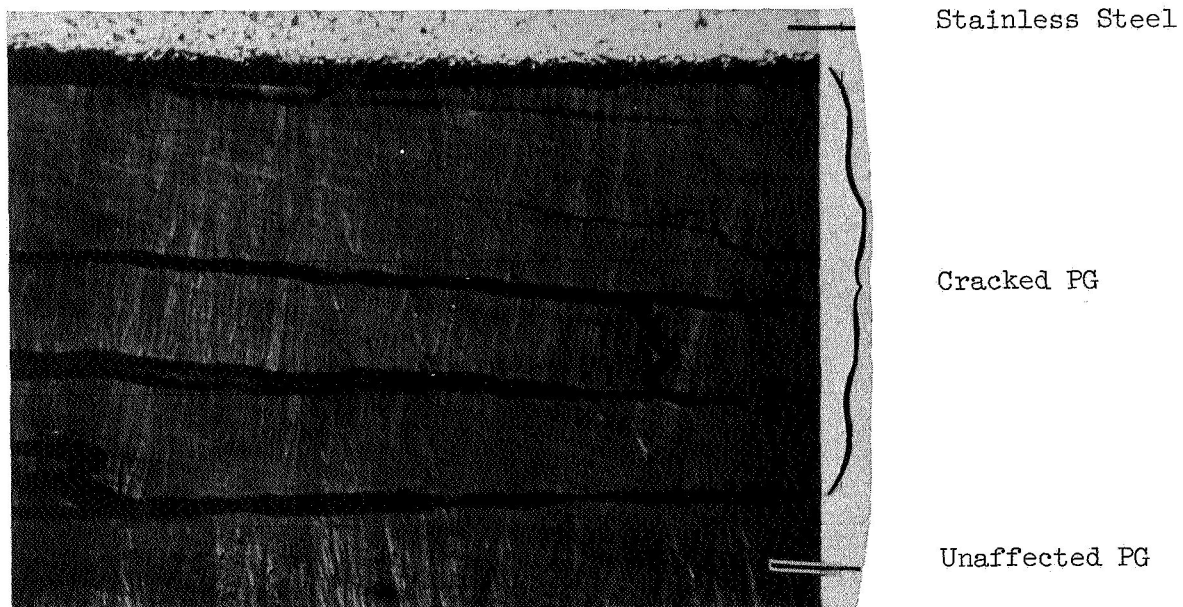
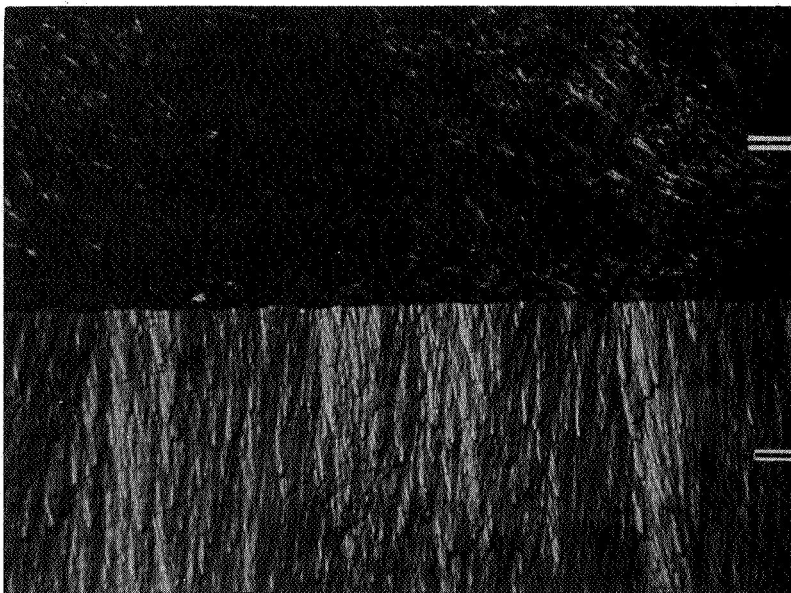


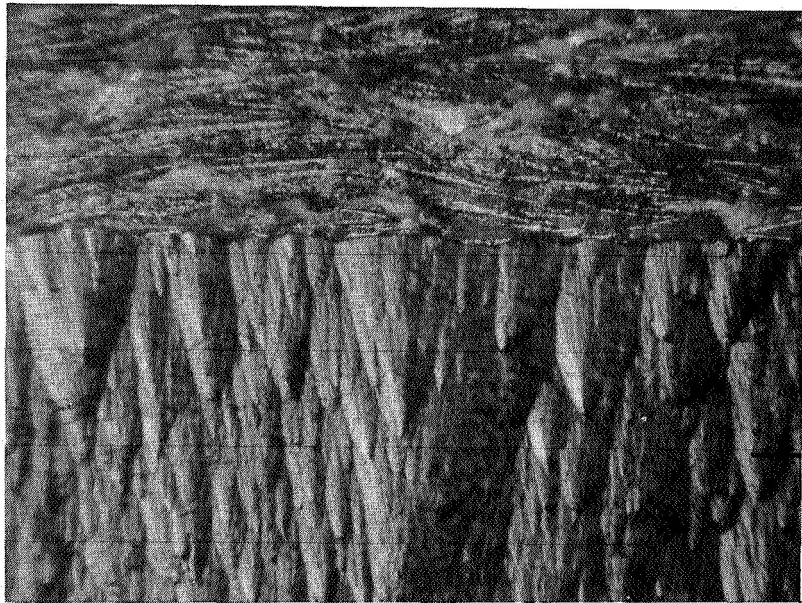
Fig. 50 Pyrolytic Graphite After Contact with Stainless Steel  
Above its Eutectic Temperature (50X magnification)



Mounting  
Material

PG

Fig. 51 Section through Pyrolytic Graphite Surface After Contact With Stainless Steel at 10°F Below its Eutectic Temperature (50X magnification)



Mounting  
Material

PG

Fig. 52 Section through Pyrolytic Graphite Surface After Contact With  
Stainless Steel at 10°F Below its Eutectic Temperature (400X magnification)



## HEATER DESIGN AND EVALUATION

For the electrically heated version of IHCOS, the heater unit will be located in the isotope fuel cavity within the liner. The heater design will duplicate the 123 watts of thermal power available from the radioisotope fuel.

Two heaters of each design were hermetically sealed in helium to simulate the capsule environment in the radioisotope fueled capsule. Helium is the end-product of the alpha particles emitted during the decay of the Pu-238 fuel form proposed for the IHCOS.

After 30 days of successful operation at a simulated liner wall temperature of 1500°F, one heater of each design was examined metallographically for post-test examination.

Temperature requirements.-Passive temperature control utilizing conventional insulation techniques is the selected approach to the IHCOS design. When passive control is used, heat losses are by thermal conduction through the insulation, and the minimum loss will be established by the 1000°F maximum temperature limit on the oxidizer surface during the no-flow depressurized cabin condition. This operational mode results in a temperature of 1500°F on the capsule liner surface.

Material requirements.-The characteristics of several high temperature heating elements were reviewed in order to select those best suited to the current application. Candidate materials include Pt, Pt-Rh, Nichrome V, and Kanthal. The criteria applied in the selection included high melting point, resistivity characteristics, availability, backlog of experience, and cost. Nichrome V and Kanthal A-1 have been used extensively by TRW Systems and are also widely used in industry. Nichrome V represents a highly developed heater material technology and appears to be the most suitable for this type application. This nickel-chromium alloy has the desired ductility and oxidation resistance while its coefficient of resistance results in an acceptable element length. Unlike refractory resistance elements, Nichrome V has a low coefficient of resistivity, as shown in Figure 53, resulting in a resistance increase of approximately 6 percent from 70°F to 1500°F. This electrical property eliminates initial high current surges which would occur during heater start up. High nickel alloys appear best suited for use in fabricating the heater leads.

The most applicable high temperature electrical insulating materials include magnesium oxide, beryllium oxide, and thorium oxide. Thorium oxide has superior insulating characteristics at extremely high temperature, but it

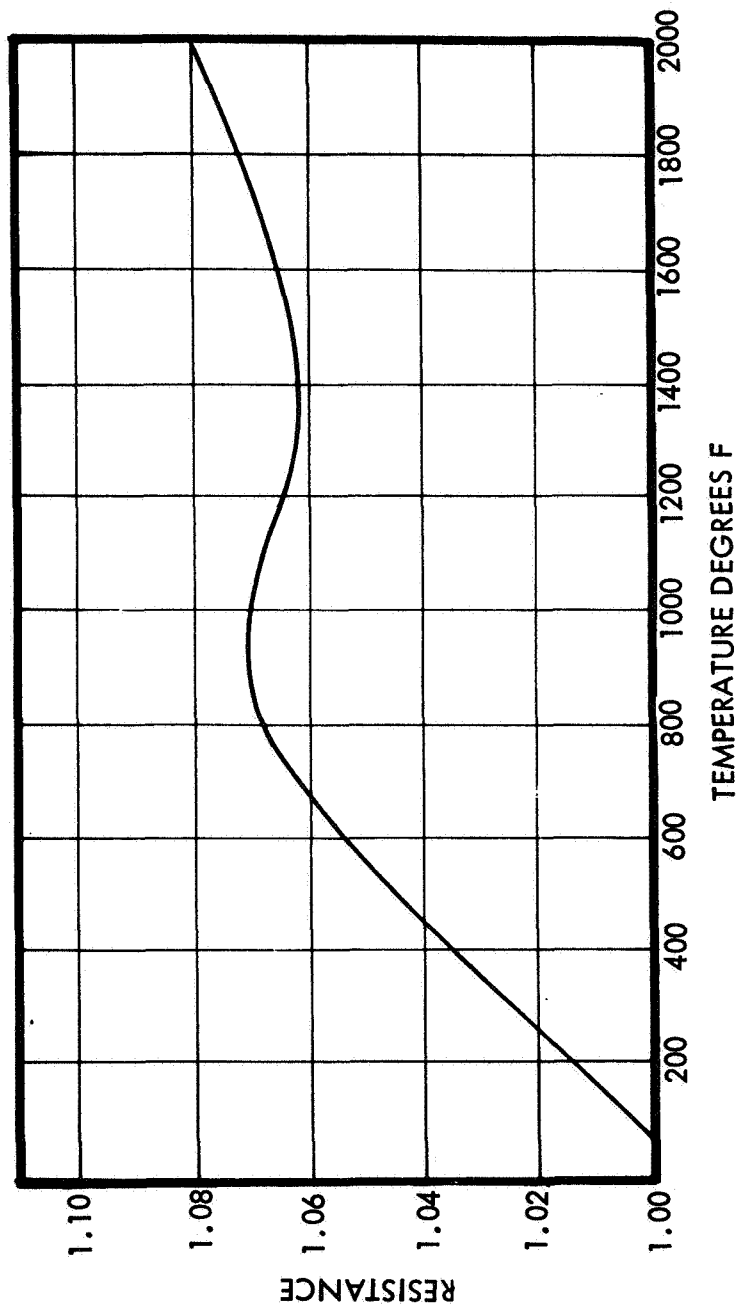


Fig. 53 Temperature Resistance Chart - Nichrome V



is rather expensive and mildly radioactive. Beryllium oxide and magnesium oxide both have excellent high temperature insulating properties but, due to availability, handling, and cost considerations, magnesium oxide has found wider application. Magnesium oxide has been extensively used by TRW in heater applications at temperatures in excess of 2000°F for long periods of time with excellent results.

Four materials, Inconel 600, Hastelloy X, Haynes 25 and T.D. Nickel were considered as candidates for the outer sheath assembly. All of these candidate materials have excellent strength and oxidation resistance at the IHCOS maximum operating temperature.

The most commonly employed heater sheath material is Inconel 600. This alloy has been used by TRW for heater applications at temperatures in the range of 1800° to 2100°F in air and vacuum environments with excellent results.

#### Candidate Heater Designs

Electrical heaters have been developed by TRW for both flight and ground applications at temperatures ranging from 1000°F to 4000°F. Many of these heaters were specifically designed to simulate the heat generated by a radioisotope fuel contained within a capsule assembly. Heaters in this category have been developed by TRW for the POODLE, DART, and SNAP 29 programs. A thorough review was performed in order to select the approach best suited to the resistively heated IHCOS design and operating requirements.

As a result of the above review, two design approaches were evaluated: one, involving a swaged cartridge element; a second based on forming a helical unit from a swaged sheathed element. Both designs are shown in Figure 54. Materials of construction chosen for both designs included Nichrome V for the resistance wire, magnesium oxide for the insulator, solid nickel lead wire, and Inconel 600 for the outer sheath. A description of each design is discussed below.

Cartridge heater.-The cartridge heater design involves winding the resistance wire on a ceramic core, carefully spacing each turn. Two nickel electrical leads are attached to the same end of the heater wires prior to swaging. The assembly is then surrounded with additional electrical insulation in powder form and swaged within a metallic tube which acts as an outer protective sheath. Thus, a rugged cylindrical heating element is produced with leads extending from one end of the assembly.

The cartridge heaters used in this task were fabricated by Rama Industrial Heater Company, San Jacinto, California. Each heater, 0.625 inch in diameter and 4.0 inches long, was wrapped with a 0.002-inch thick sheet of the capsule liner material to check compatibility of the liner and heater sheath. Interdiffusion between these two materials would be detrimental, if, during the test period, the extent were sufficient to affect the integrity of the heater. This subassembly was inserted into a stainless steel sleeve of

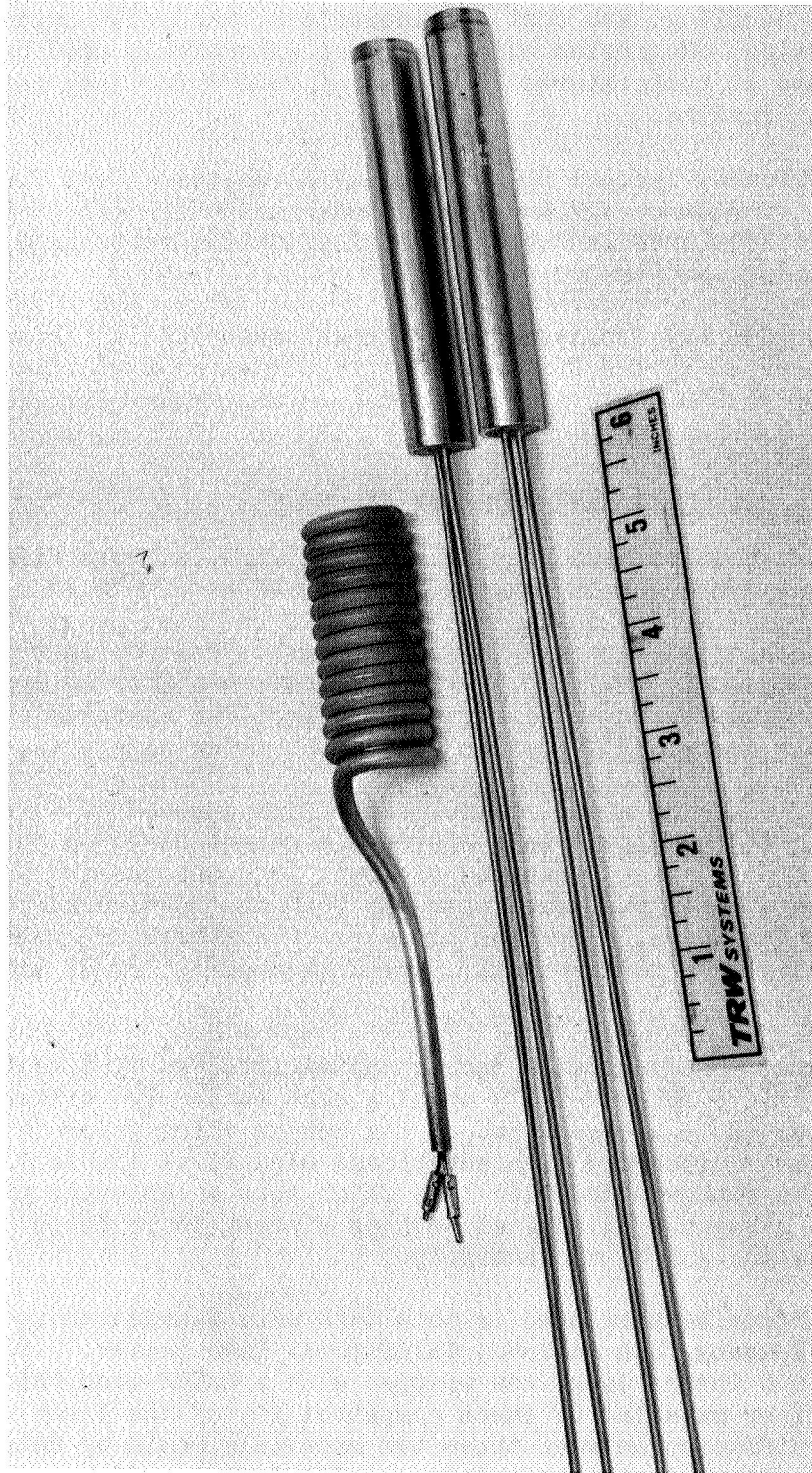


Fig. 54 Swaged Cartridge and Helical Sheathed Heaters

1.0 inch diameter. The unit was then purged and back-filled with helium gas prior to TIG welding the final closure between the heater sheath and sleeve, is shown in Figure 55.

Sheathed heater.-The sheathed design is similar to a sheathed thermocouple configuration. This type of element is made by threading refractory ceramic oxide insulators onto the resistance wires, and inserting the assembly into a metal sheath. A swaging operation reduces the diameter of the tube and compacts the insulation to produce a tough, integral assembly of wire, tube, and insulation. This relatively long, small-diameter unit can then be formed into a helical configuration with both heater leads attached to the same end.

The sheathed elements were fabricated by Semco Inc., North Hollywood, California. These elements, 3/16 inch in diameter, were formed on a 5/8 inch diameter mandrel resulting in a helical configuration with an outside diameter of 1.0 inch. The lead end was then positioned in line with the longitudinal axis of the developed heater. Each heater was wrapped with a 0.002 inch thick sheet of the capsule liner material to check compatibility of the liner and heater sheath. This subassembly, shown in Figure 56, was inserted into a stainless steel sleeve. The unit was then purged and back-filled with helium prior to TIG welding the final closure between the heater sheath and the stainless end-cap, as shown in Figure 57.

#### Heater Test Program

The prototype IHCOS electrical heaters were tested at TRW Systems. Components of the test facility included instrumentation used to monitor the electrical test parameters. A multi-channel recorder together with a millivolt potentiometer provided a redundant temperature measuring capability. Redundant chromel-alumel thermocouples were attached at each temperature measuring location. All heater assemblies were tested in air.

Each of the heater assemblies were wrapped with approximately six turns of 0.002 inch stainless steel thermal shielding which was dimpled to minimize thermal conduction losses between successive shields. A 1/2 inch thick layer of Refrasil\* was added to raise the temperature at the liner material - heater surface interface to the design temperature of 1500°F. In addition to this shielding, one of the cartridge heaters was inserted into a stainless steel structural module to simulate the IHCOS operational configuration.

Two heaters of each design were tested successfully for 30 days. At the end of this period, one heater of each design was removed from the test area for post-test examination. The remaining two heaters were tested at 1500°F for the remainder of the program. Table 14 presents test parameters recorded during these tests. The cartridge heater contained within the structural module is shown in Figures 58 and 59 and the sheathed helical heater assembly is shown in Figure 60.

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\*A high temperature SiO<sub>2</sub> thermal insulation manufactured by H. I. Thompson Co., Gardena, California

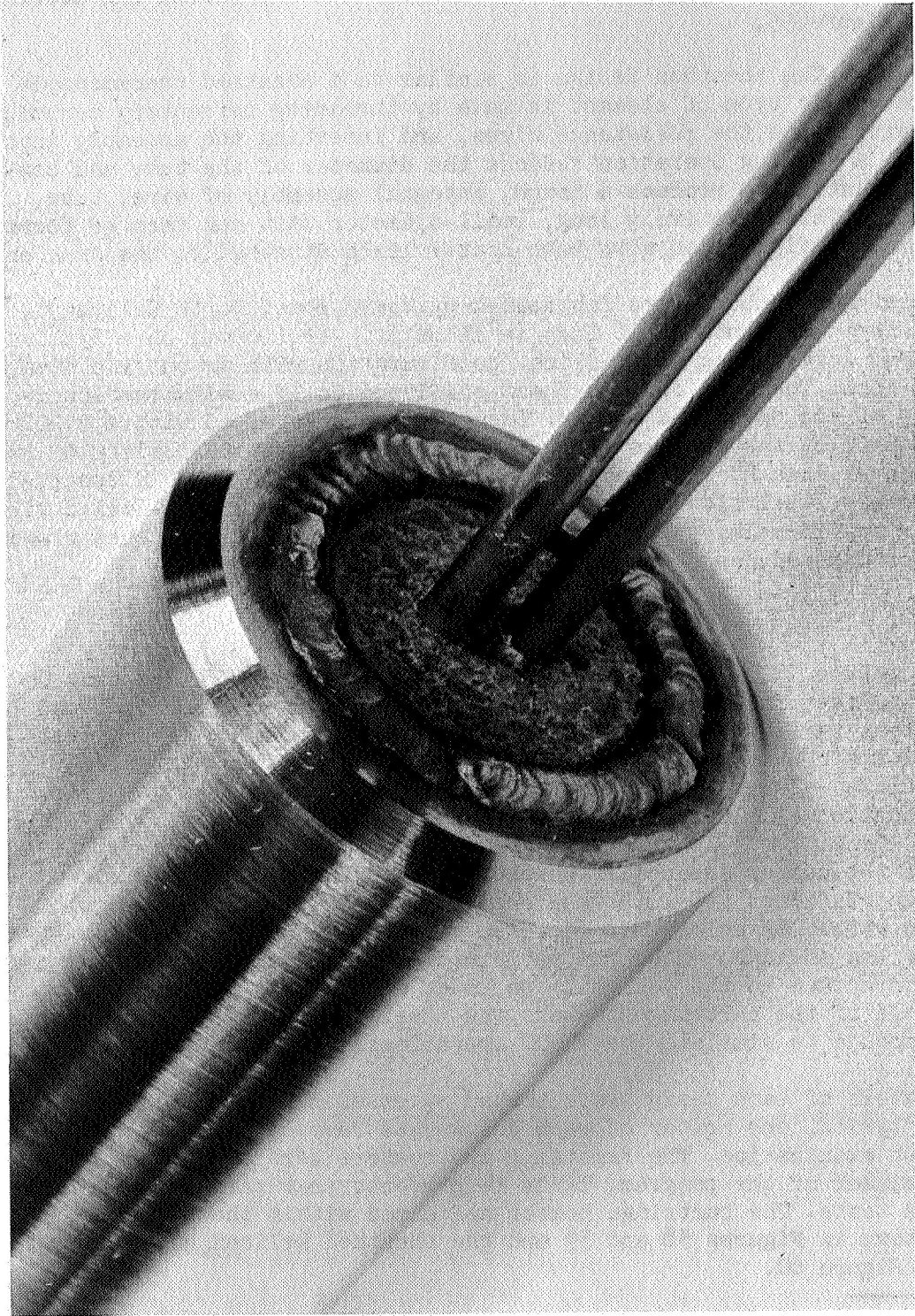


Fig. 55 Cartridge Heater Assembly

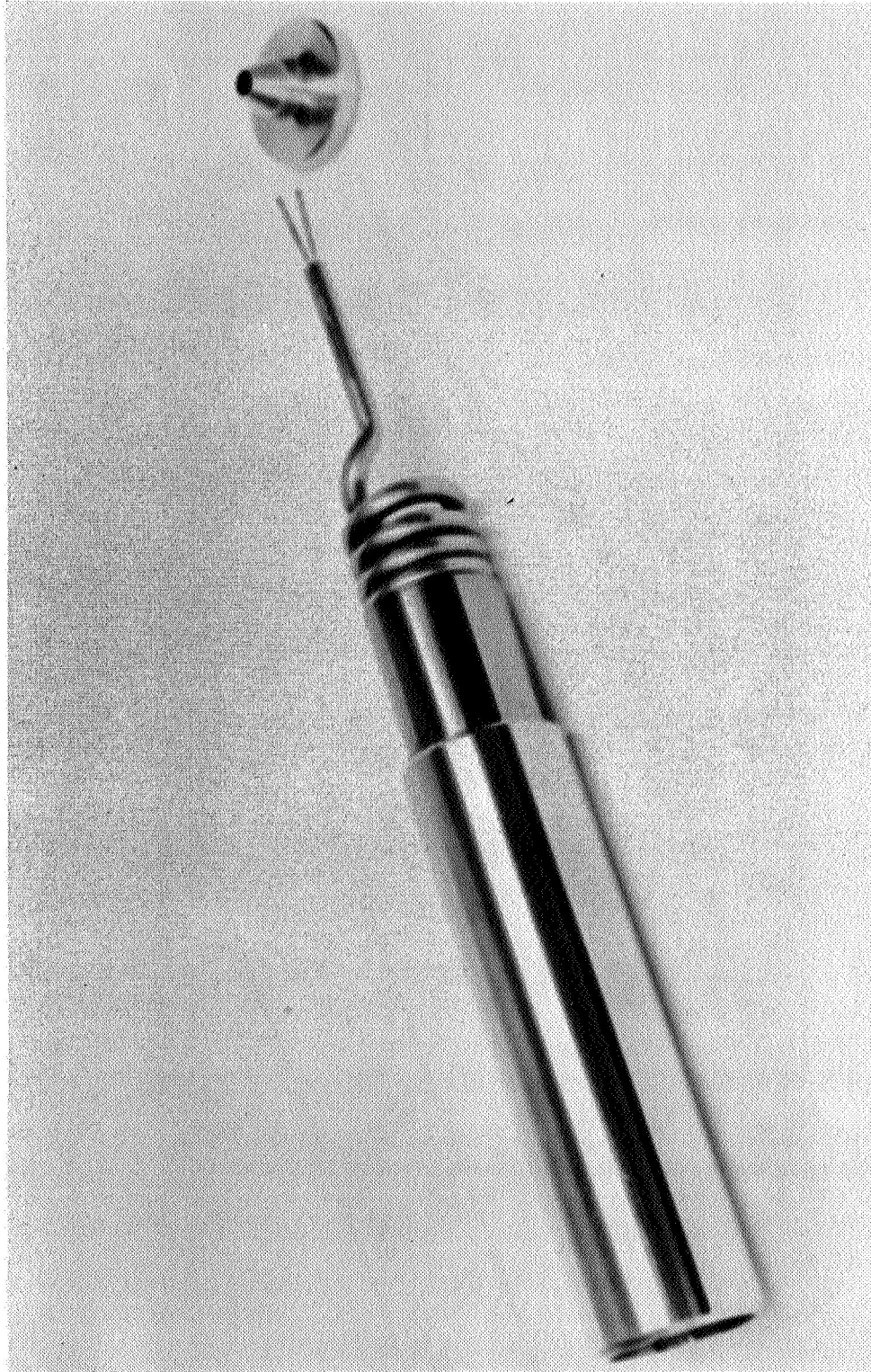


Fig. 56 Helical Sheathed Heater Subassembly

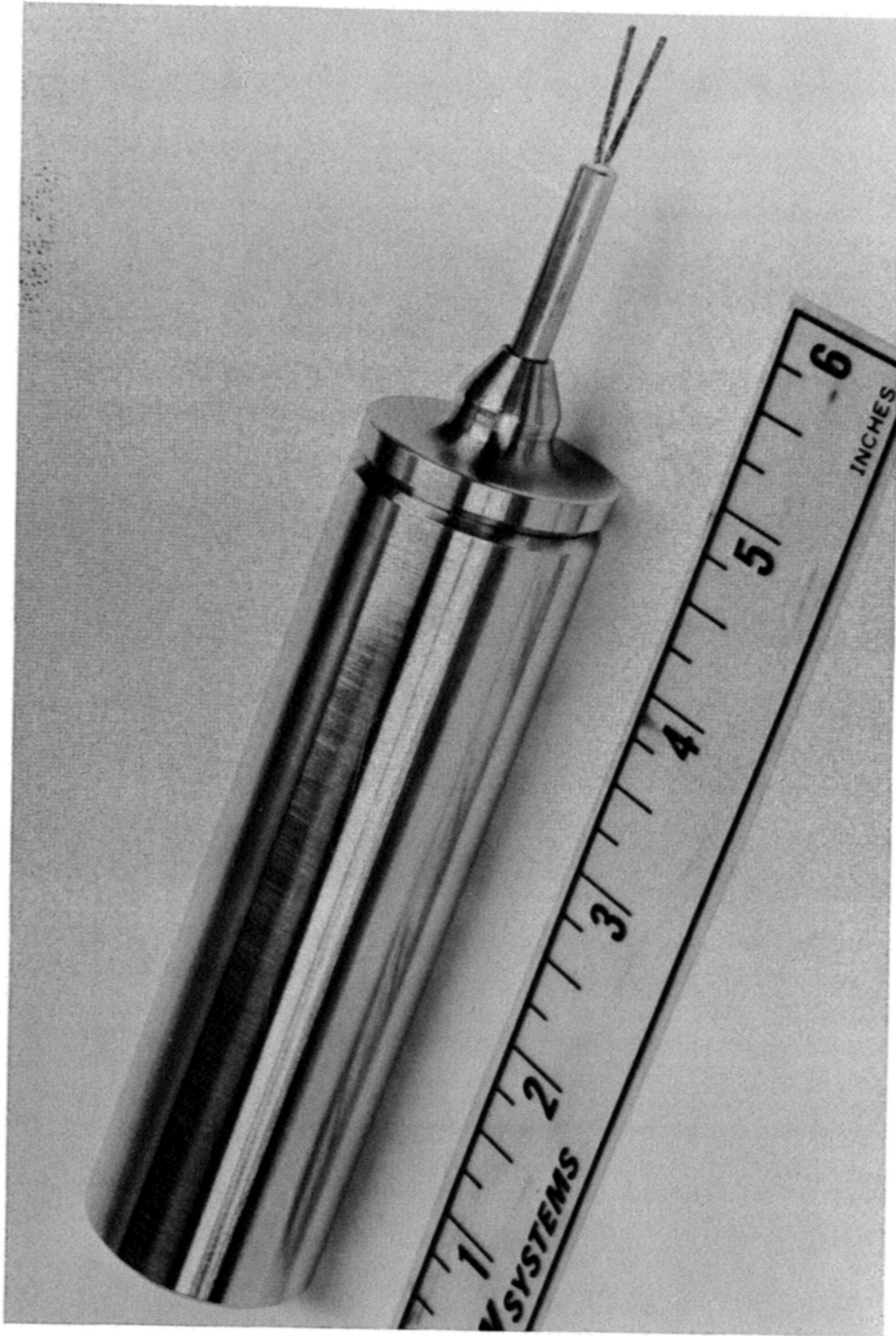


Fig. 57 Helical Sheathed Heater Assembly

TABLE 14

OPERATING EXPERIENCE-HEATER MATERIAL BUILD-UP

Heater Parameter	Units	Test Value and Heater Number (a)			
		S/N 1	S/N 2	S/N 3	S/N 4
Voltage	Volts	28.00	29.30	25.60	25.62
Current	Amperes	4.40	4.43	4.82	4.81
Resistance	Ohms	6.36	6.62	5.31	5.33
Power	Watts	123	130	123	123
Temperature	°F	1540	1500	1510	1520
Elapsed Testing Time	Hours	(b)	768	720	(b)
Heater Wire Type	-----	( ←	Nichrome V	→	(
Heater Wire Size	Inches	0.015	0.015	0.035	0.035
Insulation Material	-----	( ←	Magnesium Oxide	→	(
Sheath Material	-----	( ←	Inconel 600	→	(
Sheath Diameter	Inches	0.625	0.625	0.187	0.187
Lead Wire Material	-----	Ni	Ni	(c)	(c)
Lead Wire Size	Inches	0.090	0.090	0.035	0.035

NOTES: (a) Heaters S/N 1 and S/N 2 are the cartridge type;  
 S/N 3 and S/N 4 are the sheathed helical type;  
 Heaters S/N 2 and S/N 3 received post-test examination.  
 (b) Testing of these two heaters continuing.  
 (c) Same as heater wire type.

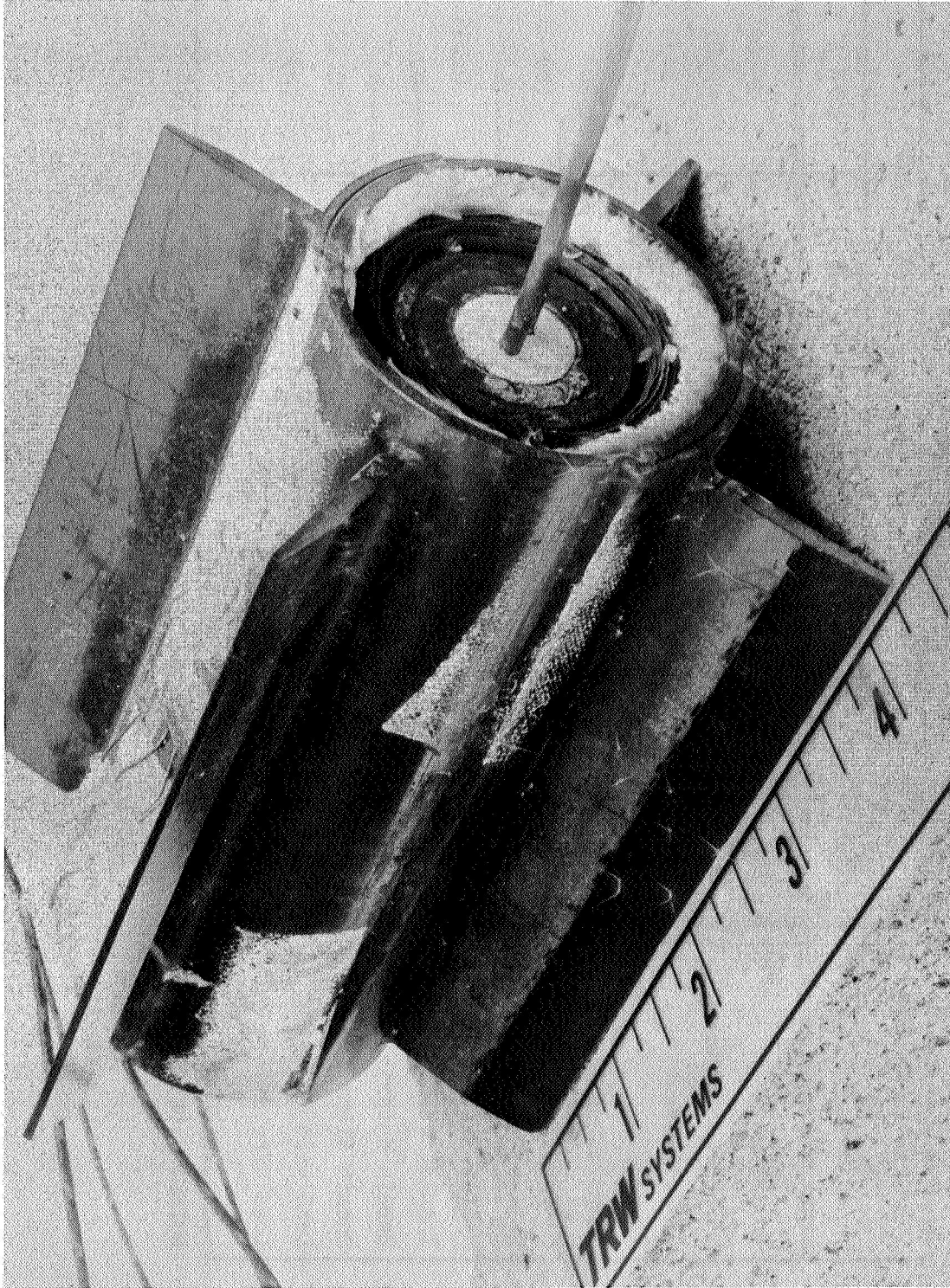


Fig. 58 Structural Module and Cartridge Heater Assembly  
(External insulation removed after test)





Fig. 59 Structural Module With Cartridge Heater Removed

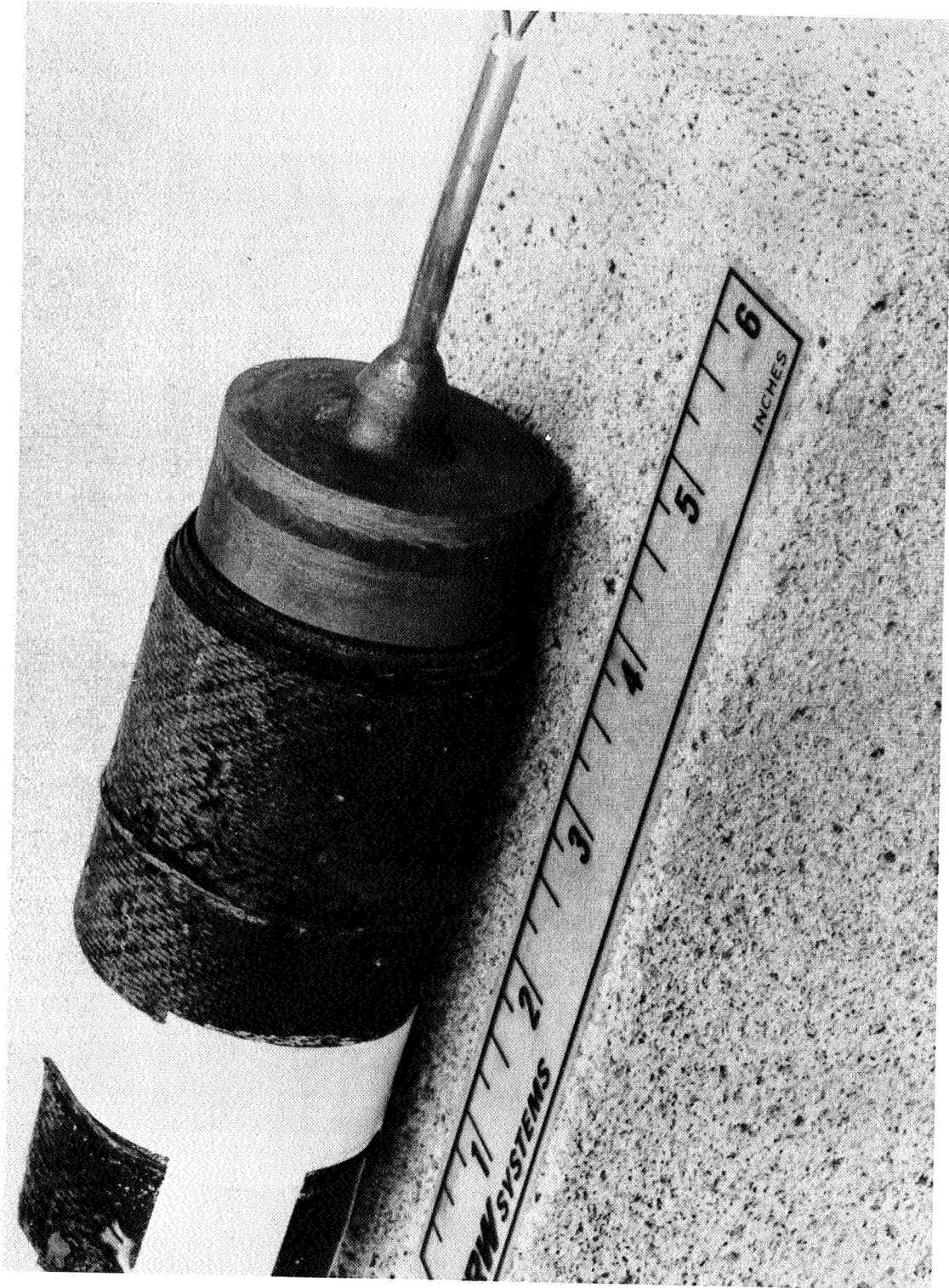


Fig. 60 Helical Sheathed Heater Assembly (Post Test)

Post-test heater examination.-At the conclusion of the 30-day test period, heaters S/N 2 and S/N 3 were sectioned and examined microscopically. The visible thickness of interdiffusion-modified material was measured with a metallograph equipped with a calibrated eye-piece. Thickness measurements were made at 400x magnification and were reproducible to within one micron (25.4 microns = 0.001 inch) at any location along the heater sheath-liner material interface. The average interdiffusion reaction zone thickness observed was approximately 0.2 mil, with a maximum of 0.8 mil after 720 hours at 1500°F. A typical reaction zone is shown in Figure 61.

### Heater Selection

The experimentation conducted during this task showed that both heaters performed satisfactorily. In addition, design techniques employed to tailor the helical sheathed heater configuration required for fabrication of an electrically-heated IHCOS were successful.

Two heater designs were tested in a simulated capsule environment for 30 days at 1500°F. An additional heater of each type was tested for 60 days at 1500°F. No deleterious effects were observed. Heaters of similar design have been successfully tested at TRW Systems for time periods in excess of 8,000 hours at 1800°F. Since a normal operational temperature in the application to the IHCOS is only 800°F, the tests performed provide a valid basis for selecting a heater that will meet the 180-day heater life requirement.

The interdiffusion rate between the liner material and the Inconel 600 heater sheath material was sufficiently low so that no detrimental effects would be expected in a heat source designed for 180-day mission. In addition, the proposed 0.020 inch wall thickness for both liner and heater sheath materials is sufficient to warrant higher temperatures for considerably longer periods of time.

Operation of both the heater and liner materials in a hermetically sealed helium environment resulted in bright and clean surfaces on these components. The lack of an oxidizing atmosphere in this simulated fuel volume was effective in maintaining the integrity of the liner material which would otherwise oxidize catastrophically.

After reviewing the test data, the sheathed helical heater unit was selected for use in the IHCOS. The reasons for this selection were:

- o The simulation of the isotope fuel weight can be more readily accomplished with this geometry.
- o The closure of the exiting leads from the structural module can be best accomplished with this heater.

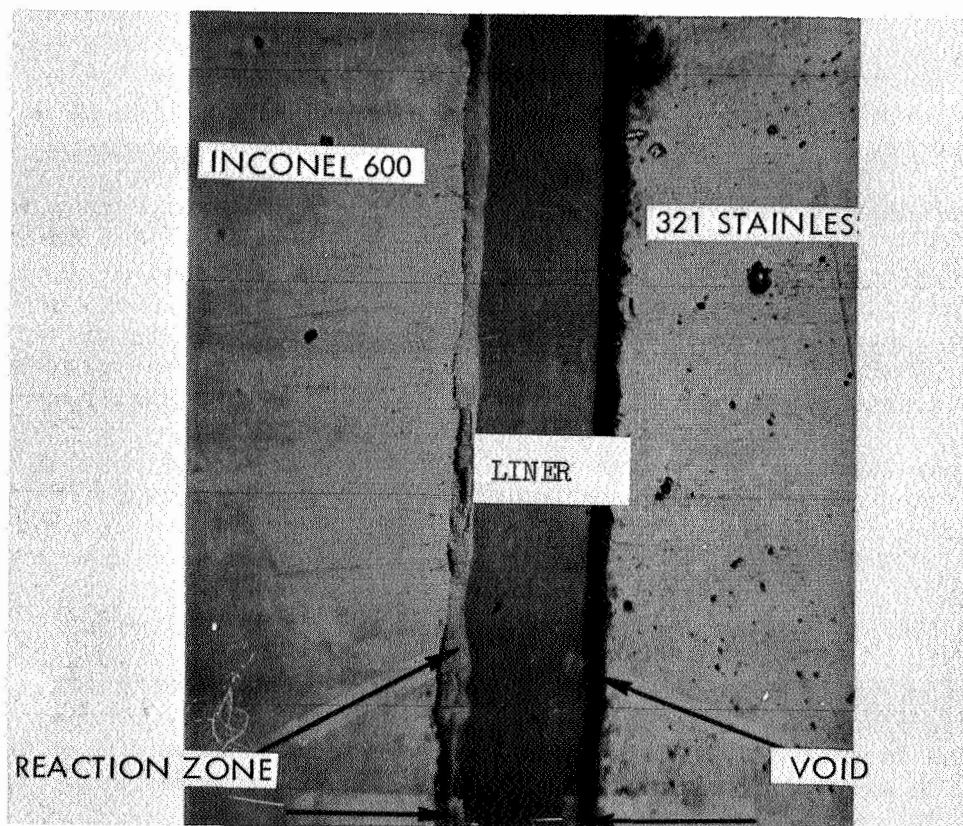


Fig. 61 Photomicrograph of Interdiffusion Zone Between Inconel 600 and Liner (400X). Tested at 1500°F for 768 Hours in a Helium Environment

## INSULATION EVALUATION

Long-term testing of the two candidate insulation concepts (vacuum and filled type) was conducted to determine which insulation type is best suited for use in IHCOS. This section presents the objectives, apparatus and procedures used, the results obtained, and a discussion of the results.

### Objective

The objective of this test was to evaluate the integrity and thermal insulation characteristics of the vacuum type insulation and the Johns Manville Min-K 1301 insulation. These test results were to be used to establish whether the IHCOS insulation shall be a vacuum or filled insulation type.

### Apparatus

The test apparatus used to obtain the performance data is shown schematically in Figure 62 and pictorially in Figure 63. The major items of equipment included:

- Heater
- Heater Power Supply
- Ammeter
- Voltmeter
- Iron Constantan Thermocouples
- Temperature Recorder
- Potentiometer
- Test Insulations

Filled insulation.-The filled insulation configuration that was tested is shown in Figure 64 and represents a simplified, but thermally accurate, version of the configuration that would be used in the final design. The simplification in geometry was in using cylindrical bored sections as opposed to a more complex and expensive molded geometry. The insulation thickness on the hot end and around the cavity that represented the catalyst canister was 1.5 inches. The thermal model was composed of six (6) pieces of insulation 7.6 inches in diameter.

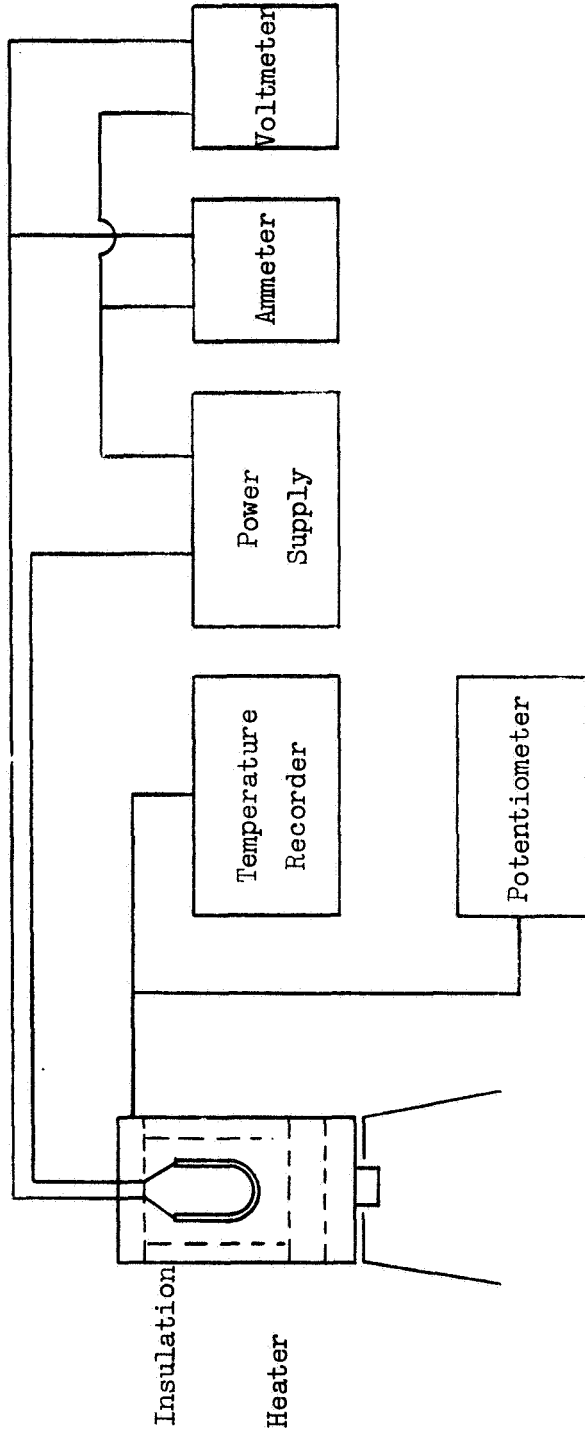


Fig. 62 Schematic of Insulation Evaluation Test Apparatus

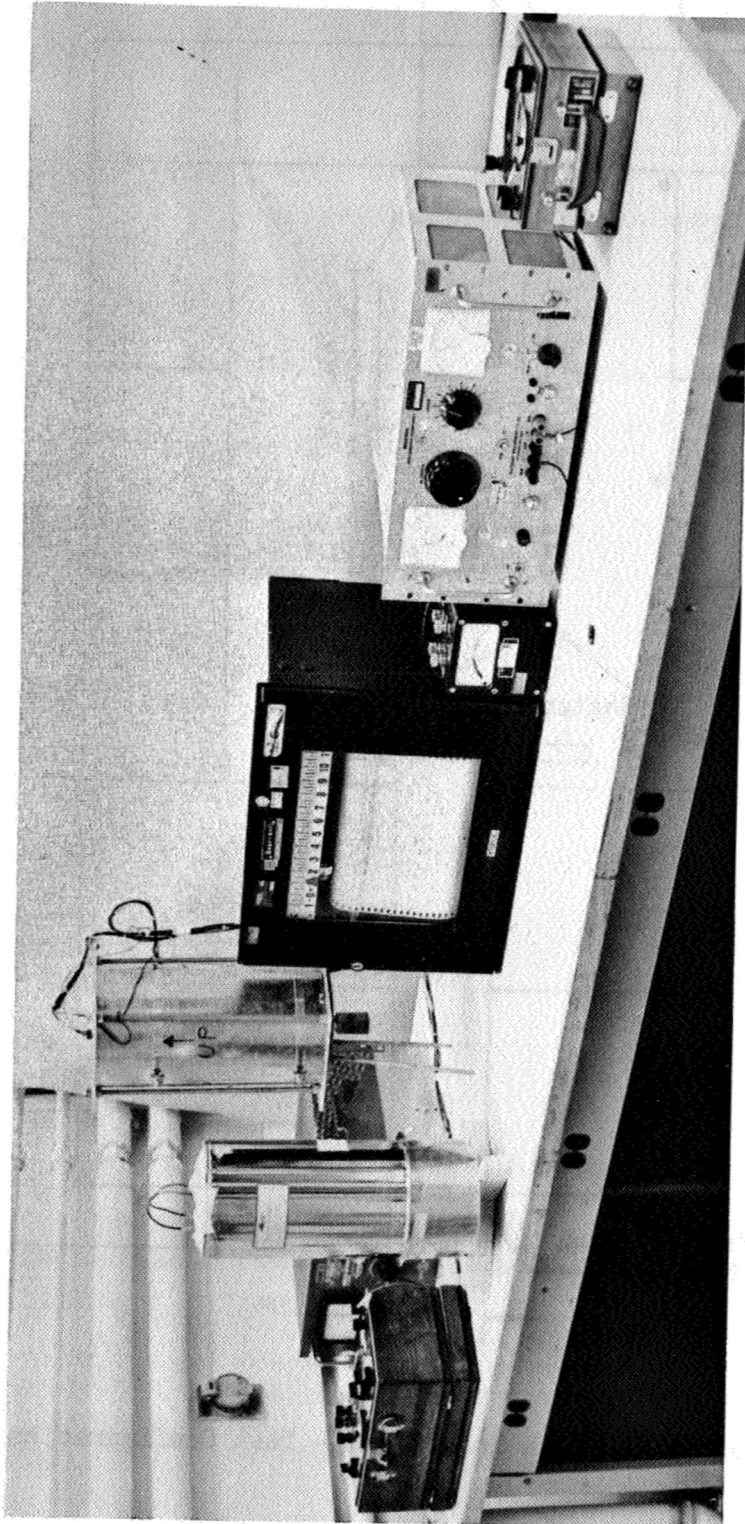


Fig. 63 Insulation Evaluation Test Apparatus

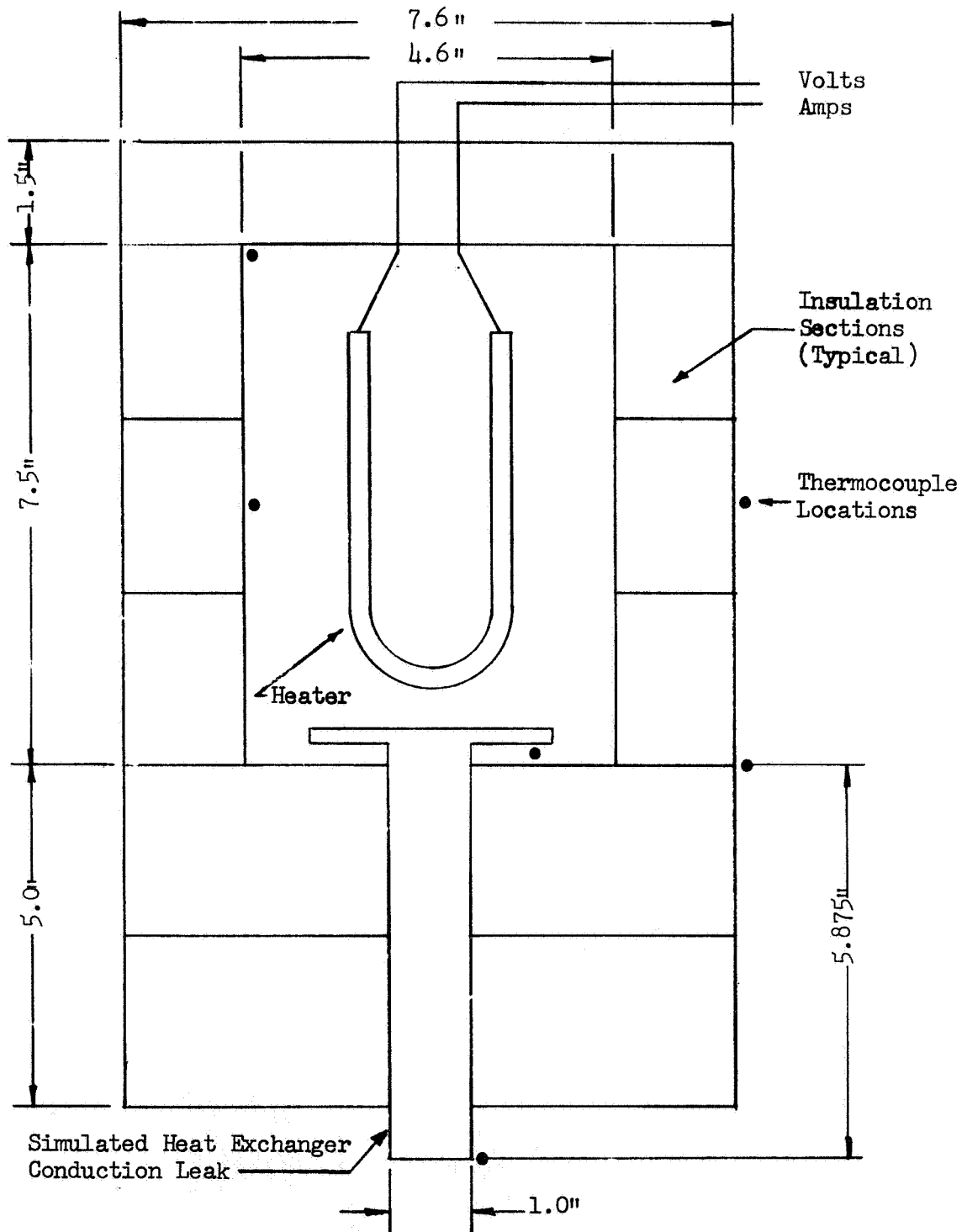


Fig. 64 Johns Manville Insulation - Test Configuration



These were:

- A solid end-cap, 1.5 inches thick.
- Three 2.5-inch thick sections with a 4.6-inch diameter hole in the center that simulated the catalyst canister cavity.
- Two 2.5-inch thick sections for the heat exchanger end with a 1.6-inch hole in the center to allow the simulated heat exchanger to pass through.

These pieces of insulation were wrapped with an aluminum outer shell and clamped firmly between two aluminum plates to prevent heat losses through the separation lines. The inner cavity had a stainless steel liner to hold the thermocouples in place. A heater rated at 200 watts was placed in the cavity and was connected with 0.050-inch nickel lead wires similar to those that will be used in the final design. A steel bar with the same cross-sectional area and length as the heat exchanger was used to simulate the heat exchanger conduction losses. The bar had a 4-inch diameter disk welded to the hot end to assure that it would be at the cavity temperature.

Vacuum insulation.-The vacuum insulation canister was manufactured by Gardner Cryogenics Co. and is shown in Figure 65. The insulation canister was fabricated from 0.04-inch stainless steel with the interior surfaces coated with gold to produce an emissivity of 0.07. The vacuum canister was evacuated to a pressure of less than  $10^{-5}$  mm/Hg. After several days of evacuation with the canister at an elevated temperature, the canister was sealed. The configuration of the canister was identical to the configuration proposed for the final design.

#### Procedure

In operation of the IHCOS, two temperature levels are of particular importance. These are 680°F which is the normal operating temperature and 1000°F which is the maximum temperature during the emergency shutdown condition. A third temperature level, 1200°F, is of interest for the filled insulation since it should be cured at this temperature to outgas possible contaminants in the binder material. However, above this temperature, an insulation additive which limits radiation begins to change its characteristic and can cause a degradation in insulation effectiveness. The majority of the testing with the filled insulation was conducted at these three temperature levels.

The simulated heat exchanger conduction leak was installed in the unit after 66 days of testing.

Testing of the vacuum insulation was first conducted at the power levels anticipated for the insulation losses at the normal and full power conditions to determine what temperature would result. Tests were then run to determine the insulation power losses at the normal operating temperature of 680°F.

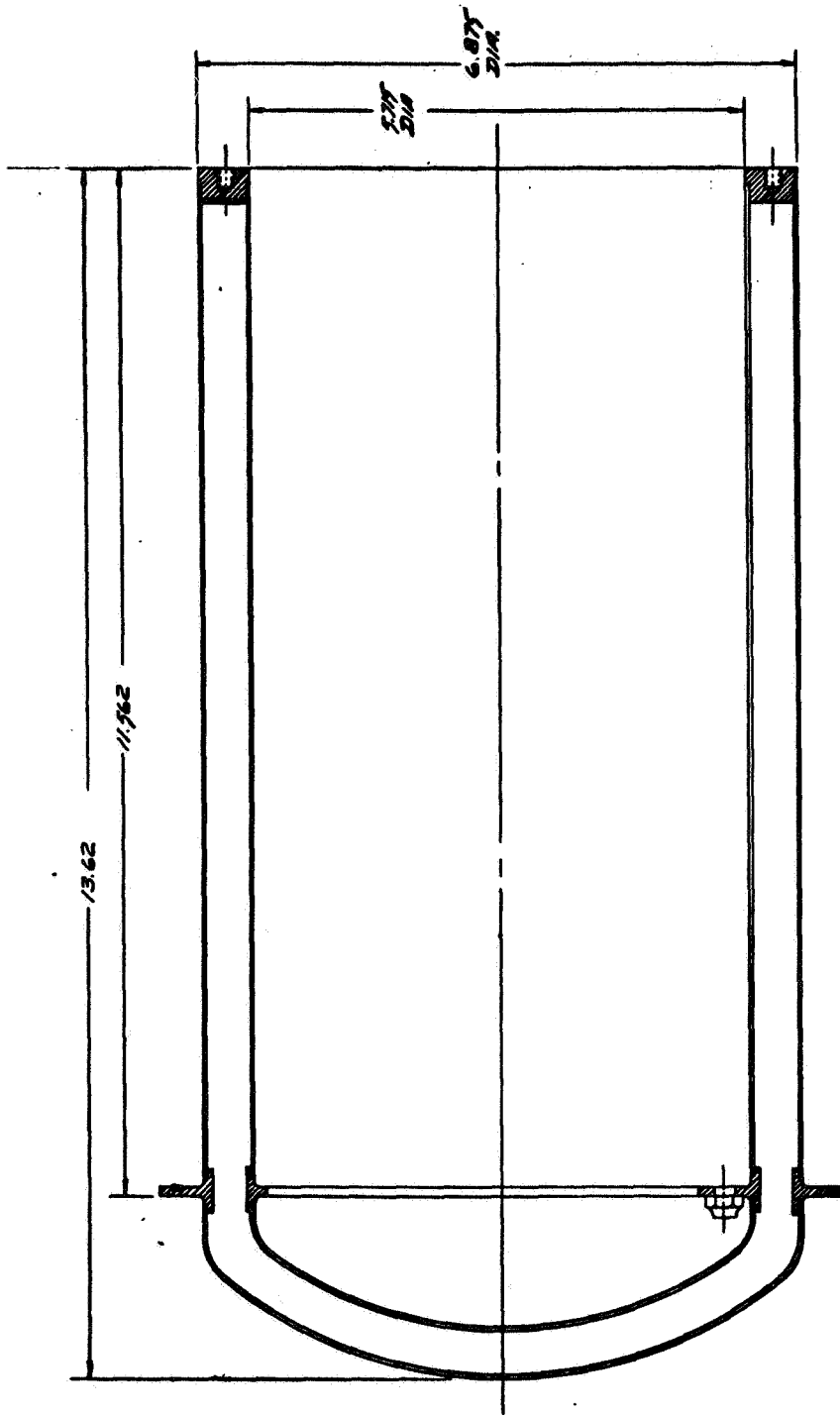


Fig. 65 Vacuum Insulation Canister

After 24 days of testing with the vacuum canister, the vacuum pinch off was opened and the canister was connected to a vacuum system and reevacuated. The vacuum system was also used to maintain the canister vacuum for the remainder of the test.

### Results

Filled insulation.-The filled insulation was tested for 180 days during which time the power level was measured for operating temperatures of approximately 680°F, 1000°F and 1200°F. These test results are shown in Figure 66. For the first thirty days of testing, the power was held at approximately 46 watts during which time the temperature remained constant at about 700°F. After this period, the power was increased approximately 70 watts and the temperature increased to slightly above 1000°F. The power was off from the 47th to the 51st day due to a heater lead failure. After the heater lead was repaired the 70 watt and 46-watt conditions were repeated to verify that the same temperatures resulted. On the 66th day, the power was turned off and the simulated heat exchanger conduction leak was installed. The unit was then cycled between the low temperature and high temperature conditions to establish these power levels which were 57 watts and 94 watts respectively. On the 104th day of testing, the power was increased to bring the internal temperature up to the recommended cure temperature of approximately 1200°F. The power was left at this level until the 118th day at which time it was returned to 57 watts to determine if any degradation had occurred due to the exposure to 1200°F. The power was left at 57 watts for the remainder of the test during which time the temperature remained constant at approximately 700°F.

Vacuum insulation.-The performance results of the vacuum insulation canister evacuation are shown in Fig. 67. This unit was initially tested at 60 watts which was the power anticipated to produce an internal temperature of 680°F. Since the temperature level was less than 680°F, the power was increased to the full power condition of 123 watts to see what temperature would result. The temperature started to increase until it reached 700°F at which time the temperature abruptly started to decrease indicating a sudden loss in performance. The power was left at 123 watts until the 24th day during which time the temperature continued to drop. On the 25th day, the power was reduced again to 60 watts and the resultant temperature was 370°F. The test was terminated because of the reduced performance of the unit and the canister was reevacuated and put back on line at 60 watts. The internal temperature returned to 600°F indicating that an increase in pressure due to a leak or outgassing caused the loss of performance. After several days at this condition, the power level was again increased to 123 watts and this time the temperature rose to 800°F and remained there. On the 45th day, the 60-watt condition was rechecked and then the power was adjusted to 85 watts to reach an internal temperature of 680°F. On the 52nd day, the test was stopped and the vacuum canister was opened to inspect the condition of the gold surfaces. The outer gold surface on the low temperature wall appeared normal with a bright gold color. The high temperature or inner canister was mottled in appearance with the bulk of the gold surface contaminated with a black

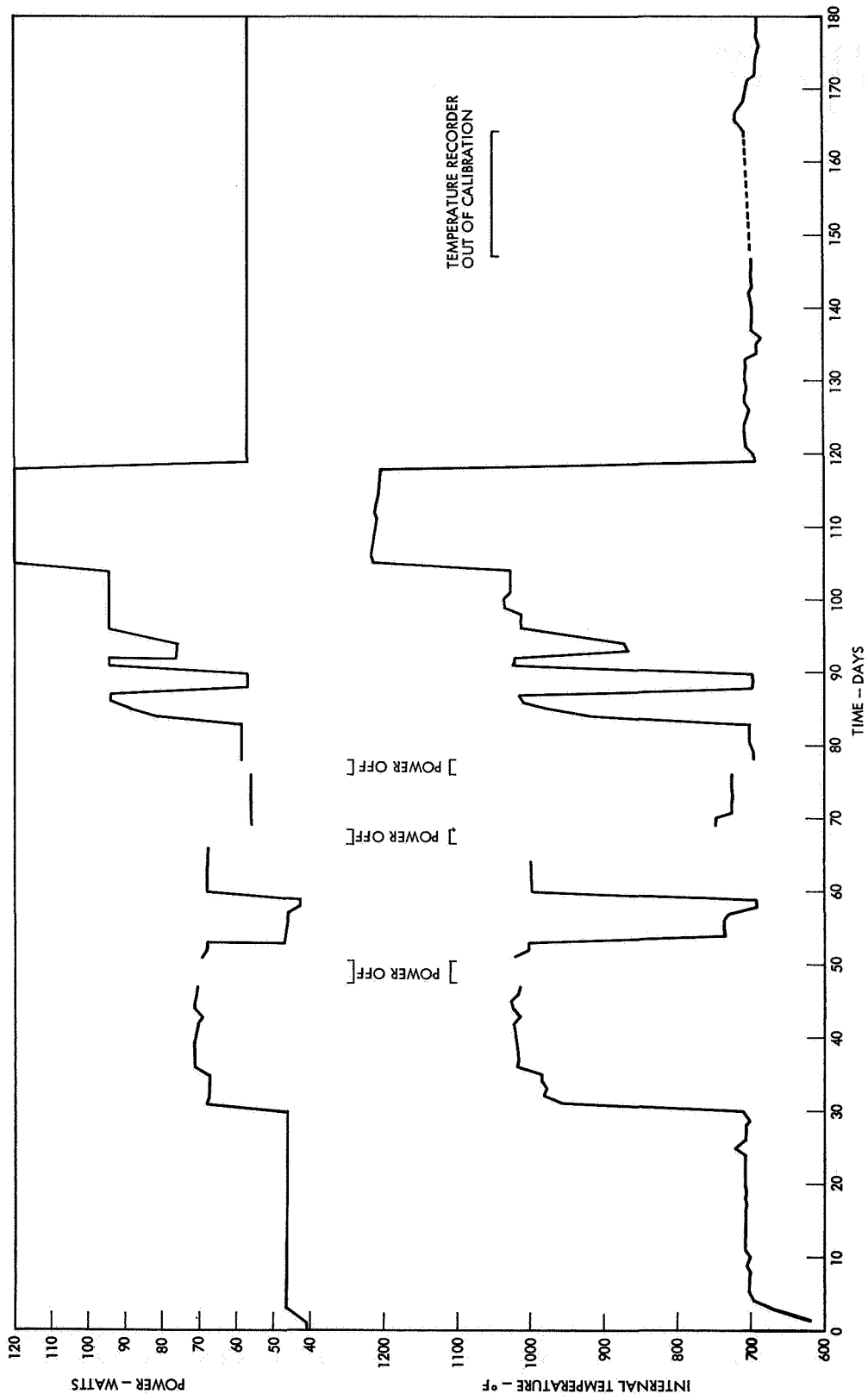


Fig. 66 Performance Results - Filled Insulation

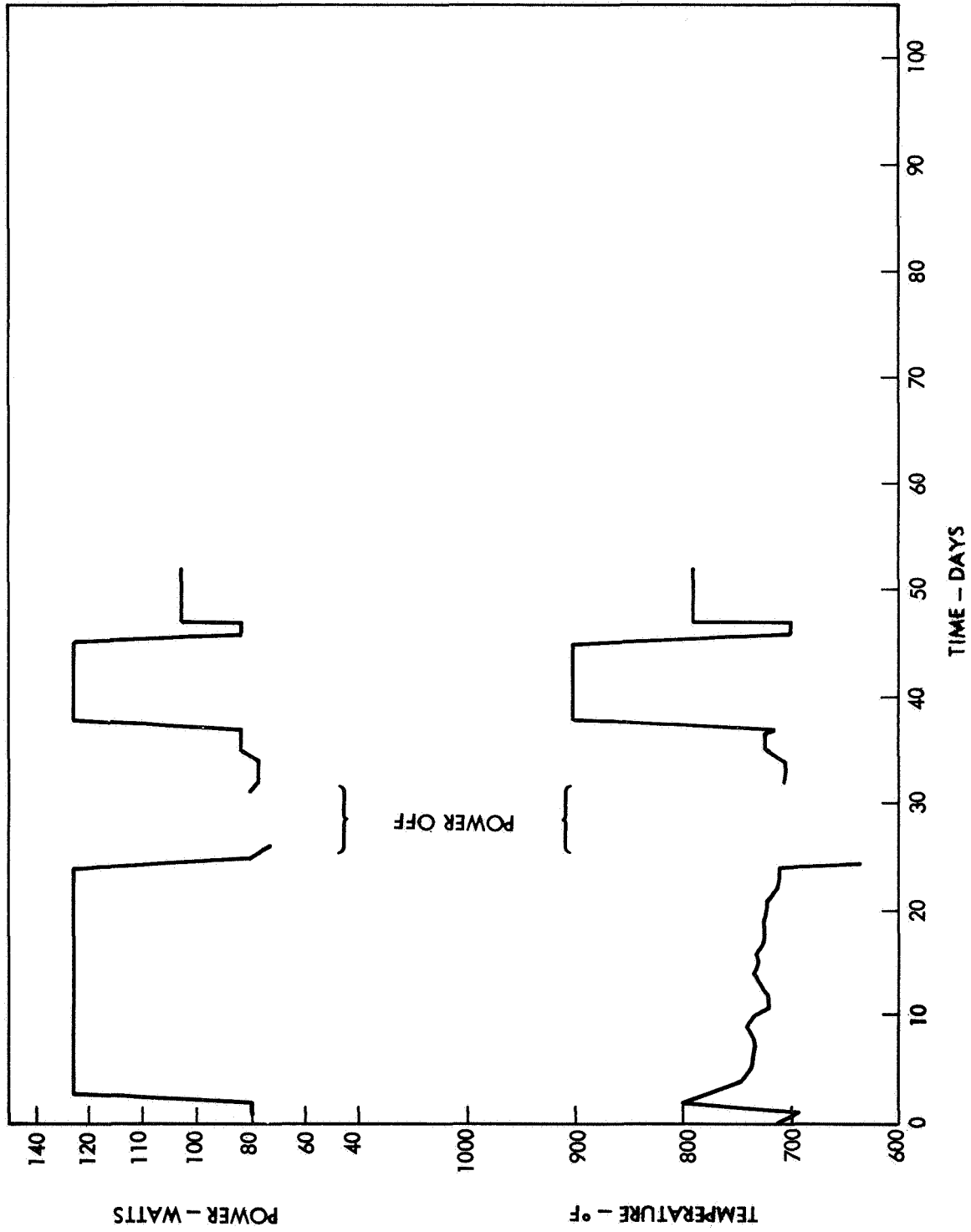


Fig. 67 Performance Results - Vacuum Insulation

coating. The black coating was analyzed by infrared spectrophotometry and the coating was identified as being a hydrocarbon material. It is probable that this contamination can be attributed to some material such as vacuum pump oil that entered the canister during the pump down period.

### Discussion

Filled insulation.-The overall thermal conductivity of the Johns Manville Min-K 1301 filled insulation was determined from the test data at various temperatures and is shown in Figure 68. This figure also shows the manufacturers data for comparison. The original IHCOS design was based on a very conservative estimate of insulation effectiveness. Results of this test indicated that the insulation effectiveness was higher than that used in the IHCOS design but lower than the manufacturers data. Using this test data and the original design constraints of a 680°F internal temperature during normal operation with a maximum allowable temperature of 1000°F during the no-flow shutdown condition, the required insulation effectiveness was re-evaluated. This analysis resulted in a reduction in required insulation thickness from 1.5 inches to 1.0 inches.

The nickel heater leads used during this test were made of the same material proposed for use in the final heater design. This type of wire had been tested at TRW for long periods of time at elevated temperatures with no problems associated with failure due to corrosion. The heater lead failure that occurred during this test was attributed to the fact that the insulation had not yet been cured to drive off contaminants present in the binder material. After replacing the heater lead, no subsequent problems were encountered with this unit and examination of the leads at the end of the test revealed that no significant corrosion had occurred.

Vacuum insulation.-The performance of the vacuum insulation was less than required throughout the test period. Also during a portion of the testing, the insulation performance degraded with time due to an internal pressure increase. These factors coupled with the initial difficulties in obtaining a satisfactory gold plating that would withstand the temperature requirements, indicated that the vacuum insulation concept was not suitable for the IHCOS insulation.

Additional development efforts might have produced a vacuum insulation canister that would have met the design requirements, however, the vacuum insulation approach is inherently less reliable than the filled insulation and appears to offer no potential advantages. For these reasons, the filled insulation was selected for the final design configuration.

### Conclusion

Another test was conducted to verify the conclusion that the uncured Min-K was the cause of the lead failure and that curing of the Min-K at 1200°F would eliminate the problem.

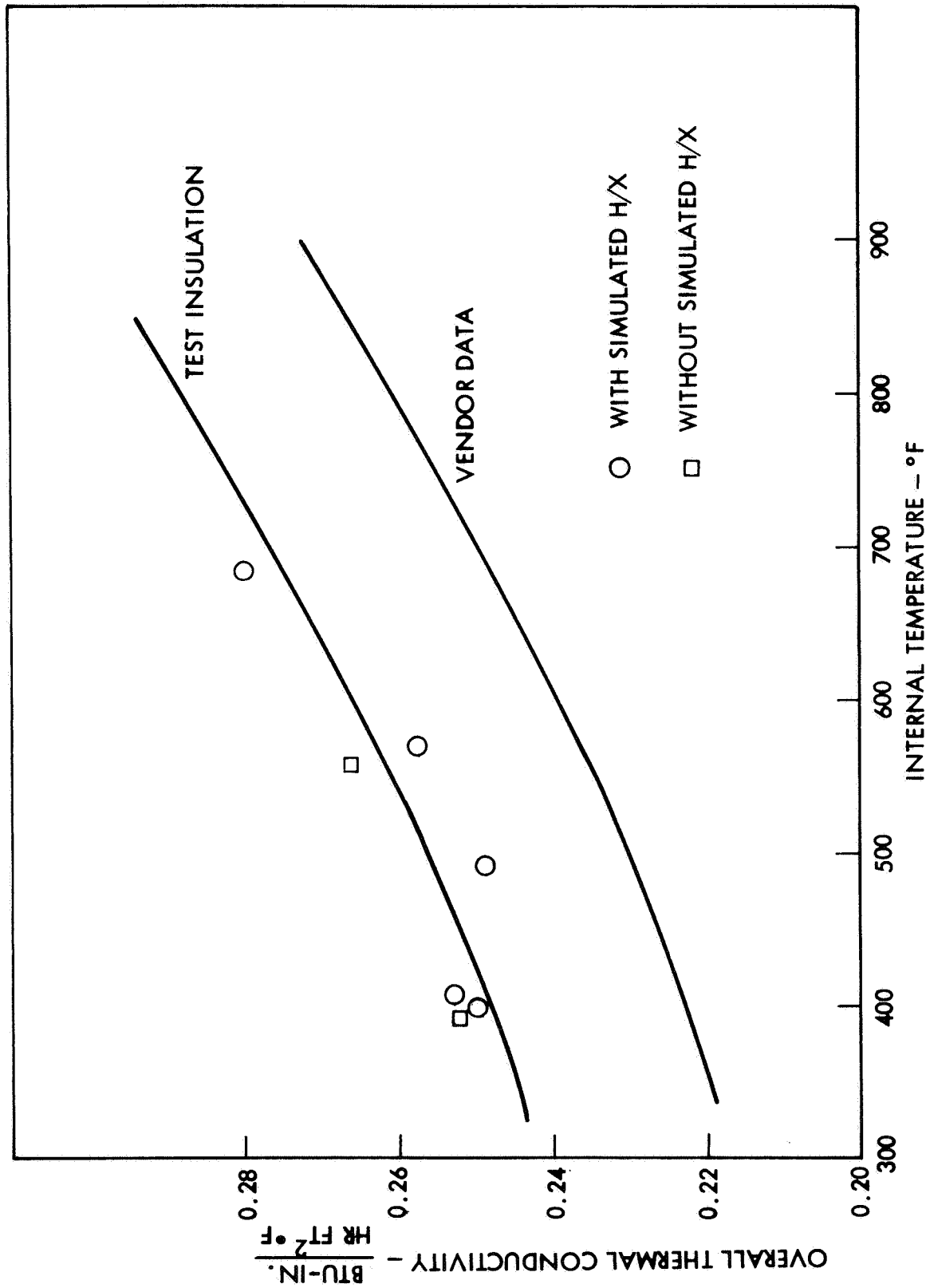


Fig. 68 Overall Conductivity of Johns Manville Min-K 1301





## SYSTEM DESIGN

The following sections describe the design characteristics of the catalytic oxidizer with an electrically heated simulated isotope, and the pre- and post-sorbent beds. This complete assembly is presented in Figure 69.

### Catalytic Oxidizer

Description.-The catalytic oxidizer assembly is shown in Figure 70 and in Appendix C with the detailed design drawings. An assembly drawing of the isotope heat source and the detailed design drawings of the liner and strength member are presented in the classified summary to this report.

The catalytic oxidizer is 14.50 inches long, excluding end fittings, and 7.62 inches in diameter. The weight of the unit is approximately 20.9 lbs. The unit consists of an outer shield, molded insulation, and an inner body. The inner body is made up of a regenerative heat exchanger, catalyst canister and radio-isotope heat source.

The regenerative heat exchanger is a 5-pass cross-counter flow, stainless steel plate fin heat exchanger. The cold end is bolted to one end of the cylindrical aluminum shield. The hot end of the heat exchanger terminates in a machined flange that mates with the catalyst canister. The gas ports are sealed with Parker metallic face seals. This heat exchanger has a very small fin and parting sheet thickness to reduce core conduction losses. In addition, the fin height is very low to obtain high heat transfer coefficients. The center fin passage on the cold side is 0.146 inches high which allows for the passage of heater wires and thermocouple instrumentation leads through the heat exchanger. This eliminates the requirement for high temperature electrical penetrations into the catalyst canister. Sintered metal plates are provided at the cold outlet and hot inlet cone face to assure good flow distribution.

The catalyst canister is a cylindrical unit that contains the 0.5 percent palladium catalyst and the radioisotope heat source. The catalyst is easily replaceable from the end opposite the heat exchanger by unbolting the end of the shield, removing the insulation section, unbolting the end of the catalyst canister, removing the screens and pouring the catalyst out. New catalyst can then be put in the unit and the unit reassembled in the reverse order. The catalyst canister body is furnace-brazed and entirely constructed of nickel. The radioisotope is mounted in the center of the catalyst canister where it is supported by posts projecting from either end of the isotope source. One post is slotted and held in place with a key to prevent rotational movement of the isotope heat source. The other post is cylindrical, and fits into a socket located on the end of the catalyst canister away from

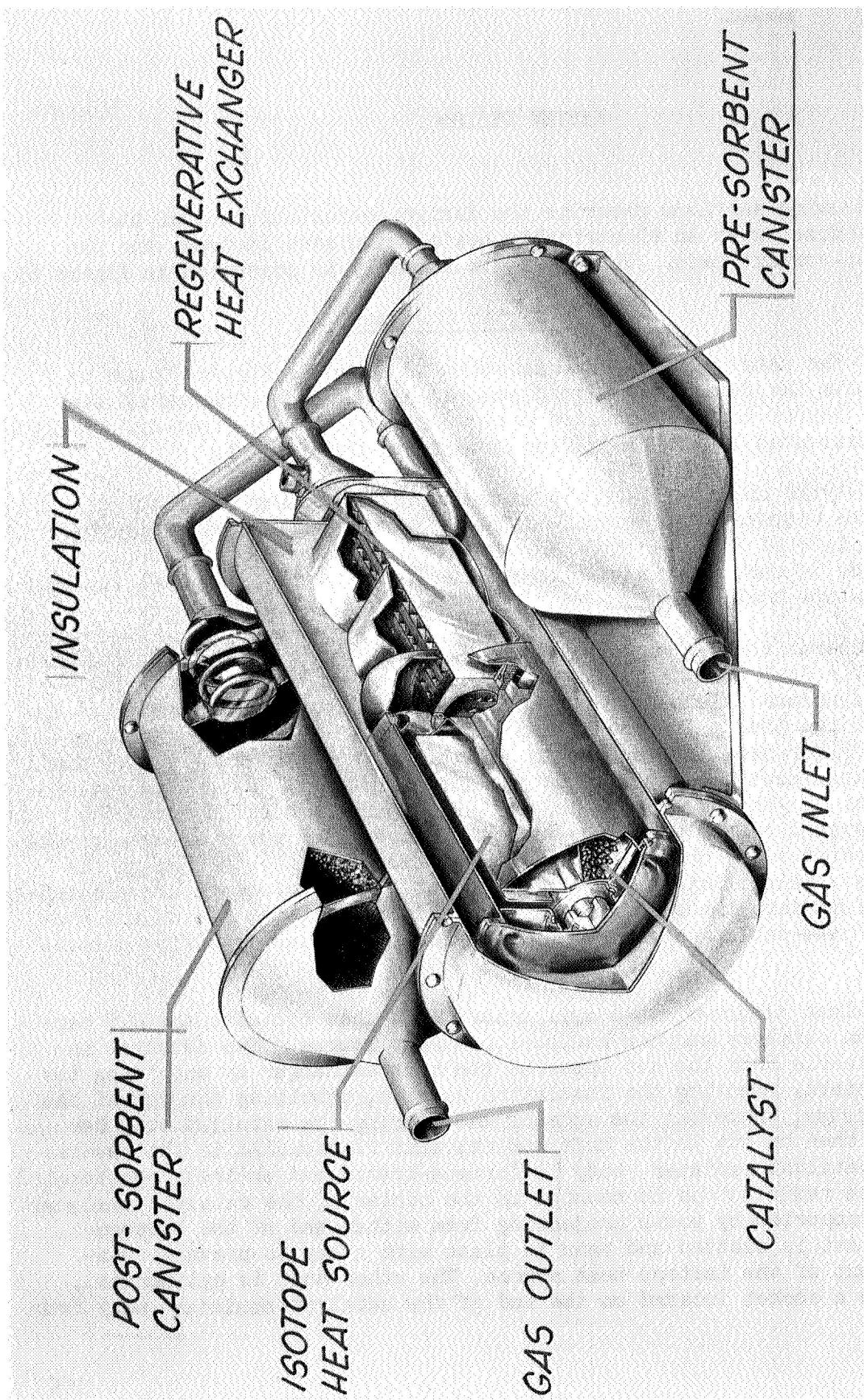


Fig. 69 Isotope Heated Catalytic Oxidizer System Assembly

QTY	ITEM NO	COOK	DATE	DESCRIPTION	MANUFACTURE OR SOURCE	REMARKS
1	BE-100-10			SCREEN		
1	22-616F			FRAMMOUNT		
1	AU-613			INSULATOR		
1	00116A111-16			INSULATOR		
1	001032X116			INSULATOR		
1	23-40949P			CONDUCTOR		
1	15% Wt of ALUMINUM			CATALYST		
1	X4032F4			INSULATOR		
1	8845-800-099			V. SEAL		
1	8817-800-0200			V. SEAL		
1	8811-800-0093			V. SEAL		
16	LSB7973-1			SCREW		
1	LS2123-8			KEY		
16	LS8869-3			NUT		
16	LSB7973-4			SCREEN		
1	MS3533-2			LOCK WASHER		
27	MS6602L			WASHER		
27	LSB7973-6			SCREEN		
1	BE-100-17			CHAMFER		
1	BE-100-18			CHAMFER		
1	BE-100-17			WATERWASHER		
1	BE-100-18			CHAMFER		
2	BE-100-13			BOOT INSULATOR		
1	BE-100-18			INSULATOR		
1	BE-100-11			INSULATOR		
1	BE-100-10			SEAL PLATE		
1	BE-100-09			DUCT ASSEMBLY		
8	BE-100-02			SCREEN LOWER		
1	BE-100-03			SCREEN UPPER		
1	BE-100-04			COVER		
1	BE-100-05			BODY		

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SUNNYVALE, CALIFORNIA

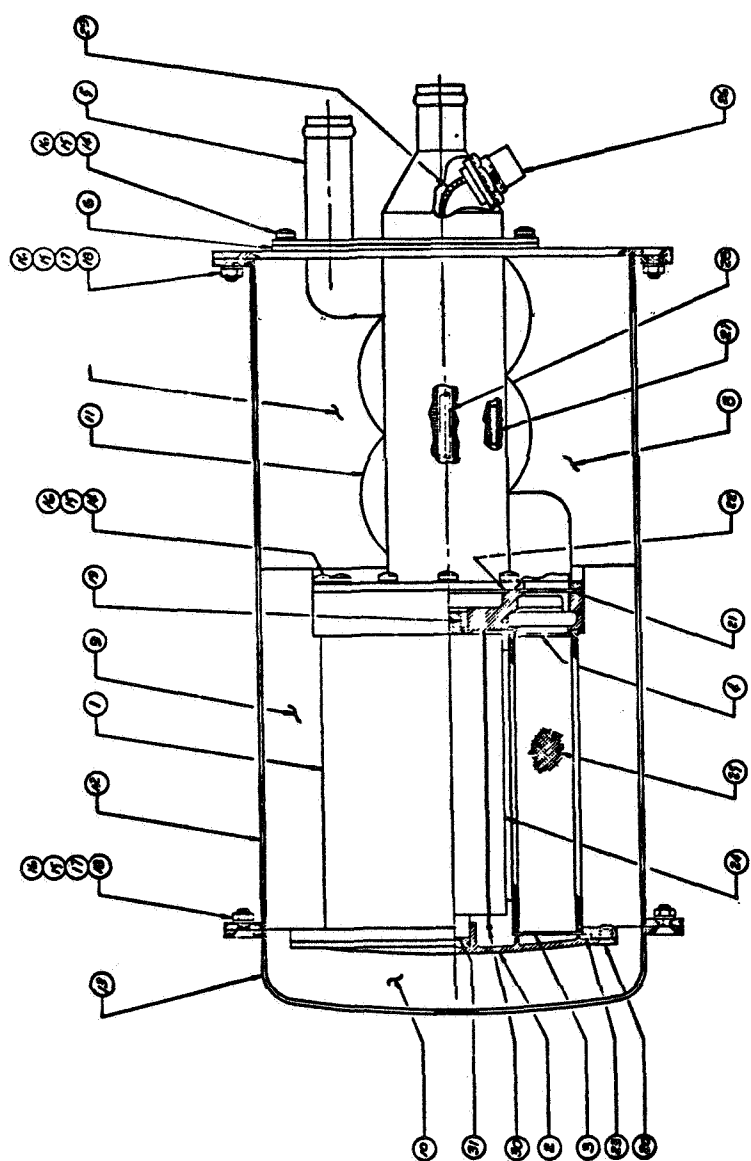
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BY: [Signature]

DESIGNED BY: [Signature]  
CHECKED BY: [Signature]  
DRAWN BY: [Signature]  
INSTRUMENTED BY: [Signature]  
TESTED BY: [Signature]

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Fig. 70 Isotope Heated Catalytic Oxidizer System

the heat exchanger. Axial movement is limited with a Belleville spring placed in this socket. This spring also allows for thermal expansion of the isotope. Work at TRW has shown that the reentry aid fins are no longer required for reentry protection, however, a heat transfer analysis has shown that these fins modified as straight radial fins reduce the maximum isotope heat source temperature. Straight fins are therefore provided on the isotope heat source.

The resistively heated source is, insofar as possible, an exact duplicate of the radioisotope-fueled heat source except that the thermal power is obtained from a resistively heated element located in the fuel cavity. All of the component closures are identical to those for the radioisotope heat source except to allow for the heater element leads and required instrumentation. The isotope heat source consists of the following components: liner, strength member, cladding, reentry member, and structural module. The liner provides a compatible container for the fuel. The strength member provides protection during impact and contains the pressure caused by the helium buildup. Cladding is provided for oxidation protection. A pyrolytic graphite shell provides aerothermal reentry protection. The structural module which contains the radial fins provides oxidation protection and heat transfer surface area.

The catalyst material is located in eight compartments located between the fins of the isotope heat source. A perforated steel plate and screen is placed at one end of the catalyst compartment and a screen is located at the other end to prevent the catalyst material from entering the heat exchanger. A machined cover is located at the end of the catalyst canister away from the heat exchanger to provide access to the isotope heat source and catalyst material. This flange is held in place with bolts and sealed with a Parker metal face seal.

The entire area between the inner body and the shield is filled with molded insulation (Johns Manville Min-K 1301). The insulation is molded in five pieces; four half cylindrical sections to insulate the catalyst canister and the heat exchanger and one to insulate the catalyst canister cover.

The aluminum outer shield separates at the catalyst cover and canister plane to allow access to the insulation and inner body of the unit. The aluminum outer shield is also attached to the cold end of the regenerative heat exchanger. The shield is painted white to provide a high emittance, and thus reduce its surface temperature.

Fitting ends on the cold end of the regenerative heat exchanger are per MS33666-12, for tube connections. An electrical feed-through is also located at the cold end of the heat exchanger for instrumentation leads and for the electrical leads of the optional electrically heated simulated isotope. The instrumentation and electrical leads pass through the inlet gas passage of the regenerative heat exchanger. Instrumentation consists of recording gas temperatures at the inlet of the catalyst bed and the heater surface temperature.

Structural analysis.-Additional structural analyses conducted on the catalytic oxidizer revealed that the metal spokes that were previously proposed to support the catalyst canister to the outer shield were not required. The major loads imposed on the unit were due to vibration which is present as a steady state acceleration, launch and space operation sinusoidal and random vibrations and shock inputs. The temperatures used in the analysis were the maximum values which were possible. Very little thermal cycling is expected which helps assure the success of the load bearing insulation concept. Vibration and thermal expansion were of primary interest in determining the workability of the load bearing insulation concept.

Since the system natural frequency is extremely dependent upon the method of mounting, a typical case of 100 g's acceleration was assumed. This value is probably higher than would occur in actual operation, but was chosen after a careful study of the vibration requirements. The 100 g vibration load produces approximately an 80 psi stress in the insulation. This stress was determined by assuming a sine load distribution between the cylinders and the insulation. A stress of this magnitude will produce no damage to the insulation, which has an approximate yield stress of 700 psi in compression.

Similarly, the shell and structure have adequate strength to withstand vibration loads, assuming a reasonable mounting configuration, such as supporting the outer shell at the heat exchanger end or supporting the outer shell at both ends.

The small thermal stresses involved should not affect the insulation if it is installed properly. The differential expansion of the internal shell, over the extremes of operating temperature, is about 0.006" radially. Since the insulation is about 1.0" thick, the radial expansion results in 0.4% radial strain. The tangential strain was not calculated but would be less than the radial strain and therefore was ignored. A 0.4% radial strain would produce only about 40 psi stress, much below the yield stress of 700 psi.

### Pre- and Post-Sorbent Canisters

Description.— The pre- and post-sorbent canisters are shown in Figures 71 and 72; the assembly and detail design drawings are presented in Appendix C. The units are constructed of 321 stainless steel and consist of a cylindrical body with a flange, housing an "O" ring seal on one end and a 45° cone outlet duct on the other end; a flanged 45° cone inlet duct is used for the cover. The flange on the cover mates and is bolted to the flange on the body. A 235 mesh screened ring is located in the outlet end of the body to retain the sorbent in the body. A screened ring backed by a compression spring is used to compress the sorbent material and keep it from channeling. The spring is compressed between the cover on one end and the screened ring and sorbent material on the other end. The mated sealing flange is used as a mounting ring. The sorbent material is environmental grade lithium hydroxide.







## CONCLUSIONS

The program for the development and design of an isotope heated catalytic oxidizer system (IHCOS) has resulted in detailed engineering drawings of a catalytic oxidizer assembly and pre- and post-sorbent beds. This system, sized for 9 men, can be successfully used to control a significant portion of the contaminants anticipated to be present in a typical early space station.

Pre- and post-sorbent beds of lithium hydroxide situated in a moist-gas stream or impregnated with lithium chloride, augmented with a main sorbent charcoal bed can effectively control those contaminants potentially poisonous to the catalyst or that will decompose to undesirable products. Testing of the catalyst at design conditions with the catalyst directly exposed to potential poisons revealed that poisoning by halogenated compounds is reversible whereas poisoning by the acid gases is irreversible. The halogenated compounds are best controlled by charcoal whose capacity for these compounds is better in a dry gas stream than a moist gas stream. The capacity of charcoal however is poor for some specific halogenated compounds such as Freon 23, and the use of these compounds in a spacecraft should be restricted. The capacity of charcoal however is poor for some specific halogenated compounds such as Freon 23, and the use of these compounds in a spacecraft should be restricted. The acid gases are controlled by lithium hydroxide and also by charcoal when they are in a moist atmosphere. Halogenated compounds will decompose in the oxidizer to acid gases which are effectively controlled by lithium hydroxide. Ammonia did not decompose in the oxidizer.

The isotope heat source configuration most suitable for the IHCOS consists of a liner for compatibility with the Pu-238 fuel form, a strength member for impact survival, noble metal cladding for oxidation protection, a pyrolytic graphite reentry member for aerothermal reentry protection, and a stainless steel structural module for oxidation protection and to provide a heat transfer surface. Testing of these materials indicated that fabrication and joining can be satisfactorily accomplished and that the materials are compatible with each other with no appreciable interdiffusion occurring over the range of expected operating conditions.

A sheathed helical heater provides the most satisfactory choice for use in an electrically heated simulated isotope heat source.

The use of Johns Manville Min-K 1301 for the IHCOS thermal insulation is superior to the use of the vacuum insulation canister that was tested.

The catalytic oxidizer requires 125 watts of power with 1-inch of insulation and is 14.5 inches long excluding end fittings and 7.6 inches in diameter. The weight of the catalytic oxidizer is 20.9 pounds. The pre-and post-sorbent canisters utilize lithium hydroxide and weigh 2.8 and 4.9 pounds, respectively.

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Appendix A  
MATERIAL SPECIFICATION  
TANTALUM ALLOY PLATE, SHEET AND STRIP





### 3. REQUIREMENTS

3.1 Manufacture. The mill products covered by this specification are formed with the conventional forging and rolling equipment normally found in primary ferrous and nonferrous plants. The ingot metal for such mill operations shall be double vacuum melted in a furnace of a type suited for reactive metals. The ingot shall be free of voids as determined by ultrasonic inspection.

3.1.1 Unless otherwise specified, material shall be furnished in the recrystallized annealed condition.

3.2 Chemical Composition. The chemical composition of tantalum alloy ingots for conversion to finished products covered by this specification shall conform to the requirements for chemical composition as prescribed in Table I. The manufacturer's ingot analysis shall be considered the chemical analysis for products supplied under this specification, except that carbon, oxygen, nitrogen and hydrogen shall be determined on the finished products and shall conform to Table I.

3.3 Hardness readings shall be as specified in Table I.

TABLE I

Hardness Requirements

<u>Alloy</u>	<u>Vickers</u>	<u>Thickness</u>	
		<u>.005 to .080"</u>	<u>over .080"</u>
Type I	230 Max.	30 N 40 Max.	Ra 60 Max.
Type II	280 Max.	30 N 50 Max.	Ra 65 Max.
Type III	310 Max.	30 N 59 Max.	Ra 70 Max.

TABLE II

CHEMICAL COMPOSITION\*

<u>Element</u>	<u>Type I</u>	<u>Type II</u>	<u>Type III</u>
Tungsten	8.5 - 11.0%	7.0 - 9.0%	9.6 - 11.2%
Hafnium		1.8 - 2.4%	2.2 - 2.8%
Carbon	50 ppm**	100 ppm	80-175 ppm
Nitrogen	50 ppm	100 ppm	100 ppm
Oxygen	100 ppm	150 ppm	100 ppm
Hydrogen	10 ppm	10 ppm	10 ppm
Boron		1 ppm	1 ppm
Cobalt		50 ppm	50 ppm
Iron	100 ppm	50 ppm	50 ppm
Molybdenum	300 ppm	200 ppm	200 ppm
Nickel	100 ppm	50 ppm	50 ppm

Vanadium		20 ppm	20 ppm
Colubium	2000 ppm	2000 ppm	2000 ppm
Tantalum	Balance	Balance	Balance

\* Maximum limits unless otherwise indicated

\*\*ppm- parts per million

### 3.4 Tensile Properties

3.4.1 Room Temperature. The annealed materials when tested at room temperature, shall have minimum tensile properties in accordance with Table III.

TABLE III

#### Tensile Properties at Room Temperature

	<u>Alloy Designation</u>		
	<u>Type I</u>	<u>Type II</u>	<u>Type III</u>
Ultimate Tensile Strength 1,000 psi	75	80	105
Yield Tensile Strength 1,000 psi	65	65	100
Elongation % in 1 inch	15	20	20

3.4.2 Elevated Temperature. When specified in the purchase order, the tensile properties of annealed material when measured at elevated temperature shall have minimum values in accordance with Table IV. These properties shall be measured at  $3000^{\circ}\text{F} \pm 25^{\circ}\text{F}$  in vacuum at a pressure not to exceed  $1 \times 10^{-4}$  mm of mercury.

TABLE IV

#### Tensile Properties at 3000°F

	<u>Alloy Designation</u>		
	<u>Type I</u>	<u>Type II</u>	<u>Type III</u>
Ultimate Tensile Strength, 1000 psi	20	15	22
Yield Strength, 0.2% offset, 1000 psi	14	13	20
Elongation, % in 1 in.	15	30	30

3.5 Ductility. Sheet or strip shall be capable of withstanding a bend of 105 degrees around a radius equal to the material thickness at room temperature without cracking as viewed under 10X magnification.

3.6 Grain Size. Grain size shall be ASTM number five or finer

### 3.7 Dimensional Tolerances

3.7.1 Thickness tolerances for sheet and strip shall conform to the following:

<u>Sheet or strip thickness, inches</u>	<u>Tolerances</u>
up to .010	$\pm$ .001
.010 to .050	$\pm$ .002
.050 to .075	$\pm$ .003
.075 to .187	$\pm$ .010

3.7.2 All other tolerances shall be in accordance with ASTM B393 except that for material greater than 24 inches in width, width tolerance shall be + 1/4", -0" and for material greater than 24 inches in length, length tolerances shall be + 1/2", -0".

3.8 Edges. Edges of sheet and strip shall be produced by slitting or shearing with a burr height not to exceed 5 percent of the material thickness.

3.9 Finish. All material shall be supplied with a cold rolled or machined finish, with a chemically clean surface. There shall be no discoloration or foreign matter on the surface.

3.10 Quality. All material shall be free from slivers, cracks, pits, blisters, and laminations. Local surface defects may be removed by buffing or spot grinding, provided that such conditioning does not violate the minimum gage tolerance. Such removal must be accomplished prior to final finish treatment. Unless otherwise specified, pits, scratches, or gouges shall be acceptable if no deeper than 0.002 inch or deeper than 10% of the thickness of the sheet whichever is smaller.

3.11 Identification of Product. Each plate, sheet and strip shall be marked with the contract or order number, ingot melt number, specification number, type and nominal dimensions. The identification shall be put on a removable adhesive sticker.

#### 4. QUALITY ASSURANCE PROVISIONS

##### 4.1 Acceptance

4.1.1 Certification. The supplier shall certify that each shipment of material made to this specification conforms to the requirements of this specification. The supplier shall submit three certified copies of a report indicating:

- (a) Chemical composition
- (b) Tensile test results at room temperature
- (c) Tensile test results at elevated temperature (when specified)
- (d) Ductility
- (e) Grain size
- (f) Hardness

This report shall include the purchase order number, heat number, material specification number, alloy type, nominal dimensions and quantity from each heat.

4.1.2 Responsibility. Unless otherwise stated on the applicable purchase order or other procurement document, acceptance testing shall be performed by the supplier. TRW Systems reserves the right to perform any of the inspections set forth in this specification where such inspections are deemed necessary to assure that the material conforms to prescribed requirements.

#### 4.1.3 Sampling

4.1.3.1 Lot. A lot shall comprise material of the same cross section dimensions fabricated from a single ingot. The samples shall be taken so as to be representative of the finished product. Unless otherwise specified the supplier shall include sufficient material (40 square inches minimum) to be used for acceptance testing.

4.1.4 Testing. Acceptance testing shall consist of the following:

- (a) Chemical composition
- (b) Hardness test
- (c) Tensile test
- (d) Ductility test
- (e) Grain size test
- (f) Visual and dimensional examination

4.1.5 Rejection and Resubmittal. Material not conforming to all of the requirements of this specification shall be subject to rejection. Resubmittal shall be accompanied by a statement providing evidence of corrective action.

#### 4.2 Test Methods

4.2.1 Chemical Composition. The methods for sampling and analysis shall be those agreed upon between TRW and the supplier.

4.2.2 Hardness Test. Hardness test Method 243.1 or 244.1 of Fed. Test Method Std. No. 151 shall be used.

#### 4.2.3 Tensile Test.

4.2.3.1 Room Temperature. Two tensile test specimens shall be prepared and tested per MAB - 192 - M. The axis of the specimens shall be transverse to the final rolling direction. The strain rate shall be maintained at  $0.005 \pm 0.001$  inch per inch per minute through the 0.2% offset yield strength and at  $0.05 \pm 0.005$  inch per inch per minute thereafter.

4.2.3.2 Elevated Temperature. Test specimens shall be tested in accordance with A.S.T.M. E 21. Strain rate shall be .05 in. per in. per minute.

4.2.4 Ductility Test. Two ductility test specimens shall be prepared and tested per Material Advisory Board Document MAB-192-M. The axis of the specimens shall be transverse to the final rolling direction.

4.2.5 Grain Size Test. Grain size shall be determined in accordance with ASTM E 112.

## 5. PREPARATION FOR DELIVERY

5.1 Packaging. All material shall be packed and packaged in a manner that will prevent damage in transit and storage. Material shall have adhesive type protective coating on each side.

## 6. NOTES

6.1 Intended Use. The alloys described by this specification are intended for use in space power systems.

6.2 Ordering Data. Procurement documents shall specify the following:

- (a) Title, number and date of this specification
- (b) Alloy type
- (c) Product size and quantity
- (d) Requirement for elevated temperature tensile testing (if applicable).

### 6.3 Definitions

6.3.1 Plate - 6" wide or over and more than 3/16" thick

6.3.2 Sheet - 6" wide or over and 3/16" thick or less

6.3.3 Strip - less than 6" wide and 3/16" thick or less



## MATERIAL SPECIFICATION

### MOLYBDENUM-0.5 PERCENT TITANIUM-0.08 PERCENT ZIRCONIUM (T2M) WROUGHT BARS

#### 1. SCOPE

1.1 Scope. This specification covers wrought bars of carbon-deoxidized T2M produced by vacuum arc-casting.

#### 2. METHOD OF PROCESSING

2.1 Bars shall be rolled, or rolled and waged, from arc-cast T2M billets produced by the consumable-electrode vacuum arc-melting process.

#### 3. CHEMICAL COMPOSITION

3.1 The chemical composition of the billets from which these bars are produced shall conform to the following limits:

<u>Element</u>	<u>Maximum Percent</u>	<u>Minimum Percent</u>	<u>Check Analysis Percent</u>
Carbon	0.03	0.01	+ 0.005
Titanium	0.55	0.40	+ 0.05
Zirconium	0.12	0.06	+ 0.02
Gaseous elements (listed below)	0.005	--	
Trace elements (listed below)	0.018	--	
Molybdenum (by difference)	--	99.25	

3.1.1 Gaseous elements as determined by vacuum fusion analysis shall conform to the following:

<u>Element</u>	<u>Maximum Weight Percent</u>
Oxygen	0.0025
Hydrogen	0.0005
Nitrogen	0.002

3.1.2 Trace elements as determined by spectrographic analysis shall conform to the following:

<u>Element</u>	<u>Maximum Percent</u>
Iron	0.008
Nickel	0.002
Silicon	0.008

4. STRUCTURAL CONDITION

4.1 The maximum grain size of recrystallized bars shall be ASTM No. 3 (ASTM E 112).

5. MECHANICAL PROPERTIES

5.1 All test specimens shall be taken from bars selected at random.

5.2 Hardness of stress-relieved bars shall be determined at the mid-radius of the bars and shall conform to the following limits:

<u>Diameter of Bar in Inches</u>	<u>DPH Hardness (10 Kg)</u>	
	<u>Minimum</u>	<u>Maximum</u>
1/8 to 7/8	260	320
Over 7/8 to 1 1/8	250	310
Over 1 1/8 to 1 7/8	245	300
Over 1 7/8 to 2 7/8	240	290
Over 2 7/8 to 3 1/2	235	285
Over 3 1/2 to 4 1/2	230	280

5.2.1 Hardness of recrystallized bars shall be determined at the mid-radius of the bars and shall conform to the following:

<u>Diameter of Bar in Inches</u>	<u>DPH Hardness (10 Kg) Maximum</u>
1/8 to 4 1/2	215

5.3 Tensile tests shall be conducted at 70 to 85°F using a strain rate of 0.002 to 0.005 in./in./min. through 0.6 percent offset and of 0.02 to 0.05 in./in./min. to failure. All tensile properties shall be determined in the longitudinal direction using test specimens taken at the center of the bar up to 1 1/8 inches in diameter and at mid-radius for over 1 1/8 inches in diameter. Test specimens shall be made in accordance with ASTM Specification E8. The gage diameter of the test bar shall be 4D for all bar diameters.

5.3.1 The properties of stress-relieved bars shall conform to the following:

<u>Diameter of Bar in Inches</u>	<u>Minimum Tensile Strength, PSI</u>	<u>Minimum Yield Strength, PSI (0.2% Offset)</u>	<u>Minimum Elongation Percent in 4D</u>
1/8 to 7/8	115,000	100,000	18
Over 7/8 to 1 1/8	110,000	95,000	15
Over 1 1/8 to 1 7/8	100,000	85,000	10
Over 1 7/8 to 2 7/8	90,000	80,000	10
Over 2 7/8 to 3 1/2	85,000	75,000	5
Over 3 1/2 to 4 1/2	80,000	70,000	5

5.3.2 If tensile properties are specified at the time of purchase, the properties of recrystallized bars shall conform to the following:

<u>Diameter of Bar in Inches</u>	<u>Minimum Tensile Strength, PSI</u>	<u>Minimum Yield Strength, PSI (0.2% Offset)</u>	<u>Minimum Elongation Percent in 4D</u>
Less than 2	80,000	55,000	20
2 to 4 1/2	75,000	45,000	10

## 6. DIMENSIONAL TOLERANCES

6.1 Bars shall be supplied to the limits shown in the following table:

<u>Diameter of Bar in Inches</u>	<u>Diameter Variation, Inches</u>	<u>Out of Round Inches</u>
1/8 to 9/32	+ 0.002 - 0.002	0.004
Over 9/32 to 13/32	+ 0.003 - 0.003	0.006
Over 13/32 to 5/8	+ 0.010 - 0.005	0.012
Over 5/8 to 7/8	+ 0.015 - 0.005	0.015
Over 7/8 to 1	+ 0.020 - 0.005	0.015
Over 1 to 1 3/8	+ 0.020 - 0.010	0.018
Over 1 3/8 to 1 1/2	+ 0.020 - 0.015	0.020
Over 1 1/2 to 1 5/8	+ 0.025 - 0.015	0.020
Over 1 5/8 to 2	+ 0.030 - 0.020	0.025
Over 2 to 2 1/2	+ 0.032 - 0.032	0.025
Over 2 1/2 to 3 1/4	+ 0.032 - 0.032	0.027
Over 3 1/4 to 3 1/2	+ 0.045 - 0.045	0.040
Over 3 1/2 to 4 1/2	+ 0.062 - 0.062	0.050

6.2 Centerless ground bars shall be supplied with a tolerance of  $\pm$  0.002 inch for 2-inch diameter and under,  $\pm$  0.003 inch for over 2-inch diameter.

6.3 The maximum variation from straightness shall be 0.050 inch per foot.

6.4 Maximum variation in cut lengths shall be + 1/4 inch, - 0.

## 7. SURFACE FINISH

7.1 All bars shall be supplied with chemically or mechanically cleaned surfaces.

7.2 Minor surface imperfections may be removed provided such removal does not reduce the dimension below the minimum permitted by the tolerance for the size specified.

7.3 Centerless ground bars shall be supplied with a surface finish of 90 RMS or better.

## 8. INSPECTION

8.1 The surface and ends shall be free of all surface defects which may be detected by dye penetrant inspection methods.

8.2 Bars of 2-inch diameter or more shall be sound, as determined by ultrasonic inspection methods.

8.3 Bars of smaller diameter than 2 inch shall be ultrasonically inspected for soundness, if requested.

## 9. MARKING

9.1 All bars shall be identified with the heat number. The characters shall be applied using a suitable marking fluid that will withstand ordinary handling.

## 10. PACKING AND SHIPPING

10.1 The material shall be packed so that damage will not occur during ordinary shipping and handling. When shipped, each box or pallet shall be conspicuously marked with the customer's name and purchase order number as well as the manufacturer's identification.

## 11. REPORTS

11.1 Each shipment shall be accompanied by a shipping memorandum and quality control report stating the specification number, the number of pieces, heat number, chemical composition of the billet, bar sizes, structural condition, mechanical properties, and net weight of each bar size.

11.2 If requested at the time of purchase, certified copies of a test report shall be furnished.

## MATERIAL SPECIFICATION

### PLATINUM-RHODIUM ALLOY TUBE, SHEET AND STRIP

#### 1. SCOPE

1.1 Scope. This specification establishes the requirements for platinum-rhodium alloy tube, sheet and strip.

#### 2. APPLICABLE DOCUMENTS

(None). Existing specifications for Pt-Rh alloys are tailored to specific applications and are generally proprietary for a given customer and fabrication.

#### 3. REQUIREMENTS

3.1 Chemical Composition. The purity of the platinum-rhodium alloy shall be determined by quantitative spectrographic procedure. The rhodium content shall be determined by either standard chemical procedure or X-ray spectrographic procedure. The concentration of any single impurity in no case shall exceed the maximum amount listed in the tabulation below. The combined total of the various groups of selected impurities shall not exceed the maximum for that group as indicated in table I.

TABLE I

#### CHEMICAL COMPOSITION

<u>Element</u>	<u>Maximum ppm</u>
Mo	100
Au	100
Fe	100
Si	100
Ag	70
Pb	70
Sn	70
Zn	70
Sb	70
As	70
Cd	70
Bi	40
Cu	150
Pd	250
Other*	200
Pt + Rh	99.90% Min.

\*No other single element shall exceed 200 ppm.

3.2 Tensile Properties.

3.2.1 Room Temperature. The annealed materials when tested at room temperature, shall have minimum tensile properties in accordance with table II and table III.

TABLE II  
TENSILE PROPERTIES AT ROOM TEMPERATURE

<u>Material</u>	<u>Tensile Strength</u>
Pt	40,000 psi
90 Pt -10Rh	77,000 psi
80 Pt -20Rh	92,000 psi
60 Pt -40Rh	107,000 psi

3.2.2 Elevated Temperature. When specified in the purchase order, the tensile properties of annealed material when measured at elevated temperature shall have minimum values in accordance with table III. These properties shall be measured at  $2000^{\circ}\text{F} \pm 25^{\circ}\text{F}$  in vacuum at a pressure not to exceed  $1 \times 10^{-4}$  mm of mercury.

TABLE III  
TENSILE PROPERTIES AT  $2000^{\circ}\text{F}$

<u>Material</u>	<u>Tensile Strength</u>
Pt	7,000 psi
90 Pt -10Rh	12,000 psi
80 Pt -20Rh	21,000 psi
60 Pt -40Rh	29,000 psi

3.3 Dimensional Tolerances.

3.3.1 Thickness tolerances for sheet and strip shall conform to the following:

<u>Sheet or Strip Thickness, Inches</u>	<u>Tolerance</u>
Up to .010	$\pm .0005$
.010 to .050	$\pm .001$
.050 to .075	$\pm .002$
.075 to .187	$\pm .005$

3.4 Edges. Edges of sheet and strip shall be produced by slitting or shearing with a burn height not to exceed 5 percent of the material thickness.

3.5 Finish. All material shall be supplied with a cold rolled or machined finish, with a chemically clean surface. There shall be no discoloration or foreign matter on the surface.

#### 4. QUALITY ASSURANCE PROVISIONS

##### 4.1 Acceptance.

4.1.1 Certification. The supplier shall certify that each shipment of material made to this specification conforms to the requirements of this specification. The supplier shall submit three certified copies of a report indicating:

- (a) Chemical composition
- (b) Tensile test results at room temperature
- (c) Tensile test results at elevated temperature (when specified)
- (d) Ductility
- (e) Grain size
- (f) Hardness

4.1.2 Responsibility. Unless otherwise stated on the applicable purchase order or other procurement document, acceptance testing shall be performed by the supplier. TRW Systems reserves the right to perform any of the inspections set forth in this specification where such inspections are deemed necessary to assure that the material conforms to prescribed requirements.

4.1.3 Rejection and Resubmittal. Material not conforming to all of the requirements of this specification shall be subject to rejection. Resubmittal shall be accompanied by a statement providing evidence of corrective action.

#### 5. PREPARATION FOR DELIVERY

5.1 Packaging. All material shall be packaged in a manner that will prevent damage in transit and storage.

#### 6. NOTES

6.1 Intended Use. The alloys described by this specification are intended for use in radioisotope heat source assemblies.

6.2 Ordering Data. Procurement documents shall specify the following:

- (a) Title, number and date of this specification
- (b) Alloy type
- (c) Product size and quantity
- (d) Requirement for elevated tensile testing (if applicable)

##### 6.3 Definitions.

6.3.1 Sheet. 6" wide or over and 3/16" thick or less.

6.3.2 Strip. Less than 6" wide and 3/16" thick or less.





# MATERIAL SPECIFICATION

## PYROLYTIC GRAPHITE

### 1. SCOPE

1.1 Scope. This specification establishes the requirements for pyrolytic graphite produced by vapor deposition.

### 2. APPLICABLE DOCUMENTS

None

### 3. REQUIREMENTS

3.1 Manufacture. The materials covered by this specification are produced by thermal decomposition of a hydrocarbon gas on a hot surface and deposits so that the direction of high strength and high thermal conductivity is always parallel to the deposition surface.

3.1.1 Unless otherwise specified and agreed to between vendor and the customer only the following physical tests and quality measurements will be performed.

3.2 Plate. Flat plate or machined shapes produced from plate.

3.2.1 Density gm/cc at  $77^{\circ}\text{F} \pm 5$  2.20  $\pm$ .02  
(as determined by differential weighing in air and CP  
carbon tetrachloride)

3.2.2 Nodules. (Conical Growth Defects) The following nodule sizes and distribution will be considered the maximum allowable in plate material:

#### MAXIMUM NODULE SIZE ALLOWABLE

Size-Round or Square	Thickness				
	<u>1/8"</u>	<u>1/4"</u>	<u>3/8"</u>	<u>1/2"</u>	<u>5/8"</u>
Up to 2"	.062	.125	.187	.250	.312
2" to 4"	.094	.156	.250	.312	.406
4" to 6"	.125	.203	.312	.406	.500
6" to 8"	.156	.250	.375	.500	.500
8" to 10"	.203	.312	.437	.500	.500
10" to 12"	.250	.375	.500	.500	.500

MINIMUM DISTANCE BETWEEN EDGES OF NODULES  
OF MAXIMUM SIZE

Size-Round Or Square	Thickness				
	<u>1/8"</u>	<u>1/4"</u>	<u>3/8"</u>	<u>1/2"</u>	<u>5/8"</u>
Up to 2"	1	1	1	1	1
2" to 4"	7/8	7/8	15/16	1	1
4" to 6"	13/16	13/16	15/16	1	1
6" to 8"	3/4	3/4	7/8	1	1
8" to 10"	5/8	5/8	13/16	1	1
10" to 12"	1/2	1/2	3/4	1	1

3.2.3 Surface flaws. The exposed surfaces of the plate parallel to the "a" direction shall be free of cracks when examined visually, and shall not be visibly marked (grooved or gouged) by measuring instruments. Final inspection for cracks shall be made by a penetrant dye check after finish machining according to the appropriate Military Specifications.

3.2.4 Delaminations. The exposed surfaces of the plates parallel to the "c" direction shall be free of major delaminations when examined visually; and shall not be visibly marked (grooved or gouged) by the measuring instruments. No delaminations shall extend more than one-fourth the length of the size on which it appears. There shall be no more than two delaminations through the thickness in any material up to 1/4" in thickness, and no more than three through the thickness in material between 1/4" and 1/2" thick. Final inspection for delaminations shall be made by a penetrant dye check after final machining according to the appropriate Military Specifications.

Bow in plate as deposited:

<u>For L</u>	<u>Thickness t</u>	<u>Maximum</u>
Up to 4"	Up to 3/8"	1°
4" to 8"	Up to 1/2"	2°
8" and above	Up to 1/2"	4°

3.2.5 Purity. Total ash content by combustion - 0.01% maximum - 0.005% typ.

3.2.6 Minimum flexural strength "a" direction - 12,000 psi (loaded parallel to the surface of deposition min. at RT).

3.3 Cylinders. Hollow cylinders and closely related shapes such as ovals, cones, etc.

3.3.1 Density gm/cc at 77°F ± 5 2.20 ± .02  
(as determined by differential weighing in air and CP carbon tetrachloride).

3.3.2 Nodules. (Conical Growth Defects) No nodule or cluster of nodules (nodules touching each other) shall be larger in diameter than equal to the cylinder wall thickness for all thicknesses up to and including 1/2" material. For cylinders with wall thicknesses greater than 1/2" the nodule size and distribution shall be agreed upon between vendor and the customer. Nodule distribution shall be such that no more than two nodules of the maximum size shall be closer than 1" apart.

3.3.3 Surface Flaws. The exposed surfaces of cylinder parallel to the "a" directions shall be free of cracks when examined visually; and shall not be visibly marked (grooved or gouged) by measuring instruments. Final inspection for cracks shall be made by a penetrant dye check after finish machining according to the appropriate Military Specifications.

3.3.4 Purity. Total ash content by combustions - 0.01% maximum - 0.005% typ.

3.3.5 Tensile Strength "a" direction 10,000 psi  
(pulled in the plane parallel to the surface of deposition  
min. at RT).

3.3.6 Compressive Strength parallel to "a" dir. 10,000 psi  
min. at RT parallel to "c" dir. 45,000 psi

3.3.7 Thermal Expansion parallel to "a" dir.  $0./ \pm 0.3$   
(x  $10^{-6}$  in./in./ $^{\circ}$ F parallel to "c" dir.  $15.0 \pm 3.0$   
at  $1000^{\circ}$ F)

3.3.8 Thermal conductivity parallel to "a" dir.  $100 \pm 50$   
 $\text{BTU-ft-ft}^2 \text{ hr}^{-1} \text{ }^{\circ}\text{F}^{-1}$  parallel to "c" dir.  $1.0 \pm 0.5$

3.3.9 Elastic Modulus min. at RT  $3 \times 10^6$  psi Plate only

3.3.10 Minimum Flexural Strength "a" direction - 12,000 psi, cylinders only. (loaded parallel to the surface of deposition min. at RT).

3.3.11 Internal residual stress-- inner fiber bending stress psi maximum - 5,000, cylinders only (A 7/8 inch wide ring from one end of each cylinder shall be provided for measuring internal residual stress before machining. The internal residual stress is a function of the thickness-to-radius ratio.)

3.3.12 Out-of-Roundness, difference in diameters  $90^{\circ}$  apart after machining in.,  $\pm .003$  to  $.005$  - cylinders only.

3.4 Dimensional Measurements. The dimensions and tolerances, including surface finish, will be held according to the customer's engineering drawings and agreed to by the vendor by acceptance of the purchase order.

3.4.1 Certification. Three copies of a Certificate of Test will be furnished upon request stating that the product conforms to the requirements of our standard tests and any other tests agreed upon mutually by the vendor and the customer. This report will include the purchase order number, furnace run number, form or part number, quantity, and dimensional measurements. Where agreed to mutually between the vendor and the customer, process data including furnace cycle times and temperatures, and types of gases used will be furnished. Detailed drawings or sketches of the furnace hardware will not be provided.

3.4.2 Identification. All parts will be clearly marked to show part number, lot number, etc. in such a way that the marking does not in any way remove or displace the parent material of the part.

3.5 Packaging. Packaging shall be accomplished to such a manner as to insure the product during shipment and storage will be protected against damage from exposure to weather or any normal hazard.

#### 4. QUALITY ASSURANCE PROVISIONS

##### 4.1 Acceptance.

4.1.1 Certification. The supplier shall certify that each shipment of material made to this specification conforms to the requirements of this specification.

4.1.2 Responsibility. Unless otherwise stated on the applicable purchase order or other procurement document, acceptance testing shall be performed by the supplier. TRW Systems reserves the right to perform any of the inspections set forth in this specification where such inspections are deemed necessary to assure that the material conforms to prescribed requirements.

4.1.3 Rejection and resubmittal. Material not conforming to all of the requirements of this specification shall be subject to rejection. Resubmittal shall be accompanied by a statement providing evidence of corrective action.

##### 4.2 Test Methods.

4.2.1 Chemical Composition. The methods for sampling and analysis shall be those agreed upon between TRW and the supplier.

#### 5. PREPARATION FOR DELIVERY

5.1 Packaging. All material shall be packed and packaged in a manner that will prevent damage in transit and storage.

## MATERIAL SPECIFICATION

### STEEL, CORROSION- AND HEAT-RESISTANT (CHEMICALLY STABILIZED) PLATE, SHEET, AND STRIP (TYPE 321)

#### 1. SCOPE

1.1 Scope. This specification covers chemically stabilized steel plate, sheet, and strip products possessing high resistance to corrosion and heat and a high order of weldability.

1.2 Classification. Materials shall be furnished to the chemical compositions indicated by table I, and designated as columbium stabilized, titanium stabilized, or columbium-tantalum stabilized. Unless otherwise specified, either type may be furnished, except that all material furnished on an individual order shall be of one type only.

#### 2. APPLICABLE DOCUMENTS

2.1 The following documents, of the issue in effect on date of invitation for bids, form a part of this specification to the extent specified herein:

##### STANDARDS

##### Federal

FED. STD. NO. 48	Tolerances for Steel and Iron Wrought Products
FED. TEST METHOD STD. NO. 151	Metals; Test Methods

##### Military

MIL-STD-163	Steel Mill Products Preparation for Shipment and Storage
MIL-STD-183	Continuous Identification Marking of Iron and Steel Products

#### 3. REQUIREMENTS

3.1 Material. The steel shall be manufactured by the electric-furnace process. Sufficient discard shall be taken from each ingot to insure freedom from injurious piping and undue segregation.

3.2 Chemical composition. The chemical composition shall be in accordance with table I.

3.3 Condition. Unless otherwise specified, materials shall be furnished in the following conditions:

- (a) Sheet: Cold-rolled, solution heat treated, and pickled (No. 20 finish).
- (b) Strip: Cold-rolled, solution heat treated, and pickled (No. 1 strip finish).
- (c) Plate: Hot-rolled, solution heat treated, and pickled.

TABLE I  
CHEMICAL COMPOSITION

Element <u>1/</u>	Composition Type		
	Cb	Ti	Cb-Ta
	(percent)	(percent)	(percent)
Carbon	0.08 (max)	0.08 (max)	0.08 (max)
Manganese	2.00 (max)	2.00 (max)	2.00 (max)
Phosphorus	0.040 (max)	0.040 (max)	0.040 (max)
Sulfur	0.030 (max)	0.030 (max)	0.030 (max)
Chromium	17.0/19.0	17.0/19.0	17.0/19.0
Nickel	9.0/13.0	8.0/11.0	9.0/13.0
Silicon	0.050-1.00	0.040-1.00	0.50-1.00
Molybdenum	1.50 (max)	1.50 (max)	1.50 (max)
Copper	0.50 (max)	0.50 (max)	0.50 (max)
Columbium	10 x carbon (min) 1.15 (max)	---	---
Titanium	---	6 x carbon (min) 0.75 (max)	---
Columbium plus tantalum	---	---	10 x carbon (min) 1.25 (max) <u>2/</u>
Iron	Remainder	Remainder	Remainder

1/ Small quantities of certain elements, which are not specified or required, may be present. The aggregate amount of such elements shall not exceed 0.5 percent.

2/ The determination of tantalum is not mandatory. However, if tantalum is determined, it shall not exceed 0.4 times the sum of the columbium plus the tantalum content.

3.4 Mechanical properties. The mechanical properties shall conform to the following requirements:

Mechanical Properties

Tensile Strength, PSI

100,000 (max)

Elongation, Percent in 2 Inches

40 (min)

3.4.1 Bending. Material shall withstand, without cracking, bending at room temperature through the angle indicated below around a diameter equal to the nominal thickness of the material, with axes of bends both perpendicular and parallel to the direction of rolling:

Nominal Thickness  
(inch)

0.249 and under

Over 0.249 to 0.749, incl.

Angle, Degrees  
(min)

180

90

3.5 Dimensions and tolerances. Dimensions shall be as specified by the contract or purchase order. Dimensional tolerances shall be as specified in Federal Standard No. 48.

3.6 Identification of product. Marking shall be in accordance with Standard MIL-STD-183. The marking legend shall include the following items:

Specification MIL-S-6721B  
Composition designation (Cb, Ti, or Cb-Ta)

3.7 Workmanship. Material shall be uniform in quality and condition, clean, sound, and free from scale and foreign materials, and free from defects (including seams, laminations, or blisters) detrimental to the fabrication or service life of parts.

#### 4. QUALITY ASSURANCE PROVISIONS

4.1 Inspection responsibility. The supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own or any other inspection facilities and services acceptable to the Government. Inspection records of the examination and tests shall be kept complete and available to the Government as specified in the contract or order. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements.

4.2 Classification of tests. All the test required herein for the testing of steel are classified as acceptance tests, for which necessary sampling techniques and methods of testing are specified in this section.

4.3 Examination of product. Sufficient spot checks shall be made to insure conformance to this specification with respect to identification, workmanship, and tolerance requirements.

#### 4.4 Chemical analysis.

4.4.1 Sampling. Samples for chemical analysis shall be selected by the inspector as described in Federal Test Method Standard No. 151, to represent each heat from which materials are presented for acceptance, at one time. A sample shall consist of not less than 2 ounces of material.

4.4.1.1 Samples for chemical analysis may be waived at the discretion of the inspector, provided that all of the material under inspection can be identified as being made from a heat previously analyzed and found to be in conformance to the chemical composition specified herein.

4.4.1.2 The method of selecting samples specified above is based on the assumption that the material is produced from ingots from the same heat at one time and is essentially homogeneous in all respects. If the material is taken from stock and is not identifiable as to heat and method of manufacture, or if the identity of any portion of the shipment is obscure in any respect, the inspector shall select the necessary additional samples to determine conformance of all portions of the shipment to this specification.

4.4.2 Method of analysis. Analysis shall be accomplished in accordance with Method 111.1 or 112.1, as applicable, of Federal Test Method Standard No. 151.

#### 4.5 Embrittlement test.

4.5.1 Sampling. Embrittlement tests will not normally be required. However, in the event the Government representative has reason to question the corrosion resistance of the materials, embrittlement tests shall be conducted on not more than two samples selected at random from each heat of steel represented. Two specimens not less than 3 inches in length by 1 inch wide shall be cut from the sample(s).

4.5.2 Method of test. The specimens shall be sensitized by heating within the temperature range of 1,020° to 1,050°F for 48 hours. After the sensitizing treatment and pickling to remove any scale which may have formed, acidified, copper sulfate solution of the following proportions:

Copper sulfate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ )	10 gm.
Sulfuric acid ( $\text{H}_2\text{SO}_4$ ) (sp.gr. 1.84)	10 ml.
Water (distilled)	90 ml.

(A reflux condenser or similar device shall be used to prevent changes in concentration of the solution.)

At least 35 ml. of the above solution shall be used for each square inch of exposed specimen surface. In all cases, however, the specimens shall be covered with solution.

#### 4.6 Tensile test.

4.6.1 Sampling. Two or more tensile test samples (one or more from each end of the coil) shall be selected from each coil. When material is produced in sheet or plate form, one or more tensile specimens shall be selected from each 100 sheets or fraction thereof of each lot of material produced under the same processing conditions, from the same heat, of the same physical condition, the same thickness, essentially uniform in all respects, and submitted for acceptance at one time.

4.6.2 Preparation of specimens. Tensile specimens shall be prepared to conform to type F1 or F2, Method 211.1 of Federal Test Method Standard No. 151, and shall be prepared with the long axis perpendicular to the direction of rolling when the width of the material will permit.

4.6.3 Method of test. Tensile tests shall be conducted in accordance with Method 211.1 of Federal Test Method Standard No. 151.

#### 4.7 Bending.

4.7.1 Sampling. Two or more bend test specimens, one longitudinal and one transverse, shall be selected from each coil, each lot of 100 sheets (or fraction thereof), or each lot of 10 plates (or fraction thereof); each lot to be of the same heat, thickness, and physical condition.

4.7.2 Method of test. Testing shall be in accordance with Method 231.1 of Federal Test Method Standard No. 151.



4.8 Rejection. Materials failing to meet all the requirements of this specification shall be rejected.

4.9 Preservation, packaging, packing, and marking. Preparation for delivery shall be inspected for conformance to section 5.

## 5. PREPARATION FOR DELIVERY

### 5.1 Preservation, packaging, and packing.

5.1.1 Level A. The material shall be properly separated by class and size when prepared for delivery. Materials shall be preserved and packaged in accordance with Standard MIL-STD-163.

5.1.2 Level C. Materials shall be packaged in accordance with commercial practice.

5.2 Marking of shipments. Marking and labeling shall be in accordance with Standard MIL-STD-163.



APPENDIX B

MATERIALS TRACEABILITY PROGRAM



## APPENDIX B

### MATERIALS TRACEABILITY PROGRAM

#### 1. PURPOSE

To establish a procedure for determining and implementing traceability requirements for identification and marking of parts and materials incorporated into TRW contract hardware items.

#### 2. GENERAL

Traceability shall be accomplished through the identification of customer supplied, Contractor purchased, or subcontractor furnished material. Material traceability will be maintained by the identification of the fabricated items or by reference to supporting documentation and records which trace the material's origin and history either by themselves or through intermediate documentation.

#### 3. POLICY

3.1 The Engineering design drawing will specify the traceability classification code for all items requiring serial numbers or lot traceability.

3.2 Where compiling lists of items requiring the application of traceability requirements, the following should be considered:

- (a) Matched items of hardware which may require replacement in sets.
- (b) Parts and/or components having a specific "life" time and cycle limitation.
- (c) All components requiring interchangeability at the black box or major assembly level.
- (d) Assemblies containing one or more serialized or lot controlled items.
- (e) Parts, components or assemblies which require unique data to be recorded and maintained.
- (f) Critical items or materials where judgement and experience indicate that defects would result in a safety hazard.

#### 4. RESPONSIBILITY

##### 4.1 Project Office

- (a) Establishes the general classifications of materials, parts and assemblies requiring traceability control.
- (b) Prepares and submits traceability control listing to the customer when required by contract.
- (c) Monitors the traceability assignments made by design organizations.

#### 4.2 Quality Assurance

- (a) Coordinates with Project Office as required on material traceability matters.
- (b) Monitors all material traceability activities to assure compliance with the requirements of this document.
- (c) Reviews, evaluates and approves vendor traceability procedures and lists.
- (d) Performs physical and chemical testing of raw materials as necessary.
- (e) Maintains surveillance over all storage, fabrication, inspection and test operations to verify continued validity of identification and traceability of materials.

#### 4.3 Functional Divisions

- (a) Establish and maintain material handling, storage, traceability accounting records, and controls and procedures to insure continued validity of prescribed traceability and identification.

### 5. PROCEDURE

#### 5.1 Project Office

- (a) The Project Office establishes and controls listing of parts and materials subject to traceability. The Project Office incorporates these determinations into a chart summary format similar to that shown in Figure B-1.

#### 5.2 Receiving Inspection

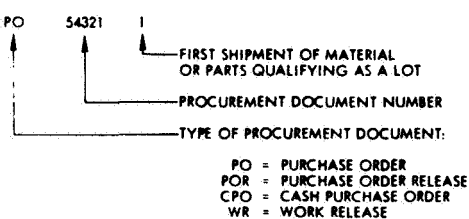
- (a) Complete quality data files are maintained on all procured contract materials whether such materials are inspected at the supplier's plant or upon receipt.
  - (1) Quality Inspection Verified Receiving Report
  - (2) Material Certificates
  - (3) Physical/Chemical Test Reports
  - (4) Non-Conforming Material Reports
  - (5) Miscellaneous Inspection and Test Data
- (b) When material traceability and identification are required by contract, lot control and serial numbers are referenced on all pertinent documentation.

#### 5.3 Fabrication and Assembly

- (a) All kits and material disbursements from stores or staging are inspected and traceability and identification verified as being noted on new or next documentation such as "Parts Accumulation List" and "Manufacturing Shop Orders".

TRACEABILITY IDENTIFICATION				REMARKS
LEVEL	REQUIREMENTS ASSIGNED BY	CLASSIFICATION DESIGNATION	PARTS	
APPLICABLE SYSTEM/ SUBSYSTEM (SPACECRAFT)	PROJECT OFFICE	SERIALIZED FLIGHT UNITS— 1001 AND UP, QUAL. UNITS— Q1001 AND UP; PROTO. UNITS— P1001 AND UP (CMP 3.2)	YES	SERIAL NUMBERS OF INDIVIDUAL PIECE PARTS, SUBASSEMBLIES WHICH MAKE UP THE END ITEM, PLUS TRACEABILITY RECORDS FROM THE ASSEMBLY LEVEL UP, COMPILED INTO THE NARRATIVE END ITEM REPORT.
MAJOR STL FABRICATED ASSEMBLIES	PROJECT OFFICE <input type="checkbox"/> ENGINEERING <input checked="" type="checkbox"/> SEE NOTE 2	SERIALIZED SERIAL NUMBER 001 AND UP (CMP 3.2)	YES	LOT CONTROL NUMBERS OF MATERIALS, SERIALIZED PIECE PARTS AND SUBASSEMBLIES WHICH MAKE UP THE ASSEMBLY, PLUS TRACEABILITY RECORDS COMPILED AND MAINTAINED BY THE QA DATA CENTER.
MAJOR SUBCONTRACTOR FABRICATED ASSEMBLIES (STL DRAWINGS)	SUPPLIER <input checked="" type="checkbox"/> SEE NOTE 2	SERIALIZED OPTIONAL (CMP 3.2)	YES	PREDELIVERY TRACEABILITY RECORDS RETAINED BY SUBCONTRACTOR, AFTER RECEIPT AT STL, TRACEABILITY MAINTAINED ON SUPPORTING TRACEABILITY DOCUMENTATION AND RECORDS. SUBCONTRACTOR'S MATERIAL TRACEABILITY SYSTEM SHALL BE APPROVED BY STL; RECORDS AND SYSTEM SUBJECT TO PERIODIC STL AUDIT.
MAJOR SUBCONTRACTOR FABRICATED ASSEMBLIES (SUPPLIER DRAWINGS)	SUPPLIER	SERIALIZED OPTIONAL (CMP 3.3)	YES	PREDELIVERY TRACEABILITY RECORDS RETAINED BY SUBCONTRACTOR, AFTER RECEIPT AT STL, TRACEABILITY MAINTAINED ON SUPPORTING TRACEABILITY DOCUMENTATION AND RECORDS. SUBCONTRACTOR'S MATERIAL TRACEABILITY SYSTEM SHALL BE REVIEWED BY STL; RECORDS AND SYSTEM SUBJECT TO PERIODIC STL AUDIT.
STL FABRICATED PIECE PARTS	QA TRACEABILITY CONTROL	SERIALIZED SEQUENTIAL NUMBERING SYSTEM	YES	TRACEABILITY RECORDS AND DOCUMENTS RETAINED BY THE COGNIZANT ACTIVITY
SUBCONTRACTOR FABRICATED PIECE PARTS (STL DRAWINGS)	QA TRACEABILITY CONTROL	SERIALIZED SEQUENTIAL NUMBERING SYSTEM	YES	PREDELIVERY TRACEABILITY RECORDS RETAINED BY SUBCONTRACTOR, AFTER RECEIPT AT STL, TRACEABILITY MAINTAINED ON SUPPORTING TRACEABILITY DOCUMENTATION AND RECORDS. SUBCONTRACTOR'S MATERIAL TRACEABILITY SYSTEM SHALL BE APPROVED BY STL; RECORDS AND SYSTEM SUBJECT TO PERIODIC STL AUDIT.
HIGH RELIABILITY SCREENED PARTS	HIGH RELIABILITY SCREENING SEC.	SERIALIZED SEQUENTIAL NUMBERING SYSTEM	YES	TEST DATA RECORDS RETAINED BY HIGH RELIABILITY SCREENING SECTION.
STANDARD HARDWARE ITEMS (AN AND MS, ETC.)	RECEIVING INSPECTION	LOT NUMBER SEE NOTE 1	NO	PART LOT CONTROL NUMBER ASSIGNED BY RECEIVING INSPECTION, TRACEABILITY IDENTIFICATION MARKED ON INDIVIDUAL CONTAINERS. LOT NUMBER NOTED ON STORES RECORDS AND TRANSCRIBED TO DISBURSEMENT DOCUMENT(S) UPON ISSUE.
COMMERCIAL OFF- THE-SHELF ITEMS	EXEMPT	N/A	NO	
RAW MATERIAL	RECEIVING INSPECTION	LOT NUMBER SEE NOTE 1	YES	RAW MATERIAL LOT IDENTIFIED UPON RECEIPT, IDENTIFICATION MAINTAINED DURING STORAGE, TRACEABILITY IDENTIFICATION TRANSCRIBED TO DISBURSEMENT DOCUMENT UPON ISSUE. TRACEABILITY MAINTAINED THROUGH INTERMEDIATE DOCUMENTS THEREAFTER.
LIMITED SHELF LIFE ITEMS	RECEIVING INSPECTION	PART LOT NUMBER AND DATE SEE NOTE 1	YES	INDIVIDUAL ITEMS AND/OR CONTAINERS LOT IDENTIFIED BY RECEIVING UPON RECEIPT. ADDITIONALLY, DATE CRITICAL LIFE WAS INITIATED AND/OR DATE USEFUL LIFE WILL BE EXPENDED IS DESIGNATED THEREON.

NOTE 1 — LOT NUMBER



NOTE 2 — SYMBOLS

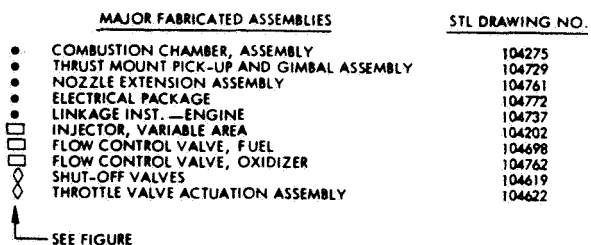


FIGURE B-1. TYPICAL TRACEABILITY SUMMARY CHART

### 5.3 Fabrication and Assembly (continued)

- (b) A Manufacturing Data Package is initiated by the preparation of Manufacturing and Quality Assurance copies of the MSO and PAL. The Quality Assurance copies of these documents are provided for inclusion in the Data Package.
- (c) All documentation initiated during the course of the manufacturing, assembly and inspection which reflect the manufacturing, configuration, serialization, or quality history of contract material are recorded on the MSO or PAL (or their equivalent). Inspection personnel concerned with or initiating such documents are responsible for the recording, verification and/or insertions of copies in the data package. Quality Assurance keeps the data package up-to-date.
- (d) The MDP must accompany the material with which it is associated at all times, including rework and Material Review.
- (e) When the last inspection operation is completed, the inspector verifies that copies of all applicable documents are accounted for, validates the MSO, and forwards the data package to Quality Assurance Data Center. Quality Inspection will attach an Acceptance Tag to the hardware signifying complete and accurate records, and perform configuration verification as required.
- (f) Throughout all operations, material removed and replaced must be documented to ensure that the line of traceability and identification is not lost or broken.



APPENDIX C

IHCOS ENGINEERING DESIGN DRAWINGS



APPENDIX C

IHCOS ENGINEERING DESIGN DRAWINGS

BE100-00 Isotope Heated Catalytic Oxidizer Assembly  
BE100-02 Screen Lower  
BE100-03 Screen Upper  
BE100-04 Cover  
BE100-05 Body Assembly  
BE100-06 Body Outer  
BE100-07 Fin  
BE100-08 Base Flange  
BE100-01 Body Inner  
BE100-09 Inlet-Outlet Duct  
BE100-10 Inlet-Outlet Duct Seal Plate  
BE100-11 H-X Insulation Upper  
BE100-12 H-X Insulation Lower  
BE100-13 Body Insulation  
BE100-14 Cover Insulation  
BE100-15 Heat Exchanger  
BE100-16 Outer Canister  
BE100-17 Canister Cover  
BE100-18 Spring  
X403254 Resistively Heated IHCOS Heat Source Assembly\*  
X402727 Member, Reentry  
X403255 Fin, Structural Module  
X403256 Sleeve, Structural Module  
X403257 Cap, End Structural Module  
X403258 Member, Transition  
X403259 Sleeve, Clad  
X403260 Cap, End-Clad  
X403261 Member, Strength\*  
X403262 Sleeve, Liner\*  
X403263 Cap, End-Liner\*  
X403264 Element, Heater  
X403553 Core, Ceramic  
BE100-19 Pre-Sorbent Canister Assembly  
BE100-20-301 Cover  
BE100-21 Spring  
BE100-22-305 Compression Plate  
BE100-23-301 Body  
BE100-24-301 Screen Lower  
BE100-25 Post-Sorbent Canister Assembly  
BE100-20-305 Cover  
BE100-21 Spring  
BE100-22-301 Compression Plate  
BE100-23-305 Body  
BE100-24-305 Screen Lower

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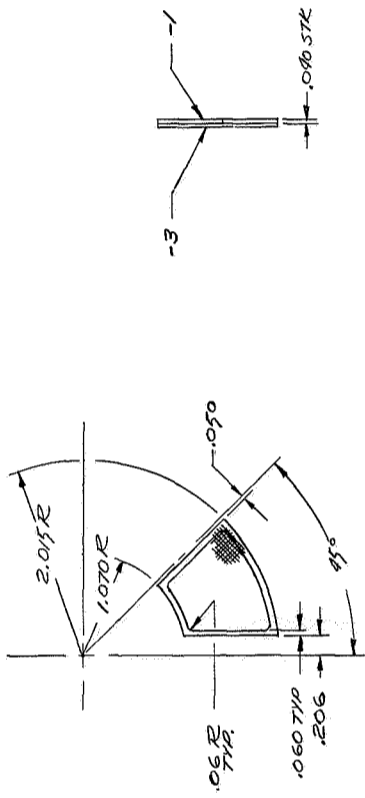
\* Presented in Classified Summary.







CHANGE EFFECT		REVISIONS		DATE		APPROVED	
MODEL	SERIAL	SYM	ZONE	DESCRIPTION	DATE	DATE	APPROVED



QTY REQD	CODE IDENT	PART OR IDENTIFYING NO.	NOMENCLATURE OR DESCRIPTION	MATERIAL OR NOTE	SPECIFICATION	ITEM NO.
8		-3	SCREEN	230 MESH CRS 316		
8		-1	FRAME	.321 CRCS .040	MIL-5-6721 COMP 21	

**LIBERTEE**  
 MISSILES & SPACE COMPANY  
 DIVISION OF LIBERTEE AIRCRAFT CORPORATION  
 SUNNYVALE, CALIFORNIA

DATE: 5-27-68  
 DR: RWJ/gy  
 CHK: SUPV  
 STRESS: VALUE ENGRG  
 RELIABILITY: APPROVED  
 APPROVED:

SCREEN-LOWER  
 14205

INTERPRET THIS DRAWING PER STANDARDS IN MIL-D-70327	UNLESS OTHERWISE SPECIFIED TOLERANCES ON:	DATE	CHK	STRESS	VALUE ENGRG	RELIABILITY	APPROVED
	FRACTIONS = ± 1/16 .X = ± .1 .XX = ± .03 .XXX = ± .010 ANGLES = ± 2°	5-27-68	RWJ/gy				

APPLICATION	USED ON	NEXT	FINAL	RECD/ASSY
BE100-00	8	8	8	8

1. SPOT WELD ITEM 14-3 PER LMC M21 CLASS C  
 SPACED APPROX. 25  
**NOTE**





CHANGE EFFECT		REVISIONS	
MODEL	SERIAL	DESCRIPTION	DATE
MODEL	SERIAL	DESCRIPTION	DATE

QTY	CODE	PART OR IDENTIFYING NO.	NOMENCLATURE OR DESCRIPTION	MATERIAL OR NOTE	SPECIFICATION	ZONE	ITEM NO.
1		-5	SCREEN	330 MESH 316 CREES			
1		-3	RING	321 CREES .0407K	MIL-S-6721 (COMP 321)		
1		-1	RING	321 CREES .0407K	MIL-S-6721 (COMP 321)		

INTERPRET THIS DRAWING PER STANDARDS IN MIL-D-70327

UNLESS OTHERWISE SPECIFIED

TOLERANCES ON:

FRACTIONS = ± 1/16

XX = ± .1

XXX = ± .03

ANGLES = ± 30

DATE 5-27-68

DR R W Joy

CHK

SUPV

STRESS

VALUE ENGRG

RELIABILITY

APPROVED

APPROVED

BE100-03

NEXT ASSY

USED ON

REGD/ASSY

06887

C

BE100-03

SCALE 1/100

SHEET 1 OF 1

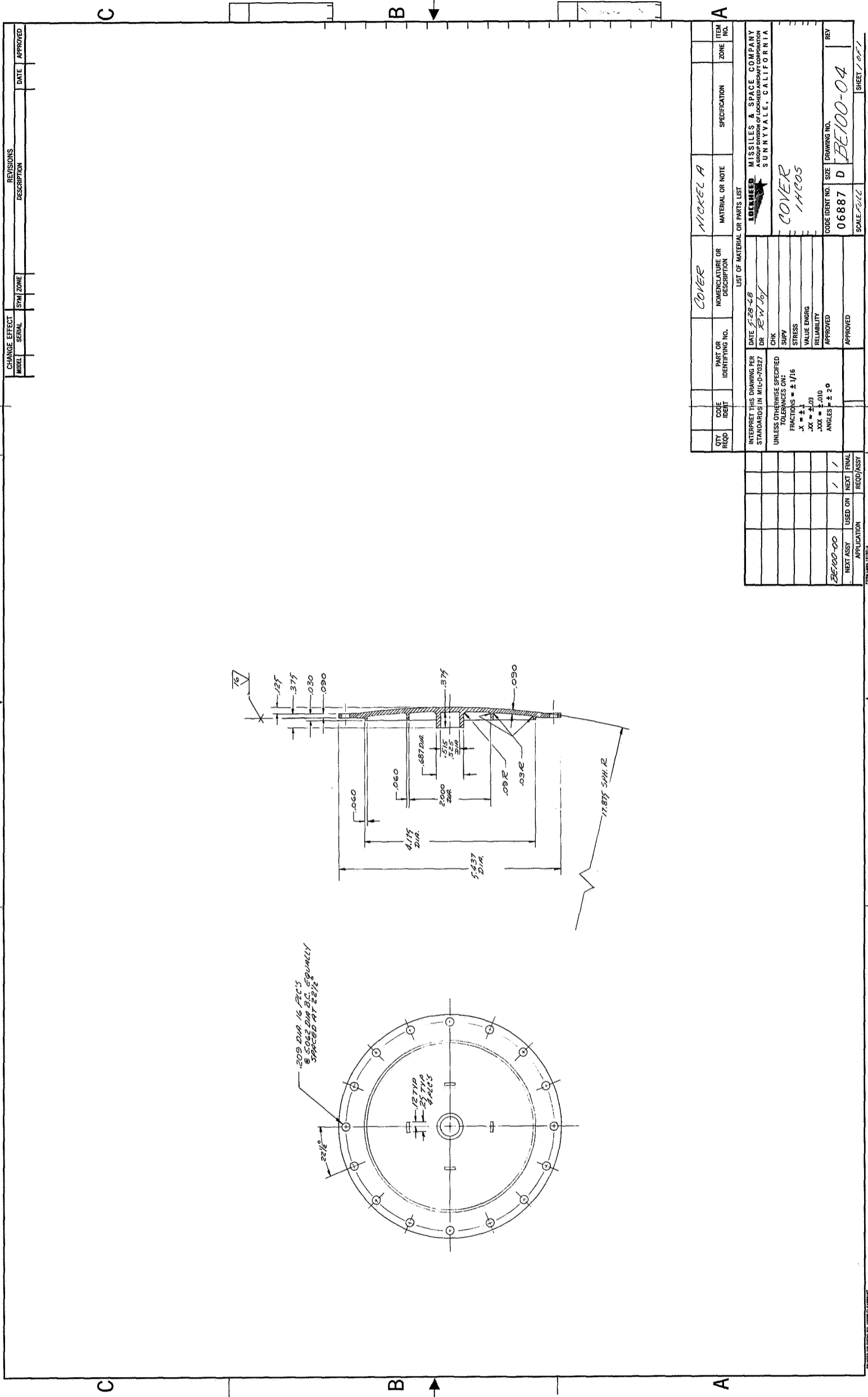
  

1. SPOT WELD TO 316-1.31.5 PER 602.101 CLASS C  
SPACED APPROX. 1.5" BETWEEN RINGS

**NOTE**

FORM LING 1113C-4 PRINTED ON WOOD GLASSPRINT





CHANGE EFFECT		REVISIONS		DATE		APPROVED	
MODEL	SERIAL	REV	ZONE	DESCRIPTION	DATE	APPROVED	

QTY	CODE	PART OR IDENTIFYING NO.	COVER	MATERIAL OR NOTE	SPECIFICATION	ZONE	TECH
REQD	IDENT		NUMBER				NO.
<p>LIST OF MATERIAL OR PARTS LIST</p> <p>LOCKHEED MISSILES &amp; SPACE COMPANY A GROUP DIVISION OF LOCKHEED AIRCRAFT CORPORATION SUNNYVALE, CALIFORNIA</p> <p>COVER 1HC05</p>							
<p>INTERPRET THIS DRAWING PER DATE 5-28-68 STANDARDS IN MIL-D-70327 DR RNY/67</p> <p>UNLESS OTHERWISE SPECIFIED</p> <p>FINISHES 94</p> <p>FRACTIONS ± 1/16</p> <p>ANGLES ± 1°</p> <p>STRESS</p> <p>VALUE ENGR</p> <p>RELIABILITY</p> <p>APPROVED</p>							
<p>CODE IDENT NO. SIZE DRAWING NO.</p> <p>06887 D BE100-04</p>							
<p>SCALE: FULL</p>							

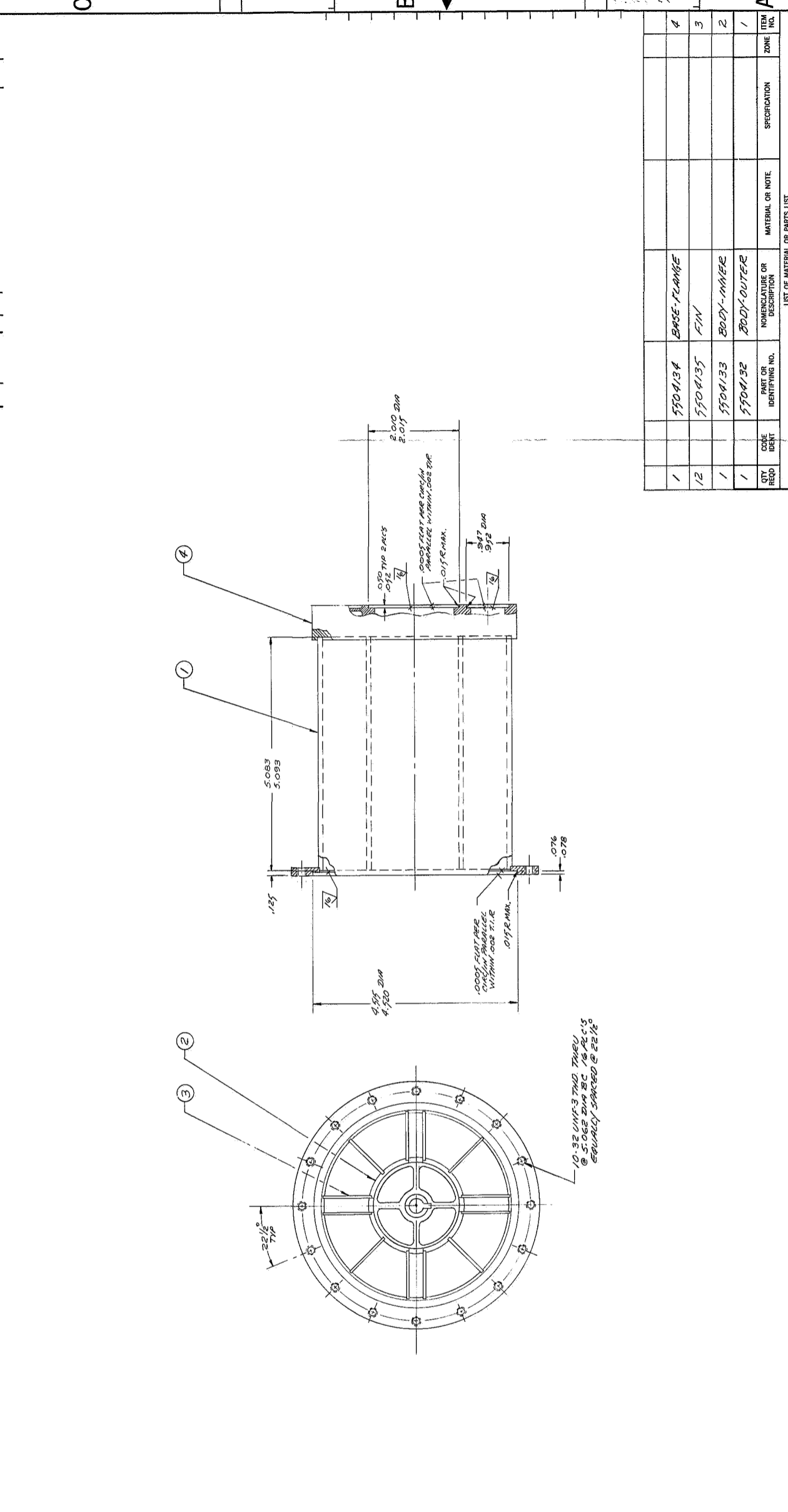
FIGURE 11522



CHANGE EFFECT		SYN ZONE		REVISIONS	
MODEL	SERIAL	SYN	ZONE	DESCRIPTION	DATE

QTY REQD	CODE IDENT	PART OR IDENTIFYING NO.	NOMENCLATURE OR DESCRIPTION	MATERIAL OR NOTE	SPECIFICATION	ZONE	ITEM NO.
1		5704134	BASE-FLANGE				4
12		5704135	FIN				3
1		5704133	BODY-INNER				2
1		5704132	BODY-OUTER				1

QTY REQD	CODE IDENT	PART OR IDENTIFYING NO.	NOMENCLATURE OR DESCRIPTION	MATERIAL OR NOTE	SPECIFICATION	ZONE	ITEM NO.
1		5704134	BASE-FLANGE				4
12		5704135	FIN				3
1		5704133	BODY-INNER				2
1		5704132	BODY-OUTER				1



QTY REQD	CODE IDENT	PART OR IDENTIFYING NO.	NOMENCLATURE OR DESCRIPTION	MATERIAL OR NOTE	SPECIFICATION	ZONE	ITEM NO.
1		5704134	BASE-FLANGE				4
12		5704135	FIN				3
1		5704133	BODY-INNER				2
1		5704132	BODY-OUTER				1

INTERPRET THIS DRAWING PER STANDARDS IN MIL-D-70327 UNLESS OTHERWISE SPECIFIED TOLERANCES ON:		DATE: 5-21-68		DR: R/W/JOY	
FRACTURES	± 1/16	CHK			
STRESS		SUPV			
VALUE ENGRG					
RELIABILITY					
APPROVED					
CODE IDENT NO.	06887	DRAWING NO.	BE100-05		
SCALE	1/2" = 1"				

QTY REQD	CODE IDENT	PART OR IDENTIFYING NO.	NOMENCLATURE OR DESCRIPTION	MATERIAL OR NOTE	SPECIFICATION	ZONE	ITEM NO.
1		5704134	BASE-FLANGE				4
12		5704135	FIN				3
1		5704133	BODY-INNER				2
1		5704132	BODY-OUTER				1

3. MARCH PER LMC 3601  
 2. MARCH AFTER BEARING  
 1. BEARING PER CAD 1126 TYPE II CLASS 3  
 NOTE

MISSILES & SPACE COMPANY  
 A GROUP DIVISION OF LOCKHEED AIRCRAFT CORPORATION  
 SUNNYVALE, CALIFORNIA

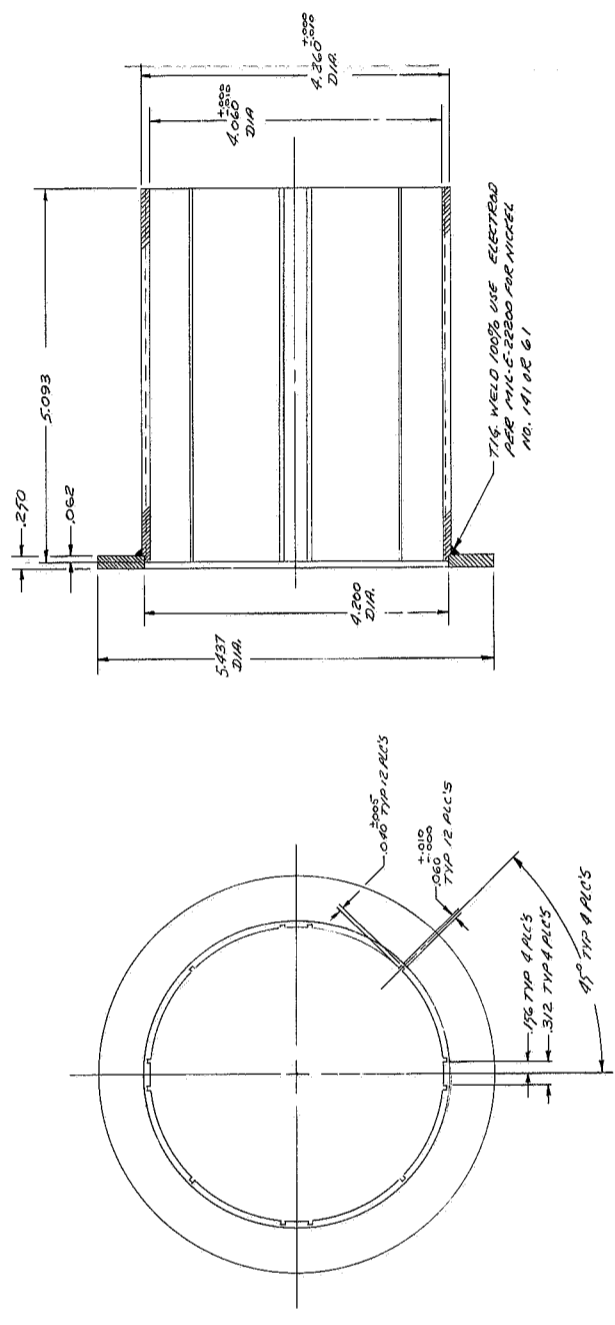
BODY ASSEMBLY  
 11005

SCALE: 1/2" = 1"

SHEET 1 OF 1



CHANGE EFFECT		REVISIONS		DATE	
MODEL	SERIAL	SWI	ZONE	DESCRIPTION	APPROVED



QTY REQD	CODE IDENT	PART OR IDENTIFYING NO.	NOMENCLATURE OR DESCRIPTION	MATERIAL OR NOTE	SPECIFICATION	ZONE	ITEM NO.
1			BODY	NICKEL A			

INTERPRET THIS DRAWING PER STANDARDS IN MIL-D-70327		DATE 5-27-68		DR R.W.J.	
UNLESS OTHERWISE SPECIFIED	STRESS SUPPLY	CHK	DATE	DR	DR
TOLERANCES ON:	VALUE ENGRG	STRESS	STRESS	RELIABILITY	APPROVED
FRACTIONS = ± 1/16	X = ± .1	REL	REL	REL	REL
DECIMALS = ± .001	XXX = ± .010	REL	REL	REL	REL
ANGLES = ± 2°					

APPLICATION	USED ON	NEXT	FRM	REQD/ASSY
BE100-07				

CODE IDENT	PART OR IDENTIFYING NO.	NOMENCLATURE OR DESCRIPTION	MATERIAL OR NOTE	SPECIFICATION	ZONE	ITEM NO.

LIST OF MATERIAL OR PARTS LIST		DATE 5-27-68		DR R.W.J.	

MISSILES & SPACE COMPANY		A GROUP DIVISION OF LOCKHEED AIRCRAFT CORPORATION		SUNNYVALE, CALIFORNIA	

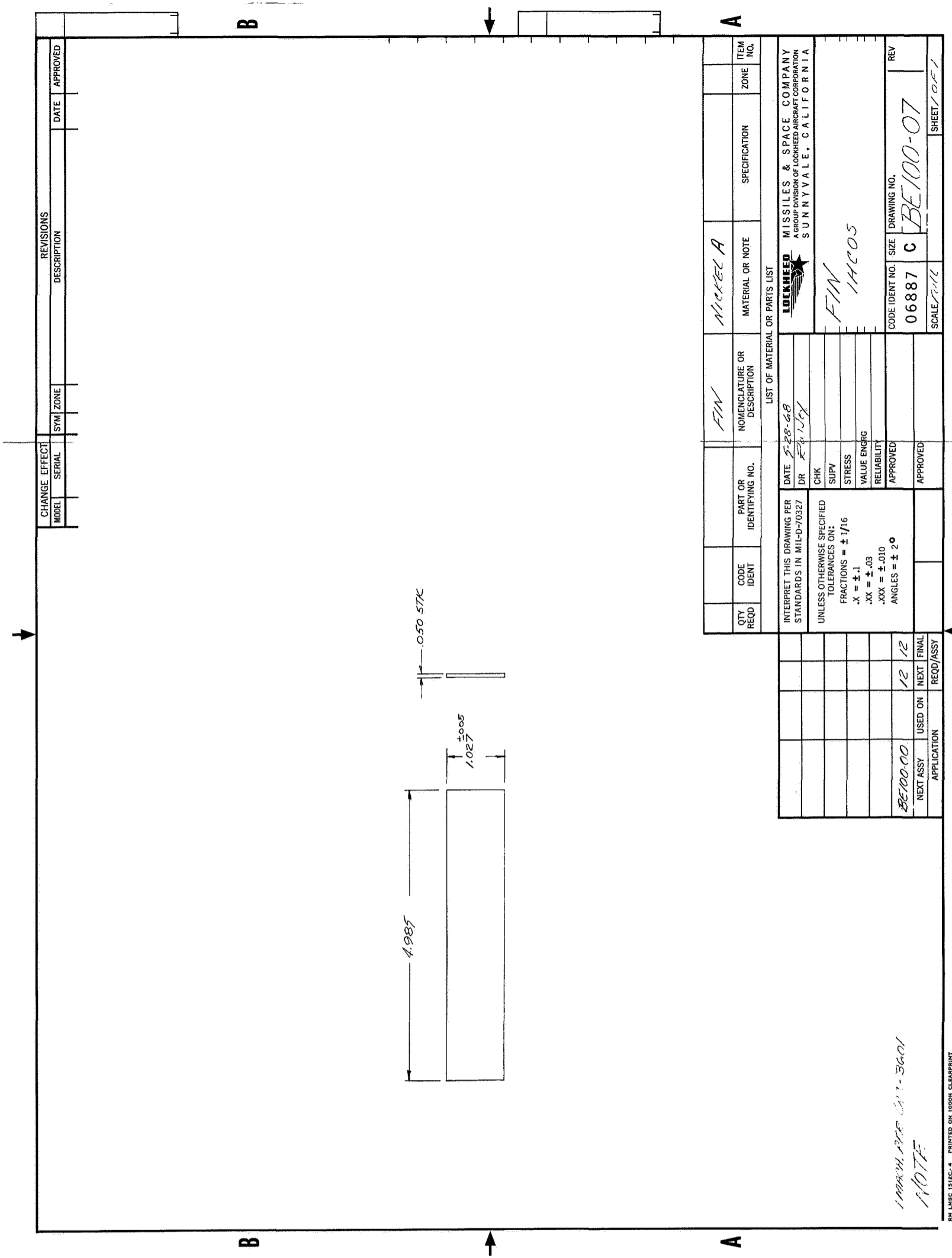
CODE IDENT NO.	SIZE	DRAWING NO.	REV
06887	D	BE100-06	

SCALE	SCALE FULL	SHEET	TOTAL

NOTE







CHANGE EFFECT		REVISIONS		DATE		APPROVED	
MODEL	SERIAL	SYM	ZONE	DESCRIPTION			

QTY REQD	CODE IDENT	PART OR IDENTIFYING NO.	NOMENCLATURE OR DESCRIPTION	MATERIAL OR NOTE	SPECIFICATION	ZONE	ITEM NO.
			FIN	Nickel A			

LIST OF MATERIAL OR PARTS LIST	
DATE	28-68
DR	R. J. J.
CHK	
SUPV	
STRESS	
VALUE ENGRG	
RELIABILITY	
APPROVED	
APPROVED	

INTERPRET THIS DRAWING PER STANDARDS IN MIL-D-70327	
UNLESS OTHERWISE SPECIFIED	
TOLERANCES ON:	
FRACTIONS = ± 1/16	
.X = ± .1	
.XX = ± .03	
.XXX = ± .010	
ANGLES = ± 2°	

APPLICATION	USED ON	NEXT	FINAL	RECD/ASSY
BE100.00	12	12	12	

CODE IDENT NO.	SIZE	DRAWING NO.	REV
06887	C	BE100-07	

SCALE	SHEET
1/16	1 OF 1

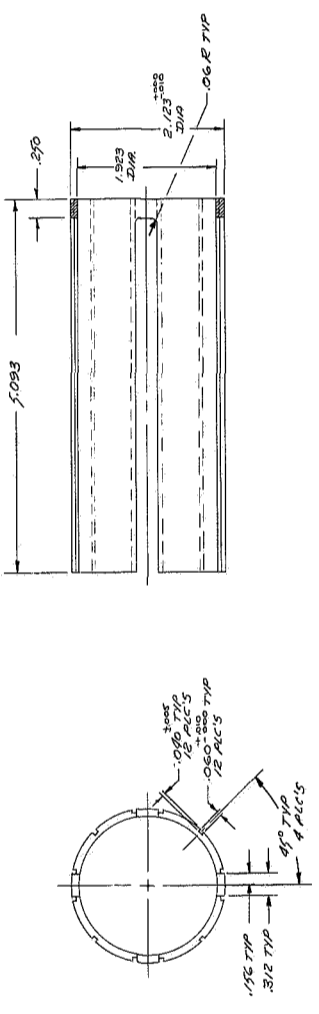
1 MAR 24 1968  
 NOTE  
 1-3601







CHANGE EFFECT		REVISIONS		DATE	
MODEL	SERIAL	SYM	ZONE	DESCRIPTION	APPROVED



QTY	CODE	IDENTIFYING NO.	PART OR IDENTIFYING NO.	NOMENCLATURE OR DESCRIPTION	MATERIAL OR NOTE	SPECIFICATION	ZONE	ITEM NO.	REVISIONS	
									DATE	DESCRIPTION
1				BODY	NICKEL A					

INTERPRET THIS DRAWING PER STANDARDS IN MIL-D-10227		DATE		LIST OF MATERIAL OR PARTS LIST	
QTY	RECD	DR	CHK	DATE	DESCRIPTION
		R-1126		5-27-68	BODY-INNER

NOTE

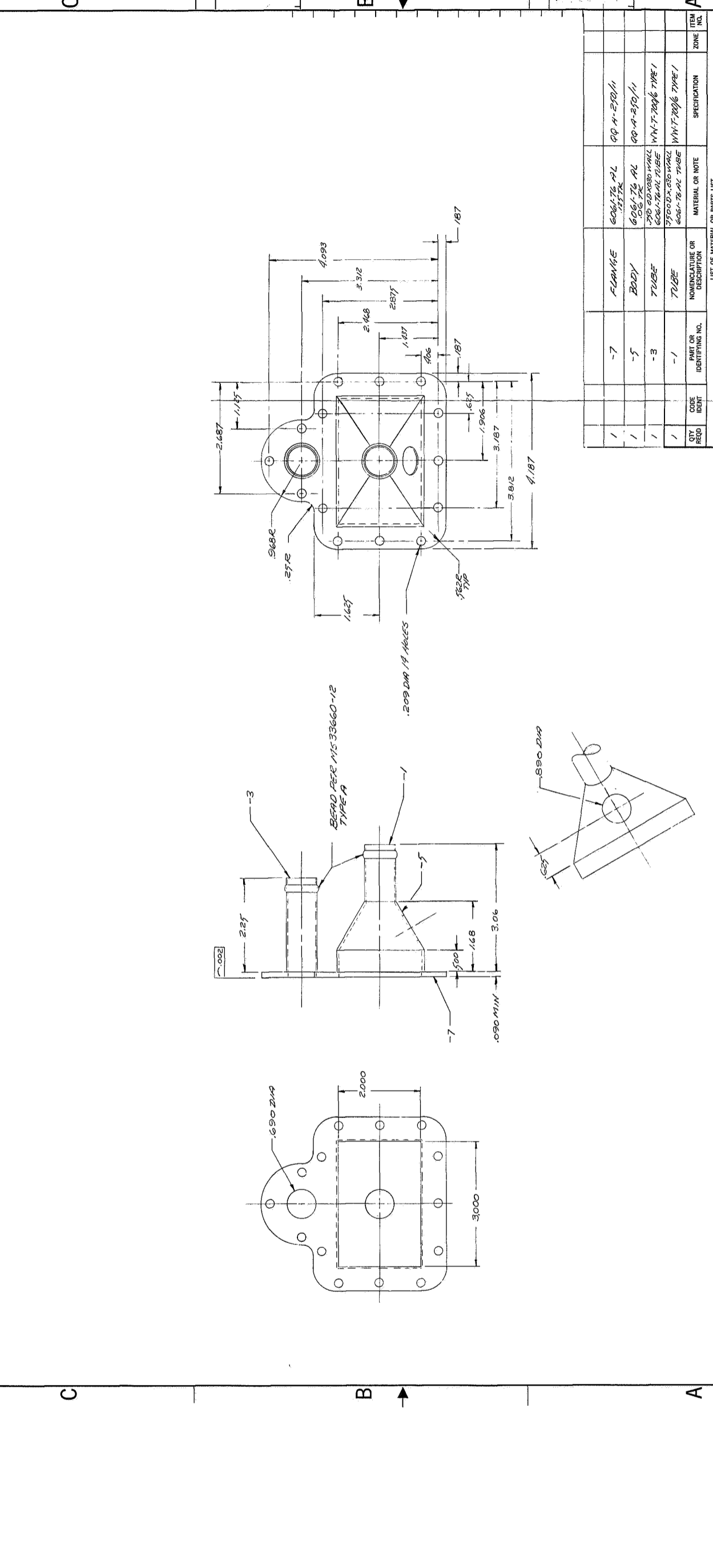
PRINTED ON RECYCLED PAPER



CHANGE EFFECT		REVISIONS		DATE	
MODEL	SERIAL	SYM	ZONE	DESCRIPTION	APPROVED

QTY	RECD	CODE IDENT	PART OR IDENTIFYING NO.	NOMENCLATURE OR DESCRIPTION	MATERIAL OR NOTE	SPECIFICATION	ZONE	ITEM NO.
1			-7	FLANGE	6061-T6 AL	QQ-A-270-11		
1			-5	BODY	6061-T6 AL	QQ-A-270-11		
1			-3	TUBE	3003-O35 AL	WM-T-7006 TYPE 1		
1			-1	TUBE	6061-T6 AL	WM-T-7006 TYPE 1		

QTY	RECD	CODE IDENT	PART OR IDENTIFYING NO.	NOMENCLATURE OR DESCRIPTION	MATERIAL OR NOTE	SPECIFICATION	ZONE	ITEM NO.
1			-7	FLANGE	6061-T6 AL	QQ-A-270-11		
1			-5	BODY	6061-T6 AL	QQ-A-270-11		
1			-3	TUBE	3003-O35 AL	WM-T-7006 TYPE 1		
1			-1	TUBE	6061-T6 AL	WM-T-7006 TYPE 1		



INTERPRET THIS DRAWING PER STANDARDS IN MIL-D-70327		LIST OF MATERIAL OR PARTS LIST	
UNLESS OTHERWISE SPECIFIED	FRACCTIONS = $\pm 1/16$	DATE 10-7-68	DR R W J
.X = $\pm .1$	.XX = $\pm .03$	CHK	
.XXX = $\pm .010$	ANGLES $\pm 2^\circ$	SUPY	
		STRESS	
		VALVE ENGRG	
		RELIABILITY	
		APPROVED	
		APPROVED	

3. FINISH PER CAC-099A TYPE I  
 2. MARK AFTER WELDING PER CAC-5601  
 1. ALL JOINTS 100% WELD PER CAC-1422

NOTE

MISSILES & SPACE COMPANY  
 A GROUP DIVISION OF LOCKHEED AIRCRAFT CORPORATION  
 SUNNYVALE, CALIFORNIA

INLET-OUTLET DUCT  
 111005

CODE IDENT NO. SIZE DRAWING NO.  
 06887 D BL100-09

SCALE 2:1

SHEET 1 OF 1

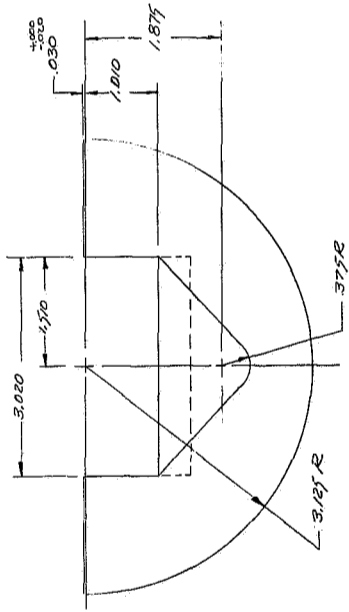
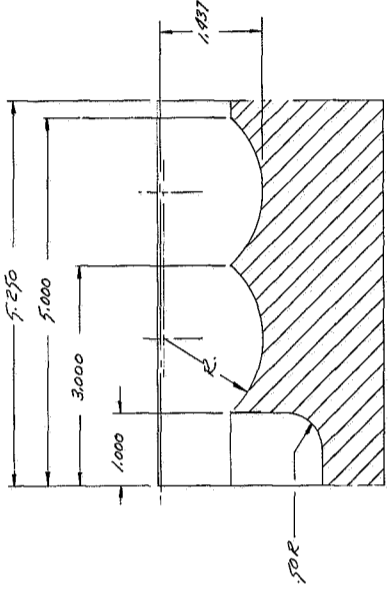








CHANGE EFFECT		REVISIONS		DATE	
MODEL	SERIAL	SYM	ZONE	DESCRIPTION	APPROVED



RECOMMENDED VENDOR JAMES MANNVILLE AEROSPACE PRODUCTS NEW YORK N.Y.  
 NOTE

QTY REQD	CODE IDENT	PART OR IDENTIFYING NO.	NOMENCLATURE OR DESCRIPTION	MATERIAL OR NOTE	SPECIFICATION	ZONE	ITEM NO.
			INSULATION	MIN-K 1301			

LIST OF MATERIAL OR PARTS LIST  
 DATE 10-9-68  
 BY R.M.J.

INTERPRET THIS DRAWING PER STANDARDS IN MIL-D-70327  
 UNLESS OTHERWISE SPECIFIED TOLERANCES ON:  
 FRACTIONS = ±.116  
 DECIMALS = ±.003  
 ANGLES = ±.2°  
 APPROVED

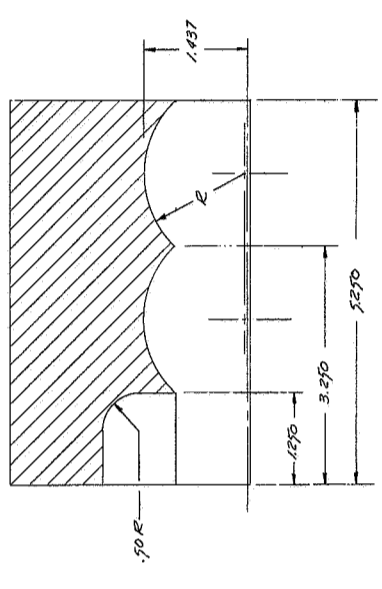
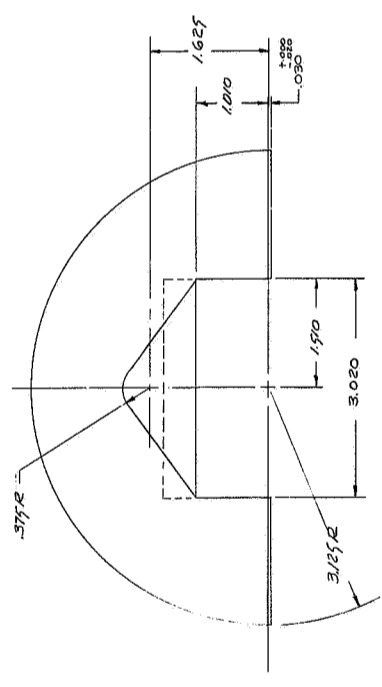
MISSILES & SPACE COMPANY  
 A GROUP DIVISION OF LOCKHEED AIRCRAFT CORPORATION  
 SUNNYVALE, CALIFORNIA

INSULATION-HEAT EXCHANGER UPPER  
 CODE IDENT NO. 06887 D  
 DRAWING NO. BE100-11  
 SCALE 1/2"=1"



CHANGE EFFECT		REVISIONS	
NO.	SERIAL	SYMBOL	DESCRIPTION

DATE	APPROVED



1. RECOMMENDED VENDOR  
 JOHNS-MANVILLE AEROSPACE  
 PRODUCTS, NEWYORK N.Y.  
 NOTE

QTY	CODE	IDENTIFYING NO.	NOMENCLATURE OR DESCRIPTION	MATERIAL OR NOTE	SPECIFICATION	ZONE	ITEM NO.
1			INSULATION	MM-K 1301			

DATE	CHK	DATE	CHK
10-9-68	DR	10-9-68	DR
	SRV		SRV
	STRESS		STRESS
	VALUE ENGRG		VALUE ENGRG
	APPROVED		APPROVED

CODE IDENT NO.	SIZE	DRAWING NO.	REV
06887	D	BE100-12	

SCALE	SHEET
1/2" = 1"	1 OF 1

MISSILES & SPACE COMPANY  
 1000 W. 10TH AVENUE  
 SUNNYVALE, CALIFORNIA



CHANGE EFFECT		REVISIONS		DATE		APPROVED	
MODEL	SERIAL	SYM	ZONE	DESCRIPTION			
ILLUSTRATION PROCESSING INFORMATION PREPARED BY: <b>NMA CR-6679</b> DATE: <b>5/7</b> DRAWING NO.: <b>133</b> LOCKHEED AIRCRAFT CORPORATION FROM: <b>LOCKHEED</b>							
<b>1</b> RECOMMENDED VENDOR: <b>JOHNS-MANNVILLE</b> <b>AEROSPACE PRODUCTS NEW YORK N.Y.</b> <b>NOTE:</b>							

QTY REQD	CODE IDENT	PART OR IDENTIFYING NO.	NOMENCLATURE OR DESCRIPTION	MATERIAL OR NOTE	SPECIFICATION	ZONE	ITEM NCL
1			INSULATION	MIN-K-1301			

LIST OF MATERIAL OR PARTS LIST	
INTERPRET THIS DRAWING PER STANDARDS IN MIL-D-70327 UNLESS OTHERWISE SPECIFIED TOLERANCES ON: FRACTIONS = $\pm 1/16$ .X = $\pm .1$ .XX = $\pm .03$ .XXX = $\pm .010$ ANGLES = $\pm 2^\circ$	DATE: <b>10-8-68</b> DR: <b>EM/ky</b> CHK: _____ SUPV: _____ STRESS: _____ VALUE ENGR: _____ RELIABILITY: _____ APPROVED: _____
NEXT ASSY: <b>BE100-00</b> APPLICATION: _____	USED ON: _____ NEXT FINAL RECD/ASSY: <b>2 2</b>

CODE IDENT NO.	SIZE	DRAWING NO.	REV
06887	C	BE100-13	

<b>LOCKHEED</b> <b>MISSILES &amp; SPACE COMPANY</b> A GROUP DIVISION OF LOCKHEED AIRCRAFT CORPORATION SUNNYVALE, CALIFORNIA	<b>INSULATION-BODY</b> <b>14005</b>
--	--

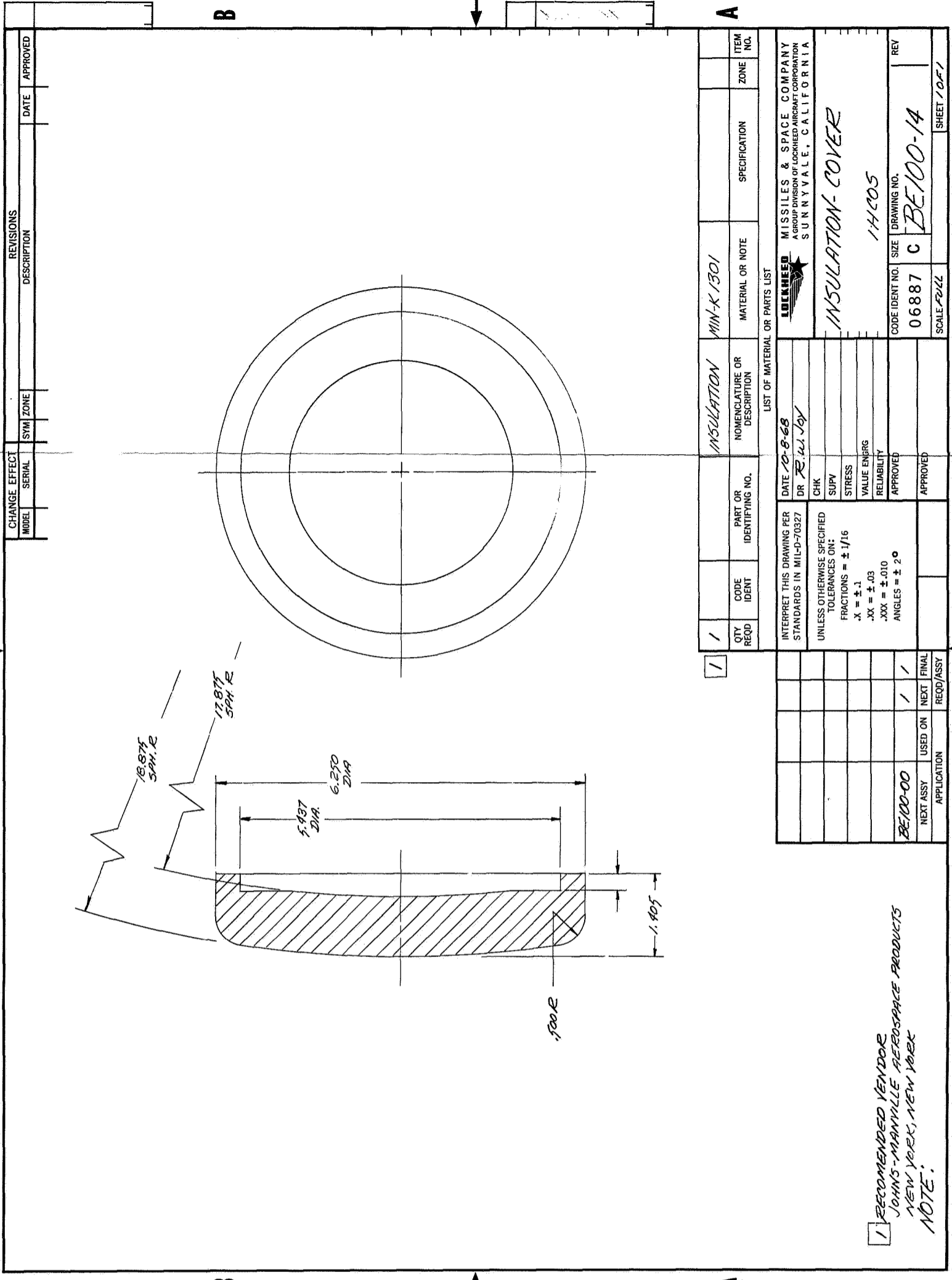
  

SCALE: <b>FULL</b>	SHEET: <b>1 OF 1</b>
--------------------	----------------------

FORM LAMC 1514C-4 PRINTED ON 100% RECYCLED PAPER







CHANGE EFFECT		REVISIONS	
MODEL	SERIAL	SYM	ZONE

DATE	APPROVED

QTY REQD	CODE IDENT	PART OR IDENTIFYING NO.	NOMENCLATURE OR DESCRIPTION	MATERIAL OR NOTE	SPECIFICATION	ZONE	ITEM NO.
1			INSULATION	MIN-K 1301			

DATE	CHK	STRESS	VALUE ENGRG	RELIABILITY	APPROVED
10-8-68	DR R. L. Joy				

QTY REQD	CODE IDENT	PART OR IDENTIFYING NO.	NOMENCLATURE OR DESCRIPTION	MATERIAL OR NOTE	SPECIFICATION	ZONE	ITEM NO.
1			INSULATION	MIN-K 1301			

MISSILES & SPACE COMPANY  
 A GROUP DIVISION OF LOCKHEED AIRCRAFT CORPORATION  
 SUNNYVALE, CALIFORNIA

INTERPRET THIS DRAWING PER  
 STANDARDS IN MIL-D-70327  
 DR R. L. Joy  
 UNLESS OTHERWISE SPECIFIED  
 FRACTIONS = ± 1/16  
 .X = ± .1  
 .XX = ± .03  
 .XXX = ± .010  
 ANGLES = ± 2°

INSULATION COVER  
 11/105  
 CODE IDENT NO. 06887 C  
 DRAWING NO. BE100-14  
 SCALE PULL  
 SHEET 1 OF 1

1 RECOMMENDED VENDOR  
 JOHN'S-MANNVILLE AEROSPACE PRODUCTS  
 NEW YORK, NEW YORK  
 NOTE:

FORM LK6C 1515C-4 PRINTED ON 100% RECYCLED PAPER

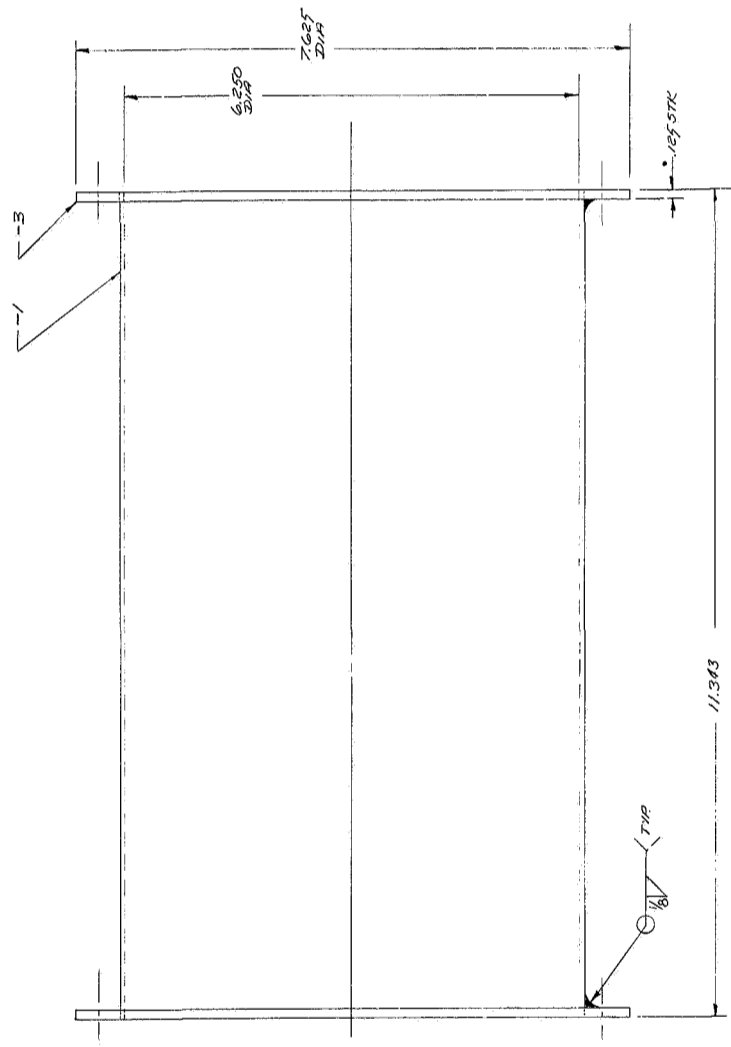
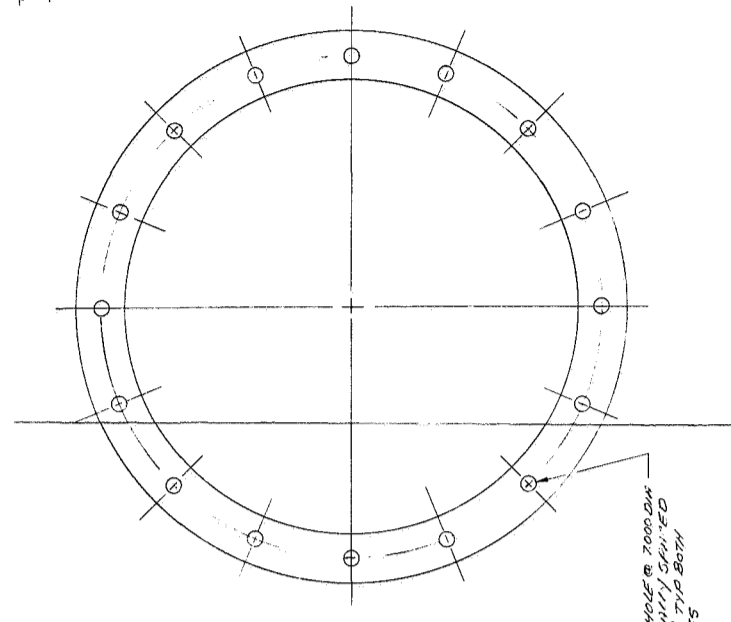






CHANGE EFFECT		REVISIONS		DATE APPROVED	
MODEL	SERIAL	SYM	ZONE	DESCRIPTION	DATE

ILLUSTRATION PROCESSING INFORMATION  
 DRAWING NO. 06887  
 NRSR 02-14-61  
 DATE 2/1/61  
 VNA  
 MISSILES & SPACE COMPANY  
 SUNNYVALE, CALIFORNIA



QTY REQD	CODE IDENT	PART OR IDENTIFYING NO.	NOMENCLATURE OR DESCRIPTION	MATERIAL OR NOTE	SPECIFICATION	ZONE	ITEM NO.
2		-3	FLANGE	6061-T6 AL 1/2 STK	QQ-A-250/11		
1		-1	BODY	6061-T6 AL 1/2 STK	QQ-A-250/11		

INTERPRET THIS DRAWING PER STANDARDS IN MIL-D-10327 UNLESS OTHERWISE SPECIFIED		DATE	CHK
TOLERANCES ON:		10-2-58	DR RWJ
FRACTIONS	$\pm 1/16$		
X	$\pm .1$		
XX	$\pm .03$		
XXX	$\pm .010$		
ANGLES	$\pm 2^{\circ}$		

APPROVED	DATE	BY

APPROVED	DATE	BY

APPROVED	DATE	BY

APPROVED	DATE	BY

APPROVED	DATE	BY

APPROVED	DATE	BY

APPROVED	DATE	BY

APPROVED	DATE	BY

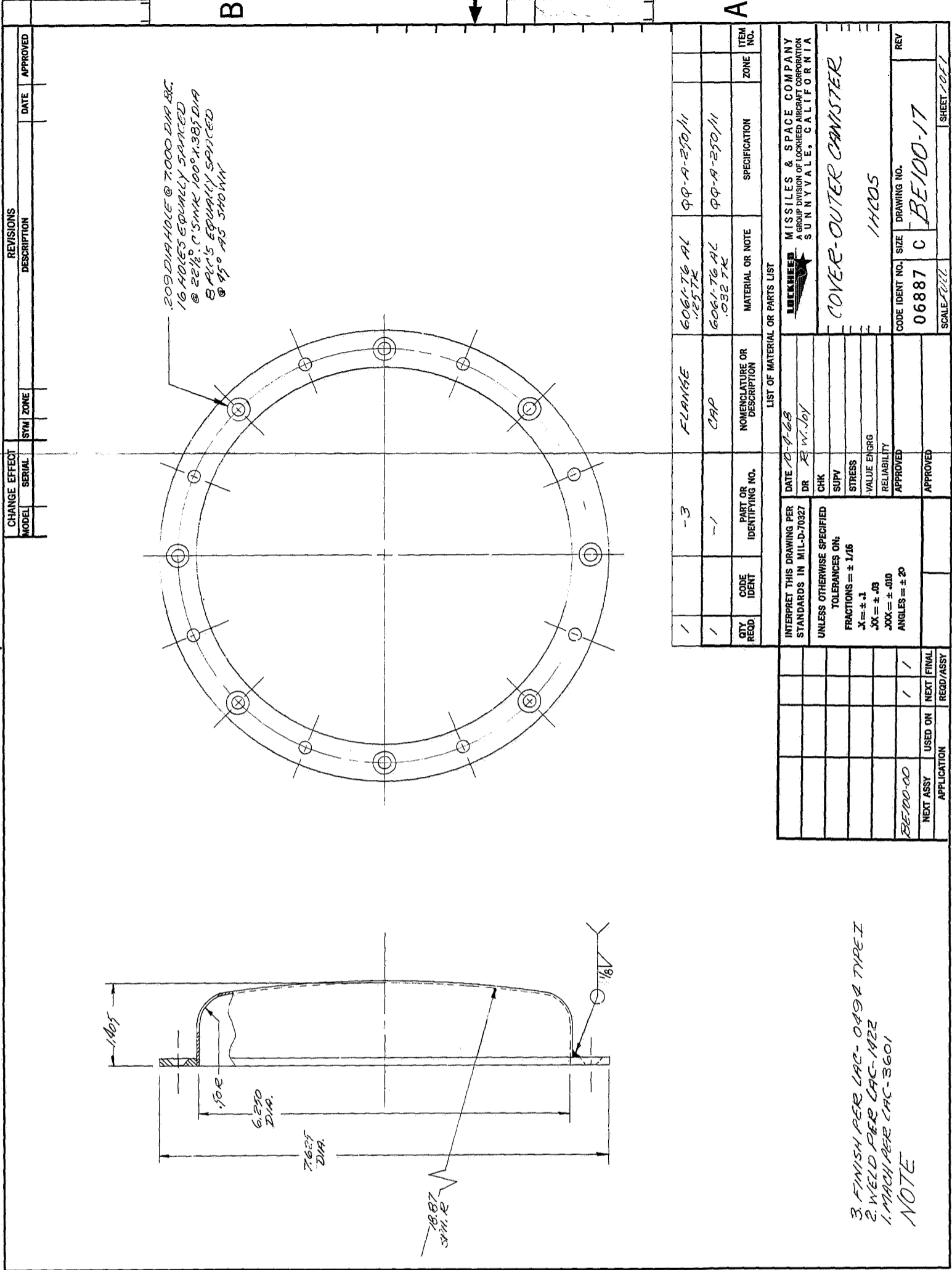
APPROVED	DATE	BY

APPROVED	DATE	BY

3. FINISH PER LAC-0994 TYPE I  
 2. MACH PER LAC-3601  
 1. WELD PER LAC-1422  
 NOTE

MISSILES & SPACE COMPANY  
 SUNNYVALE, CALIFORNIA  
 OUTER CANISTER  
 14805  
 CODE IDENT NO. 06887  
 SIZE D  
 DRAWING NO. BE100-16  
 SCALE 2:1  
 SHEET 1 OF 1





CHANGE EFFECT		REVISIONS		APPROVED	
MODEL	SERIAL	DESCRIPTION	DATE	DATE	DATE

QTY	CODE IDENT	PART OR IDENTIFYING NO.	NOMENCLATURE OR DESCRIPTION	MATERIAL OR NOTE	SPECIFICATION	ZONE	ITEM NO.
1		-3	FLANGE	6061-T6 AL .032 TK	99-A-250/11		
1		-1	CAP	6061-T6 AL .032 TK	99-A-250/11		

INTERPRET THIS DRAWING PER STANDARDS IN MIL-D-70327 UNLESS OTHERWISE SPECIFIED

TOLERANCES ON:  
 FRACTIONS = ± 1/16  
 X = ± .1  
 XX = ± .08  
 XXX = ± .010  
 ANGLES = ± 20

DATE 10-9-68  
 DR R. W. JOY  
 CHK

MISSILES & SPACE COMPANY  
 A GROUP DIVISION OF LOCKHEED AIRCRAFT CORPORATION  
 SUNNYVALE, CALIFORNIA

COVER-OUTER CANISTER  
 111005

CODE IDENT NO. 06887  
 SIZE C  
 DRAWING NO. BE100-17

SCALE 1:1

SHEET 1 OF 1

APPLY	USED ON	NEXT FINAL	RECD/ASSY
BE100-00	1	1	1

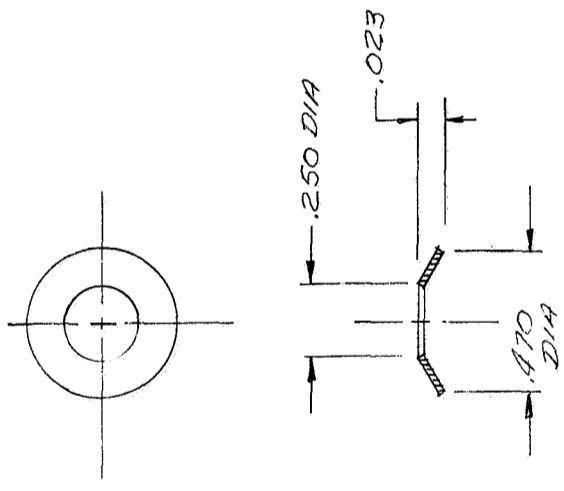
FORM LACS 1183C-4 PRINTED ON ISO CLEARPRINT





CHANGE EFFECT		REVISIONS	
MODEL	SERIAL	SYM	ZONE

DESCRIPTION	DATE	APPROVED



QTY REQD	CODE IDENT	PART OR IDENTIFYING NO.	NOMENCLATURE OR DESCRIPTION	MATERIAL OR NOTE	SPECIFICATION	ZONE	ITEM NO.
6			SPRING	UDIMET 700 .015 TK			

LIST OF MATERIAL OR PARTS LIST

<b>LOCKHEED</b> MISSILES & SPACE COMPANY A GROUP DIVISION OF LOCKHEED AIRCRAFT CORPORATION SUNNYVALE, CALIFORNIA	
DATE	10-24-68
DR	R.W. Joy
CHK	
SUPV	
STRESS	
VALUE ENGRG	
RELIABILITY	
APPROVED	
APPROVED	

INTERPRET THIS DRAWING PER STANDARDS IN MIL-D-70327

UNLESS OTHERWISE SPECIFIED TOLERANCES ON:  
 FRACTIONS = ± 1/16  
 X = ± .1  
 .XX = ± .03  
 .XXX = ± .010  
 ANGLES = ± 2°

CODE IDENT NO.	06887	SIZE	B	DRAWING NO.	BE100-18	REV	
SCALE		NONE		SHEET		1 OF 1	

APPLICATION	USED ON	NEXT	FINAL
BE100-00	6	6	6







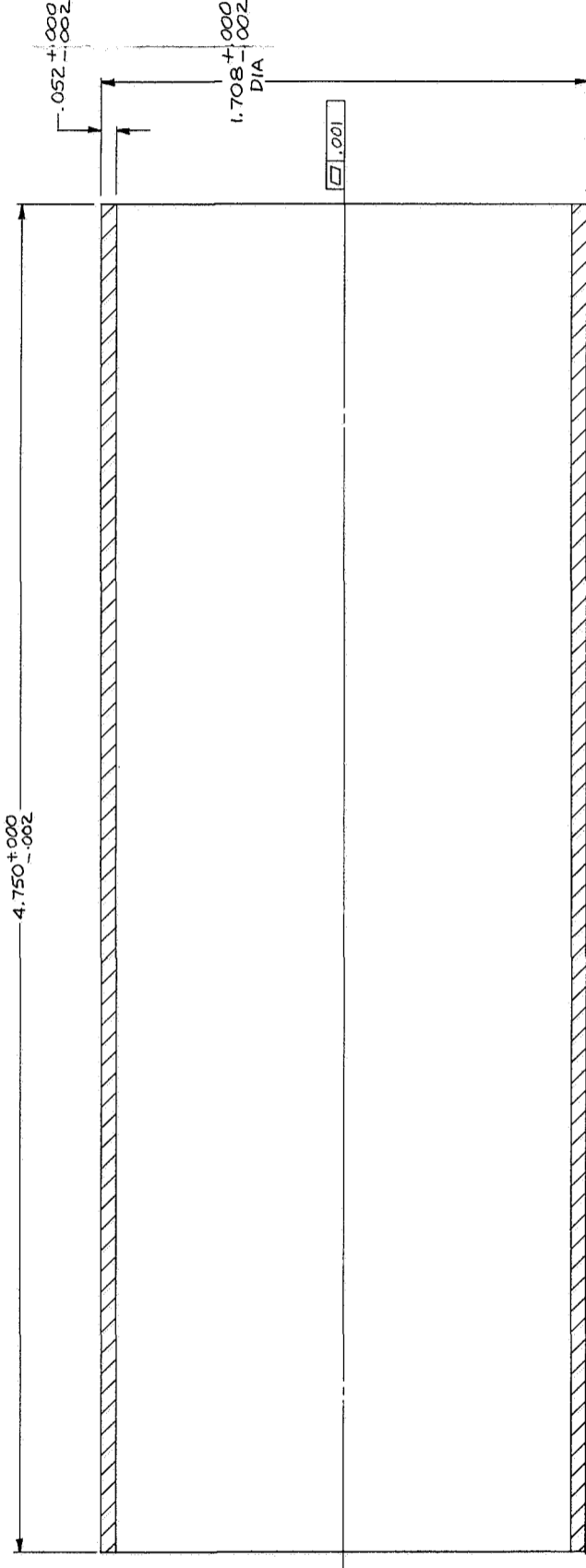




8 7 6 5 4 3 2 1

THE INFORMATION AND TECHNICAL DATA DISCLOSED BY THIS DOCUMENT MAY BE USED WITHOUT RESTRICTION BY AND FOR THE UNITED STATES GOVERNMENT WHEN DELIVERED UNDER GOVERNMENT CONTRACT EXCEPT WHERE A LIMITED RIGHTS LEGEND PER ASPR 4802 APPEARS ON THIS DOCUMENT, AND MAY BE USED BY OTHER CUSTOMERS OF THE SYSTEM GROUP WHEN THE INFORMATION AND TECHNICAL DATA DISCLOSED BY THIS DOCUMENT ARE PROPRIETARY TO THE SYSTEM GROUP AND MAY NOT BE USED, REPRODUCED, OR DISSEMINATED TO OTHERS EXCEPT WHERE NECESSARY TO COMPLY WITH A SYSTEM GROUP CONTRACT OR AS AUTHORIZED IN WRITING BY THE SYSTEM GROUP.

ZONE	LTR	DESCRIPTION	DATE	APPROVED



X403256

QTY REQD PER ASSY CONFIGURATION	PART OR IDENTIFYING NO.	PART NAME	STOCK	NOMENCLATURE OR DESCRIPTION	MATERIAL	SPEC	ELEC REF DES	CODE IDENT	ZONE	ITEM NO.
	-1	SLEEVE		1.750 O.D. X 0.083 WALL	TYPE 321 CRES TUBING	MIL-T-8808				

FINISH: PASSIVATE PER PR 2-9  
HEAT TREAT

UNLESS OTHERWISE SPECIFIED:  
 1. INTERFERE FIT UNLESS NOTED  
 2. DIMENSIONS ARE IN INCHES  
 3. SURFACE TEXTURE: 63  
 4. ALL DIMS TO APPLY BEFORE PLATING  
 5. REMOVE BURRS & SHARP EDGES  
 6. TOLERANCES ON DECIMALS:  
 .005 ± .002  
 .01 ± .003  
 .02 ± .004  
 .03 ± .005  
 .04 ± .006  
 .05 ± .007  
 .06 ± .008  
 .07 ± .009  
 .08 ± .010  
 .09 ± .011  
 .10 ± .012  
 .125 ± .015  
 .15 ± .018  
 .175 ± .021  
 .20 ± .024  
 .25 ± .030  
 .30 ± .036  
 .375 ± .045  
 .50 ± .056  
 .75 ± .084  
 1.00 ± .125  
 1.50 ± .188  
 2.00 ± .250

APPROVALS:  
 CHECKED: R. T. ...  
 SUPERVISOR: ...  
 ENGINEER: R. T. ...  
 DESIGNER: ...  
 DATE: 11-1-88

SCALE: 4/1

TRW  
 FITTING DIVISION  
 ONE BRACE PARK - REDWOOD BEACH, CALIFORNIA

SLEEVE, STRUCTURAL MODULE  
 CODE IDENT: D 11982  
 SIZE: X403256

1. IDENTIFICATION MARKING PER PR 12-1  
 TYPE II CLASS IG PART NUMBER

NOTES: UNLESS OTHERWISE SPECIFIED

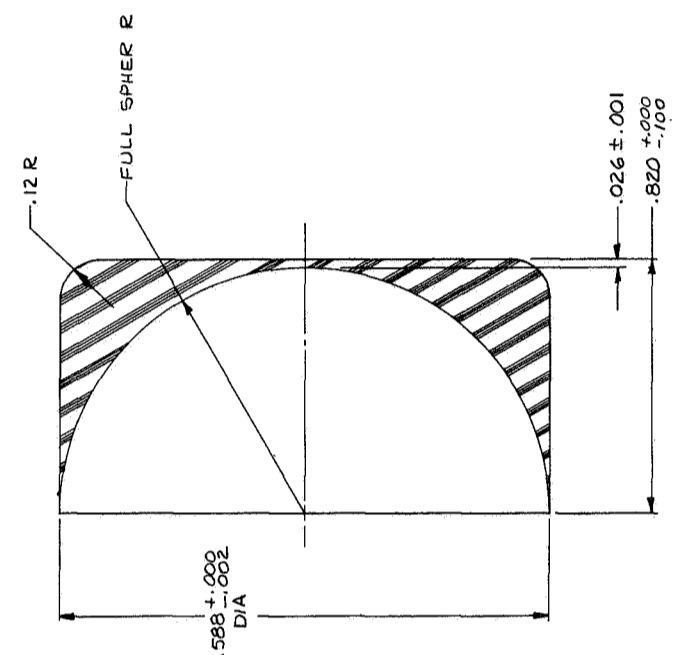
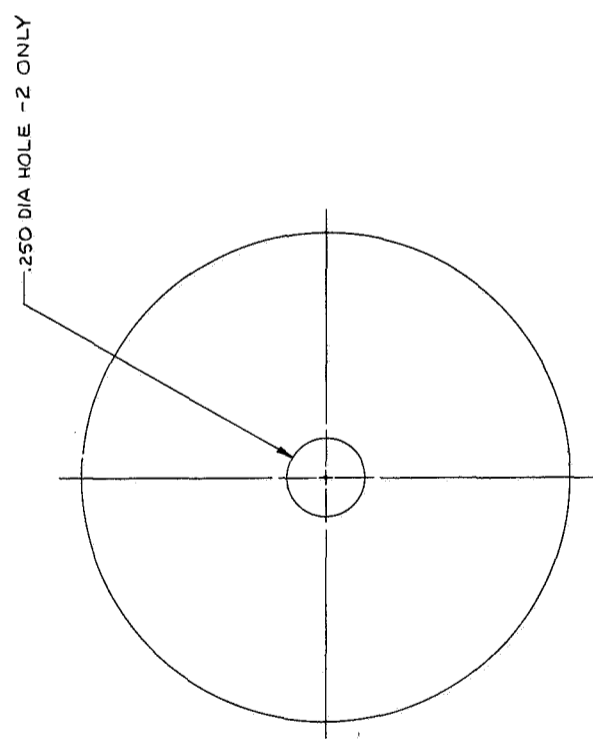








THE INFORMATION AND TECHNICAL DATA DISCLOSED BY THIS DOCUMENT MAY BE USED WITHOUT RESTRICTION BY AND FOR THE UNITED STATES GOVERNMENT WHEN DELIVERED UNDER GOVERNMENT CONTRACT EXCEPT WHERE A LIMITED RIGHTS LEGEND PER APRI 5301 ALSO APPEARS ON THIS DOCUMENT, AND MAY BE USED BY OTHERS EXCEPT WHERE NECESSARY TO COMPLY WITH A SYSTEMS GROUP CONTRACT OR AS AUTHORIZED IN WRITING BY THE SYSTEMS GROUP.



-1 MEMBER - SHOWN  
 -2 MEMBER - IDENTICAL EXCEPT AS SHOWN

ZONE LTR	REVISIONS DESCRIPTION	DATE	APPROVED

X403258

QTY REQD PER ASSY CONFIGURATION	PART OR IDENTIFYING NO.	PART NAME	STOCK	NOMENCLATURE OR DESCRIPTION	MATERIAL	ITEM NO.	ZONE
	-2	MEMBER		1.625 DIA X .820	ATJ GRAPHITE	2	
	-1	MEMBER		1.625 DIA X .820	ATJ GRAPHITE	2	

PARTS LIST	
UNLESS OTHERWISE SPECIFIED	DO NOT SCALE DRAWING
1. DIMENSIONS ARE IN INCHES	CHECKED <i>R. Traylor</i> F-13-8
2. DIMENSIONS ARE IN INCHES	STRUCTURES
3. SURFACE TEXTURE	EXPANSION
4. ALL DIMS TO APPLY BEFORE PLATING	TEMPER
5. REMOVE BURRS & SHARP EDGES	FINISH
6. TOLERANCES ON DECIMALS	OTHER
7. STD HOLE TOLERANCES:	APPROVALS
.001 - .010	
.010 - .030	
.030 - .060	
.060 - .125	
.125 - .250	
.250 - .500	
.500 - 1.000	
1.000 - 2.000	
2.000 - 3.000	
3.000 - 4.000	
4.000 - 5.000	
5.000 - 6.000	
6.000 - 7.000	
7.000 - 8.000	
8.000 - 9.000	
9.000 - 10.000	

FINISH	HEAT TREAT	APPLICABLE SPECIFICATIONS	APPLY	APPLY	APPLY
		PR17-1			

QTY REQD PER ASSY CONFIGURATION	PART OR IDENTIFYING NO.	PART NAME	STOCK	NOMENCLATURE OR DESCRIPTION	MATERIAL	ITEM NO.	ZONE

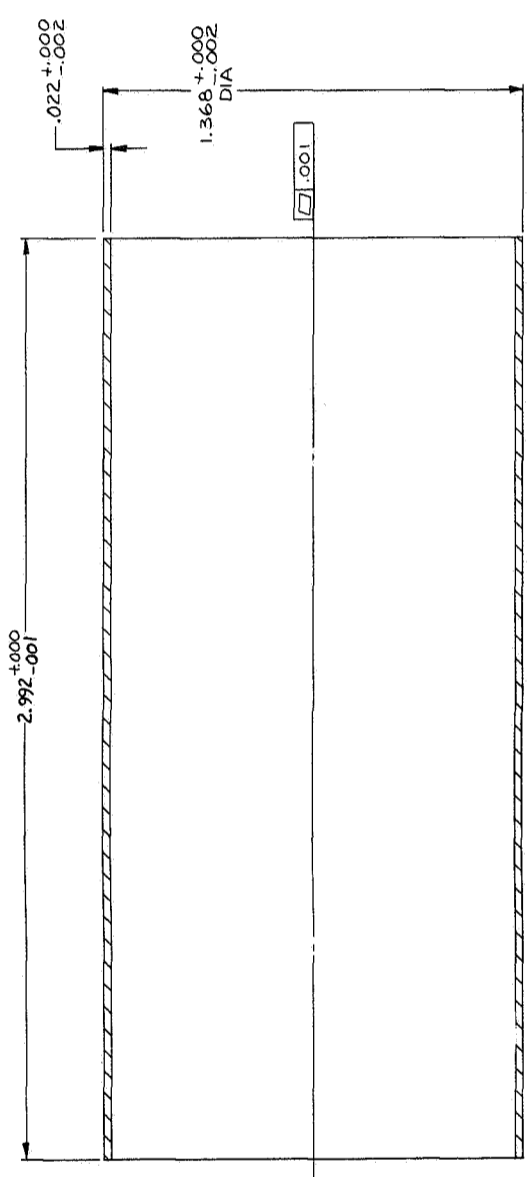
2. MATERIAL SPECIFICATIONS LISTED IN IHCOS DESIGN AND DEVELOPMENT TASK I  
 1. IDENTIFICATION MARKING PER PR 12-1  
 TYPE III CLASS IIS - PART NUMBER  
 NOTES: UNLESS OTHERWISE SPECIFIED

MEMBER, TRANSITION  
 D 11982 X403258  
 SCALE



8 7 6 5 4 3 2 1

THIS INFORMATION AND TECHNICAL DATA DISCLOSED IN THIS DOCUMENT MAY BE USED WITHOUT RESTRICTION BY AND FOR THE UNITED STATES GOVERNMENT WHEN DELIVERED UNDER GOVERNMENT CONTRACT, EXCEPT WHERE A LIMITED RIGHTS LEGEND PER ASPR 480 ALSO APPEARS ON THIS DOCUMENT, AND MAY BE USED BY OTHER CUSTOMERS OF THE UNITED STATES GOVERNMENT WITHOUT RESTRICTION. THE INFORMATION AND TECHNICAL DATA DISCLOSED IN THIS DOCUMENT ARE PROPRIETARY TO THE SYSTEMS GROUP AND MAY NOT BE USED, REPRODUCED, OR DISSEMINATED TO OTHERS EXCEPT WHERE NECESSARY TO COMPLY WITH A SYSTEMS GROUP CONTRACT OR AS AUTHORIZED IN WRITING BY THE SYSTEMS GROUP.



ZONE	LTR	DESCRIPTION	DATE	APPROVED

REVISIONS	DESCRIPTION	DATE	APPROVED

X403259

QTY REQD PER ASSY CONFIGURATION	PART OR IDENTIFYING NO.	PART NAME	STOCK	NOMENCLATURE OR DESCRIPTION	MATERIAL	ITEM NO.
	-1	SLEEVE	1.368 DIA X .022 WALL	Pt-20 R.H. SEAMLESS TUBING		

FINISH	HEAT TREAT	APPLICABLE SPECIFICATIONS
		PR12-1

USED ON	NEXT ASSY	APPLICATION
IHCOS	X403254	(1)

DO NOT SCALE DRAWING	THE FOLLOWING LOG HAVE BEEN ATTACHED TO THIS PRINT

TRW	ONE SPACE MARK - RECORD BEACH, CALIFORNIA

2. MATERIAL SPECIFICATIONS LISTED IN IHCOS DESIGN AND DEVELOPMENT TASK I

1. IDENTIFICATION MARKING PER PR 123 TYPE II CLASS LOG PART NUMBER

NOTES: UNLESS OTHERWISE SPECIFIED

SLEEVE, CLAD  
D 11982 X403259  
SCALE 4/1

SYSTEM FORM REV. 4-67 SHEET 4/1

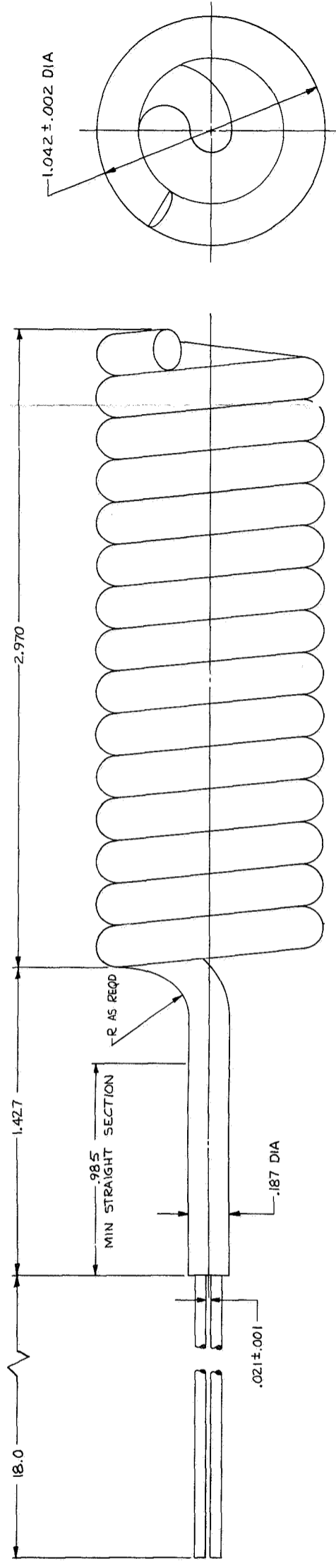








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ZONE LTR	REVISIONS DESCRIPTION	DATE	APPROVED

CITY REQD PER ASSY CONFIGURATION	PART OR IDENTIFYING NO.	PART NAME	STOCK	NOMENCLATURE OR DESCRIPTION	MATERIAL	ITEM NO.
		ELEMENT				

DO NOT SCALE DRAWING	THE FOLLOWING EDS HAVE BEEN ATTACHED TO THIS PRINT

UNLESS OTHERWISE SPECIFIED	DO NOT SCALE DRAWING
1. INTERPRET PER MIL-STD-883C	
2. DIMENSIONS IN INCHES	
3. SURFACE TEXTURE	
4. ALL DIMS TO APPLY BEFORE PLATING OR CONVERSION COATING	
5. DIMENSIONS TO SHARP EDGES	
6. TOLERANCES ON DECIMALS	
7. STD HOLE TOLERANCES	
8. ANGLES UNLESS OTHERWISE SPECIFIED	
9. DIMENSIONS TO CENTER UNLESS OTHERWISE SPECIFIED	
10. DIMENSIONS TO SURFACE UNLESS OTHERWISE SPECIFIED	
11. DIMENSIONS TO CENTER UNLESS OTHERWISE SPECIFIED	
12. DIMENSIONS TO SURFACE UNLESS OTHERWISE SPECIFIED	

FINISH	PLATING	PLATING SPECIFICATIONS	OTHER APPROVALS

APPROVALS	DATE	APPROVED

1. IDENTIFICATION MARKING PER PR 12-1  
 TYPE II CLASS 15 PART NUMBER  
 NOTES: UNLESS OTHERWISE SPECIFIED

X403264

ELEMENT, HEATER

D 11982 X403264

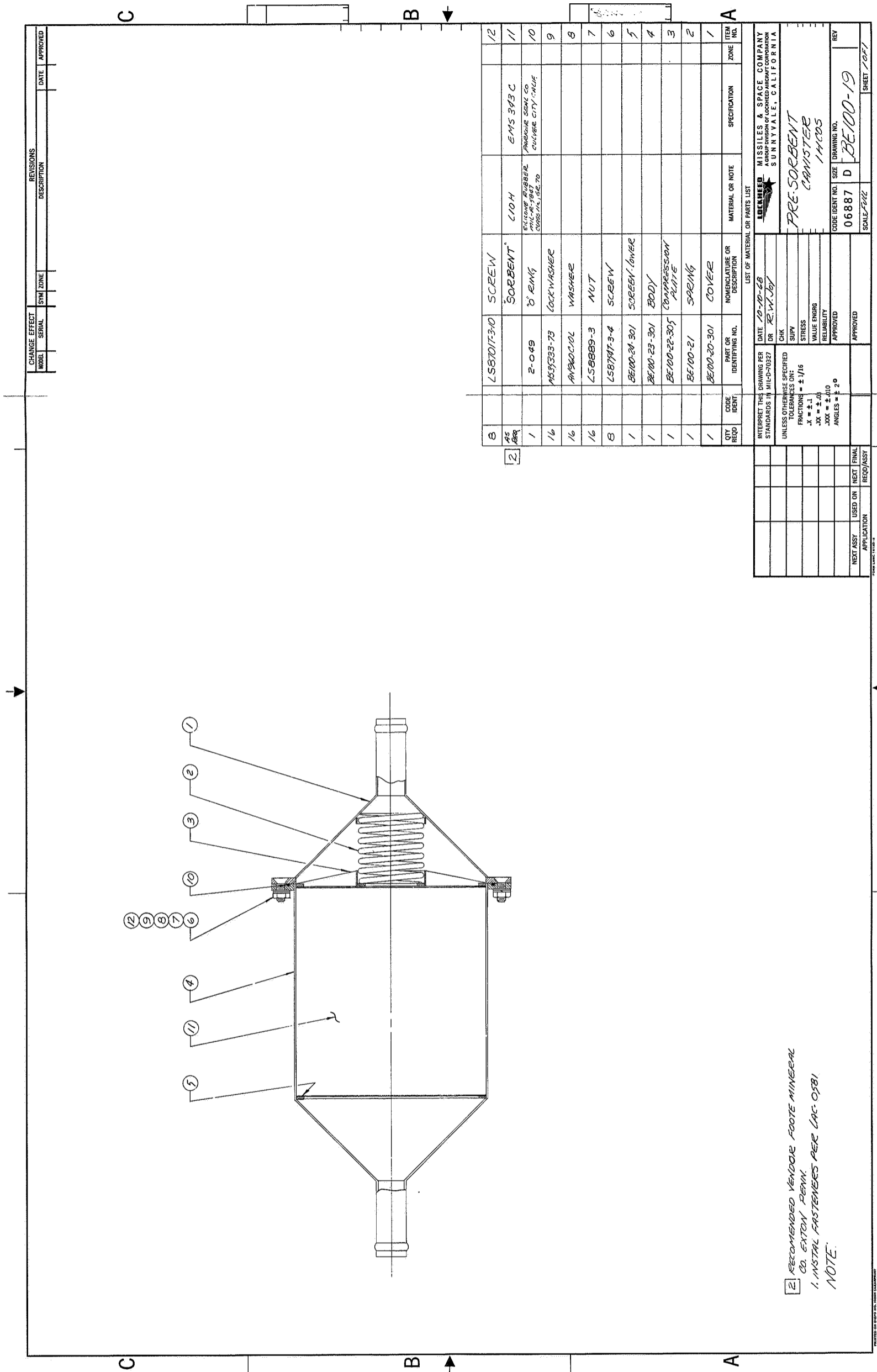
SCALE 4/1

SHEET 1









CHANGE EFFECT		SYMBOL		REVISIONS		DATE	
MODEL	SERIAL	SYM	ZONE	DESCRIPTION	DATE	APPROVED	

QTY REQD	CODE IDENT	PART OR IDENTIFYING NO.	NOMENCLATURE OR DESCRIPTION	MATERIAL OR NOTE	SPECIFICATION	ZONE	ITEM NO.
12		LS87017-3-10	SCREW				12
11		2-O-49	SORBENT		EMS 343 C		11
10		M53333-3	O RING	SILICONE RUBBER AMERICAN RUBBER CO CULVER CITY, CALIF MIL-PRC-131-10			10
9		AM920-01	LOCK WASHER				9
8		LS8889-3	WASHER				8
7		LS8889-3	NUT				7
6		BE100-24-301	SCREW				6
5		BE100-23-301	SCREEN COVER				5
4		BE100-22-301	BODY				4
3		BE100-21-301	COMPRESSION PLATE				3
2		BE100-20-301	SPRING				2
1		BE100-19-301	COVER				1

LIST OF MATERIAL OR PARTS LIST	
INTERPRET THIS DRAWING PER STANDARDS IN MIL-D-70327 OR R.W. 101	MISSILES & SPACE COMPANY A GROUP DIVISION OF LOCKHEED AIRCRAFT CORPORATION SUNNYVALE, CALIFORNIA
UNLESS OTHERWISE SPECIFIED FRACTIONS = 1/16 X = ±.1 XX = ±.05 XXX = ±.010 ANGLES = 2°	PRE-SORBENT CANNISTER 14005
NEXT ASSY. APPLICATION	CODE IDENT NO. SIZE DRAWING NO. 06887 D BE100-19
NEXT FIN. REQ/ASSY.	APPROVED
SCALE: 1/2" = 1"	SHEET 1 OF 1

RECOMMENDED VENDOR: FORT MINERAL CO. EXTON, PENN.  
1. INSTALL FASTENERS PER LAC-0381.  
NOTE.



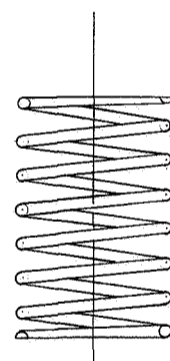






CHANGE EFFECT		REVISIONS		DATE		APPROVED	
MODEL	SERIAL	SYM	ZONE	DESCRIPTION	DATE	APPROVED	APPROVED



**SPRING DATA**

1. FREE LENGTH 2.50
2. NO. ACTIVE COILS 7
3. WIRE DIA. .128
4. MEAN DIA. 1.50
5. TOTAL COILS 9
6. END GROUND SQ. FLAT
7. WIRE MAT. 17-7PH CRCS

QTY REQD	CODE IDENT	PART OR IDENTIFYING NO.	NOMENCLATURE OR DESCRIPTION	MATERIAL OR NOTE	SPECIFICATION	ZONE	ITEM NO.
SEE SPRING DATA TABLE							

INTERPRET THIS DRAWING PER STANDARDS IN MIL-D-70327		DATE 10-1-68		DR R.W.V. / J.S.Y.	
UNLESS OTHERWISE SPECIFIED	CHK				
FRACTIONS = ± 1/16	SUPV				
.X = ± .1	STRESS				
.XX = ± .03	VALUE ENGRS				
.XXX = ± .010	RELIABILITY				
ANGLES = ± 2°	APPROVED				
	APPROVED				

APPLICATION	USED ON	NEXT	FINAL	REQD/ASSY

MISSILES & SPACE COMPANY		AEROSPACE DIVISION OF LOCKHEED AIRCRAFT CORPORATION		SUNNYVALE, CALIFORNIA	
<b>LOCKHEED</b>					
SPRING					
PREF. POST-SORBENT CANISTER					
1 H205					
CODE IDENT NO.	06887	SIZE	C	DRAWING NO.	BE100-21
SCALE	FULL	REV			
SHEET 1 OF 1					



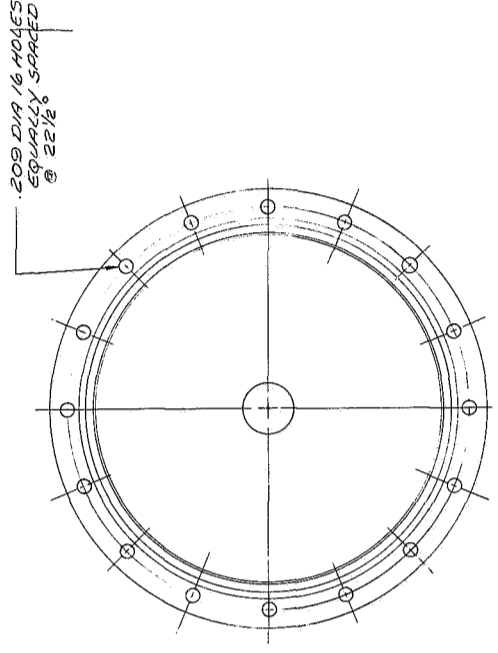
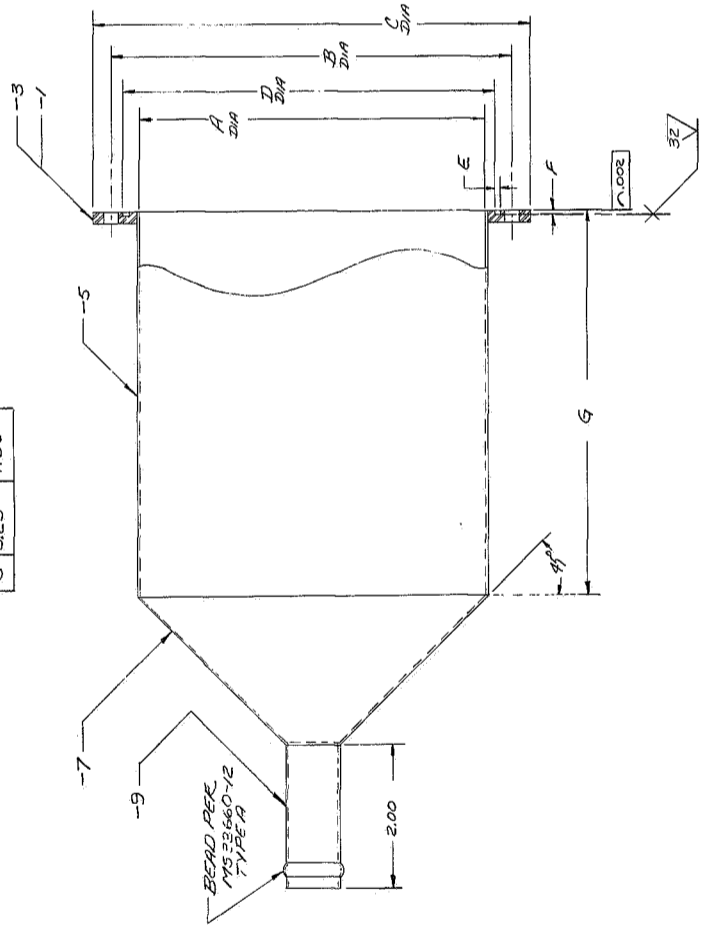




CHANGE EFFECT		REVISIONS		DATE	
NO.	SERIAL	SYMBOL	DESCRIPTION	DATE	APPROVED

-301	-305
A	4.750 5.500
B	5.500 6.375
C	6.000 6.875
D	5.000 5.737
E	$\frac{+0.005}{-0.005}$ .140
F	$\frac{+0.002}{-0.002}$ .081
G	5.25 7.95

ILLUSTRATION PROCESSING INFORMATION  
 MISSILES & SPACE COMPANY  
 5000 W. 10TH AVENUE  
 DENVER, COLORADO 80202  
 DRAWING NO. BE100-23  
 DATE 11/1/68  
 BY VJ  
 CHECKED VJ  
 APPROVED VJ



ITEM NO.	ZONE	SPECIFICATION	MATERIAL OR NOTE	DESCRIPTION	IDENTIFYING NO.	PART OR IDENTIFYING NO.	CODE IDENT	REMARKS
1			750004288V 321 CRES TUBE	TUBE	-9			
2			MIL-5-6721 COMP321	TRANSITION	-7			
3			321 CRES	BODY	-5			
4			321 CRES	FLANGE	-3			
5			321 CRES	FLANGE	-1			
6			MIL-5-6721 COMP321	NOMENCLATURE OR DESCRIPTION				

LIST OF MATERIAL OR PARTS LIST	
DATE	11-1-68
DR	R W Joy
CHK	
SUPP	
STRESS	
VALUE ENGR	
RELIABILITY	
APPROVED	

INTERPRET THIS DRAWING PER STANDARDS IN MIL-D-20327	
UNLESS OTHERWISE SPECIFIED	
TOLERANCES ON	
FRACTIONS	± .016
DECIMALS	± .01
ANGLES	± .03
ANGLES	± .03
ANGLES	± .03

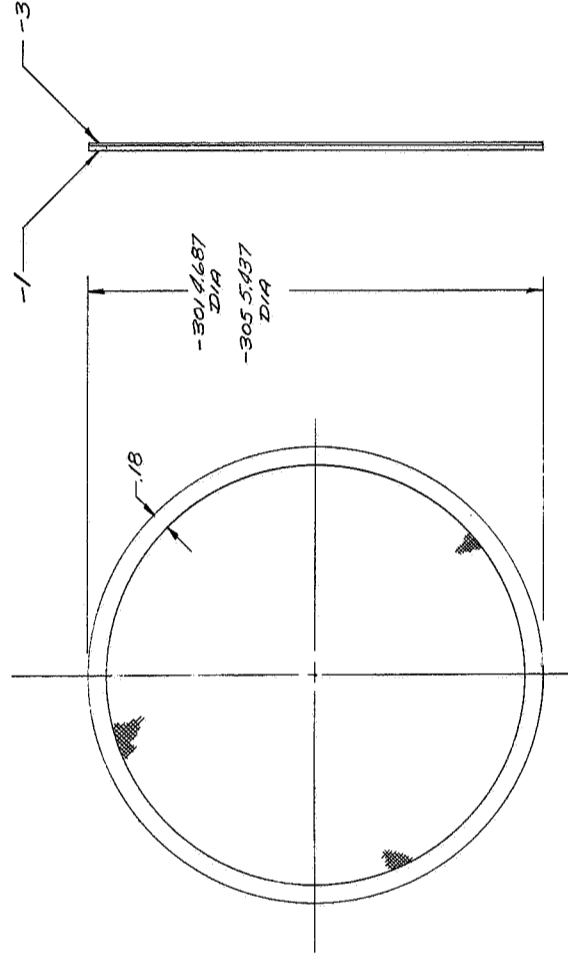
APPLICATION	USED ON	NEXT FINAL	REC'D/ASSY
BE100-23			
BE100-79			

2 MACH PER LAC-3601  
 1 ALL JOINTS WELDED 100%  
 PER LAC-1422  
 NOTE

FORM NO. 11-1



CHANGE EFFECT		REVISIONS		DATE		APPROVED	
MODEL	SERIAL	SYM	ZONE	DESCRIPTION	DATE	DATE	APPROVED



-301 SHOWN

QTY REQ	CODE IDENT	PART OR IDENTIFYING NO.	NOMENCLATURE OR DESCRIPTION	MATERIAL OR NOTE	SPECIFICATION	ZONE	ITEM NO.
1		-3	SCREEN	230 MESH 316 CREES			
1		-1	RING	321 CREES .063 TK	MIL-S-671 COMP 321		
305							

INTERPRET THIS DRAWING PER STANDARDS IN MIL-D-70327		DATE	DR	CHK	SUPY	STRESS	VALUE ENGR	RELIABILITY	APPROVED
		10-14-68	RWJ						

UNLESS OTHERWISE SPECIFIED TOLERANCES ON:	FRACCTIONS = ± 1/16	.X = ± .1	.XX = ± .03	.XXX = ± .010	ANGLES = ± 2°

APPLICATION	USED ON	NEXT	FINAL	REQD/ASSY

CODE IDENT NO.	SIZE	DRAWING NO.	REV
06887	C	BE100-24	

SCALE	SHEET
FULL	1 OF 1

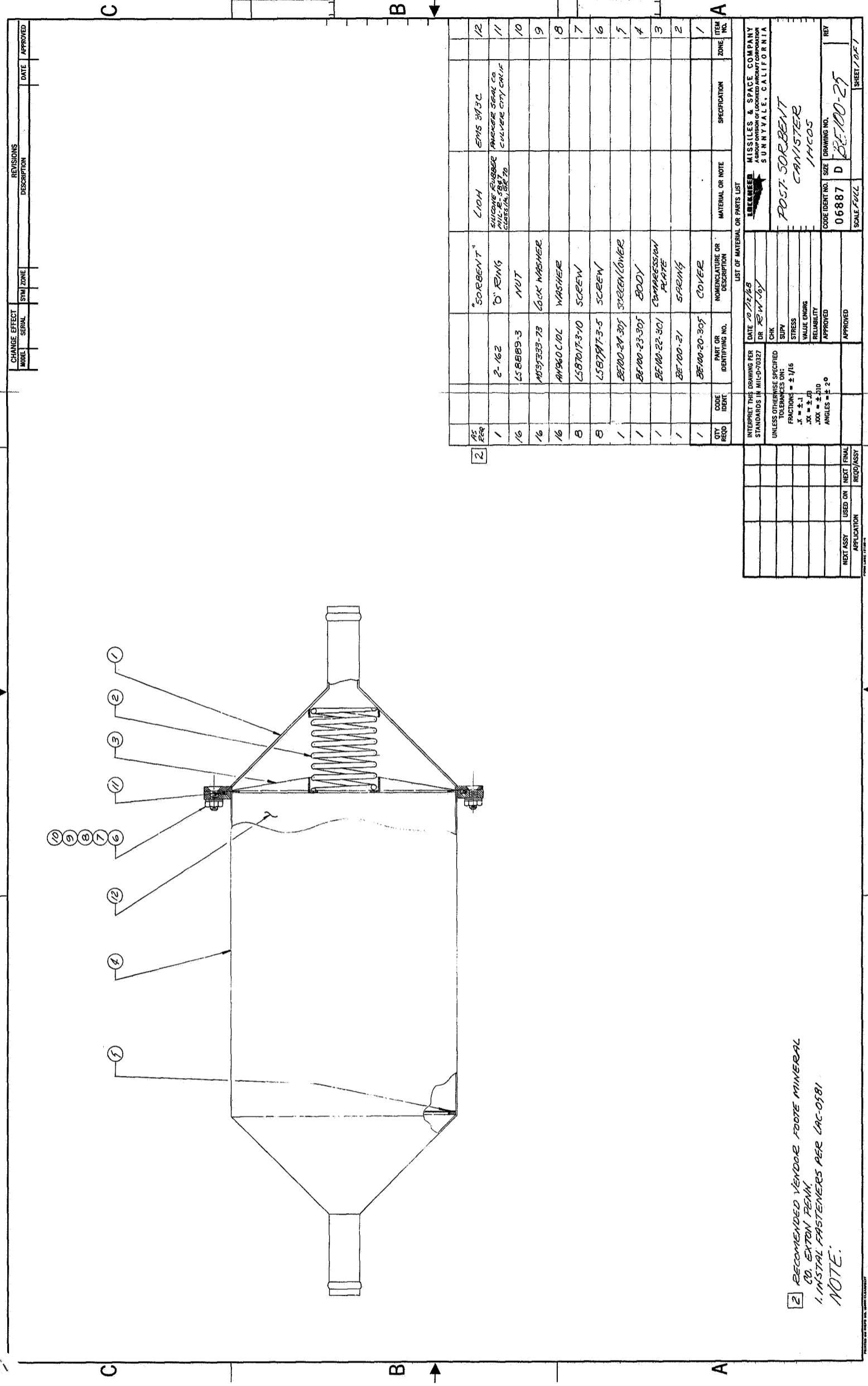
MISSILES & SPACE COMPANY  
A GROUP DIVISION OF LOCKHEED AIRCRAFT CORPORATION  
SUNNYVALE, CALIFORNIA

SCREEN-LOWER  
PREP POST SORBENT CANISTER  
14C05

1. SPOT WELD -1 TO -3  
PER LAC-ME1 CLASS C  
NOTE







CHANGE EFFECT		REVISIONS	
MODEL	SERIAL	SYM	ZONE

DATE	APPROVED

QTY REQD	CODE IDENT	PART OR IDENTIFYING NO.	NOMENCLATURE OR DESCRIPTION	MATERIAL OR NOTE	SPECIFICATION	ZONE	ITEM NO.
2	AS PER		*SORBENT	L10H	EMS 313C		12
1		2-162	O-RING	SILICONE RUBBER MIL-R-5887 CLASS 1A, 58E 70	ANCHOR SEAL CO CHRYSLER CITY CNL/F		11
1/6		LS8889-3	NUT				10
1/6		MS3333-73	LOCK WASHER				9
1/6		AN960C10L	WASHER				8
8		LS87017-310	SCREEN				7
8		LS87917-3-5	SCREEN				6
1		BE100-24-305	SCREEN COVER				5
1		BE100-23-305	BODY				4
1		BE100-22-301	COMPRESSION PLATE				3
1		BE100-21	SPRING				2
1		BE100-20-305	COVER				1

INTERPRET THIS DRAWING PER STANDARDS IN MIL-D-70327  
 UNLESS OTHERWISE SPECIFIED TOLERANCES ON:  
 FRACTIONS = ± 1/16  
 .X = ± .1  
 .XX = ± .03  
 .XXX = ± .010  
 ANGLES = ± 2°

DATE: 20/12/28  
 DR: R/W/S/Y  
 CHK: SUPP  
 STRESS: VALVE ENGINE  
 RELIABILITY: APPROVED  
 APPROVED:

MISSILES & SPACE COMPANY  
 SUNNYVALE, CALIFORNIA

POST-SORBENT  
 CANISTER  
 1MCO5

CODE IDENT NO.: 06887  
 DRAWING NO.: BE100-27  
 SCALE: FULL  
 SHEET 1 OF 1

2. RECOMMENDED VENDOR: FODTE MINERAL CO, EXTON PENN.  
 1. INSTALL FASTENERS PER IAC-0581  
 NOTE:



#### LIBRARY CARD ABSTRACT

This report describes the development and design of an isotope-heated catalytic oxidizer trace contaminant control system. The program included establishing pre- and post-sorbent bed designs to control potentially poisonous contaminants and to control contaminants that might produce undesirable products. A 180-day evaluation test of the pre-sorbent bed, catalytic oxidizer, and post-sorbent bed system was conducted. Tests were conducted to establish the fabrication and joining techniques and the compatibility between the isotope heat source materials of construction. Additional tasks included evaluation of candidate electric heater concepts for use in a simulated isotope heat source and evaluation of thermal insulation concepts for IHCOs. Detailed design drawings of the system including catalytic oxidizer, pre-sorbent bed and post-sorbent bed were prepared.